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Recovery of copper from pre-concentrated printed circuit boards (PCBs) by catalyzed acidic leaching

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Abstract: This study aimed to extract copper from waste printed circuit boards (WPCBs) through a sequential process involving physical pre-concentration via a shaking table, followed by acid leaching. A shredder and hammer mill were utilized to fragment the various components of the PCB into particles smaller than 1 mm. The shaking table pre-concentration tests revealed that the heavy fraction exhibited a copper grade of 56.4 % with a yield of 89.6 %. Subsequent leaching of the copper concentrate using a solution containing 1 M H₂SO₄ and 4 % (v/v) H₂O₂ at 50 °C for two hours resulted in a copper extraction efficiency exceeding 95 % with a solid ratio of 2 % wt./v.

Keywords: printed circuit board; copper; leaching; recovery; shaking table.

INTRODUCTION

The rapid pace of development in today's world has led to a significant accumulation of waste electrical and electronic equipment (WEEE), commonly referred to as E-waste due to the short lifespan of these products. In recent years, the growing global concern over environmental issues has prompted researchers implement various recycling strategies for extracting metals from E-waste. The main driving force behind the development of these recycling strategies is the increasingly stringent policies and regulations governing industries and landfill areas. Improper management of WEEE can lead to environmental issues due to the presence of hazardous inorganic substances like mercury (Hg) and lead (Pb), as noted in the literature.¹⁻³

WEEE is a valuable source of base metals like copper (Cu), as well as precious metals such as gold (Au), silver (Ag), platinum (Pt), and palladium (Pd), which are comparable in content to those found in ores and concentrates. As such, recycling WEEE is crucial for both environmental preservation and economic gain, as noted

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in literature.⁴⁻⁶ Additionally, recycling WEEE has been highlighted as an important measure for both environmental and economic benefits.⁷⁻⁹

Printed circuit boards (PCBs), computers, and other forms of electronic waste (E-waste) contain a high concentration of base metals, particularly copper (Cu). In fact, the weight percentage of copper obtained from PCBs can range from 16-30 %, which is significantly higher than any other metal found in e-waste. By comparison, copper found in ores typically only ranges from 0.5-1 %.¹⁰

Various researchers have proposed physical,¹¹⁻¹⁵ pyrometallurgical,¹⁶⁻¹⁸ hydrometallurgical¹⁹⁻²³, and biometallurgical^{24,25} methods based on conventional techniques for recovering metals from e-waste. While physical and pyrometallurgical processes are widely used in industrial applications of e-waste recycling, recent attention on metal recovery has shifted towards hydrometallurgical processing. In general, pre-treatment steps, such as separating different parts of electronic and electrical products, are necessary for metal recovery from e-waste through size reduction and hydrometallurgical processes. In industrial processes, e-waste is typically fed into chemical process for metal recovery immediately after physical separation and pyrometallurgical process.^{3,26}

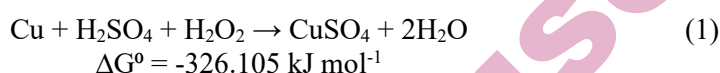
Furthermore, various leaching studies have also been conducted using many different reagents.²⁷⁻²⁸ According to Montero *et al.*²⁹ during column leaching of waste printed circuit boards using cyanide solutions, copper was found to dissolve simultaneously at a rate of 77 %. However, direct cyanidation led to increased cyanide consumption and decreased recovery of precious metals. To address this issue, pre-leaching is recommended to dissolve or remove copper and other base metals before extracting precious metals using cyanide, thiosulfate, thiourea, or chloride media. Quinet *et al.*,²¹ Oh *et al.*²⁰ and Kamberović *et al.*³⁰ have suggested an acidic sulfate leaching with an appropriate oxidant prior to extracting precious metals.

The investigation on various inorganic acids and oxidants used for metal recovery revealed that the highest Cu recovery was obtained with nitrate and chloride solutions.²² Mecucci and Scott¹⁹ reported > 95 % dissolution of Cu from circuit boards using HNO₃. Kinoshita *et al.*³¹ found similar results with Cu recovery of > 90 %. Although the use of nitric acid can be costly due to its oxidative nature, sulfuric acid with additives such as H₂O₂, O₂, and Fe³⁺ can be a more affordable and favorable option over other processes.

In the literature, leaching methods with two chemicals were reported to extract both base and precious metals from waste printed circuit boards (WPCBs). They utilized sulfuric acid with hydrogen peroxide for the first group of metals, while they used thiourea with the ferric ion in sulfuric acid medium for the second group of metals.³² The optimal conditions for copper extraction were found to be 2M H₂SO₄ (98 % w/v), 5 % H₂O₂, 85 °C temperature, 1/10 solid-to-liquid ratio, and

200 rpm. Various investigators³³⁻³⁷ have utilized distinct oxidizing agents, including H₂O₂, O₂, and Fe³⁺, for leaching copper in H₂SO₄.

Sulfuric acid and hydrogen peroxide have both a role of oxidizing and reducing agent in metal component dissolution, and their standard-state free energy (ΔG°) can be expressed as follows⁶:

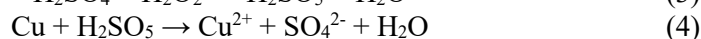
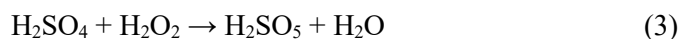


The second reaction represents the decomposition of the hydrogen peroxide (H₂O₂) molecule into water (H₂O) and oxygen (O₂) gas, and it can be accelerated using catalysts such as manganese dioxide (MnO₂) or potassium iodide (KI).



Sulfuric acid forms peroxydisulfuric acid (Caro's acid, H₂SO₅) on reaction with hydrogen peroxide (Eqs. 2 and 3). This is a strong oxidizer for copper and assists in the dissolution of other metals. Copper dissolution with H₂SO₅ at the controlled-experimental conditions is shown in Eq. 4.³⁸

It can be prepared from concentrated hydrogen peroxide and concentrated sulfuric acid. However, it is generated and consumed immediately at the point of use because of its exothermic nature. Therefore, with the use of diluted sulfuric acid and hydrogen peroxide solutions at higher temperatures than 10 °C, the primary oxidation mechanism with hydrogen peroxide involves electron transfer, stemming from the weak H-O bond dissociation, resulting in oxygen serving as an electron acceptor.



The focus of this study is on the chemical recovery of copper, concentrated from a physical treatment process, specifically gravity concentration of PCBs. A shaking table was chosen for the physical separation step, while acidic leaching was preferred for the hydrometallurgical process.

MATERIAL AND METHOD

In this study, chemical leaching methods were conducted on the concentrate gained by the shaking table method. Similar gravimetric enrichment method of waste PCBs after removal of electronic components (resistor, capacitor, diode, transistor etc.) was explained in the study of Gok and Sen.³⁹

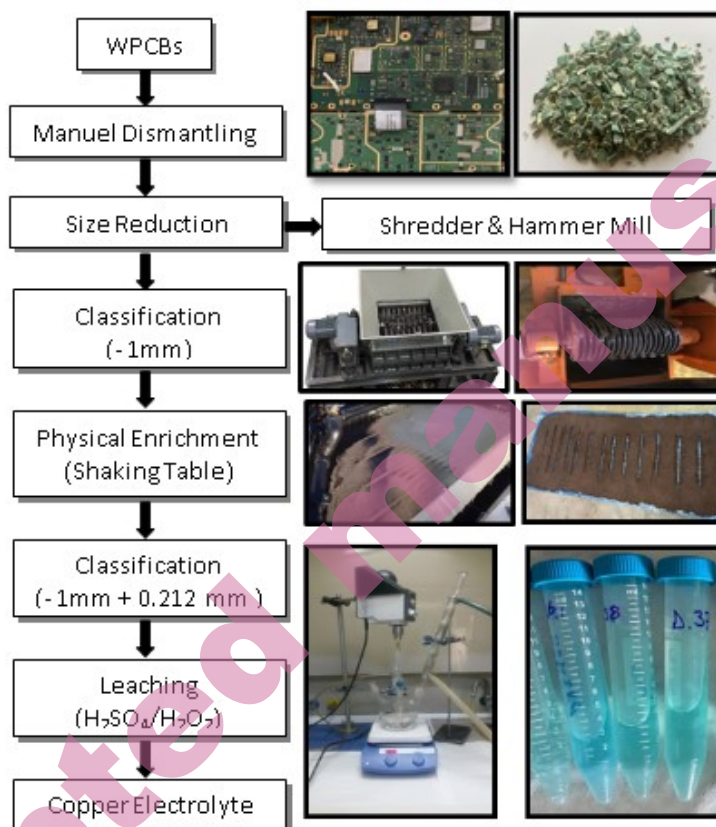


Fig.1. Gravimetric enrichment flowsheet of the waste PCBs

The essential purpose of the crushing process in WPCB recycling is to separate metals from non-metals, playing a vital role in the recovery of metals from WPCBs. This process is closely tied to the subsequent selective recovery of metals.⁴⁰ Pre-concentration methodology includes comminution and gravimetric concentration using shaking table. Employing a two-step crushing approach prior to the pre-concentration step (shaking table) has proven to be effective in dismantling WPCBs. A single shaft shredder knife crusher was used to break down WPCB plates into smaller particles (-10 mm). Subsequently, these smaller particles undergo further crushing in a hammer grinder for WPCBs, featuring 1 mm diameter screen holes. Closed circuit pulse-jet bag filters collected and recycled the dust throughout the comminution. Particle classification and size control was conducted using a vibratory sieve.

The graphical flowsheet of process steps is illustrated in Fig. 1.

Pre-concentration of copper particles was carried out using a Wilfley shaking table to recover the copper particles, presenting a promising technique to mitigate dust-related issues. This technique involves the separation of materials based on particle density on inclined planes with smoothed or grooved surfaces, achieved through the back-and-forth vibration of the pulp material while wash water flows. The underlying principle relies on segregating rapidly moving coarse light particles from slowly moving small dense particles within the flowing water film

through longitudinal vibration. The optimal settings of concentration with shaking table are presented in Table I.

TABLE I. Optimal operating parameters and results of Shaking Table Enrichment

Particle Size, mm	-1
Wash Water Flowrate, Lpm	12
Deck Angle, °	2
Motion Frequency, Hz	60
Cu Grade, %	56.40
Cu Yield, %	89.56

Chemical dissolution of the sample in aqua regia and spectroscopic analyses of the electrolytes on Analytik Jena NovaA 300 AAS indicated that major component was copper. Table II presents the chemical components of PCB feed material (-1 mm) and concentrate for four size fractions. Apart from the metal fraction indicated for the samples, it was observed that the remaining parts consisted of polymer and ceramic. Sieve analyses and fractioning were conducted using a vibratory laboratory sieve shaker for 20 minutes. Three different fractions of the concentrate samples were used at leaching tests: -1000+500 μm ; -500 + 300 μm ; and -300+212 μm (Table I). -212 μm fraction consisted of higher metallic content such as Fe, Zn, Pb, Ni, Mn than larger size fractions. Thus, this smallest size fraction was not included in leaching experiments.

TABLE II. Chemical analysis of PCB feed and concentrate samples

Sample	Weight, %	Component $d_{\text{particle, } \mu\text{m}}$	Cu	Ni	Pb	Zn	Fe	Mn
Feed	100	-1000	16,78	1,13	1,40	0,59	0,10	0,15
	30.95	-1000+500	67.42	1.69	2.85	0.96	2.35	0.27
Concentrate	30.60	-500+300	63.13	1.33	1.91	1.18	2.40	0.22
	25.62	-300+212	47.38	1.19	1.78	1.51	3.32	0.44
	12,83	-212	20.43	3.35	5.56	5.07	14.83	2.21

Rigaku Miniflex II diffractometer with Cu $K\alpha$ radiation was used for X-Ray Diffraction analyses of samples. XRD analysis of PCB concentrate revealed the presence of polymer, ceramic, solder and copper as major components. The microscope analysis given in Fig 2. also supported XRD analysis. The microscopic analysis of PCB concentrate revealed the presence of liberated copper particles smaller than 1 mm, as well as particles containing traces of Fe, Ni, and Pb. Additionally, black-colored solder particles, yellow-colored glass fibers, and green-colored resin particles smaller than 1 mm were observed.



Fig. 2. Optical microscope pictures of the shaking table concentrate at 8× magnification

The main purpose of the research was to investigate optimal parameters of the acid leaching of copper from pre-concentrated PCBs. A 5 g of PCBs concentrate were immersed in leaching reagent saturated with an oxidant. Leaching tests were performed in a 0.50-L four-necked glass reactor equipped with a condenser. The temperature was controlled within ± 2 °C using a glass-coated temperature sensor in conjunction with a hot plate. Typically, the vessel was charged with an electrolyte containing H_2SO_4 (0.5-1.5 M) and H_2O_2 (2-8 % v/v) and sample at three particle size fractions (1000-500 μm , 500-300 μm , 300-212 μm) with 1-8 % wt./v solid ratio. The initial temperatures of the solution were selected as 30 °C, 50 °C, 70 °C, and the reaction was sustained for 2 h. The solution was mixed with a mechanical stirring rate of 10 s^{-1} by a magnetically driven twin impeller. 5 ml of pregnant solutions were withdrawn to determine copper concentration using Analytik Jena NovaA 300 AAS.

RESULTS AND DISCUSSION

The dissolution efficiency of copper from PCB concentrate was established in the presence of an oxidant with various pulp ratios, particle sizes, temperatures and acid concentrations. Except for the experiments designed to determine the effect of solid ratio, all tests were conducted with an optimum solid ratio of 2 % wt./v.

Effect of particle size

Experiments conducted with three different particle size fractions (1000-500 μm , 500-300 μm , 300-212 μm) in 1M H_2SO_4 and 4 % H_2O_2 solution at 30°C to determine leaching time have revealed no significant difference in leaching efficiency after 90 min. (Fig. 3). In the particle size range of 300-212 μm , the finest particle size interval, a leaching efficiency of over 90 % copper has been achieved in one hour. The results indicate that after two hours, the recovery is around 85 % for the 1000-500 μm fraction, and 90 % for the 500-300 μm fraction. After 2 hours of leaching, a 15 % difference in copper recovery was observed between the largest

and smallest sizes of PCB samples. In the fine particle size fraction, the reaction rate increases due to the higher percentage of liberated copper particles. This leaching method represents a classic heterogeneous reaction system, and therefore it is obvious that the larger surface area due to the smaller particle size has a beneficial effect. It is evident that grinding to finer sizes incurs additional costs. However, as seen from the graph, the copper recovery achieved within 1 hour at the 300-212 μm size was attained only after 2 hours for the 1000-500 μm particle size.

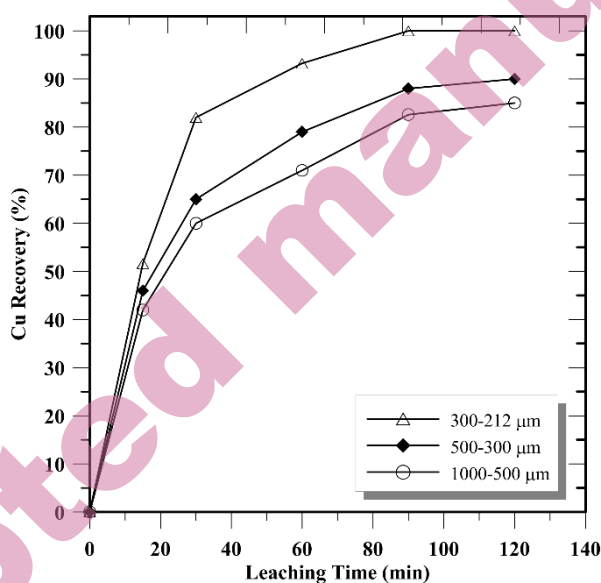


Fig 3. Effect of the particle size on copper recovery. Initial electrolyte conditions: $C_{\text{H}_2\text{SO}_4} = 1\text{M}$, $C_{\text{H}_2\text{O}_2} = \% 4$, $T = 30^\circ\text{C}$.

Effect of acid concentration

The influence of the acid concentrations of the copper recovery was investigated with various particle size fractions (1000-500 μm , 500-300 μm , 300-212 μm) using electrolyte containing 4 % v/v H_2O_2 at 50 $^\circ\text{C}$. The values in Fig. 4 reveal that high levels of copper extraction, typically around 90 %, were achieved with 1.5 M H_2SO_4 and 300-212 μm fraction at 50 $^\circ\text{C}$. The leaching experiments conducted with 1.5 M H_2SO_4 demonstrated that the coarser particle size fractions resulted in lower recovery rates. Specifically, the recovery for the 1000-500 μm fraction was approximately 65 %, while the recovery for the 500-300 μm fraction was around 80 %. Although copper recovery improved with increasing concentration, the rate of improvement diminished for smaller size fractions at concentrations exceeding 1 M H_2SO_4 . The solvent effect of H_2SO_4 has been observed to be less pronounced in larger particle sizes compared to finer sizes.

Researchers explained that this type of decrease in efficiency usually was indicative of a reaction controlled by diffusional effects. Hence, as the particle size increases, the leaching kinetics decrease.

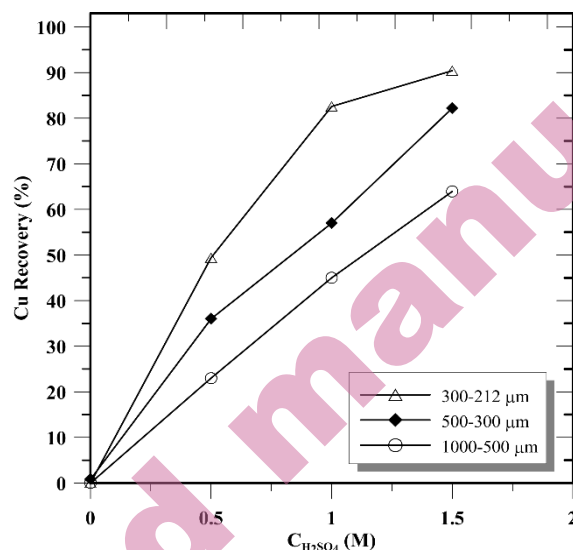


Fig 4. Effect of the acid concentration on copper recovery. Initial electrolyte conditions: $C_{H_2O_2} = \% 4$, $T = 50$ °C.

Effect of oxidant concentration

The effect of H_2O_2 concentration (2–8 % v/v) on the copper leaching was elucidated with three size fractions (1000-500 μm , 500-300 μm , 300-212 μm) using in the solutions containing 1 M H_2SO_4 at 50 °C. The contribution of an oxidant is revealed in Fig.5. The oxidizing effect created at a concentration of 6 % H_2O_2 has been observed to facilitate the leaching of all copper with the smallest size fraction. With this amount of hydrogen peroxide used, the copper recovery for the finest size fraction was found to be 25 % higher than that obtained for the coarsest size fraction. The results obtained with 8 % H_2O_2 revealed a recovery rate of 85 % for the 1000-500 μm particle size fraction, while the recovery for the 500-300 μm fraction exceeded 90 %. At higher concentrations of H_2O_2 , there is a reduced requirement for fine particle size. The decomposition rate of hydrogen peroxide is quite high. Therefore, it is thought that the oxidation of copper by peroxide in coarser samples, such as those in the 1000-500 μm range, occurs more slowly due to the lower diffusion rate.

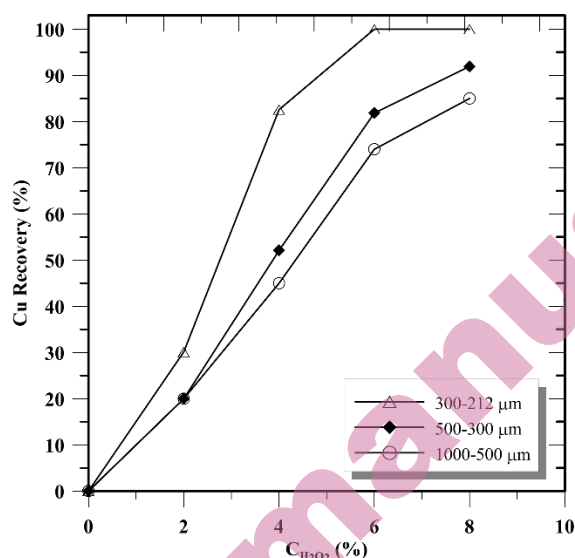


Fig 5. Effect of the oxidant concentration on copper recovery. Initial electrolyte conditions: $C_{H_2SO_4} = 1\text{M}$, $T = 50\text{ }^\circ\text{C}$.

Effect of the temperature

The temperature effect on the results of copper extraction in the electrolyte containing 4 % v/v H_2O_2 and 1 M H_2SO_4 at various temperatures (30 $^\circ\text{C}$, 50 $^\circ\text{C}$, 70 $^\circ\text{C}$) are shown in Fig.6. The leaching tests indicated that with 300-212 μm size fractions copper recovery at 30 $^\circ\text{C}$ was completed within 90 min. In comparison, the recovery rate reached approximately 90 % at 50 $^\circ\text{C}$ and exceeded 80 % at 70 $^\circ\text{C}$. It has been observed that within the fine particle range, a 20 % increase in leaching efficiency occurs as the reaction temperature decreases. Thus, temperature increase had adverse effect. It was noted that temperature increase led the rapid decomposition of H_2O_2 , which caused a decrease in the oxidant behavior. Temperatures above 50 $^\circ\text{C}$ do not significantly affect leaching efficiency.

Effect of solid ratio

A solid ratio of 2 % and low acid/oxidant concentration (1M H_2SO_4 , 4 % H_2O_2) have yielded efficiencies exceeding 95 % with 300-212 μm fraction at 50 $^\circ\text{C}$. Effect of the solid ratio on copper recovery is shown in Fig. 7. Experiments conducted with an 8 % solid-to-liquid ratio resulted in copper recoveries of approximately 80 % for the 300-212 μm size fraction, 70 % for the 500-300 μm fraction, and around 60 % for the 1000-500 μm fraction. The observed variation in the curvature trend in fine particle sizes is interpreted as the diffusion becoming more challenging with an increase in pulp ratio in larger particle sizes.

The copper recovery results obtained in this research was found similar with the studies in the literature^{6,20,34,41,42} on the copper leaching from PCBs with higher

$\text{H}_2\text{SO}_4 + \text{H}_2\text{O}_2$ concentrations. It was thought that this was because of the pre-concentration step in which plastic part preventing the dissolution is eliminated.

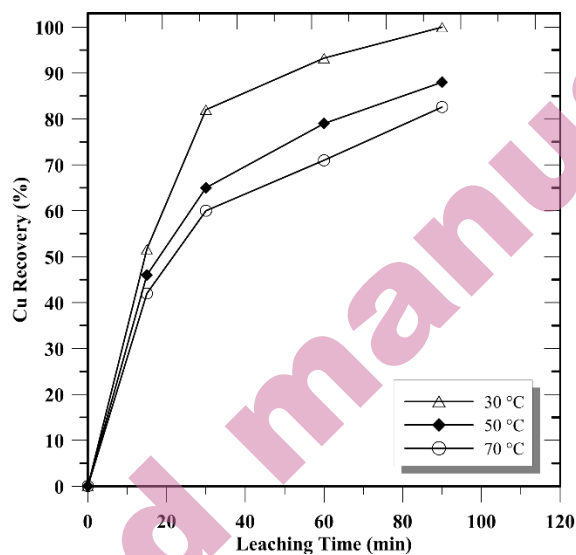


Fig 6. Effect of the temperature on copper recovery. Initial electrolyte conditions: $C_{\text{H}_2\text{SO}_4} = 1\text{M}$, $C_{\text{H}_2\text{O}_2} = \% 4$, $d_p = 300\text{-}212 \mu\text{m}$.

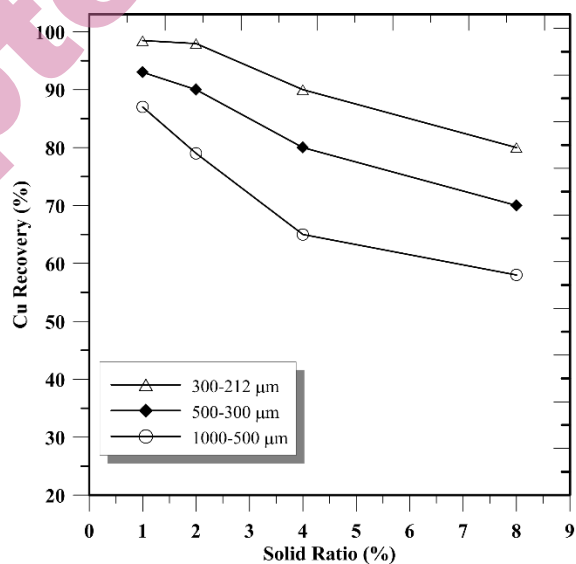


Fig 7. Effect of the solid ratio on copper recovery. Initial electrolyte conditions: $C_{\text{H}_2\text{SO}_4} = 1\text{M}$, $C_{\text{H}_2\text{O}_2} = \% 4$, $T = 50 \text{ }^\circ\text{C}$.

Kinetic analysis

The primary consideration in understanding a leaching system is its mechanism, with an in-depth knowledge of the kinetics of the rate-controlling processes and the solid reaction products being essential for a thorough comprehension of the system. Kinetic analysis was conducted to elucidate the reaction mechanism of a non-catalytic heterogeneous reaction. The kinetic parameters and activation energy of the copper dissolution in oxidized sulfuric acid electrolytes was evaluated. The reaction between solid particles and solution may have its rate-controlling step attributed to one of the following factors: external diffusion of the reactant through the boundary layer of the fluid enveloping the particle (film diffusion controlled), surface reaction between the fluid reactant and the solid (chemical reaction controlled) or internal diffusion of the reactant through the reaction products on the particle (ash diffusion controlled).^{43, 44}

*Film diffusion control dense constant size small particles – all geometries

$$X = K_c.t \quad (5)$$

*Chemical reaction control dense constant size or shrinking spheres

$$1 - (1 - X)^{1/3} = K_c.t \quad (6)$$

*Ash diffusion control dense constant size-spherical particles

$$1 - 3(1 - X)^{2/3} + 2(1 - X) = K_c.t \quad (7)$$

where reaction rate constant, K_c / min^{-1} , time, t / min , and fraction reacted of copper, $X / \%$.

TABLE III. Apparent rate constants and correlation coefficients for shrinking core models

T/ K	Chemical Reaction Controlled		Ash Diffusion Controlled	
	K_c / min^{-1}	R^2	K_c / min^{-1}	R^2
303	0,0111	0,9447	0.0111	0.9954
323	0,0064	0,9194	0.0059	0.9965
343	0,0055	0,9108	0.0046	0.9961

The data obtained from the kinetic analysis at different temperatures were tested by the equations of the shrinking core models (5-7). As seen in the Table III and Fig. 8, the regression coefficient in the chemically reaction-controlled kinetic model was found to be closer to 1 only for 30 °C. For higher temperatures, the values appear to be less-linearized. The result data fitted with the ash diffusion controlled shrinking spheres model; i.e., $1 - 3(1 - X)^{2/3} - 2(1 - X) = K_c.t$. This model indicated the highest value of regression coefficient R^2 and the constant value of K_c was obtained. The reaction rate constant at 30 °C was determined to be 0.0111 min^{-1} , and the reaction rate decreased as the temperature increased. Although the PCB solid samples were concentrated by shaking table method, a polymeric matrix

was still present during leaching. This may have caused the diffusion of the electrolyte in the leaching process to become the rate-determining step. Since the chemical reaction-controlled model does not exhibit a linear trend at high temperatures, the ash-controlled reaction model was preferred for activation energy calculations. As shown in Figure 8, the reaction rate constant values tend to decrease with increasing temperature. This is attributed to the rapid decomposition of hydrogen peroxide and the subsequent loss of the oxidant, which prevents the copper leaching rate from reaching sufficiently high reaction rates.

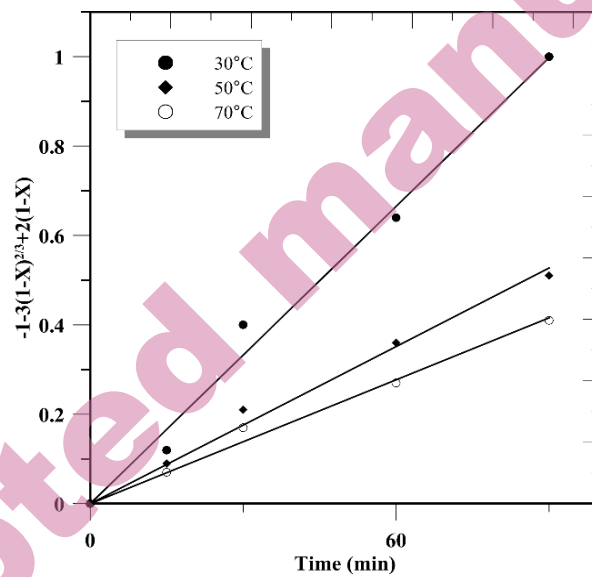


Fig. 8. Plot of the ash diffusion controlled shrinking core model for the effect of temperature on the copper recovery reaction rate

The activation energy, $E_a = -19.17 \text{ kJ mol}^{-1}$, was determined from the slope of straight line plotted between $\ln k$ and $10^3/T$ as seen on Fig 9. Thus, the determined activation energy indicates that the copper leaching from PCBs using $\text{H}_2\text{SO}_4\text{-H}_2\text{O}_2$ in the range of 303–343 K occurs in constant zone.

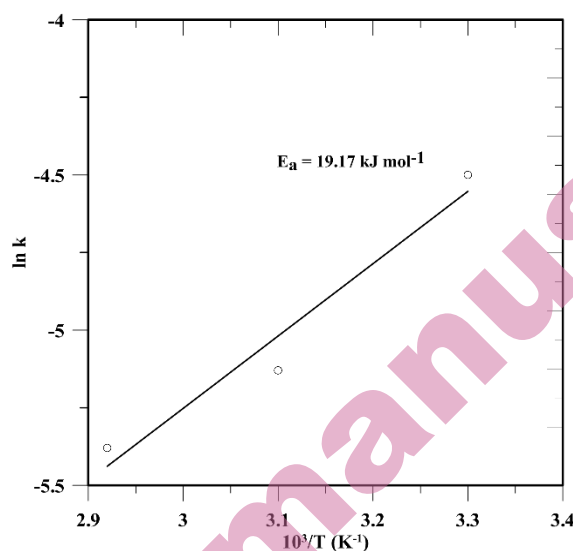


Fig. 9. Plot of parabolic leaching rate constants vs. inverse of temperature (Arrhenius plot)

CONCLUSION

- The investigation involved the dissolution of copper in an oxidized acidic electrolyte ($H_2SO_4-H_2O_2$) under varying conditions such as particle size, solid ratios, temperatures, and oxidant concentrations to elucidate the leaching kinetics of copper in Waste Printed Circuit Boards (WPCBs).
- The chemical, X-ray Diffraction (XRD), and optical analyses collectively suggested that the disassembly of electronic components in WPCBs, coupled with a two-step comminution process and physical pre-concentration, facilitates the copper dissolution process.
- Copper pre-enrichment was achieved through a shaking table, reducing particle size to under 1 mm, with a wash water flow rate of 12 Lpm, a 2° deck angle, and a motion frequency of 60 Hz. The resulting copper concentrate exhibited a grade and yield of 56.40 % and 89.56 %, respectively.
- A 90 % copper recovery rate was achieved within 60 minutes at 30 °C in a 1 M sulfuric acid solution containing 4 % v/v hydrogen peroxide. The incorporation of oxidant and size reduction promoted the disproportionate dissolution of copper in plastic under all experimental conditions, with temperature variations showing reverse impact on copper recovery.
- Copper leaching from silicate-rich WPCBs was determined to be governed by ash diffusion. The dissolution rate constant was calculated as 0.0111 min^{-1} at 30 °C, with a regression constant of 0.9954. The activation energy (E_a) for the process was estimated to be $19.17 \text{ kJ mol}^{-1}$.

- The proposed process flowsheet outlined in this study can be readily adapted for industrial plant applications. Despite the apparent economic impracticality of using oxidant in large-scale operations due to its high cost, this method holds promise as an alternative to existing pyrometallurgical and hydrometallurgical applications, including those involving aggressive reactant.

ИЗВОД

ЕКСТРАКЦИЈА БАКРА ИЗ ПРЕД-КОНЦЕНТРИСАНИХ ШТАМПНИХ ПЛОЧА (PCB)
ПУТЕМ КАТАЛИЗОВАНОГ КИСЕЛИНСКОГ ИЗЛУЖИВАЊА

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Ова студија је за циљ имала издвајање бакра из отпадних штампаних плоча (WPCB) секвенцијалним процесом који укључује физичку пред-концентрацију на тресећем столу, а затим киселинско излуживање. За фрагментацију различитих компоненти штампаних плоча на честице мање од 1 мм коришћени су дробилица и чекићни млин. Тестови пред-концентрације помоћу тресећег стола су показали да тешка фракција има садржај бакра од 56,4 % са приносом од 89,6 %. Након тога је излуживање бакра из концентрата извршено третирањем раствором који садржи 1 М H₂SO₄ and 4 % (v/v) H₂O₂ на 50 °C током два сата, што је резултирало ефикасношћу екстракције бакра већом од 95 % са односом чврсте материје од 2 % теж./вол.

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