

Recovery of Critical Metals from E-Waste Printed Circuit Boards (PCBs) Via Acid Leaching

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Abstract

In this research, the study focused on recovering critical metals from E-waste Printed Circuit Boards (PCBs) using a hydrometallurgical approach that involves leaching in a chloride-based solution. The investigation examined how various leaching parameters, such as acid concentration, reaction temperature, particle size, and leaching duration, influenced the efficiency of dissolving critical metals. A kinetic model was developed to represent how these variables affected the leaching rate. The raw HP waste computer desktops (HP WPCBs) primarily consisted of Propylene, (C₃H₆) n {54-1936}; Sasaite, {031-0020; Silicon oxide, SiO₂ {82-1558} and Impure copper oxide CuO {10-1329}. The results obtained from the Energy-Dispersive X-ray Fluorescence (EDXRF) analysis supported the findings of X-ray Diffraction (XRD). Under the optimized leaching conditions, 81.8% of the critical metals dissolved within 120 minutes, and the leaching process was predominantly controlled by a surface layer mechanism. The estimated activation energy (E_a) of 55.79 kJ/mol further supported this dissolution mechanism.

1. Introduction

The rise in development and industrialization has enhanced people's living standards by introducing various electronic devices. The field of information technology has seen significant advancements, making our lives more convenient with the use of electrical and electronic equipment (EEE). This progress, however, has resulted in not only increased extraction of raw materials but also a concerning surge in electronic waste production. The consumption of Electrical and Electronic Equipment (EEE) plays a pivotal role in driving global economic development. In today's modern society, electronic devices have become indispensable for daily life. On average, global EEE consumption increases by approximately 2.5 million metric tons (Mt) annually. Unfortunately, this has led to electronic waste (E-waste) becoming one of the fastest-growing categories of solid waste due to its short lifespan. In 2019, an estimated 53.6 Mt of electronic waste was generated per capita, and projections suggest that this figure could reach 74 Mt by 2030 [1,2]. At a global level, E-waste is increasing rapidly, at a rate of 2 Mt per year. However, only 17.4% of this E-waste is currently being recycled [3-5]. The majority of E-waste contains valuable yet hazardous metals, including Al, Ag, Be, Au, As, Bi, Cr, Cd, Cu, Ni, Hg, Pt, Fe, Sb, Zn, and Si, as well as organic chemicals and flame retardants [2].

E-waste poses a significant threat to both the environment and human well-being due to the presence of toxic elements and various metals. When electronic waste components containing heavy metals, and organic and

inorganic substances enter bodies of water, they contaminate water resources [6]. In the past, natural ore deposits contained over 5.0% copper content, but current ore grades typically have less than 1.0% metal content. E-waste, when assessed for its metal content, is often referred to as an urban mine. Properly processed, it has the potential to serve as an alternative source for resource recovery [7-9]. Improper disposal of E-waste includes landfill dumping and open-air burning. This results in the production of dioxins and furans, as well as runoff of hazardous waste residues that contaminate the soil, water, and air. The leaching of metals from electronic waste is crucial not only for sustainable waste management but also for meeting the growing demand for these metals while conserving rapidly depleting natural resources [10,11]. Industrial systems designed to leach metals from E-waste employ pyrometallurgical and hydrometallurgical techniques. However, pyrometallurgical methods have some limitations. They can only partially separate metals, mainly focusing on base metals, and are inefficient for leaching precious metals [12]. One of the most intriguing and practical approaches lies in the hydrometallurgical recycling route. In recent decades, hydrometallurgy has made significant strides in the recovery of precious metals from E-waste printed circuit boards. These methods offer precise control, environmental friendliness, reduced capital costs, and, most importantly, higher metal recovery compared to pyrometallurgical processing.

Recently, there has been a growing interest in using hydrometallurgical techniques to treat electronic waste (E-waste). Researchers have found that strong acids, such as sulfuric acid, hydrochloric acid, nitric acid, aqua regia, citric acid, ascorbic acid, malic acid, and oxalic acid, are effective in leaching metals from E-waste [13-25]. Metals are vital in modern society and are closely linked to industrial development and improved living standards. Society can obtain metals from the Earth's crust and also from discarded metal products. Despite the large amounts of E-waste, including computer desktops, being discarded, some of these materials can be recovered. However, the leaching process for E-waste printed circuit boards is more complex than natural resources due to the various metals, intrinsic structure, and complex manufacturing materials. This complexity makes leaching precious metals challenging, as multiple competing reactions consume reagents. To address the increasing global demand for metals, this study explores the use of hydrochloric acid to extract critical and precious metals from E-waste printed circuit boards. The hydrochloric acid system demonstrates good selectivity for copper and precious metals.

2. Materials and Method

2.1 Materials

The HP E-waste computer printed circuit boards treated in the present work were collected in Kabba, Kogi State, Nigeria. Principally, it was composed of spent materials of HP E-waste computer printed circuit boards (HP WPCBs). The chemical reagents used in this work were hydrochloric acid (HCl, AnalaR product) and distilled water.

2.2 Physical Processing and Pulverization

The HP E-waste computer printed circuit boards (HP WPCBs) were broken down into smaller pieces using a hammer mill acquired from the Department of Geology and Mineral Sciences at the University of Ilorin in Ilorin, Nigeria. The processed E-waste was further ground into powder at Hikmah Plastic Grinding in Mabushi, Abuja, Nigeria, using a high-density H-180 model recycling machine. The resulting crushed and finely ground samples were sieved through American Standard Sieves (ASTM) of various sizes, including -63+56 μm , -75+63 μm , and -90+75 μm fractions.

2.3 Sample Characterization

The HP E-waste computer printed circuit boards were analyzed for their elemental composition, mineralogical purity, and morphological structures. The analysis was performed using a MINI PAL 4 EDXRF spectrometer, EMPYREAN X-ray diffractometer, and scanning electron microscopy (SEM) model Leo1450 with LaB6 filament. The samples were carbon coated and viewed at 10 kV and 13 mm working distance. Elemental analysis was conducted using a Bruker X-Flash detector and Esprit 1.82 software.

2.4 Leaching Process

The leaching process was conducted in a 250 ml glass reactor equipped with a mechanical stirrer and temperature control unit. The reactor was filled with hydrochloric acid (HCl) solution, which was heated to the desired temperature upon adding 10 g/L of HP WPCBs to the reactor. Leaching experiments were performed using HCl solutions with concentrations ranging from 0.1 mol/L to 8.0 mol/L, and various time intervals up to 120 minutes were examined. Preliminary tests were conducted to find the optimal leaching conditions. The concentration of HCl that resulted in maximum dissolution was chosen for further optimization of leaching

parameters such as reaction temperature and particle size. Arrhenius plots were used to determine activation energy (E_a) and other constants. The fraction of dissolved HP WPCBs was calculated based on the initial difference in weight, measured at different time intervals up to 2 hours after filtration, washing, and oven drying at approximately 60 °C [26-28]. The mass balance, representing the disappearance of critical metals in the solution as well as the corresponding residual solid during the dissolution process, is expressed by Equation 1, where α = fraction of critical metals reacted after time, t (min.), w_1 = initial critical metals mass before leaching, w_2 = mass of undissolved critical metals after leaching.

$$\alpha = \frac{w_1 - w_2}{w_1} \quad (1)$$

The remaining material after the ideal leaching process (conducted at 80°C with 4.0 mol/L HCl for 2 hours) was examined using X-ray diffraction (XRD) and scanning electron microscopy (SEM) methods. The metallic ions in the leaching solution were identified through Inductively Coupled Plasma-Mass Spectrometry (ICP-MS) using the Agilent HP 7700 ICP-MS model and the BUCK Scientific ACCUSYS 211 Atomic Absorption Spectrophotometer.

3. Results and Discussion

3.1 Characterization

The elemental composition of the HP WPCBs was analyzed using the EDXRF technique, as shown in Table 1. The HP WPCBs primarily contain Al_2O_3 (5.3%), SiO_2 (11.3%), CuO (52.46%), Fe_2O_3 (10.8%), TiO_2 (12.8%), and minor amounts of K_2O (0.73%), CaO (0.36%), Pb (1.35%), V_2O_5 (0.28%), Cr_2O_3 (0.73%), MnO (0.36%), and ZnO (0.32%), with concentrations ranging from low to trace levels ($\leq 2\%$).

Table 1 Elemental composition of the HP E-waste computer printed circuit boards

Compound	Composition (%)
Al_2O_3	5.3
SiO_2	11.3
K_2O	0.73
CaO	0.36
Pb	1.35
TiO_2	10.8
V_2O_5	0.28
Cr_2O_3	0.73
MnO	0.36
Fe_2O_3	10.8
CuO	52.46
ZnO	0.32
Other	13.11

The XRD spectrum of the HP E-waste computer printed circuit boards (HP WPCBs) (Figure 1) consists primarily of (1) Propylene, $(C_3H_6)_n$ {54-1936} (2) Sasaite, {031-0020} (3) Silicon oxide, SiO_2 {82-1558} (4) Impure copper oxide, CuO {10-1329}.

The SEM analysis of the HP WPCBs sample, depicted in Fig. 2, revealed a diverse range of particle shapes and sizes. The particles displayed variations in size, ranging from several hundred nanometers to tens of micrometers, taking on forms such as cylindrical and thin plate-like structures. Furthermore, the sample exhibited pores and cracks that were filled with additional gangue material, while the surface appeared notably clear and crystalline. Li *et al.*, [13], Yujin *et al.*, [16] and Guo *et al.*, [20] also have similar results from their XRF, ICPMS, XRD analysis. Indicated that copper, silicon, iron and aluminum as the major phase of the material.

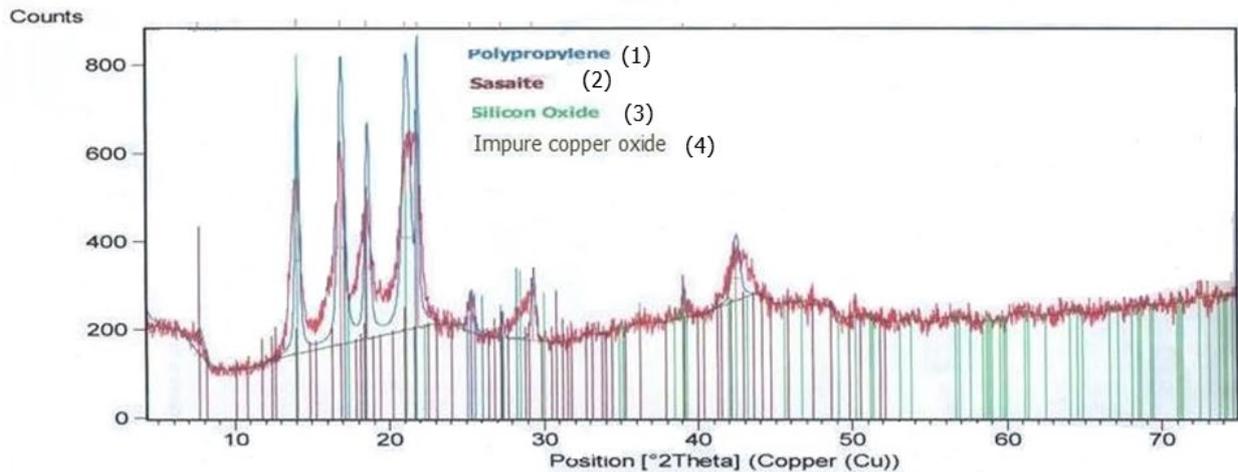


Fig. 1 XRD pattern of HP E-waste computer printed circuit boards (HP WPCBs) showing the identified compounds with their respective Joint Committee on Powder Diffraction Standard (JCPDS) file number used in peaks attribution

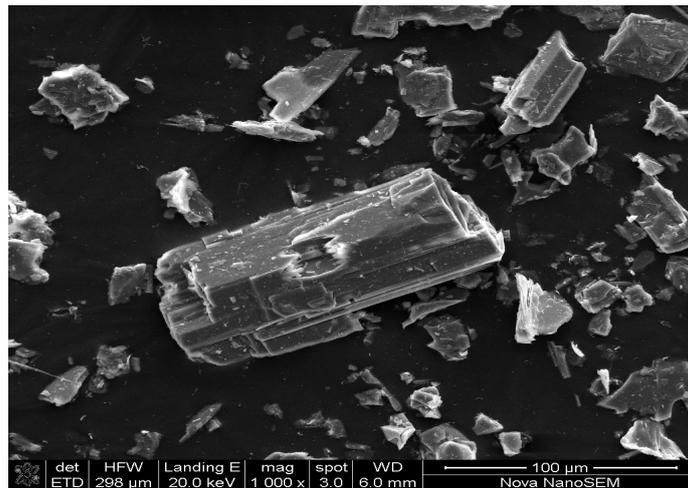


Fig. 2 SEM image of raw HP E-waste computer printed circuit boards (HP WPCBs)

3.2 Leaching Studies

3.2.1 Effect of HCl Concentration

The effect of HCl concentration (0.1–8.0 mol/L) on the HP WPCBs dissolution at 55 °C at various leaching times with moderate agitation was studied. The results, illustrated in Fig. 3, reveal a significant correlation between HCl concentration and the rate of dissolution. As the concentration of HCl increases, the dissolution rate escalates sharply during the initial leaching period and stabilizes after approximately 30 minutes, indicating a noteworthy influence of HCl concentration on HP WPCBs leaching. Notably, at a 4.0 mol/L HCl solution and 55°C, a 49.8% recovery efficiency was achieved within 120 minutes, establishing 4.0 mol/L as the optimal HCl concentration for WPCBs leaching. Further escalation of leachant concentration beyond 4.0 mol/L HCl solution resulted in decreased dissolution efficiency, emphasizing the importance of maintaining the acid concentration within this optimal range. The study also observed that the leaching efficiency of HP WPCBS exhibited a non-linear response to HCl concentration; initially rising before declining. The process of metal leaching generated hydrogen, which adsorbed onto the HP WPCBS surface, diminishing leaching efficiency. Additionally, high metal leaching efficiencies led to increased leaching solution viscosities, impeding the leaching process. Consequently, the research determined that the optimum concentration of HCl for effective WPCBs leaching was 4.0 mol/L.

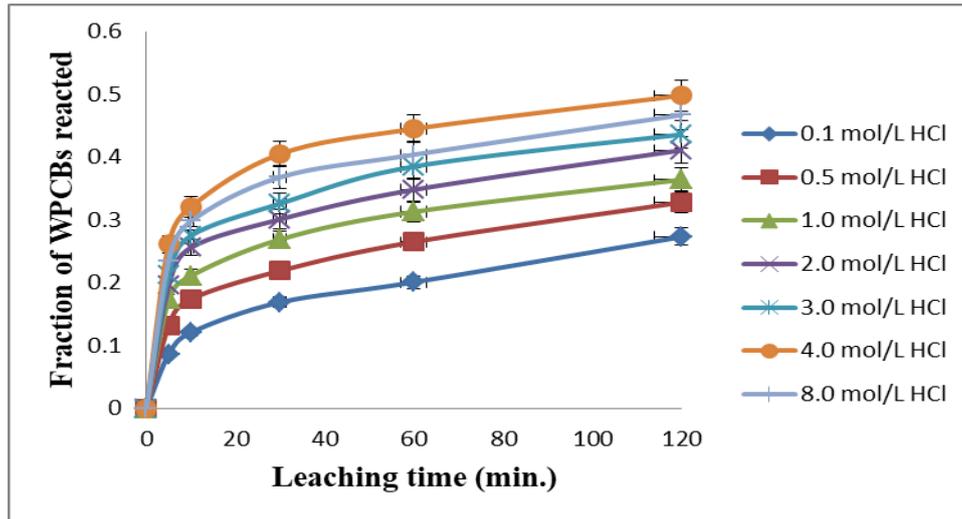


Fig. 3 Effect of HCl concentration on HP WPCBs. Experimental conditions: HCl concentration = 0.1–8.0 mol/L, Temperature = 55 °C, Particle size = -56+63 μ m, Solid/liquid ratio = 10 g/L with moderate stirring

3.2.2 Effect of Reaction Temperature

The study explored the impact of varying reaction temperatures (28–80 °C) on the dissolution of HP WPCBs. The results, illustrated in Fig. 4, clearly demonstrate the high sensitivity of leaching rates to temperature changes, with rates escalating as the temperature rises. Notably, at 80 °C, significantly higher and more consistent rates were observed, suggesting a more uniform product yield. This led to a substantial increase in the dissolved amount of HP WPCBs. Within the 120-minute leaching period, elevating the temperature from 28 to 80 °C resulted in a rise in relative dissolution output from 22.20% to 81.80%. This is directly correlated with a higher extraction rate of HP WPCBs with increasing temperature. The heightened reactivity of metals at higher temperatures facilitated the leaching of other active metals, competing with copper [30]. Consequently, 80 °C was determined as the optimum temperature for this process.

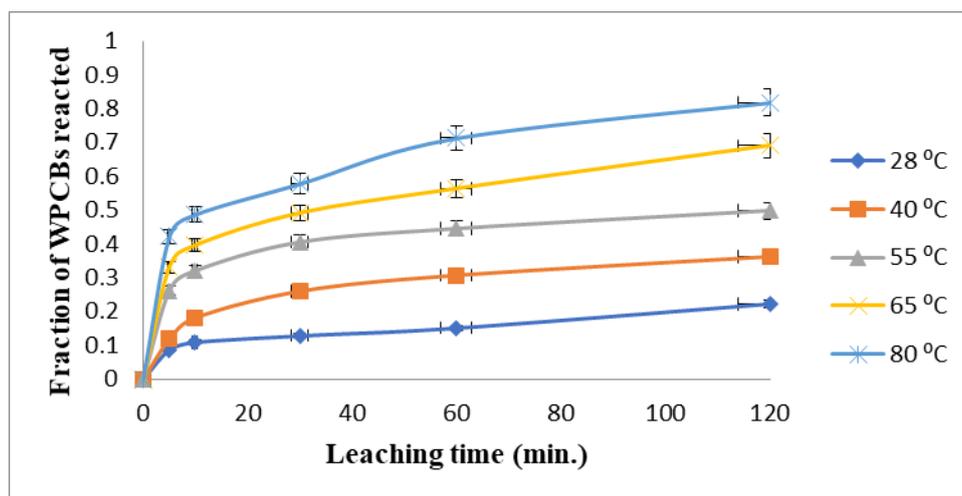


Fig. 4 Effect of temperature on HP WPCBs dissolution at various leaching times. Experimental conditions: HCl concentration = 4.0 mol/L, Temperature = 28 °C–80 °C, Particle size = -56 + 63 μ m, Solid/liquid ratio = 10 g/L with moderate stirring

3.2.3 Effect of Particle Size

The study examined how the dissolution rate of HP WPCBs varied with different particle sizes (-56+63 μ m, -75+63 μ m, -90+75 μ m) in a 4.0 mol/L HCl solution. The results, illustrated in Fig. 5, revealed that 52.12%, 69.70%, and 81.80% of critical metals reacted with particle sizes -56+63 μ m, -75+63 μ m, and -90+75 μ m, respectively, under the specified experimental conditions. This indicates that as particle size decreased, the dissolution rate of critical metals increased. Smaller particles led to enhanced liberation of metal and non-metal

materials, as depicted in Fig. 5, facilitating greater contact between the metal and leaching solution. This enhanced contact promoted the leaching of critical and precious metals [20, 30, 31].

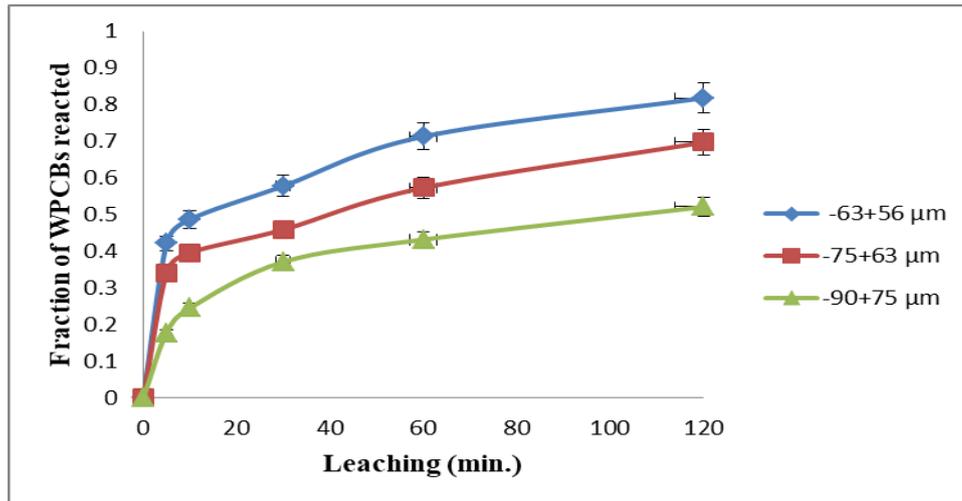


Fig. 5 Effect of particle size on hydrochloric acid HP WPCBs dissolution at various contact times. Experimental conditions: [HCl] = 4.0 mol/L, Temperature = 80 °C, Solid/liquid ratio = 10 g/L with moderate stirring

3.3 Leaching Kinetics of HP WPCBs

In the process of extracting valuable metals from HP WPCBs using hydrochloric acid, the speed of the reaction is influenced by several factors: the external movement of the leaching agent through the boundary layer, the internal diffusion within the solid layer, and the chemical reactions occurring on the particle surface. These factors jointly determine the reaction kinetics, as indicated in previous research [32]. In this study, the leaching behavior, dictated by the step with the slowest kinetics, was described using the shrinking core model (SCM) [33-35]. The equations of the SCM model were simplified to represent the rate-limiting stages of the chemical reactions, internal diffusion, and their combination, denoted as Equations (2), (3), and (4) respectively, where x is the leaching rate of HP WPCBs (%), t is leaching time (min), and k_1 , k_2 , and k_3 are rate constant (min^{-1}), respectively.

$$1 - (1 - \alpha)^{\frac{1}{3}} = k_1 t \tag{2}$$

$$1 - \frac{2}{3} \alpha - (1 - \alpha)^{2/3} = k_2 t \tag{3}$$

$$1 - (1 - \alpha)^{\frac{1}{3}} - \frac{1}{3} \ln(1 - \alpha) = k_2 t \tag{4}$$

The reaction constants at different temperatures follow the Arrhenius equation is presented as in Equation (5) [36,37].

$$k = A \exp[-E_a / (RT)] \tag{5}$$

where A is the pre-exponential factor, k is the reaction rate constant (min^{-1}), E_a is the apparent activation energy (kJ/mol), T is the thermodynamic temperature (K), and R is the molar gas constant (8.314 J/(mol·K)). Hence, Fig. 3-5 data were accordingly treated with the shrinking core model (SCM). The experimental rate constants, k_d , were determined from the slope in Fig. 6. In this particular study, it was found that the speed at which dissolution occurs is primarily influenced by the presence of a chemical on the surface, potentially resulting in a higher degree of metal release when the leachant concentration is increased. To illustrate this relationship, a graph depicting the natural logarithm of the rate constant ($\ln k_c$) against the natural logarithm of the hydrochloric acid concentration ($\ln[\text{HCl}]$) was generated and is displayed in Fig. 7. The calculated reaction order concerning HCl concentrations within the range of 0.1 mol/L to 8.0 mol/L is approximately 0.8692, which

is close to 1. This suggests that the reaction order for the examined HCl concentrations can be considered a first-order relationship.

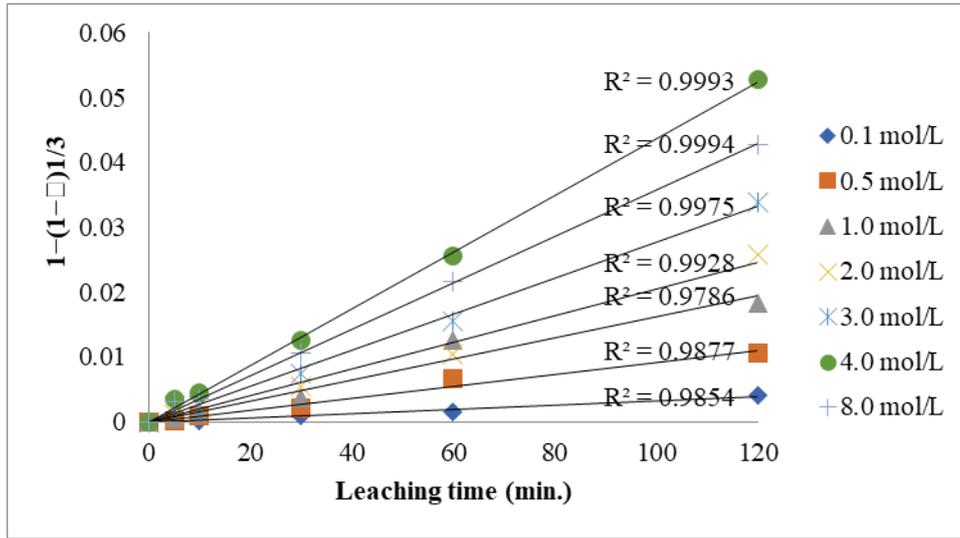


Fig. 6 Plot of $1-(1-\alpha)^{1/3}$ versus leaching time at different HCl concentrations (Experimental conditions: Same as in Fig. 3)

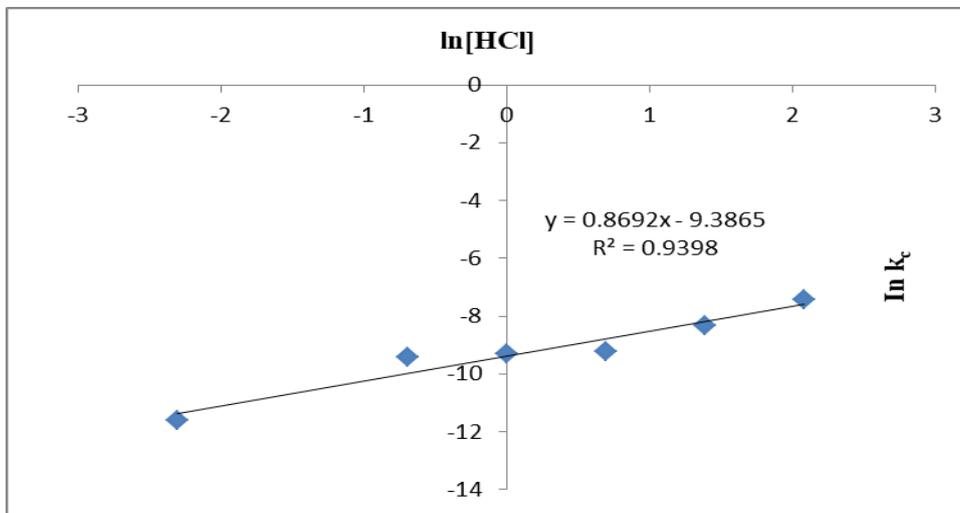


Fig. 7 Plot of $\ln kc$ versus $\ln[HCl]$

The experimental results, when analyzed using Equation (2), demonstrate a strong correlation coefficient ($R^2=0.9398$). This suggests that the leaching process of critical metals from HP WPCBs is primarily governed by chemical reactions.

It can be seen from Figure 8 that when $1 - (1-\alpha)^{1/3}$ is plotted as a function of leaching time t at different temperatures, $R^2 > 0.9$, as shown in Table 2. As shown in Fig. 9, the activation energy E_a of the chemical-reaction-controlled step shown in Equation (5) is 55.79 kJ/mol (> 42 kJ/mol), thereby proving that the HP WPCBs leaching process is controlled by chemical reactions [16, 24]. In summary, the kinetic equation for HP WPCBs leaching is formulated as Equation (6):

$$1 - (1-\alpha)^{1/3} = \exp\left(12.032 - \frac{6710.7x}{T}\right) t \tag{6}$$

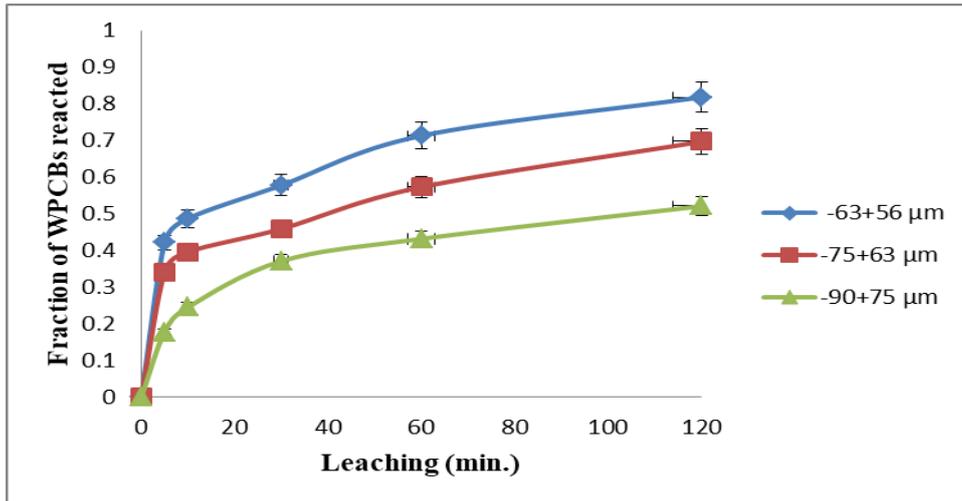


Fig. 8 Plot of $1-(1-\infty)^{1/3}$ versus leaching time at different temperatures (Experimental conditions: Same as in Fig. 4)

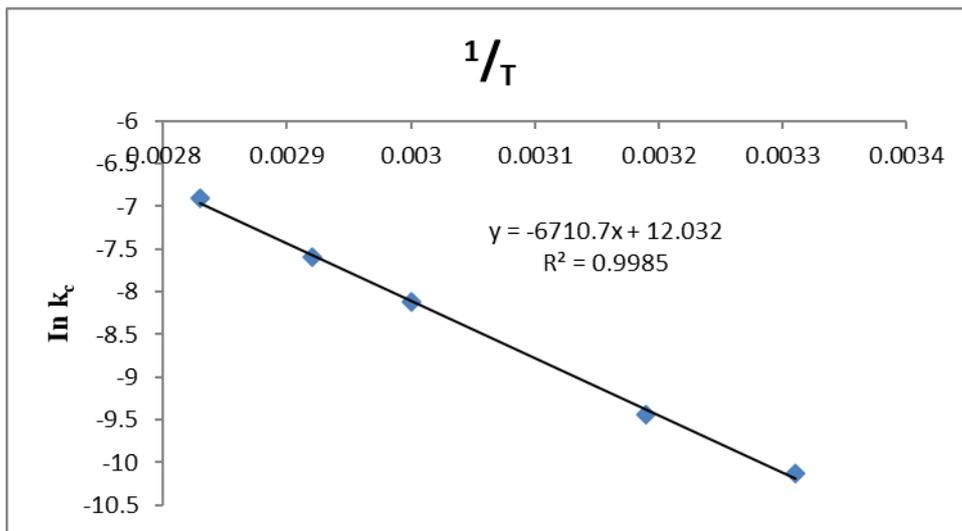


Fig. 9 Arrhenius plot for HP WPCBs leaching in chemical reaction control

Table 2 Reaction rate constants at different temperatures

Temperature (°C)	k/min ⁻¹	R ²
28	0.000456	0.9879
40	0.000812	0.9777
55	0.003970	0.9806
65	0.005010	0.9701
80	0.016890	0.9960

3.4 Residual Product Analysis

After the leaching process was optimized, XRD analysis of the residual product revealed the presence of impurities such as quartz (SiO₂) {65-0466}, polypropylene (C₃H₆)_n {54-1936}, and graphite (C) {74-2328}, as shown in Fig. 10. These impurities, along with the leached metals, were removed from the leach liquor. Additionally, SEM images of the leached residue obtained under optimal leaching conditions, depicted in Fig. 11, displayed scattered and eroded surface grains. These images indicate the successful leaching of critical metals from the surface of HP WPCBs.

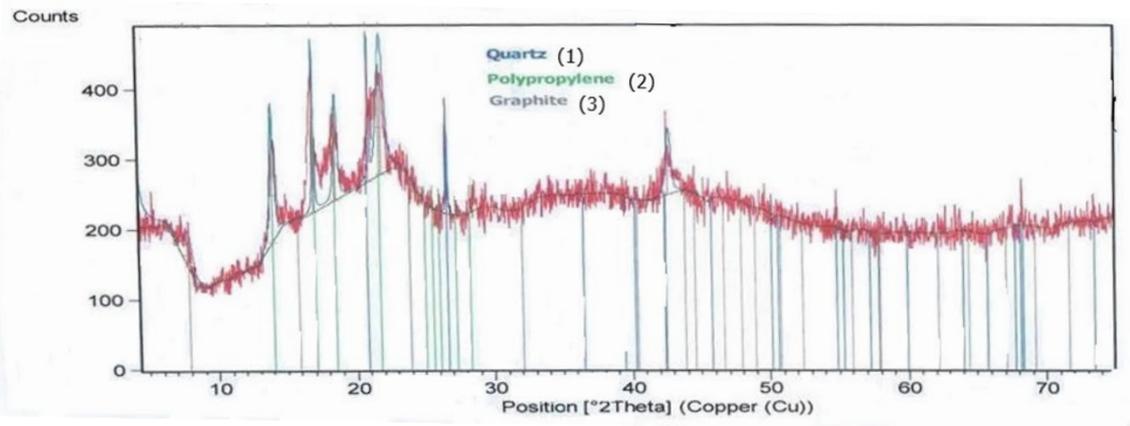


Fig. 10 XRD pattern of leached residual products showing the identified compounds with their respective Joint Committee on Powder Diffraction Standard (JCPDS) file number used in peaks attribution

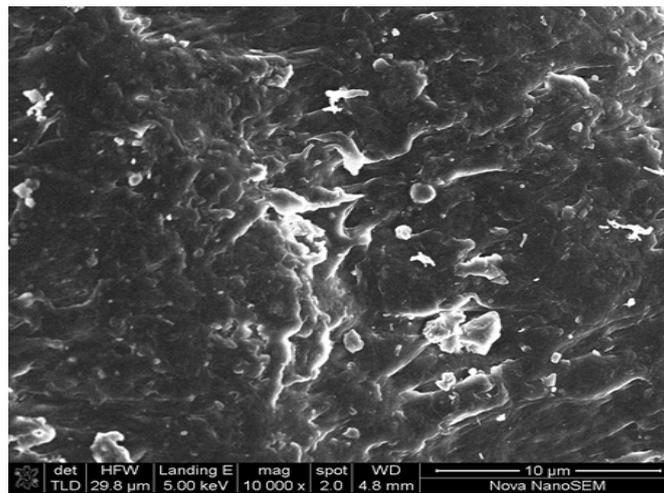


Fig. 11 SEM image of the hydrochloric acid leached product

4. Conclusion

Hydrochloric acid was evaluated for its effectiveness in leaching critical metals from E-waste HP printed circuit boards. The study analyzed the impact of hydrochloric acid concentration, reaction temperature, and particle size on the dissolution rate. A detailed characterization of the raw E-waste HP printed circuit boards was compared with the leached products obtained under optimal conditions. The dissolution kinetics of critical metals increased with higher hydrochloric acid concentration, elevated reaction temperature, and reduced particle size. Under specific experimental conditions, a dissolution rate of 81.8% was achieved using 4.0 mol/L HCl within 120 minutes at 80 °C. Additionally, analysis of the dissolution data revealed that the process followed a chemical control mechanism.

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Conflict of Interest

Authors declare that there is no conflict of interests regarding the publication of the paper.

Author Contribution

The authors confirm contribution to the paper as follows: **study conception and design:** Akor, Joel. E & Balogun, Ayo. F; **data collection:** Balogun, Ayo. F; **analysis and interpretation of results:** Balogun, Ayo. F; **draft manuscript preparation:** Akor, Joel. E & Balogun, Ayo. F. All authors reviewed the results and approved the final version of the manuscript.

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