

RESEARCH PAPER

# Advanced Approaches to Acid Leaching Optimization of Copper from Printed Circuit Board Wastewater Sludge

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## Abstract

The most challenging issue associated with recycling the sludge generated from printed circuit boards (PCBs) is the separation of copper (Cu) using different leaching methods. The aim is to mitigate environmental impacts while effectively reclaiming valuable resources. The goal was to enhance the acid leaching process by carefully adjusting sulfuric acid concentrations, liquid-to-solid ratios, and leaching periods to maximize copper extraction from PCB waste. The research utilized an innovative approach that integrated a factorial experimental design with multiple linear regression (MLR) and sophisticated analysis of variance (ANOVA) to thoroughly evaluate the intricate interactions among these essential components. Furthermore, the study implemented predictive analytics to enhance both accuracy and efficiency, while also refining process parameters. Therefore, we found that mean square error (MSE) for MLR is better than the factorial design (FD) and has a good result. The results were impressive, exhibiting considerable improvements in leaching efficacy with an R-squared ( $R^2$ ) value of 87.58%, an adjusted R-squared of 84.33%, and a minimal MSE of 1.02 highlighting the strength of the reanalysis. MLR not only improved predicted accuracy but also provide deeper statistical formal validation technique. Future research areas include applying these enhanced procedures to a larger spectrum of e-waste materials and diving deeper into the environmental effects of leachate byproducts. The study also advises the incorporation of machine learning models to further optimize leaching operations in real-time, potentially revolutionizing recycling procedures in the electronics industry and driving a shift towards a more sustainable and economically practical circular economy.

**Keywords:** Machine Learning in Industrial Processes; Sustainability in Recycling; Factorial Experimental Design; Sulfuric Acid Optimization; Acid Leaching; Electronic Waste.

## 1. Introduction

### 1.1 Background

The advancement of the technologies and the development of society, the lifespan of electronic and electrical equipment has been foreshortened. This can increase the number of e-wastes all over the world. In the developing world, this problem creates both environmentally and economically losses, like Environmental risk due to toxic e-waste particles and economically the loss of valuable recoverable metals. So, PCBs contain abundant high-value metals (e.g., gold, silver, copper, etc.). Improper PCB disposal not only causes the loss of precious resources but also creates major environmental problems from the toxic chemicals let free during inappropriate disposal techniques [1]. Additionally,

environmental and sustainability concerns are also causing growing attention on the conventional metals recycling technologies such as pyrometallurgical and hydrometallurgical operations. Moreover, these antiquated technologies often use a lot of energy and may release hazardous chemicals, which underlines the demand for more efficient and ecofriendly and sustainable alternatives [2]. Recent technical advances are making innovations in the various acid leaching processes possible, which is one of the possible solutions for enhancing the efficiency of copper recovery from e-waste. Well known for its low environmental effect and great recovery rate potential is acid leaching, or the application of acidic solutions to extract metals from solid waste. This method also calls for meticulous parameter modification to boost recycling efficiency and reduce environmental effect [3]. Additionally, variables including acid concentration, liquid-to-solid ratio, and leaching time control leach efficiency; yet, this requires some work on how these variables interact and what the ideal circumstances are. These issues are successfully resolved by the factorial experimental design, which allows for the simultaneous investigation of multiple components and their interactions. Furthermore, by using this Innovative experimental design, scientists may identify optimum conditions and understand how each part is mostly affects the leaching process [4]. Furthermore, a substantial research gap exists regarding the combined effects of environmental factors like pH and temperature, which may primarily affect the kinetics and thermodynamics of the leaching reactions [5]. By integrating these extra environmental factors into a comprehensive factorial experimental design, the current study aids in resolving the problems that arose in the earlier research. Along with learning more about the acid leaching process, it also aims to offer a more realistic model for optimizing copper recovery from PCBs. Additionally, this study aims to establish a solid basis for future environmental and technological initiatives and significantly advance the field of sustainable e-waste management.

## 1.2 Literature Review

Copper's high electrical conductivity makes it widely usable. The high demand for electronic products in modern life has rapidly expanded the production of printed circuit boards (PCBs), resulting in the release of a massive amount of unwanted solid and liquid waste containing hazardous materials. Additionally, temperature also a major cause of creating an environmental issues due to e-waste like hot areas with in the Kingdom of Saudi Arabia (KSA) [6], there is a proper need to dispose the e-waste with in time limit. Furthermore, processing e-waste using outdated techniques like metallurgical treatment is insufficient [7]. Furthermore, one another article shows 93.56% of leaching efficiency using an ionic liquid (IL) and HO system in a closed-loop leaching process for copper recovery from WPCBs [8]. On the other hand, bioleaching optimization using *Acidithiobacillus thiooxidans* and achieved an 85% coper recovery efficiency [9]. So, acid leaching is one of the good alternatives for the recovery of copper because of its high efficiency and lesser environmental impact when compared to more traditional procedures such as melting and direct landfilling. Some of the important acids like Sulfuric acid ( $H_2SO_4$ ), Hydrochloric acid (HF) and nitric acid ( $HNO_3$ ) are frequently employed in these operations, with varied degrees of efficacy depending on the material treated and operating conditions [4]. Furthermore, the most recent research articles are adjusting pH levels, and varying temperature settings to optimize the leaching process [10]. FD is commonly employed to understand the complex interactions between multiple variables in industrial processes. This statistical approach allows researchers to systematically analyze the effects of several factors at the same time and discover the ideal conditions for desired results. Acid leaching depends on variables such as acid content, liquid-to-solid ratio, and leaching duration [11]. These studies often reveal that no single factor controls the efficiency of the leaching process; rather, it is the interaction between factors that plays a significant role.

After a wide overview of current research articles, which are discussing the different copper recovery processes such as chemical leaching, bioleaching, and advanced hydrometallurgical processes. Furthermore, major finding from 2021 to 2024, which is highlighting notable developments such as

the utilization of citric acid, acetic acid, and hydrogen peroxide combinations to enhance leaching efficiency, and new closed-loop systems attaining up to 93.56% leaching rates. Additionally, one of the noticeable facts is that methods like organic chemical leaching, bioleaching with *Penicillium simplicissimum*, and the use of environmentally friendly materials like metal-organic frameworks (MOFs) highlight the shift towards more sustainable and efficient recovery techniques. All the current studies explaining the major environmental and industrial concerns by demonstrating the combination of innovative materials, experimental methods, and analytical models to push the boundaries of copper recovery technology (Table 1).

### **1.3 Research Motivation and Contribution**

The need for this study is motivated by the emerging need to address environmental concerns connected with the disposal of PCBs, which account for a large portion of electronic waste. PCBs contain important metals such as copper, which is disposed to contribute not only to the loss of natural resources but also to environmental deterioration through harmful leachate [25]. Conventional copper recovery technologies, such as pyrometallurgical procedures, are energy-intensive and environmentally harmful [26]. Acid leaching is an appealing choice because of its potential for enhanced efficiency and lower environmental impact [27]. Furthermore, leaching technique for the e-waste from PCBs is poorly optimized, which is particularly in terms of acid concentration, liquid-to-solid ratios, and leaching times, limiting its efficiency [28]. Therefore, current research is mostly motivated by the desire to improve all these parameters to optimize copper recovery while minimizing environmental and economic costs as well. Furthermore, this study uses an innovative FD to investigate copper recovery from PCB sludge. FD using a 33 complete FD, which finds out the relationship between acid content, liquid-to-solid ratio, and leaching time. This technique not only improves our understanding of the individual and combined effects of these elements, but it also determines the best circumstances for optimum leaching efficiency. Thus, using the desirability function included into RStudio software, the study evaluates the results' practical applicability, ensuring that the improved parameters are not only statistically significant but also feasible in industrial settings. This improves the scalability of acid leaching processes, increasing their use in large-scale operations. It also streamlined the leaching process; this study helps to reduce the environmental impact of metal recovery from e-waste. The findings provide a pathway to more sustainable practices in the electronics recycling business, harmonizing with global efforts to reduce e-waste and improve resource recovery.

### **1.4 Research Objectives**

This work systematically analyzes novel strategies for optimizing copper extraction from PCB sludge, focusing on three critical goals. Specifically, the study aims (i) Optimization of Leaching Conditions: The primary goal is to optimize the acid leaching process for extracting copper from PCB sludge by investigating the effects of three key variables: acid concentration, liquid-to-solid ratio, and leaching time; and (ii) Statistical Analysis Using Factorial Design: To systematically evaluate the interactions among the three variables and determine their combined impact on the efficiency of copper leaching, utilizing a full factorial experimental design; and (iii) Model Validation with Desirability Function: Assess the compatibility of the optimized conditions with the desired outcomes by employing the desirability function in Minitab software, ensuring the model's effectiveness and applicability in real-world scenarios. Additionally, after achieving these goals this technique not only improves our understanding of the individual and combined effects of FD and MLR, but it also determines the best circumstances for optimum leaching efficiency. Furthermore, it also contributes to offer scalable insights for advancing global e-waste recycling and circular economy initiatives.

**Table 1:** Brief overview of relevant research.

Study focus	Key findings	Method used	Methodology	Year	Reference
Copper leaching from waste PCB	The leaching efficiency of copper has been improved by utilizing a combination of citric acid, acetic acid, and hydrogen peroxide	Organic chemical leaching	Hydrometallurgy	2023	[12]
Innovative closed loop copper recovery	The results indicate that the ILsH <sub>2</sub> O <sub>2</sub> system effectively leaches copper, reaching a maximum leaching rate of 93.56%	Chemical leaching	Hydrometallurgy	2024	[8]
Metal recovery from waste PCB through bioleaching and biosorption techniques	Bioleaching is effective for extracting Cu, Ni, and Zn from WPCBs, while biosorption is more suitable for recovering Au	Bioleaching and biosorption	Hydrometallurgy	2024	[13]
Metal leaching from PCB by using Penicillium simplicissimum	Key finding shows the mechanism for leaching aluminum and nickel was acidolysis, whereas the main mechanism for copper leaching was complexolysis mechanism	Bioleaching ANOVA	Hydrometallurgy	2022	[14]
Eco-friendly recovery of metal from PCB	In, lead, and copper are extracted in sequence as tin oxide (solid), lead nitrate (solid), and copper nitrate (aqueous), achieving efficiencies of 77%–97%, 51%–85%, and 100%, respectively	Chemical leaching	Hydrometallurgy	2021	[15]
Targeted extraction of copper from electroplating sludge	The copper leaching efficiency reached 85% after 5 hours when using 70 mM EDTA, a liquid-to-solid ratio of 120:1, and 10 mM citric acid	Chemical leaching	Hydrometallurgy	2022	[16]
Biological leaching of sewage sludge for copper recovery	Under optimal conditions of S/L = 0.16% (w/v), S = 8.2 g/L, and pH 1.4, the maximum copper recovery achieved was 85.3%	Bioleaching ANOVA	Hydrometallurgy	2024	[9]
Extraction of Cd, Cu, Ni, and Zn from soil amended with sewage sludge	The findings indicated that incorporating SS reduced the leaching of Cd and Cu, while it had no impact on Ni and Zn leaching. Additionally, the presence of NPs and zeolite was effective in minimizing heavy metal leaching	Chemical leaching	Hydrometallurgy	2021	[17]
Co-treatment of electroplating sludge, copper slag, and spent cathode carbon	Under optimal conditions, the recovery ratios for Cr, Ni, and Cu were 75.56 wt%, 98.41 wt%, and 99.25 wt%, respectively, showing improvements of 40%, 5%, and 5% compared to the method currently in use	Solidification and leaching	Solidification and leaching	2021	[18]

Copper recovery from electroplating sludge	Under optimal experimental conditions, 96.4% of Cu <sup>2+</sup> was extracted from the electroplating sludge, with 65.4% being recovered in the form of foil	Electrodialysis and electrodeposition	Electrodialysis and electrodeposition	2023	[19]
Investigating battery black mass leaching performance	The model demonstrated that leaching temperature was the most influential factor and revealed greater copper reduction efficiency compared to using equal molar amounts of H <sub>2</sub> O <sub>2</sub>	Chemical leaching DOE Regression	Hydrometallurgy	2024	[20]
Oxidative-reductive leaching	The extraction of copper was influenced by experimental conditions that promoted the reduction of chalcopyrite while facilitating the oxidation of chalcocite	Leaching Factorial design	Hydrometallurgy	2022	[21]
Characteristics of pore structure and the leaching mechanism of Cu and Cr in sustainable porous ceramsite	The leaching of copper and chromium exhibited a positive correlation with the effective pore volume of pores greater than 1 μm, while showing a negative correlation with the fractal dimension	Leaching	Hydrometallurgy	2024	[22]
Decomplexation of copper compounds from synthetic and actual electroplating wastewater	The Fe <sub>3</sub> O <sub>4</sub> coating decreased the copper leaching concentration in the TCLP test, reducing it from 272.0 mg/L for CuO to 80.1 mg/L for CuO@Fe <sub>3</sub> O <sub>4</sub>	Decomplexation	Pyrometallurgy and hydrometallurgy	2024	[23]
Extraction of cupric oxide from copper-rich wastewater sludge	Copper extraction surpassed 90% within 30 minutes, and the oil residue was eliminated from the sludge	Chemical leaching	Hydrometallurgy	2012	[24]

**2. Materials and Methods**

A scientific framework for this research is to recover the copper from the PCBs sludge by using the acid leaching approach. Furthermore, this approach starts with experimental stages, which include sludge characterization, acid-leaching experiments, and experimental design. Additionally, all these factors are collectively set the stage for defining the Research Objectives of the study. Also, this method further explains the framework through the Materials and Methods section, which covers the sludge qualities, and the specific acid-leaching experiments performed. Thus, followed by a comprehensive overview of the experimental approach, which includes the use of FD and MLR to investigate the effects of different treatments and levels. FD setup and MLR analysis used to evaluate the experimental data. Subsequent sections, such as ANOVA Results, interaction graphs, and Residual Analysis, provide a quantitative and qualitative evaluation of the treatments, demonstrating the research findings’ robustness (Figure 1). This systematic strategy not only ensures a thorough analysis of the research issue but also lays the groundwork for future studies to build on the findings presented.

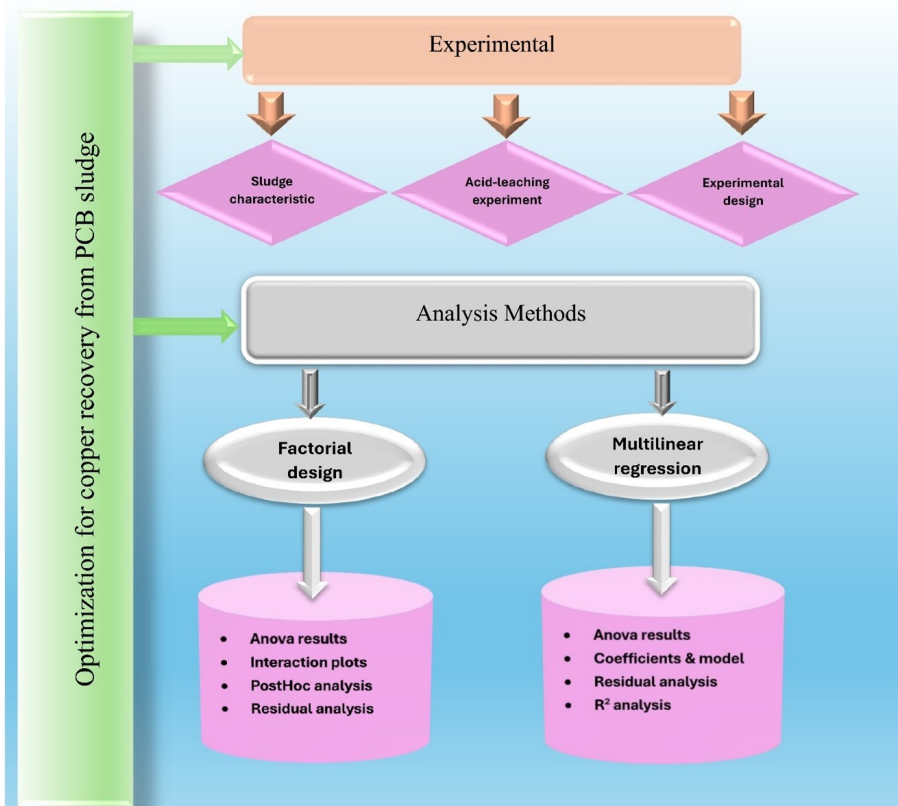


Figure 1: Methodological frameworks of the current study.

**2.1 Sludge Characteristics**

After alkaline precipitation has completed then dewatered sludge had obtained from a PCB facility in Thailand’s wastewater treatment plant for further processing of sludge. After air drying and

grinding to achieve homogenization, the sludge was run through a 10-mesh (2.0 mm) screen for sieve analysis. The sludge was sieved to obtain particle size  $<9.5$  mm and  $<150\mu$  m, to ensure that the sludge material has uniformity for subsequent procedures of experimental design [29]. And then stored prior to the experiment at room temperature until utilization [29], [30]. A glass electrode was used to measure the sludge's pH and moisture content in a 1:5 (W/V) suspension of sludge in water that has been distilled [31]. A 1.0 g dry sludge sample was digested using a solution of  $\text{HNO}_3$  and  $\text{H}_2\text{SO}_4$  in according to the method described by Rice et al., section 3030 G, in order to determine the copper concentration [30]. To further analyze the composition of sludges, samples were designed as aqua regia ( $\text{HCl} : \text{HNO}_3 = 1 : 3$ ), to ensure heavy metal comprehensive determination such as Cu, Ni, Pb, and Zn, [32]. The design filtrate was measured using inductively Coupled Plasma (ICP) spectrometry to quantify metal concentration with precision, conducting measurements of triplicate for showing data reliability [33]. After digestion the suspensions were cooled, and filtered, and used a Cu analysis technique by Flame Atomic Absorption Spectroscopy (Flame AAS, Perkin Elmer, Model AAAnalyst200). The initial copper concentration (CI) on a wet weight basis was calculated by using Equation (1) to the quantity of acid extractable copper, or CAE, in the sludge. In addition, microwave-assisted acid extraction techniques were conducted as a complementary method to enhance copper recovering from sludges [34]. Experimental parameters like concentration of acid, sludge to liquid proportion, and microwave power were varied to optimize heavy metal extraction efficiency. To confirm copper as the principal metal in the sludge, an X-Ray Fluorescence Spectrometer (Brüker SRS 3400, Germany) captured the sludge's XRF spectra. For mineralogical investigation of the sludge structure, crystallinity was analyzed using X-Ray Diffraction (XRD). This approach provided critical insights into the phases present within the raw sludge, complementing the elemental analysis. The following equation 1 was applied to determine the Acid- extractable copper concentration in the sludge (mg/g). Whereas CAE is the amount of acid- extractable copper concentration in equation 1, which accounts for measurement of the copper concentration(A), correction of blank (B), volume of acid (C), and sample weight (W).

$$C_{AE} \left( \frac{\text{mg}}{\text{g}} \right) = \frac{(A - B) \times C}{W} \quad (1)$$

## 2.2 Acid Leaching Experiments

Copper extraction from the sludge was investigated using a batch leaching approach for reliability. In each experiment, approximately 10 mg of dry wastewater sludge sample was added to flasks containing a predetermined volume of acidic solution, adjusted for achieving the required liquid-to-solid (L/S) ratio of the sludge. The mixture was agitated in an incubated rotary shaker at 200 rpm and maintained at  $60^\circ\text{C}$  for a specified duration of time to achieve the final desirable ratio. The effectiveness of acid leaching is impacted by the Ph of the waste water sludge and the partition characteristics of heavy metals, as lower Ph quantity leading the dissolution of heavy metal hydroxides and exchange reactions with proton in the mixture [35]. After the leaching time, all suspensions were centrifuged at 3000 rpm for 5 minutes of time and filtered through Whatman 42 filter paper. The copper concentration in the filtrate was then examined using Flame Atomic Absorption Spectroscopy (FAAS) [36]. The leaching efficiency of copper (Y) was calculated applying the following equation, whereas the (Y) is the percentage of copper that leached, (CI) is the initial concentration of copper in the sludge, as expressed in the equation 2 [31].

$$Y = \left( \frac{C_L}{C_I} \right) \times 100 \quad (2)$$

Before conducting further experiments, the leaching efficiency of three acids—sulfuric acid ( $\text{H}_2\text{SO}_4$ ), hydrochloric acid (HCl), and nitric acid ( $\text{HNO}_3$ ) was compared to one another to ensure

the process efficiency. The acid presenting the highest copper-leaching efficiency was selected as the optimal extracted agent. High concentrations of acid not only improve copper leaching effectiveness but also influences the fate of other heavy metals as well, for instance Ni, Pb, and Zn, through dissolution processes [35]. Further, three different concentrations of the selected acid were analyzed as part of the experimental design to optimize the leaching process for achieving the desire value of copper from sludge. Therefore, the experimental ranges and levels for essential parameters in optimizing acid leaching for copper recovery from printed circuit boards. The table divides three key variables into three categories based on stated ranges: low, center, and high. The variable 'A' denotes the concentration of sulfuric acid, which ranges from 0.2 to 1.0 M. Variable 'B' describes the liquid-to-solid (L/S) ratio, which ranges from 10:1 at the lowest level to 60:1 at the midpoint and 100:1 at the highest level. Finally, variable 'C' covers the leaching times, starting from 5 minutes at the low end, 40 minutes at the middle point, and reaching up to 80 minutes at the highest setting. These parameters were meticulously chosen to explore their influence on the efficiency of copper leaching under controlled experimental conditions (Table 2).

**Table 2:** *Experimental ranges and level of experimental factors.*

Coded variables	Description	Experimental field		
		Low (1)	Central point (0)	High (+1)
A	Concentrations of sulfuric acid (M)	0.2	0.6	1.0
B	L/S ratio (mL/g)	10:1	60:1	100:1
C	Leaching times (min.)	5	40	80

### 2.3 Data Pre-processing

The data presented in this table was extracted from a study that explores various variables (Y, A, B, and C) under different conditions [31]. In this data set Y is the response variable, which is used as a dependent variable being analyzed. The remaining columns, labeled A, B, and C, serve as the predictor or independent variables, which are the factors influencing or explaining the variation in the response. This dataset captures the interplay between the different factors, offering insights into the study's objectives and contributing to the analysis (Table 3).

#### 2.3.1 Test Assumptions; Residual Analysis for FD

**Constant variance:** The residuals should be randomly distributed around the horizontal zero line. This indicates that the ANOVA model is appropriate and does not have systematic error patterns (Figure 2).

**Formal Test:** The Breusch-Pagan test is used to discover heteroscedasticity in a regression model, which arises when the residual variance does not match across the range of explanatory variables. In the reported results, the test statistics are 33.332 with 26 degrees of freedom, yielding a p-value of 0.1527. This p-value exceeds the conventional significance threshold (e.g., 0.05), indicating that there is insufficient evidence to reject the null hypothesis of homoscedasticity. As a result, we conclude that the variance of the residuals remains nearly constant across varied predictor values. This suggests that the fundamental assumptions required for effective linear regression analysis are met, allowing for credible statistical inference based on the model's outputs.

**Normality:** The plot you provided is a Normal Q-Q (Quantile-Quantile) Plot. This type of plot is used to compare the distribution of your sample data to a theoretical normal distribution. If the data follows a normal distribution, the points should lie approximately along the blue diagonal line (Figure 3).

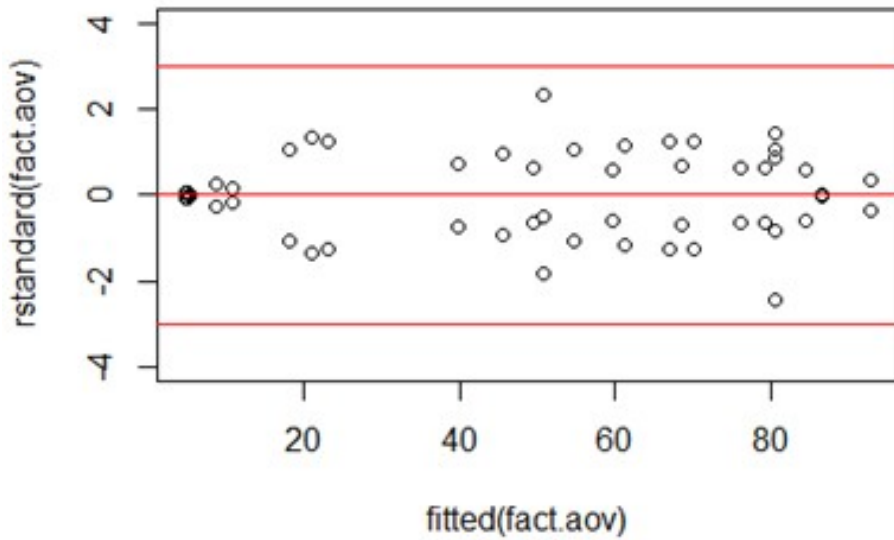


Figure 2: Constant variance plot.

### Normal Q-Q Plot

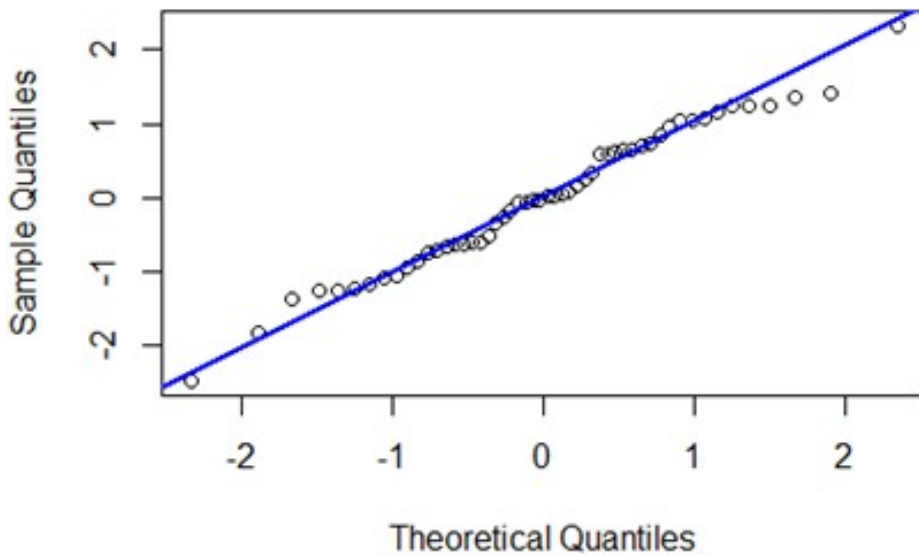


Figure 3: Q-Q plot for normality.

**Formal Test:** The Shapiro-Wilk Normality Test is a commonly used method for determining the normality of data distribution. The  $W$  statistic value for this test is 0.9843, which corresponds to a  $p$ -value of 0.72. The  $p$ -value is much higher than the customary threshold (e.g., 0.05), indicating that there is insufficient evidence to reject the null hypothesis that the data has a normal distribution.

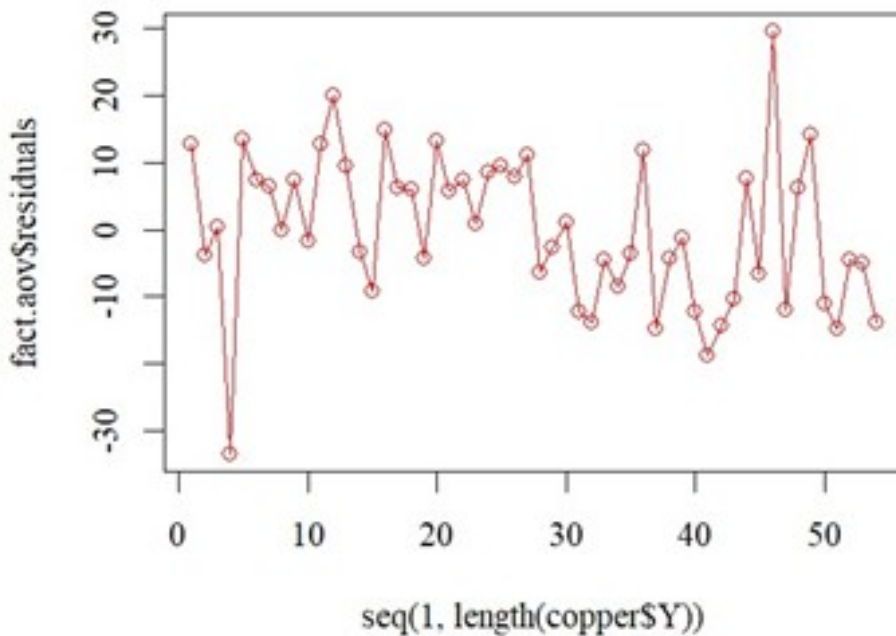
As a result, we can assume that the data follow a normal distribution. This conclusion is critical because many statistical tests and procedures rely on normalcy to get correct and meaningful results. Thus, the test results support the use of parametric approaches that rely on the normal assumption.

**Table 3:** *Data used in this study.*

Y	A	B	C	Y	A	B	C
55.09	-1	0	0	35.86	-1	0	0
4.47	-1	-1	0	5.74	-1	-1	0
5.09	-1	-1	1	5.76	-1	-1	1
51.79	1	1	1	35.62	1	-1	1
34.63	0	-1	0	7.35	0	-1	0
65.68	0	0	-1	53.61	0	0	-1
47.51	1	-1	0	32.39	1	-1	0
12.64	-1	1	-1	9.08	-1	1	-1
92.72	1	1	1	97.14	1	1	1
82.6	1	0	-1	69.6	1	0	-1
88.96	1	0	0	71.8	1	0	0
65.39	-1	1	0	44.18	-1	1	0
29	0	-1	-1	7.31	0	-1	-1
44.79	1	-1	-1	29.5	1	-1	-1
11.5	-1	0	-1	6.42	-1	0	-1
79.71	0	0	0	54.43	0	0	0
4.16	-1	-1	-1	5.6	-1	-1	-1
55.94	-1	0	1	43.28	-1	0	1
85.38	1	1	-1	77.8	1	-1	-1
35.93	0	-1	1	10.71	0	-1	1
86.27	1	1	0	86.8	1	1	0
89.23	1	0	1	96.11	1	0	1
90.54	0	1	1	78.49	0	1	1
72.93	-1	1	1	49.56	-1	1	1
75.51	0	1	-1	61.34	0	1	-1
85.72	0	1	0	72.87	0	1	0
82.65	0	0	1	57.67	0	0	1

**Independence:** Random Distribution of Residuals: Ideally, residuals should be randomly distributed around the zero line on the y-axis. This indicates that the model is fitting the data well and there are no systematic errors (Figure 4).

**Formal test:** The Durbin-Watson test, applied here, evaluates the presence of autocorrelation at lag 1 in the residuals of a regression model. The obtained Durbin-Watson statistic is 1.775, with a p-value of 0.592. This statistic is close to 2, suggesting minimal evidence of first-order autocorrelation. Since the p-value significantly exceeds the common significance level (e.g., 0.05), it further indicates that there is no statistically significant autocorrelation present. Therefore, the residuals can be considered independent, upholding one of the crucial assumptions necessary for the reliability of linear regression analysis.



**Figure 4:** *Data Independence plot.*

### 2.3.2 Residual Analysis for MLR

The normality of residuals, a critical assumption in regression analysis, was assessed using both informal and formal methods. The Q-Q plot (Figure 5) demonstrates that the residuals align closely with the reference line, indicating approximate normality with minor deviations at the extremes, which are not substantial enough to impact the analysis. The Shapiro-Wilk test further supports this observation, with a p-value of 0.6975, far exceeding the typical significance threshold of 0.05. This result confirms that the null hypothesis of normal residuals cannot be rejected. Together, the visual and statistical analyses validate the assumption of normality, ensuring the reliability of the regression model's inferences.

The homoscedasticity assumption, which requires constant variance of residuals, was assessed using both informal and formal methods. The residuals vs. fitted values plot (Figure 6) provides an informal visual examination of variance constancy. In this plot, the residuals appear randomly scattered without a discernible pattern, suggesting no significant heteroscedasticity. However, there are minor deviations that necessitate formal confirmation. The Breusch-Pagan test, a formal statistical method for testing homoscedasticity, yielded a p-value of 0.04355. Since the p-value is less than the commonly used significance level of 0.05, the null hypothesis of constant variance is rejected. This result indicates the presence of heteroscedasticity in the residuals, despite the relatively minor deviations observed in the visual inspection. These findings suggest that the assumption of constant variance is violated, and remedial measures such as weighted least squares, or robust standard errors may be required to address this issue.

Assessing a residual independence Durbin Watson and a time series plot were used, which is the important assumption for the precise regression analysis. The Durbin-Watson test investigates the presence of autocorrelation in the residuals. A p-value of 0.3948, which is more than the standard significance threshold of 0.05. Therefore, there is insufficient evidence to reject the null hypothesis of no autocorrelation, suggesting that the residuals are independent. Furthermore, the time series

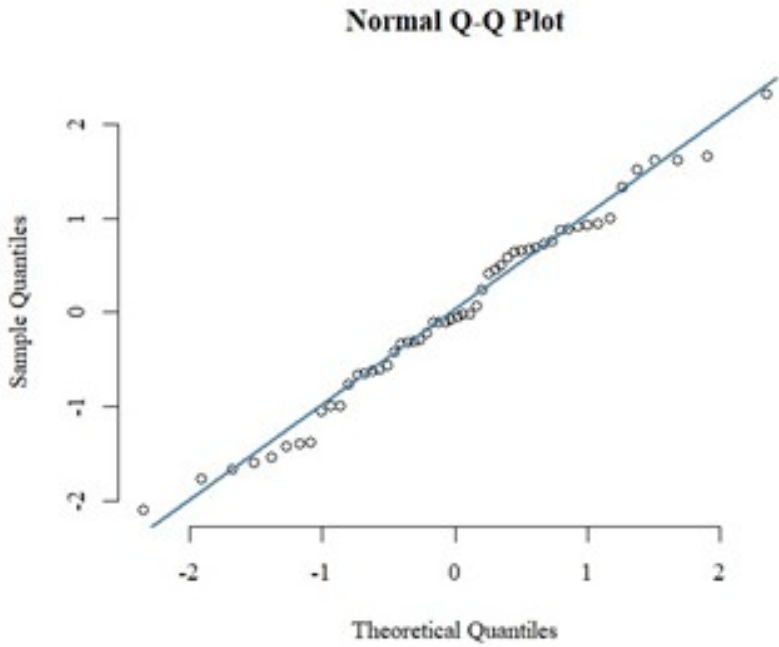


Figure 5: Normal Q-Q Plot for Residuals.

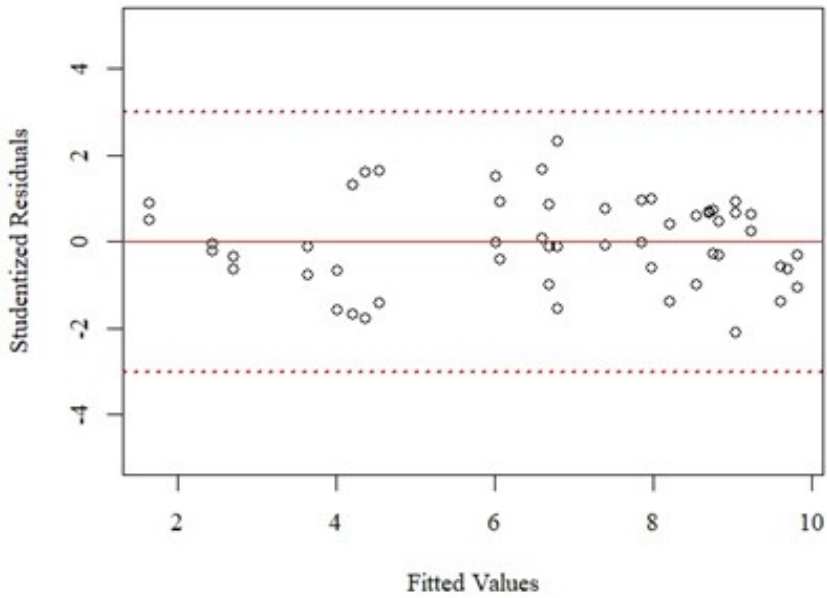
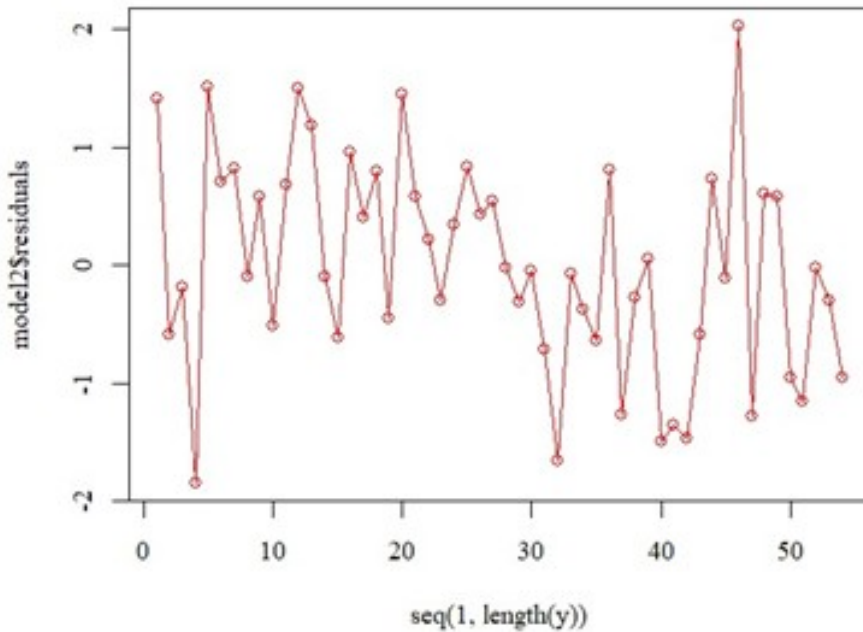


Figure 6: Residuals versus fitted values plot. Residuals versus fitted values plot.

plot of residuals (Figure 7) represents a visual inspection of independence. The residuals are randomly distributed across the data, with no discernible patterns or trends that would indicate autocorrelation. This further supports the conclusion from the Durbin–Watson test. Overall, both the statistical and visual analyses confirm that the independence assumption is satisfied for this regression model.



**Figure 7:** Residuals versus fitted values plot. Residuals versus fitted values plot.

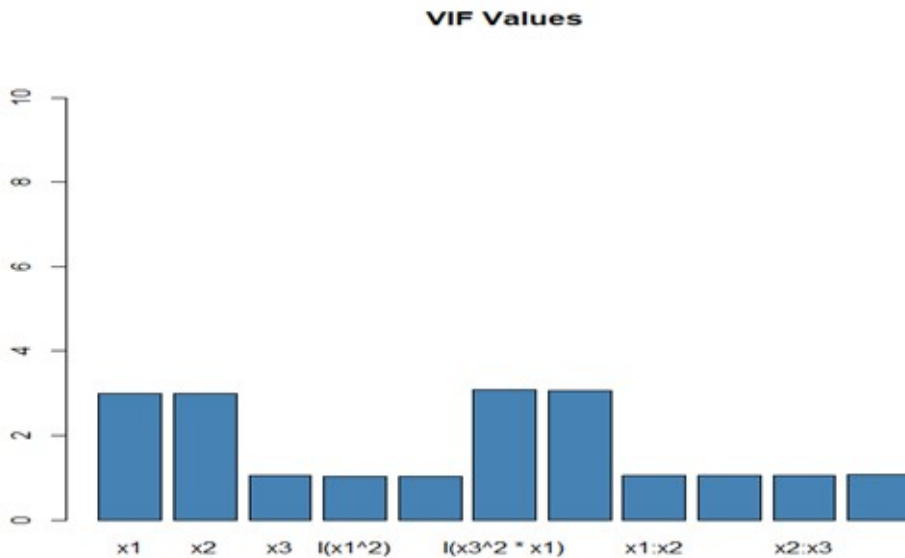
The variance inflation factor used to predict the multicollinearity between the independent variables as shown in figure 8. VIF calculated how much the variance of a regression coefficient is inflated due to multicollinearity with other predictors. Generally, VIF values below 5 are considered acceptable, indicating a low risk of multicollinearity, while values above 10 suggest severe multicollinearity that may compromise the reliability of the regression coefficients. Therefore, all predictors exhibit VIF values below the critical threshold of 10, with the highest observed VIF value being moderate. Furthermore, it also indicates that multicollinearity is not a significant concern in the model, and the estimated coefficients are reliable for interpretation and inference. Thus, the result shows that the predictors are sufficiently independent of each other, ensuring the robustness of the regression model.

## 2.4 Methods

### 2.4.1 Factorial Design

The FD for the copper leaching is done through different steps. The first stage of the FD to bring in the libraries required for analysis in the R-studio software. Then, a smooth workflow is made possible by this phase, which guarantees that all necessary functions and packages are available for the following processes. In the second step, view the data after loading it from an Excel file. This is one

of the essential steps that the data has been imported accurately and is prepared for analysis. Third, to get the data ready for analysis, change the character type of a few columns (A, B, and C). This guarantees that the factors are appropriately identified in the following stages. Fourth, to evaluate the main effects and interactions of the factors, do an ANOVA for the  $3^3$  FD. Understanding the importance of each component and how they interact with the response variable is aided by this phase. Fifth, to see how factors relate to one another and comprehend how they affect the response variable collectively, make interaction plots. To see how the degree of one element affects the effect of another, interaction graphs are crucial. Sixth, to find significant changes between factor levels, perform repeated comparisons with post-Hoc tests using the LSD (Least Significant Difference) approach. After identifying significant effects in the ANOVA, this step is crucial to determining precisely which factor levels differ from one another. Finally, carry out residual analysis to check if the assumptions were right. Furthermore, for checking the copper leaching efficiency, three level, three factor full FD had employed to investigate the relationships between different parameters. Therefore, three important elements have been checked: Acid Concentration (A), Liquid-to-Solid Ratio (L/S ratio; B) and Leaching Time (C). There are 3 FD levels that have been checked: low (-1), medium (0), and high (+1). The experimental runs were carried out using R software, 54 ( $2 \times 3^3 = 54$ ) including duplicates. Thus, copper-leaching efficiency (Y) is the dependent variable, and the significance of the main and interaction effects was determined and analyzed with 95% confidence intervals.



**Figure 8:** *VIF Values for Predictors.*

#### 2.4.2 Multi Linear Regression Model

The MLR is the core and one of the important statistical method that analyzes the relationship between the one dependent (Y) and several independent variables [37]. Furthermore, this method helps researchers to quantify the effect of each factor individually while simultaneously accounting for interactions between factors. Therefore, regression models are very important in experimental design, particularly in complex factorial studies, as they allow for both the assessment of factor significance and the optimization of responses [38]. These models not only improve our understanding of the

system being studied but also provide predictive capabilities for enhancing process performance under various conditions (Equation 3).

$$Y = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_4 X_1^2 + \beta_5 X_2^2 + \beta_6 X_3^2 + \beta_7 X_1 X_2 X_3 \quad (3)$$

The current study, Y is the output variable which represents process efficiency, such as copper-leaching efficiency, while the independent variables (A, B, and C) denote the experimental factors under investigation. Additionally, variable A represents acid concentration, which significantly influences the availability of hydrogen ions necessary for the reaction. Moreover, Variable B represents the L/S ratio, determining the extent of interaction between the liquid reagent and the solid material, affecting the leaching process. Furthermore, variable C represent the leaching time, which controls the duration of the reaction, impacting the degree of material dissolution. These variables are expressed in coded units (-1, 0, +1) to normalize their ranges and facilitate statistical analysis. The interactions and quadratic effects of these variables are also considered capturing complex relationships in the process.

### 3. Results

#### 3.1 Factorial Design Results

##### 3.1.1 ANOVA Analysis

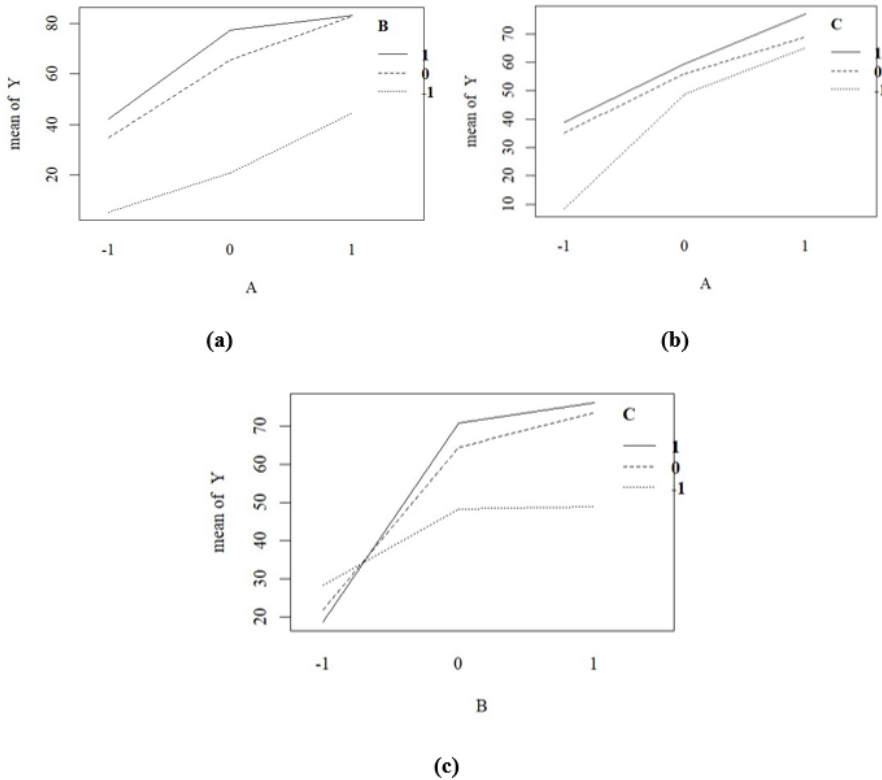
The ANOVA analysis reveals that Acid Concentration (Factor A) and Liquid-to-Solid Ratio (Factor B) have highly significant effects on copper leaching efficiency. This is confirmed from the  $p$  value which is very low  $1.04e - 12$  and  $4.53e - 13$  respectively. Additionally, the F value exceeding 100 which is further confirmed the significance of factor A and B. These factors show extremely low  $p$ -values, indicating their strong impact on the response variable. Leaching Time (Factor C) also significantly influences copper leaching, though its effect is smaller compared to Factors A and B. The interactions between these factors are generally significant, demonstrating that the combined levels of these factors impact the response variable. On the other hand, when we are talking about the Quadratic interaction then the term  $B * A^2$  shows a significant non-linear relationship affecting the response with  $p$  value 0.000444. These quadratic effects are important as they capture curvature in response to that linear terms cannot. Furthermore, the three-way interaction between Acid Concentration, Liquid-to-Solid Ratio, and Leaching Time is marginally significant with  $p$  value 0.075 shows the potential higher-order complexity that might be explored further in future studies (Table 4).

##### 3.1.2 Interaction Plots

The interaction plot demonstrates that the influence of variable A on the mean value of Y is conditional on the level of variable B or other words: the effect of factor A on variable Y is not constant, but depends on the value of factor B. This means that A and B interact in a way that affects the result of Y (Figure 9a). The effect of factor A on variable Y is not constant, but depends on the value of factor C. This means that A and C interact in a way that affects the result of Y (Figure 9b). The graphic depicts the interaction of factors B and C and their effect on the response variable Y. Each line shows a distinct level of factor C and demonstrates how Y changes with B at these values. Thus, the response increases as B shifts from -1 to 1, but the pattern varies by C level. At  $C = -1$ , the increase in Y is gradual. At  $C = 0$ , there's a sharp rise from  $B = -1$  to 0, then it levels off up to  $B = 1$ . At  $C = 1$ , the increase from  $B = -1$  to 0 is most pronounced, followed by a more moderate rise to  $B = 1$ . This indicates a strong interaction effect in which the influence of B on Y is regulated by the level of C, implying potential synergy or antagonism depending on the combination of factor levels (Figure 9c).

**Table 4:** ANOVA analysis

Source	Df	Sum Sq	Mean Sq	F value	Pr(>F)
A	2	16612	16612	100.502	1.04e-12 ***
B	2	17556	17556	106.213	4.53e-13 ***
C	2	1505	1505	9.104	0.004320 **
I(A^2)	1	399	399	2.414	0.127737
I(BA^2)	1	2889	2889	17.478	0.000444 ***
I(CA^2 * A)	1	375	375	2.269	0.139483
I(AA^2 * B)	1	871	871	5.270	0.026755 *
A:B	4	14	14	0.084	0.773606
A:C	4	1407	1407	8.515	0.005638 **
B:C	4	682	682	4.123	0.048659 *
A:B:C	8	550	550	3.327	0.075265
Residuals	27	6942	165		



**Figure 9:** (a) Interaction plot between AB and (b) plot between AC (c)Plot between BC.

### 3.1.3 Post-Hoc Analysis

The Fisher LSD post-hoc analysis compares the means of the different levels of factors A, B and C, as well as their interactions, with a 95% confidence level. Here is a summary of the most significant results (Table 5).

**Table 5: Post-Hoc Analysis.**

Factor/Interaction	Comparison	Difference	Significance
Factor A	0 vs 1	27.25	Highly significant (p <0.0001)
	1 vs -1	42.96	Highly significant (p <0.0001)
Factor B	0 vs -1	37.60	Highly significant (p <0.0001)
	1 vs -1	44.17	Highly significant (p <0.0001)
Factor C	0 vs -1	10.19	Significant (p = 0.0415)
	1 vs -1	12.85	Significant (p = 0.0119)
Interactions A:B	0:0 vs -1:-1	61.21	Highly significant (p <0.0001)
	1:0 vs -1:-1	78.64	Highly significant (p <0.0001)
Interactions A:C	1:-1 vs -1:-1	62.63	Highly significant (p <0.0001)
	1:0 vs -1:-1	60.24	Highly significant (p <0.0001)
Interactions B:C	0:0 vs -1:-1	39.32	Highly significant (p <0.0001)
	1:0 vs -1:-1	47.78	Highly significant (p <0.0001)
Interactions A:B:C	1:1:0 vs -1:-1:-1	81.66	Highly significant (p = 4.5e-06)
	1:0:1 vs -1:-1:-1	87.79	Highly significant (p = 1.4e-06)
	1:0:-1 vs -1:-1:-1	71.22	Highly significant (p = 3.2e-05)
	1:1:-1 vs -1:-1:-1	80.50	Highly significant (p = 8.9e-05)
	1:0:0 vs -1:-1:-1	75.50	Highly significant (p = 1.4e-05)
	0:1:0 vs -1:-1:-1	74.42	Highly significant (p = 1.7e-05)
	1:1:0 vs -1:-1:-1	81.66	Highly significant (p = 4.5e-06)

### 3.2 Multi Linear Regression

#### 3.2.1 Explanation of ANOVA Table

The analysis of variance (ANOVA) provides a statistical breakdown of the effects of the independent variables ( A, B, C ), their interactions, and quadratic terms on the response ( Y ). The first factor, x1 (acid concentration), shows an extremely significant effect on the response, as evidenced by a p-value of  $1.718 \times 10 - 131.718 * 10^{-13}$ . This underscores the importance of acid concentration in driving the process efficiently. Similarly, x2(L/S ratio) has a highly significant impact with a p-value of  $1.477 \times 10 - 131.477 * 10^{-13}$ , indicating its critical role in creating an optimal reaction environment. While x3 (leaching time) is less significant than the other two factors, it still exhibits a notable effect on the response, with a p-value of 0.0034791 , demonstrating the importance of sufficient leaching time for effective material interaction (Table 6). The quadratic terms provide insight into the non-linear effects of the factors. The term  $X1^2$ , representing the squared effect of acid concentration, is significant, with a p-value of 0.0462013 , suggesting curvature in the response relationship. Similarly,  $X2^2$ , the squared effect of L/S ratio, is highly significant, with a p-value of  $2.708 \times 10 - 52.708 * 10^{-5}$ , highlighting its pronounced non-linear effect on the process. On the other hand,  $X^2 * X1$  and  $X_1^2 * X2$  show marginal significance with pvalues of 0.0801136 and 0.0509323 , respectively, indicating weak but notable non-linear interactions involving these variables.

The interaction effects between the factors reveal additional complexity in the system. The interaction between x1(acid concentration) and x2 (L/S ratio) is statistically significant, which influencing the copper leaching efficiency individually and combination with a p-value of 0.0389331. This result highlights the synergistic effect of these two factors, suggesting that optimizing both can lead to substantial improvements in process efficiency. Similarly, the interaction between x1 (acid concentration) and x3 (leaching time) is highly significant, with a p-value of 0.0008633, emphasizing the combined influence of these variables. The interaction between x2(L/S ratio) and

x3 (leaching time) is also significant, with a p-value of 0.0489700, showing the joint effect of these two parameters. The three-way interaction term x1:x2:x3 has a marginally significant p-value of 0.0500241, suggesting a weak combined effect of all three factors on the response. On the other hand, interaction between the quadratic terms reveal non-linear behaviors, suggesting that beyond certain thresholds, increasing these factors does not result in proportionally higher recovery. The interaction between x1 and x3 reflect the strong effect when high acid levels are applied for extended periods with p value 0.0008633. This result indicates that while the individual and two-factor interactions are more pronounced, the simultaneous variation of all three factors may still contribute to changes in process efficiency. The residuals, which represent unexplained variation in the model, have a mean square value of 1.030, indicating a relatively good fit of the model to the data.

**Table 6: ANOVA Table**

Term	Df	Sum Sq	Mean Sq	F value	Pr(>F)
x1	1	116.503	116.503	113.1513	1.718e-13
x2	1	117.647	117.647	114.2624	1.477e-13
x3	1	9.874	9.874	9.5904	0.0034791
I(x1^2)	1	4.345	4.345	4.2203	0.0462013
I(x2^2)	1	22.848	22.848	22.1903	2.708e-05
I(x3^2 * x1)	1	3.312	3.312	3.2162	0.0801136
I(x1^2 * x2)	1	4.158	4.158	4.0383	0.0509323
x1:x2	1	4.678	4.678	4.5438	0.0389331
x1:x3	1	13.256	13.256	12.8744	0.0008633
x2:x3	1	4.233	4.233	4.1114	0.0489700
x1:x2:x3	1	4.192	4.192	4.0718	0.0500241
Residuals	42	43.244	1.030		

**3.2.2 Regression Coefficients and Model Explanation**

The multiple regression model derived for the experimental data quantifies the relationships between the independent variables (A: acid concentration, B: liquid-to-solid ratio, and C: leaching time) and the response (Y: efficiency). The model incorporates linear effects, interaction terms, and quadratic effects to capture both direct and combined influences of the factors on the response. The fitted regression equation, based on the coefficients provided in Table 7.

$$\begin{aligned}
 Y = & 7.9689 + 1.3700X_1 + 2.2283X_2 + 0.5751X_3 - 0.5870X_1^2 \\
 & - 1.3651X_2^2 + 0.6655X_3^2X_1 - 0.5991X_1^2X_2 \\
 & - 0.3132X_1X_2 - 0.7656X_1X_3 + 0.4019X_2X_3 \\
 & - 0.5346X_1X_2X_3
 \end{aligned}
 \tag{4}$$

This equation expresses the combined effects of the three factors and their interactions on the process efficiency (Equation 4). The constant term, 7.9689, represents the base efficiency when all variables are at their coded zero levels. The model’s overall performance, as measured by the R<sup>2</sup> value, is strong, with a multiple R<sup>2</sup> of 0.8758 and an adjusted R<sup>2</sup> of 0.8433, indicating that approximately 87.58% of the variation in the response is explained by the model. The residual standard error, a measure of the model’s prediction accuracy, is relatively low at 1.015, demonstrating good model reliability. The significance of the regression coefficients (as shown in Table 1) was assessed using their associated p-values. Smaller p-values indicate that a term has a statistically

significant contribution to the model. For instance, the coefficients of the linear terms for A, B, and C are all highly significant, with p-values of 3.02e-05, 1.99e-09, and 0.001799, respectively. This confirms the substantial individual impact of each factor on the response. The quadratic terms for A2 and B2 provide insight into the non-linear effects of acid concentration and liquid-to-solid ratio. Additionally, A2 has a key borderline effect with a p-value of 0.053537, which illustrates some curvature in the relationship between A and the response. Furthermore, B2 term is highly significant, with a p-value of 3.63e-05, indicating a pronounced non-linear effect of the liquid-to-solid ratio on efficiency. Additionally, which suggests that B plays a critical role in optimizing the interaction between the liquid reagent and the solid material. Therefore, the interaction effects between the factors are also captured in the model. The interaction between A and C (represented by the AC term) is highly significant, with a p-value of 0.000863, highlighting a strong combined influence of acid concentration and leaching time on the response. Also, the BC term (interaction between liquid-to-solid ratio and leaching time) has a marginally significant p-value of 0.066489, indicating a weaker, but notable, interaction between these variables. The three-way interaction term ABC has a p-value of 0.050024, indicating a borderline significant combined effect of all three factors on the response. In summary, the regression analysis underscores the importance of both individual and interactive effects of the factors on the response. The significant coefficients for linear, quadratic, and interaction terms reveal the nuanced relationships within the system, providing a robust basis for optimization. These findings, as detailed in table 7, demonstrate the utility of the regression model in explaining and predicting process efficiency, while also offering critical insights for process optimization strategies.

**Table 7: Regression Coefficients.**

Term	Estimate	Std. Error	t value	Pr(> t )
(Intercept)	7.9689	0.3099	25.718	<2e-16
x1	1.3700	0.2929	4.677	3.02e-05
x2	2.2283	0.2929	7.607	1.99e-09
x3	0.5751	0.1725	3.333	0.001799
l(x1^2)	-0.5870	0.2955	-1.986	0.053537
l(x2^2)	-1.3651	0.2955	-4.619	3.63e-05
l(x3^2 * x1)	0.6655	0.3635	1.831	0.074229
l(x1^2 * x2)	-0.5991	0.3618	-1.656	0.105191
x1:x2	-0.3132	0.2123	-1.475	0.147642
x1:x3	-0.7656	0.2134	-3.588	0.000863
x2:x3	0.4019	0.2133	1.884	0.066489
x1:x2:x3	-0.5346	0.2649	-2.018	0.050024

### 3.2.3 Coefficient of Determination (R<sup>2</sup>)

This study revealed the effectiveness of a complete strategy to develop regression models for predicting outcomes in acid leaching processes. As seen in table 8, the inclusion of both linear and interaction factors in the regression equation significantly increased the model's explanatory power. The error estimate is a low 1.02, indicating great precision in anticipated values. The model obtains an impressive R<sup>2</sup> of 87.5839%, explaining nearly 88% of the variability in the response variable through the included predictors. Therefore, it confirms that the proposed regression model can explain the majority of variability in copper recovery outcomes, demonstrating strong predictive power. Furthermore, a small drop with the adjusted R<sup>2</sup> (which adjusts for the number of predictors in the model) remains strong at 84.33% which proof that interaction and quadratic terms improves model

performance without overfitting. This modification is critical because it offers a more precise measure of model fit, especially when more terms are included. Integrating interaction terms results in a significant increase in  $R^2$  values, highlighting the interdependence of factors affecting the leaching process. This enhanced modeling technique not only improves forecast accuracy, but it also provides deeper insights into the dynamics of acid leaching, allowing for more educated and effective e-waste management decisions.

**Table 8:** *Estimates of regression equation*

Regression equation	Error Estimate	$R^2$ (%)	$R^2$ Adjusted (%)
Linear terms + all interaction terms	1.02	87.5839	84.33

#### 4. Discussion

Most of the studies investigate the recovery of copper from PCBs through various leaching techniques such as the use of inorganic acids, organic acids, bioleaching, and ionic liquid-assisted extraction. Some studies using bioleaching techniques and achieved near 85% copper recovery [9]. Additionally some articles recover copper and iron through flotation tailings by acid leaching and achieve 60% to 70% recovery [39]. The study on improving acid leaching for copper recovery from PCB waste found that sulfuric acid, at optimal concentrations and temperatures, considerably improves copper solubility. Our factorial experimental design clearly showed the vital role of acid concentration and leaching period in maximizing recovery efficiency, indicating that the acid concentration interaction has a significant impact on leaching outcomes while the study had not perform the experimental design part only perform the MLR [31]. In the current study we uses the formal test as well in the residual analysis which support the analysis but the study had not perform the formal test [40]. This is consistent with current research, which recognizes sulfuric acid's aptitude for strong leaching performance because of its stability and cost-effectiveness. In the current study we check the quadratic or square effect in the analysis of experimental design which improve the model performance but the study had not performed [31] [40]. Furthermore, few articles showing high recovery percentages, failed to account for residual diagnostic validation, thus raising concerns about model reliability, overfitting. On the other hand, this study shows strong performance metrics, with an  $R^2$  of 87.58%, an adjusted  $R^2$  of 84.33%, and a mean square error (MSE) of just 1.02. Additionally, residual analysis also confirms the model reliability like Q-Q plot shows the linear trend, which shows normally distributed residuals, and the Shapiro-Wilk test result ( $p > 0.05$ ) supported this visual conclusion. Furthermore, formal residual validation such as Shapiro-Wilk, Breusch-Pagan, and Durbin-Watson tests with multiple regression framework is also a key achievement of this study. Additionally, the triple validation approach and quadratic effects shows and excellent modeling architecture than the typically e-waste leaching approaches. The study's findings were statistically confirmed using ANOVA, highlighting the importance of these variables. When compared to older technologies such as pyrometallurgy, acid leaching is not only more efficient, but it also provides significant environmental benefits by lowering toxic emissions and energy consumption. In the current research we perform the quadratic or we can say the square interaction effect in the analysis which improve the model performance but the study had not used to check this [41]. In the current study check the multicollinearity by performing VIF test but the study had not checked [31]. However, the study noted shortcomings, including its focus on a specific type of PCB waste and the lack of long-term environmental effect assessments of leachate treatments, indicating opportunities for future research. Overall, this study advances the field of e-waste management by demonstrating a scalable, ecologically acceptable approach for copper recovery, hence encouraging more sustainable recycling practices in the sector. The limitation of this study is that, this study only limited with three variables and not

included most influential factors like particle size distribution, pH stability and mixing speed were not considered and could be explored in future work. This limitation will not decrease the value of this study but highlighting the areas for future exploration and enhancement. This work helps to refine e-waste recycling procedures, promoting a shift to more sustainable ways in the electronics recycling business.

## 5. Conclusion

This study contributes greatly to sustainable electronic waste management by enhancing acid leaching procedures for copper recovery from PCB waste. Using a FD and MLR experimental approach, we carefully examined the impacts of acid content, temperature, and leaching period, adding both linear and higher order interaction effects into our regression models which make it unique approach from the previous research. Furthermore, Shapiro–Wilk, Breusch–Pagan, and Durbin–Watson strengthens the statistical validity of our results. Additionally, optimizing the usage of HCL is an affordable and industrially viable leachant, which is greener alternative conventional leaching methods, which reduced the energy demand and environmental emissions. On the other hand, this method is scalable and one of the global priorities like minimizing the waste, resource recovery and circular economy. These findings not only demonstrate the economic and environmental benefits of improved sulfuric acid leaching but also lay the groundwork for future research. On the other hand, these methods have some limitation like this experiment is conducted in the control environment in the laboratory, which may not fully show the variability in the real-world e-waste stream. Additionally, this study mainly focused on the copper recovery without demonstrating of the other co metals like zinc, lead and nickel in the leachate. Further research could broaden this technique to a wider spectrum of e-waste materials and examine the environmental implications of leachates, improving the scalability and sustainability of copper recovery processes. Additionally, applying the machine learning optimization tools for further improvement in industrial scale application. This work helps to refine e-waste recycling procedures, promoting a shift to more sustainable ways in the electronics recycling business.

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