Research Article

Optimization Studies on Recovery of Metals from Printed Circuit Board Waste

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1. Introduction

Recycling of e-waste is an important subject not only from the point of waste treatment but also from the recovery aspect of valuable materials [1–4]. Among the resources in e-waste, metals contribute more than 95% of the materials market value. Hence, the recovery of valuable metals is the inherent motive in e-waste disposal. In the past decades, many techniques for recovering valuable metals from e-waste have been developed such as gravity separation, magnetic separation, and electrostatic separation [5] or synthesis of CuCl with e-waste, separation of PCBs with organic solvent method [6, 7], cyanide and noncyanide lixiviants leaching methods, ammonium persulfate leaching bioleaching methods [8–10], or a combination of these approaches. Among those methods, hydrometallurgical methods are more accurate, predictable, and controllable [11]. Therefore, hydrometallurgical techniques are most active in the research of valuable metal recovery from electronic scraps in the past two decades. However, traditional hydrometallurgical methods are acid dependent, time-consuming, and inefficient for simultaneous recovery of precious metals. Remarkably, a large amount of corrosive or toxic reagents, such as aqua regia, nitric acid, cyanide and halide, are consumed, producing large quantities of toxic and corrosive fumes or solution [12, 13]. Therefore, it is necessary to seek a more environmental friendly method for the recovery of valuable metals from e-wastes. Hydrometallurgical methods are used in the upgrading and refining stages of the recycling chain [14–16]. In this research article, the recovery of lead and copper metals from e-waste is widely investigated. The PCBs were converted into fine ash powder and subjected to electrowinning process for the recovery of metals. The experimental results were determined by EDS and AAS, respectively. Furthermore, the experimental results are validated through RSM software at different parameters like acid-base concentration, electrode combination, and leaching time [17–22].
pyrolysis to avoid side reaction in the leaching process with the electrolyte solution. The optimum condition of the pyrolysis reactor was 500°C in atmospheric pressure for 1 h where the epoxy resins and polymers were volatilized at the temperature less than 500°C. The volatilized contents were condensed and collected separately. The ferrous materials present in the obtained ash were separated by a magnetic separator.

2.2. Electrowinning Process. The fine ash powder was treated with aqua regia solution (3 : 1 ratio of HCl and HNO₃) in the incineration chamber in order to avoid the liberation of toxic fumes. Then the precipitated salts obtained from the leaching was analyzed by EDS to determine the composition of metal present in the salts (Figure 1). The electrowinning setup consists of bath arrangement and amplifier. The bath having two slots for the anode and cathode fixing and the electrode is connected with amplifier, and the current density was varied through the amplifier (Figure 2).

2.3. Extraction Process of Lead. About 25 g of incinerated fine ash was added into the acid bath followed by the addition of ammonical electrolyte solution. The current density was set to 1 to 10 (A/dm²). The solution was agitated at regular interval to get an effective electrodeposition:

\[ \text{Pb} + 4\text{HCl} \rightarrow \text{PbCl}_4 + 2\text{H}_2 \]  

(1)

After the stipulated time of operation, pure lead was deposited on lead cathode. The deposited elements were scrapped and stored in an air tight container. The recovered lead quantitated from the EDS method. The spent acid left with mud filtered at pH 6–10 was stored in a glass container for further treatment.

2.4. Extraction Process of Copper. About 25 g of incinerated fine ash was added into the acid bath followed by the addition of ammonical electrolyte solution. The current density was set to 1 to 10 (A/dm²). The solution was agitated at regular intervals to get an effective electrodeposition. After the stipulated time of operation, pure copper (cupric) was deposited on the cathode and impure copper (cuprous ion) were deposited on the anode. The deposited elements were scrapped and stored in an air tight container. The recovered copper quantitated from the EDS method. The spent acid left with mud (nonleached elements) was filtered (pH–8.4) and were stored in a glass container for further treatment (Figure 3):

\[ 2\text{Cu}^{2+} (aq) + 2\text{H}_2\text{O} (l) \rightarrow 2\text{Cu} (s) + 4\text{H}^+ (aq) + \text{O}_2 (g) \]  

(2)

The spent solution collected from the electrodeposition was neutralized to 6.9 for the safe disposal as per the standard. Moreover, the presence of any metal in the spent solution was analyzed by Fourier-transform infrared spectroscopy. The results (Figure 4) show that the metallic traces were found to be absent which confirms that all the metals recovered from the ashes deposited on the electrode.

3. Results and Discussion

3.1. RSM for Lead. The response surface methodology (RSM) is a statistical modeling technique employed for multiple regression analysis using quantitative data obtained from designed experiments to solve multivariable equations (Table 1). The response surfaces can be visualized as three-dimensional plots that exhibit the response as a function of two factors while keeping the other factors constant. In this above plot, the red zone corresponds to the extract percentage above 85%, yellow zone shows 60 to 70%, and the blue zone confirms below 40% extraction of lead (Figures 5 and 5(a)). The regression equation for the RSM data plots for the lead is

\[
\text{Extract} = 66.36 + 9.0175 \times A + 7.37375 \times B + 6.42375 \times C + 1.235 \times AB + 0.17 \times AC + (-7.1525 \times BC) + (-18.6187 \times A^2) + (-1.67125 \times B^2) + (-1.81625 \times C^2).
\]

(3)

The model as a function of coded factor could be utilized to predict the response of each parameter within the given limit. Here, the maximum limit of process parameters (factors) is termed (coded) as +1 and minimum limit is terms (coded) as –1. The modified equation or coded equation is very much useful in order to find the comparative effect of the process parameters by relating the coefficient of factors. The final equation in terms of actual factors is

\[
\text{Extract} = -70.2665 + 5.06454 \times CD + 0.201188 \times \text{solvent} + 25.6766 \times \text{time} + 0.00914815 \times \text{CD} \times \text{solvent} + 0.0125926 \times \text{CD} \times \text{time} + (-0.0317889 \times \text{solvent} \times \text{time}) + (-0.229861 \times CD^2) + (-7.42778e-05 \times \text{solvent}^2) + (-0.807222 \times \text{time}^2).
\]

(4)

Equation (4) in terms of process parameters could be utilized to predict the response for the provided levels of each parameter (Table 2). In this equation, the original units of each parameters should be considered for each levels. In order to evaluate the comparative effect of each factor, the above equation should not be considered since the coefficients are balanced to embrace the units of each parameters. Also, the intercept does not fall at design space center.

3.2. Analysis of Variance (ANOVA). Analysis of variance is used to determine the significant effects of process variables on current efficiency (Table 3) along with the factor coding. The sum of squares is found to be Type III—partial derived from the ANOVA quadratic model. The model F value of 4.43 implies the model is significant. A minimum value of 3.12% is possible for the F value due to noise. p values less
than 0.0500 indicate model terms are significant. In this case A, A² are significant model terms. Values greater than 0.1000 indicate the model terms are not significant. If there are many insignificant model terms (not counting those required to support hierarchy), model reduction may improve the model. The lack of fit F value of 63.27 implies the lack of...
fit is significant. There is only a 0.08% chance that a lack of fit $F$ value could be large that could occur due to noise. The coefficient represents the expected change in response per unit change in the factor value, when all remaining factors were constant. The intercept in an orthogonal design is the overall average response of all the runs. The coefficients are adjustments around the average factor settings. When the factors are orthogonal, the variance inflation factors (VIFs) are 1; VIFs greater than 1 indicate multicolinearity; the higher the VIF, the more severe the correlation of factors. As a rough rule, VIFs less than 10 are tolerable. Hence, from the data obtained (Table 4), the VIF values of lead are found to be tolerable.

### 3.3. Model Terms

For a standard deviation of 1, the power calculations are performed using response type “continuous,” and parameters are $\Delta = 2$ and $\sigma = 1$. The power is evaluated over $-1$ to $+1$ coded factor space. From (Table 5), the standard errors should be similar to each other in a balanced design. The ideal VIF value should be 1, VIFs above 10 are cause for concern, and VIFs above 100 are cause for alarm, indicating coefficients are poorly estimated due to multicolinearity, where ideal $R^2$ is 0.0. High $R_i^2$ means terms are correlated with each other, possibly leading to poor models. If the design has multilinear constraints, then multicolinearity will exist to a greater degree. This inflates the VIFs and the $R_i^2$, rendering these statistics would not perform well. Hence, FDS could be used. Power is an inappropriate tool to evaluate response surface designs. Use prediction-based metrics provided in this program via fraction of design space (FDS) statistics.

### 3.4. Fit Statistics

A negative predicted $R^2$ implies that the overall mean may be a better predictor of the response than the current model. In some cases, a higher order model may also predict better. Adeq. precision measures the signal to noise ratio. A ratio greater than 4 is desirable. The ratio of 5.915 indicates an adequate signal. This model can be used to navigate the design space. The optimization of current efficiency is shown in Figure 6. From the results, it is observed that 69% of lead extract is obtained at current density = 10 A dm$^{-2}$, solvent ratio = 5 : 2, and the electrolysis time = 4 hours (Figures 7 and 8). The significance of regression coefficients were analyzed using the $p$-test and $t$-test. The $p$ values are used to check the effect of interaction among the variables. A larger magnitude of $t$-value and a smaller magnitude of $p$ value are significant in the corresponding coefficient term. The coefficient of current efficiency and the corresponding $t$ and $p$ values are shown in Table 6. Finally, the coefficients in the interaction terms for current density-electrolysis time is significant compared to current density-solvent ratio, and current density-electrolysis time.

### 3.5. RSM for Copper

The regression equation for the RSM data plots for the copper is in terms of coded factors form as follows:
The model (Equation 5) as a function of coded factor could be utilized to predict the response of each parameter within the given limit. Here, the maximum limit of process parameters (factors) is termed (coded) as +1 and minimum limit is termed (coded) as −1. The modified equation or coded equation is very much useful in order to find the comparative effect of the process parameters by relating the coefficient of factors (Table 7).

The final equation in terms of actual factors is

\[ \text{Extract (E)} = 48 + 24 \times A + 8.25 \times B + 15.5 \times C + 0.5 \times AB + 7.5 \times AC + 2.5 \times BC + (-6.5 \times A^2) + 8 \times B^2 + 1.5 \times C^2. \]  

\[ (5) \]
Equation (5) in terms of process parameters could be utilized to predict the response for the provided levels of each parameter. In this equation, the original units of each parameter should be considered for each level. In order to evaluate the comparative effect of each factor, the above equation should not be considered since the coefficients are balanced to embrace the units of each parameter. Also, the intercept does not fall at design space center (Table 8). In this contour plot, the red zone indicates extract percentages above 85%. And yellow and blue zones indicate 60 to 70% and below 40% extraction of copper (Figures 9 and 9(a)).

3.6. Analysis of Variance (ANOVA). Analysis of variance is used to determine the significant effects of process variables on current efficiency along with the factor coding. The sum of squares is found to be Type III—partial derived from the ANOVA quadratic model. The model $F$ value of 155.08 in the Table 9 implies the model is significant. A minimum value of 0.01% is possible for the $F$ value due to noise. $P$ values less than 0.050 indicate model terms are significant.

$$
\text{Extract} = 191.503 + 2.32716 \times \text{CD} + (-0.773056 \times \text{solvent}) \\
+ (-11.3333 \times \text{time}) + 0.000555556 \times \text{CD} \times \text{solvent} \\
+ 0.833333 \times \text{CD} \times \text{time} + 0.025 \times \text{solvent} \times \text{time} \\
+ (-0.0802469 \times \text{CD}^2) + 0.0008 \times \text{solvent}^2 \\
+ 1.5 \times \text{time}^2.
$$

Equation (6)
In this case, A, B, C, AC, A₂, and B₂ are significant model terms. Values greater than 0.1000 indicate the model terms are not significant. If there are many insignificant model terms (not counting those required to support hierarchy), model reduction may improve the model. If the lack of fit F value is nil, that implies the lack of fit is significant. The coefficient represents the expected change in response per unit change in factor value, when all remaining factors were constant. The intercept in an orthogonal design is the overall average response of all the runs. The coefficients are adjustments around the average factor settings. When the factors are orthogonal, the VIFs are 1; VIFs greater than 1 indicate multicollinearity; the higher the VIF, the more severe the correlation of factors. As a rough rule, VIFs less than 10 are tolerable. Hence, from the data obtained (Table 10), the VIF Values of lead are found to be tolerable.

3.7. Model Terms. For a standard deviation of 1 the power calculations are performed using response type “continuous,” and the parameters are $\Delta = 2$ and $\sigma = 1$. The power is evaluated over $-1$ to $+1$ coded factor space (Table 11). The standard errors should be similar to each other in a balanced design. The ideal VIF value should be 1, VIFs above 1 indicate multicollinearity; the higher the VIF, the more severe the correlation of factors. As a rough rule, VIFs less than 10 are tolerable. Hence, from the data obtained (Table 10), the VIF Values of lead are found to be tolerable.

Table 6: Fit statistics.

| Std | Run | Factor 1 A: CD A/dm² | Factor 2 B: solvent ml | Factor 3 C: time Hrs |
|-----|-----|----------------------|------------------------|----------------------|
| 1   | 12  | 1                    | 400                    | 2                    |
| 2   | 14  | 19                   | 400                    | 2                    |
| 3   | 2   | 1                    | 600                    | 2                    |
| 4   | 4   | 19                   | 600                    | 2                    |
| 5   | 18  | 1                    | 500                    | 1                    |
| 6   | 13  | 19                   | 500                    | 1                    |
| 7   | 16  | 1                    | 500                    | 3                    |
| 8   | 11  | 19                   | 500                    | 3                    |
| 9   | 8   | 10                   | 400                    | 1                    |
| 10  | 1   | 10                   | 600                    | 1                    |
| 11  | 6   | 10                   | 400                    | 3                    |
| 12  | 7   | 10                   | 600                    | 3                    |
| 13  | 5   | 10                   | 500                    | 2                    |
| 14  | 9   | 10                   | 500                    | 2                    |
| 15  | 17  | 10                   | 500                    | 2                    |
| 16  | 3   | 10                   | 500                    | 2                    |
| 17  | 15  | 10                   | 500                    | 2                    |

Table 7: RSM parameters for copper extraction.

| Std | Run | Factor 1 A: CD A/dm² | Factor 2 B: solvent ml | Factor 3 C: time Hrs |
|-----|-----|----------------------|------------------------|----------------------|
| 1   | 12  | 1                    | 400                    | 2                    |
| 2   | 14  | 19                   | 400                    | 2                    |
| 3   | 2   | 1                    | 600                    | 2                    |
| 4   | 4   | 19                   | 600                    | 2                    |
| 5   | 18  | 1                    | 500                    | 1                    |
| 6   | 13  | 19                   | 500                    | 1                    |
| 7   | 16  | 1                    | 500                    | 3                    |
| 8   | 11  | 19                   | 500                    | 3                    |
| 9   | 8   | 10                   | 400                    | 1                    |
| 10  | 1   | 10                   | 600                    | 1                    |
| 11  | 6   | 10                   | 400                    | 3                    |
| 12  | 7   | 10                   | 600                    | 3                    |
| 13  | 5   | 10                   | 500                    | 2                    |
| 14  | 9   | 10                   | 500                    | 2                    |
| 15  | 17  | 10                   | 500                    | 2                    |
| 16  | 3   | 10                   | 500                    | 2                    |
| 17  | 15  | 10                   | 500                    | 2                    |
3.8. **Fit Statistics.** A predicted $R^2$ implies that the overall mean may be a better predictor of the response than the current model. In some cases, a higher order model may also predict better. Adeq. precision measures the signal to noise ratio. A ratio greater than 4 is desirable. A ratio of 44.9 indicates an adequate signal. If the model can be used to navigate the design space, the optimization of current efficiency is shown in Figure 10. The optimum extraction of 69% Cu is obtained at current density $19 \text{ A dm}^{-2}$, solvent ratio = 5:2, and electrolysis time = 4 hour (Figures 11 and 12). The significance of regression coefficients was analyzed using the $p$-test and $t$-test. The $p$ values are used to check the effect of interaction among the variables. A larger magnitude of $t$-value and a smaller magnitude of $p$ value are significant in the corresponding coefficient term. The coefficient of

### Table 9: ANOVA quadratic model for copper.

| Source   | Sum of squares | DOF | Mean square | $F$ value | $p$ value |
|----------|----------------|-----|-------------|-----------|-----------|
| Model    | 7763.50        | 9   | 862.61      | 155.08    | <0.0001   |
| A-CD     | 4608.00        | 1   | 4608.00     | 828.40    | <0.0001   |
| B-solvent| 544.50         | 1   | 544.50      | 97.89     | <0.0001   |
| C-time   | 1922.00        | 1   | 1922.00     | 345.53    | <0.0001   |
| AB       | 1.0000         | 1   | 1.0000      | 0.1798    | 0.6827    |
| AC       | 225.00         | 1   | 225.00      | 4.045     | 0.0002    |
| BC       | 25.00          | 1   | 25.00       | 4.49      | 0.0668    |
| $A^2$    | 184.36         | 1   | 184.36      | 33.14     | 0.0004    |
| $B^2$    | 279.27         | 1   | 279.27      | 50.21     | 0.0001    |
| $C^2$    | 9.82           | 1   | 9.82        | 1.77      | 0.2206    |
| Residual | 44.50          | 8   | 5.56        |           |           |
| Lack of fit | 44.50      | 3   | 14.83       |           |           |
| Pure error | 0.0000     | 5   | 0.0000      |           |           |
| Total    | 7808.00        | 17  |             |           |           |

### Table 10: Coefficients in terms of coded factors for copper.

| Factor   | Coefficient estimate | DOF | Standard error | 95% CI low | 95% CI high | VIF |
|----------|----------------------|-----|----------------|------------|-------------|-----|
| Intercept| 48.00                | 1   | 0.9629         | 45.78      | 50.22       |     |
| A-CD     | 24.00                | 1   | 0.8339         | 22.08      | 25.92       | 1.00 |
| B-solvent| 8.25                 | 1   | 0.8339         | 6.33       | 10.17       | 1.00 |
| C-time   | 15.50                | 1   | 0.8339         | 13.58      | 17.42       | 1.00 |
| AB       | 7.50                 | 1   | 1.18           | 4.78       | 10.22       | 1.00 |
| AC       | 2.50                 | 1   | 1.18           | 0.2193     | 5.22        | 1.00 |
| BC       | 1.50                 | 1   | 1.18           | 1.10       | 4.10        | 1.00 |
| $A^2$    | −6.50                | 1   | 1.13           | −9.10      | −3.90       | 1.02 |
| $B^2$    | 8.00                 | 1   | 1.13           | 5.40       | 10.60       | 1.02 |
| $C^2$    | 1.50                 | 1   | 1.13           | −11.0      | 4.10        | 1.02 |

### Table 11: Model terms in RSM for copper.

| Term | Standard error | VIF | $R_i^2$ | Power (%) |
|------|----------------|-----|---------|-----------|
| A    | 0.3536         | 1   | 0.0000  | 69.8      |
| B    | 0.3536         | 1   | 0.0000  | 69.8      |
| C    | 0.3536         | 1   | 0.0000  | 69.8      |
| AB   | 0.5000         | 1   | 0.0000  | 42.1      |
| AC   | 0.5000         | 1   | 0.0000  | 42.1      |
| BC   | 0.5000         | 1   | 0.0000  | 42.1      |
| $A^2$| 0.4787         | 1.01852 | 0.0182 | 95.4      |
| $B^2$| 0.4787         | 1.01852 | 0.0182 | 95.4      |
| $C^2$| 0.4787         | 1.01852 | 0.0182 | 95.4      |
current efficiency and the corresponding $t$ and $p$ values are shown in (Table 12). Finally, the coefficients in the interaction terms for current density-electrolysis time is significant compared to current density-solvent ratio and current density-electrolysis time.

### 4. Conclusion

The ammonia-lead nitrate and ammonia-copper sulphate system have been employed as a leaching agent for recovery of lead and copper from scraped printed circuit board wastes. A two-stage leaching was employed, wherein the first stage consisted of leaching the scrap board with 0.1 M Pb (NO$_3$)$_2$ and 0.1 M CuSO$_4$ which results in the selective
dissolution of lead and copper leaching rate, and other metals was found in lower amounts, respectively. The undissolved residue portion from the leaching stage containing nickel, tin, and silica were leached out in respective treatments. The current efficiency was found to increase with current density and concentration ratio with the contact time in acid bath. Hence, 73.29% lead and 82.17% copper have been successfully recovered from the electrolysis process. And, also by RSM Software prediction, the recovery of lead and copper are as 78.25% and 89.1%, respectively. In addition to the quadratic model equation, ANOVA, model terms, and fit statistics were also tested for the experimental conditions.

**Data Availability**

All the data used to support the findings of this study are included within the article.

**Conflicts of Interest**

The authors declare that they have no conflicts of interest.

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