Indian Journal of Chemical Technology
Vol. 29, March 2022, pp. 157-165

Effect of clay minerals on copper reclamation from leached solution

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Received 18 June 2021; accepted 3 November 2021

The present investigation focus on the recovery of copper (Cu) ions from printed circuit boards (PCBs) by applying simultaneous treatment of leaching and adsorption, as a novel approach. The PCBs are subjected to chemical leaching using aqua regia resulted in a Cu recovery of 97.06%. The leached solution is treated for removal of Cu with activated bent clay as an adsorbent. The optimum condition of process variables is found through central composite design-response surface methodology (RSM-CCD). The maximum %Cu removal of 97.33% is obtained at the optimum operating conditions of adsorbent size of 0.04 mm, adsorbent dosage of 3.5 $g.L^{-1}$ and the temperature of 80°C with 0.845 desirability. This investigation is found to be an eco-friendly way to recover copper ions and does not cause any environmental issues.

Keywords: Aqua regia, Bentonite Clay, EDXs, E-waste, Leached solution, Response surface methodology

Electronic waste, or E-waste, is usually designated as discarded electrical or electronic devices that are intended for disposal. Recent innovations, market expansion, economic growth, and the short life of electrical and electronic equipment (EEE) have led to significant growth in the waste EEE (WEEE). PCBs are the main component of WEEE, which consists of 40% metals, 30% ceramics, and 30% plastics[1-3]. The metallic composition in the E-waste consists primarily of copper (Cu) which account for 10-30% and meager amounts of various heavy metals such as Tin (Sn), Zinc (Zn), Lead (Pb), Nickel (Ni), Iron (Fe), Silver (Ag), Cadmium (Cd) and Gold (Au)[4]. Informal processing of e-waste in developing countries can lead to adverse effects on health and environmental issues[5]. In 2016, 44.7 million metric tons of e-waste were produced globally[6]. If the e-waste is directly disposed of through landfilling, it would create land and water pollution. Exposure to metals such as Pb and Cd affects reproductive health, development, mental instability, and damage to human DNA[7-9]. Health symptoms like headache, dizziness, irritation in the eyes, nose, mouth, etc. are caused by exposure to Cu [10,11]. The traditional methods that can be used to recover metals from PCBs are essentially physical-mechanical and chemical separations. Several studies on the feasibility of metal recovery from PCBs have been investigated in the last decade. Hydrometallurgical procedures, such as leaching, are gaining importance as per the recent studies. Several leaching reagents demonstrate major improvements in metal recovery. When treated with different acidic media, HNO_3, HCl, and H_2SO_4, PCBs were cut to extract Cu^{2+} ions, and the recovery % of Cu^{2+} was good.

Heavy metals have been identified as potentially dangerous to human health and the environment. Many studies have demonstrated that even at low doses, these metals are hazardous. When levels of these dangerous metals exceed the tolerance limit, they can induce accumulative poisoning, liver cancer, and brain damage. The hydrometallurgical method is of great concern to researchers because it involves low consumption of reagents, energy, and less environmental pollution. Many studies were performed and reported the impact of operating conditions on the recovery of heavy metals such as Ag, Au, Ni, and Cu found in PCBs. Significant recovery rates of copper through chemical leaching were reported in our previous research[12-14]. In a column leaching utilizing NaCN reagent, the cyanidation of PCBs recovers 47.9% Au, 51.6 % Ag, 48.1 % Ni, and 77.2 % Cu, whereas the activated carbon adsorption procedure recovers 97.3 % Au, 99.3 % Ag, 98.2 % Ni, and 80.7 % Cu. The deposition of extracted metals possesses different dendritic growth with respect to the leaching reagent used. The copper recovered by leaching of PCBs with H_2SO_4 solution presented a fine dendritic structure with branches of about 80-100 m in length[15,16].

Heavy metal recovery methods such as electrowining, electro refining, and ion exchange procedures were used by the researchers to recover the metals from leached solutions. These approaches have various drawbacks, such as the emission of secondary pollutants[17]. As a result, natural adsorbents were tried as an alternative. For heavy metal adsorption, two locally accessible adsorbents, bentonite clay and roasted date pits, were utilized. The date pits were roasted in an oven at 130°C for 4 h before being crushed into a powder for testing. A surface area analyser was used to examine the two adsorbents. The lowest metal ion removal effectiveness by adsorption using bentonite clay and roasted date pits was 97%[18]. 15g of mobile PCB samples were leached in a 250 mL solution containing the prescribed quantity of ammonium thiosulfate and copper sulfate at various *p*H levels in a 500 mL glass beaker. All leaching studies were conducted at a speed of 250 rpm and a temperature of 250°C. The solution was withdrawn and filtered after 8 h of leaching to separate the remaining PCBs from the solution. The residue was then dried in a vacuum oven at 130°C for 2 h to eliminate all moisture from the sample before being weighed and the weight of the residue computed. Under optimal circumstances, 0.1M ammonium thiosulfate, stirring speed of 250 rpm, and room temperature for 8 h, 56.7 % of gold could be leached. In the instance of a full PCB unit, the highest gold leaching was 78.8 % in an 8 h time period at 0.1M thio sulfate, copper sulfate of 40 mm, *p*H 10–10.5, stirring speed 250 rpm at room temperature[19].

To solve the shortcomings of earlier researchers' copper-recovery approaches from diverse wastes, the researchers used a novel method such as adsorption by Bentonite clay. Clays and clay minerals are valuable because of their distinctive qualities, such as hardness, durability, strong flexibility, and plasticity, which make them perfect for industrial applications. Clays have small particle sizes and high surface areas due to their diverse forms. They have been identified as one of the best low-cost adsorbents. Bentonite is a montmorillonite-derived aluminum phyllosilicate adsorbent. It is a sedimentary rock largely consisting of clays, with a characteristic 2:1 layer structure and significant concentrations of Na^+, Ca^{2+}, and K^+ ions found between the layers. It is a sedimentary rock largely consisting of clays, with a characteristic 2:1 layer structure and significant concentrations of Na^+, Ca^{2+}, and K^+ ions found between the layers. The application of acid to clay minerals has extra mineralogical and mineralogical effects on a mineralogical system. Because of its cation capacity, larger surface area, and adsorption ability for diverse organic and inorganic ions, acid-activated bentonite has long been used to remove metal ions. The present study mainly deals with the copper recovery from PCB leached solution with treated bentonite clay. The extraction of copper ions (Cu^{2+}) from PCBs has been carried out by two-stage leaching technique. The adsorption process has been optimized using response surafe methodology (RSM).

Experimental Section

Sample Collection and Preparation

The waste PCBs are obtained from e-waste disposal units in Erode, Tamilnadu, India. For experimental use, 500 gm of PCB scraps were broken into 15–20 cm size and shredded into small pieces around 50 × 50 mm to 30 x 30 mm, using pliers and four blade cutting shredder[12,17,20]. Metals and non-metals are separated by different separation methods, such as pneumatic separation, magnetic separation, filtering, eddy current separation, electrostatic separation, etc., are used to enrich metals and non-metals [12, 15-17].

The crushed PCBs obtained from the crusher are then pulverized and further exposed to milling operations for better size reduction using a ball mill, and particles of different mesh sizes are analyzed. The weight fraction of crushed PCBs obtained from the lower screens of jaw crushers with a capacity of 80 kg hr^{-1} and a clearance of 10 mm is much lower, making better ion recovery impossible. Thus, it is subjected to 5 mm of clearance in the same jaw crusher, yielding samples weighing 65, 53, 48, and 36 gm for sieves with mesh sizes of 0.3, 0.18, 0.05 mm, and pan, respectively, when screened using a rotary sieve shaker at a speed of 60 rpm with a power of 0.25 HP and a single-phase 80 volt supply. As the reduction in size increases the rate of recovery of metal ions, the resulting crushed samples are processed into powder form using a pulverizer with a disk diameter of 175 mm, operated by a 3-phase motor at 1400 rpm on a 225–445 V supply. The resulting powder samples are screened under different mesh sizes and the weight fraction of the bottom products (Sieves from 52 B.S.S. to pan) is increased but not adequate for the anticipated recovery. The pulverized PCB powder is milled in a ball mill having a ball weight of 500 g at a speed of 60-120 rpm with a mill diameter of 200 mm, driven by a 0.25 HP 3 phase motor, which results in a size reduction and the highest weight fraction is obtained at the lowest sieves. The weight fractions obtained at each sieve are collected separately and subjected to leaching (Fig. 1).

Chemical leaching experimentation with Aqua regia

Aqua regia preparation

The metal recovery from PCBs is carried out by two stages of leaching media (first stage HCl and HNO_3 and second stage HCl and H_2SO_4). It is prepared by mixing HCl and HNO_3 in a 3:1 ratio under specified conditions of temperature, time, and surrounding conditions. In previous studies (Table.1), with aqua regia as a leaching reagent, copper was extracted from PCBs with a high recovery rate[15,16,19,21,22].

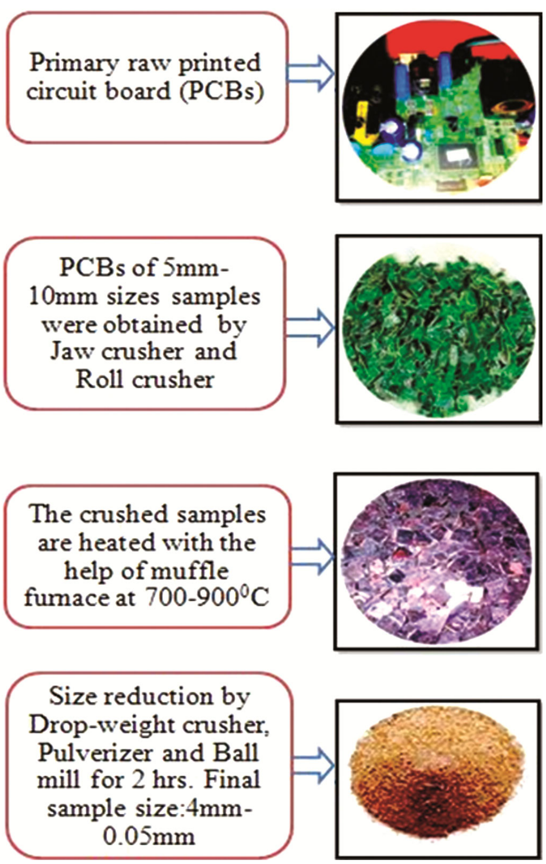

Fig. 1 — Schematic diagram of primary PCBs in to stepwise size reduction under the various mechanical operations

Aqua regia preparation involves the mixing of strong acids. The two concentrated acids were mixed in a 3:1 ratio (HCl: HNO_3), with concentrated HCl (35%), and HNO_3 (65%). The solutions should be kept away from organic contaminants because they lead to vigorous or violent reactions and a low temperature should be maintained.

Experimentation with various parameters on copper recovery

All the tests were carried out in a conical flask equipped with a temperature-controlled shaker. At 80°C, 20 gm of PCB samples are mixed with 0.5 L of leaching medium and agitated in a mechanical shaker at a speed of 120 rpm for 3 h. The shaker is turned off at the conclusion of this contact time, and the solutions in the conical flask are filtered using filter paper. The metal composition retained is evaluated after thorough filtering. The rate of leaching is affected by a number of factors, including shaking intensity, size, contact duration, pulp density, and temperature. By adjusting these parameters, various values for the recovery rate and heavy metal content are produced. After that, the samples are examined, and the findings are studied over time to establish the recovery rate.

Copper ion reclaimation by adsorption

Adsorption operations are extensively applicable in chemical operations for the reclamation of copper ions from the leached solution. Some other techniques have been used in previous studies, like precipitation, cementation process, liquid membrane techniques, and ion exchange process. Each of these methods has its own specific advantages and disadvantages. Precipitation methods were used to convert reagents such as carbonate, sulfide, and hydroxide from leached solutions into insoluble forms. The disadvantage of this precipitation method is the formation of a huge

Table 1 — Recovery data of copper with different leaching agents

Leaching media used	Cu recovery %	References
$H_2SO_4 + H_2O_2$	96.72	15
HNO_3 and HCl+ HNO_3	86.9	16
NaCN	77.7	19
$(NH_4)_2S_2O_3$ and $CuSO_4$	78.8	21
$H_2SO_4 + NH_3$	88.6	22

quantity of sludge that contains toxic compounds[23,24]. To overcome this defect, the copper metal reclaiming process was done by another method called the cementation process, which involves metal displacement reactions. The disadvantage of this process is the need for high contact time. The copper solution necessitates a slow flow rate.

To overcome all these downsides, a new technique has to be developed for the separation of copper ions (Cu^{2+}) from the leached solution. The stability of adsorption operations compared to other separation operations is the major reason for the recent renovation for selective separation and recovery of copper ions from the leached solution. Therefore, a suitable technique has to be selected so that the highest rate of copper recovery can be achieved. Cu^{2+} ions are recovered more effectively with this A-Bent adsorbent.

Adsorbent activation

The bent clay samples were subjected to physical and chemical activations. The adsorbent was placed in the thermal crucible and dried for about 5 h for thermal activation at 600°C. The bentonite clay is rinsed with distilled water and dried in the sun. The concentrated HCl and 85% HNO_3 solutions were combined in a 1:2 by mass ratio and stirred well for 2 h. The sample is kept in a muffle furnace and heated at 550°C for 2 h. Natural cooling reduces the temperature to room temperature. The adsorbent samples were then crushed to less than 1 m in size, weighted and rinsed with 0.1 mol L^{-1} HCl to remove surface ash. The activated adsorbents are shown in Fig. 2a and b.

Then, the adsorbent samples were washed with de-ionized water to remove the HCl and dried for 24 hrs at 150°C. After drying, both samples were ground and sieved. The prepared samples were tested with the help of the Scanning Electron Microscope (SEM-FEI-Quanta FEG 200F), which is shown in Fig. 3a & b.

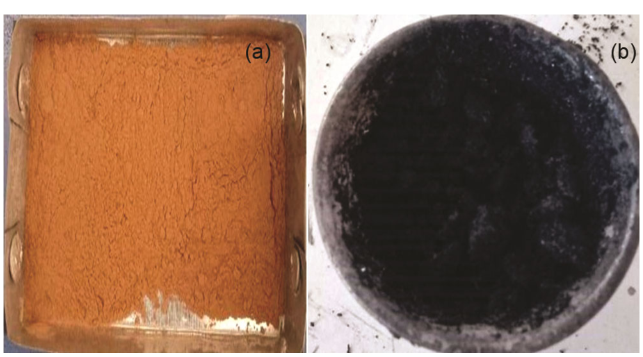

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Fig. 2 — (a) Schematic diagram for Bentonite Clay thermal activation at 600°C and (b) Chemical activation by concentrated HCl and HNO_3

Adsorption experiments

The feed for adsorption is copper solution recovered by leaching. Copper ion adsorption on bent was studied in a batch system using both activated and NA-adsorbents. The adsorbent was added to 20 ml of the leached solution in a conical flask. The mixture was shaken at 200 rpm for 5 hrs at 80^0C. After adsorption, the samples were filtered and the copper concentration was analyzed by using EDX, which is used for the analysis of the elemental characterization of a sample in conjunction with SEM. The energy of the beam current is typically in the range of 100 Na, the Schottky emitter ranges between (-200v to 30 kV), the magnification range is 12X-105X, and the resolution is 2 nanometers (gold nano-particles suspended on carbon substrate). Then, the adsorption efficiency of the beny clay was determined by the following equation: 1.

$$\text{Removal Efficiency (\%)} = (C_o - C_e) / C_o \times 100 \quad \ldots(1)$$

C_o is the initial concentration of metal ions from leached samples. C_e is the metal ions concentration after adsorption operation[25].

Response surface methodology (RSM)

Statistical modeling methods were used to evaluate the multiple regression of the experiments designed to

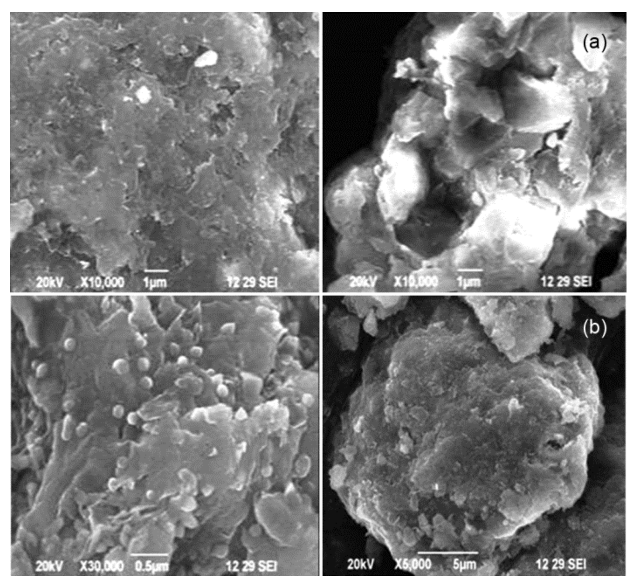

Fig. 3 — (a) SEM images of Bentonite Clay thermal activation at 600°C and (b) Chemical activation by concentrated HCl and HNO_3

determine the multivariable equation. RSM concept data plots collected in the final regression equation in terms of coded recovery variables for the response[25]. The final equation in terms of the coded factors can be used to make predictions about the response for given levels of each factor. By default, the high levels of the factors are coded as +1 and the low levels are coded as -1. The coded equation is useful for identifying the relative impact of the factors by comparing the factor coefficient.

Studies were conducted in order to obtain the optimum values for various parameters from the recovery of copper ions from leached solution by Response Surface methodology. The influence of various parameters (size of adsorbent, adsorbent dosage, and temperature) was studied for copper ion recovery. In this analysis, input parameters that were taken into account were temperature, adsorbent dosage, and temperature. Based on the ideal experimental conditions for the shaking intensity and dimensions of the metals optimum recovery %, the leaching variable input parameters were calculated (Table 2).

Results and Discussion

Sample analysis of PCBs (Sizes and metal elements)

The graphical representation of the size analysis reveals that, subject to the size decrease sequence, the fraction of samples generated on the screens with larger mesh sizes has decreased. The total weight collected in the sieves is, however, maintained at a similar level with marginal loss. The sample collected at the ball mill is much less than 0.05 mm from the analytical data of each procedure. Numerous experiments have used a shredded sample dimension of less than 0.5 mm, contributing to an elevated copper recovery rate[26]. The present findings consist of 0.05 mm of the sample held above the pan for the liquidation used on the particle scale. EDXs have been used to analyze the copper concentrations of preliminary samples and the final composition of metals by weight was (Cu 3.15%, Sn 42.4%, Zn 1.16%, Pb 27.81%, and other metals 25.48%) (Fig. 4(a)).

Copper recovery of leaching

The leaching tests were conducted by varying the operational parameters i.e time, temperature and pulp

Table 2 — Levels of different process variables in coded and un-coded form used for adsorption of copper ions (Central composite design)

Variable	Name of the process variable	Range and levels		
		-1	0	1
A	Temperature, °C	40	60	80
B	Adsorbent dosage gm L^{-1}	2	3	5
C	Adsorbent size µm	0.4	2	5

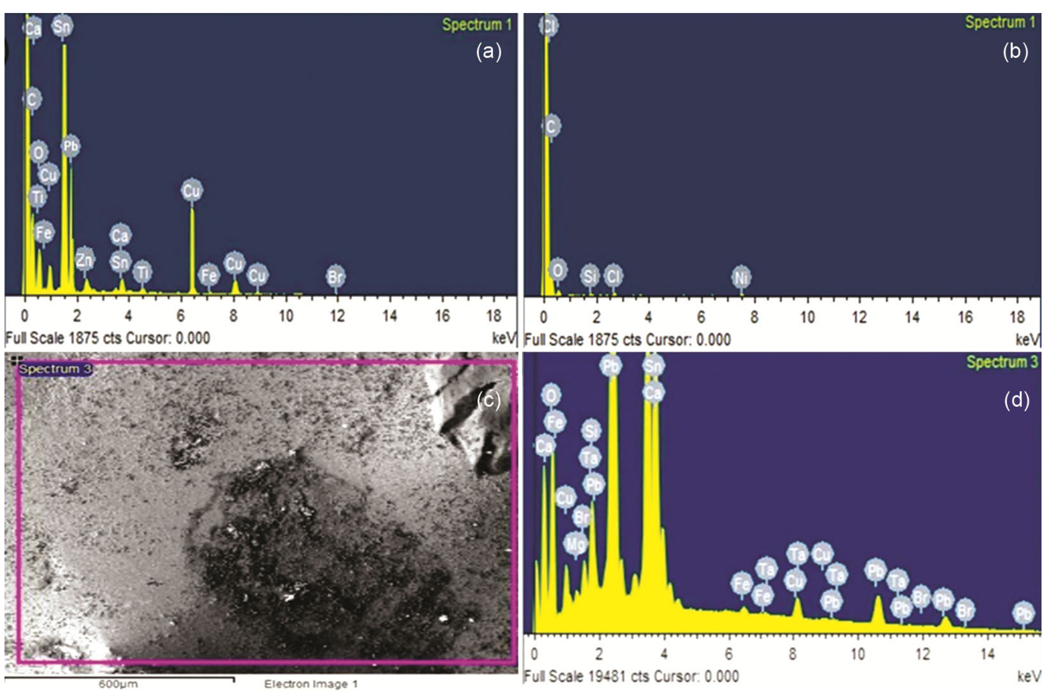

Fig. 4 — (a) EDX_S of PCB sample taken (b) EDX_S of Leached solution and (c) SEM image of the bent clay after adsorption (d) EDX_S of the bent clay after adsorption

density. Metal compositions present in the PCBs after leaching (Cu 0.09 wt %) are shown in EDX results at the optimum condition (Fig. 4(b)). The results obtained under optimal conditions show that the copper recovered rate is 97.06% at a time of 5 hrs, temperature of 90^0 C, and a pulp density of 25 gm L^{-1}.

Adsorption studies for copper recovery from leached solution

Adsorption studies for the recovery of copper from leached solutions using bentonite clay as an adsorbent are described. The influencing conditions are the concentration, size, temperature, and time[28]. The one factor at a time studies showed the best conditions for adsorption as 4g of adsorbent dosage, 0.05 mm adsorbent particle size, 80°C temperature, and 4 hrs of contacting time. The results show that at these conditions, the copper recovery is 97 %. This is the most favorable condition for obtaining the maximum recovery of copper ions, which was initially found to be 3.119 wt % of copper and after copper was present in adsorbent 3 wt %, copper was recovered 97.33 %.

Optimization of parameters by Design of Experiments (DOE)

By analyzing operating parameters and reducing the number of tests, you can optimize and evaluate individual process variables for higher recovery rates. By using RSM-CCD for Cu adsorption, the optimized operationg conditions were calculated. The experimental and predicted copper adsorption results shown in (Table 3) were analyzed.

Table 3 — Experimental and predicted results from CCD with optimal parameters for copper adsorption

Run no.	A	B	C	Experimental	Predicted
1.	1	-1	0	92.88	92.85
2.	-1	1	0	89.24	89.15
3.	1	1	0	94.16	93.96
4.	-1	0	-1	92.16	92.19
5.	1	0	-1	90.23	90.06
6.	-1	0	1	88.34	88.43
7.	1	0	1	96.24	96.33
8.	0	-1	-1	94.88	96.33
9.	0	1	-1	93	92.71
10.	0	-1	1	96.8	96.33
11.	0	1	1	94.6	94.49
12.	0	0	0	86.2	86.37
13.	0	0	0	91.36	91.47
14.	0	0	0	93.7	93.9
15.	0	0	0	96.17	96.46
16.	0	0	0	97.33	96.33

RSM for copper reclaimation from leached solution

The multiple regression of the experiments designed to determine the multivariable equation was evaluated using statistical modeling methods. RSM concept data plots in terms of coded recovery variables for Cu recovery were collected in the final regression equation. The high levels of the factors are coded as +1 by default, while the low levels are coded as -1. By comparing the factor coefficients, the coded equation can be used to determine the relative importance of the factors.

$$\% \text{ Cu} = +96.33+0.7688\times A+0.7388\times B+1.10\times C-0.4750\times A\times B +1.03\times A\times C-0.287\times B\times C+1.09\times A^2-3.96\times B^2-3.86\times C^2$$

The model predicted the response of each parameter within the boundaries of the coded factor. The maximum and minimum coded factors are denoted as +1 and -1 in this case. The response surface was depicted in three-dimensional plots that show two factor functions while holding the other variables constant. According to the projected design plots, the above red zones indicate 97.33% of Cu, the above yellow zones confirm 93% of Cu, and the above blue hues confirm 88.95% of Cu. It is depicted in Fig.5

Evaluation of the model

Model terms are significant if the P-value is less than 0.0500. A, B, C, AC, A^2, B^2, C^2 are crucial model terms in this situation. Table 4 shows how the analysis of variance corresponded to the experimental data. The model's F-value of 34.28 indicates that it is significant. An F-value this big might arise owing to noise just 0.01% of the time. Furthermore, the acceptable and fair value of lack of fit with probability (>0.05) suggests the method's appropriateness for effective experimental data presentation. It was implied that the model was correct. Furthermore, the acceptable and fair value of lack of fit with F-value of Cu 0.02322, with probability (>0.05) suggests the method's appropriateness for good presentation of data.

As seen in Table 5, the model's high R^2 value of 0.9778 suggests that the experimental and predicted findings were in good agreement. Furthermore, the projected R^2 value of 0.9353 was in fair agreement with the corrected R^2 value of Cu 0.9493. The signal-to-noise ratio is measured by Adeq Precision. A ratio larger than 4 is preferred. This model can help you find the way around the design area. The predicted values of the response from the model were in

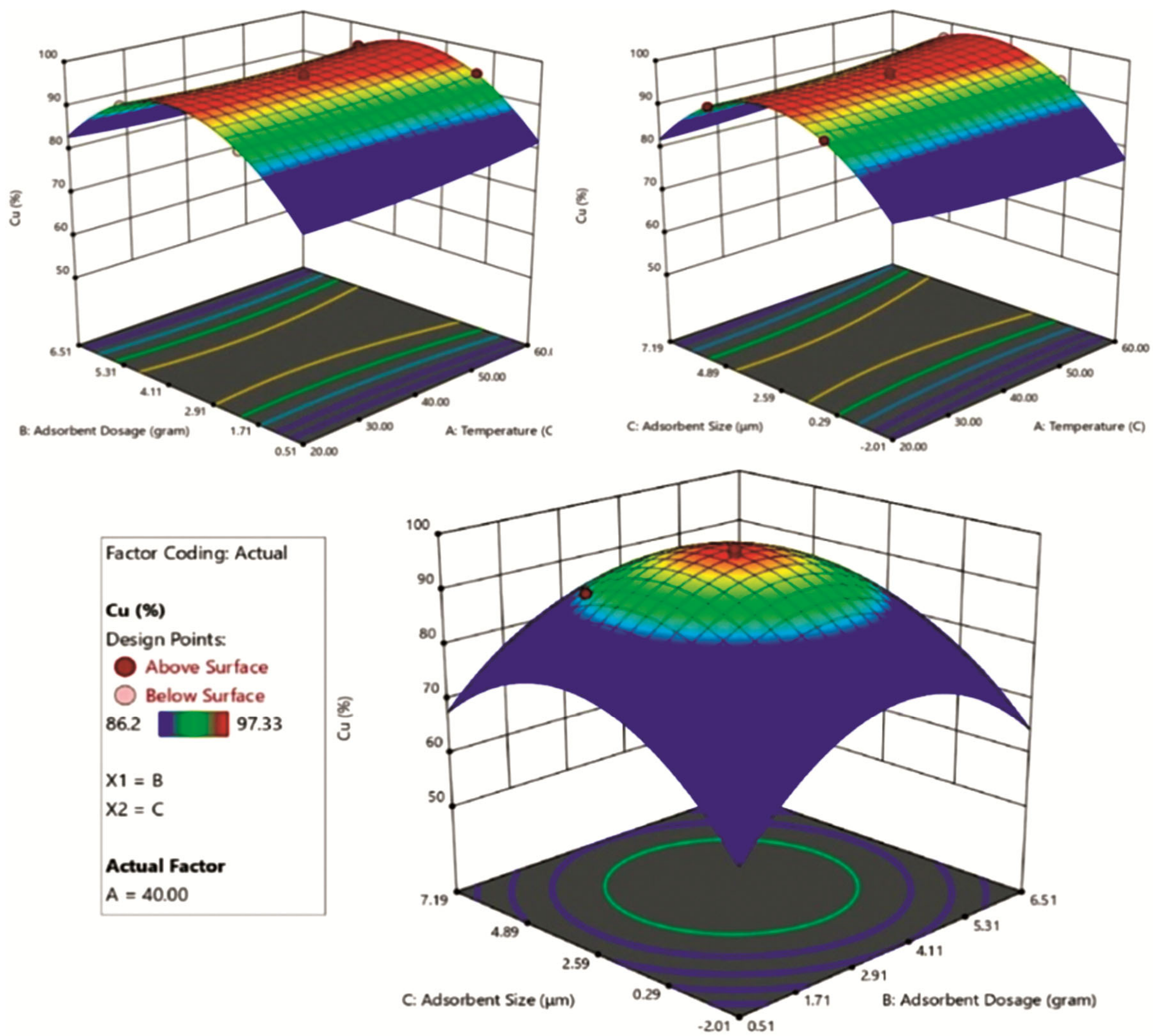

Fig. 5 — RSM plots and Interactions between the temperature, adsorbent dosage and size of adsorbent by Cu recovery

Table 4 — ANOVA table for model to predict % of leaching of copper

Source	Sum of squares	df	Mean square	F Value	p-value Prob> F
Model	162.29	9	18.03	34.28	< 0.0001
A-Temperature	4.73	1	4.73	8.99	0.02
B-Adsorbent dosage	4.37	1	4.37	8.3	0.0236
C-Adsorbent size	9.72	1	9.72	18.49	0.0036
AB	0.9025	1	0.9025	1.72	0.2316
AC	4.26	1	4.26	8.11	0.0248
BC	0.3306	1	0.3306	0.6285	0.4539
A^2	4.98	1	4.98	9.47	0.0179
B^2	66.11	1	66.11	125.68	< 0.0001
C^2	62.9	1	62.9	119.57	< 0.0001
Residual	3.68	7	0.526	-	-
Lack of fit	0.3458	3	0.1153	0.1382	0.9321
Pure error	3.34	4	0.831	-	-
Cor total	165.98	16	-	-	-

Table 5 — Quality of the quadratic model for the adsorption of copper

Parameters	Cu
Standard Deviation (SD)	0.7253
Mean	93.16
Coefficient of Variation (CV%)	0.7785
Predicted residual error sum of squares (PRESS)	3.68
R-Squared(R^2)	0.9778
Adj R-Squared (R^2)	0.9493
Pred R-Squared (R^2)	0.9353
Adequate precision (AP)	18.125

agreement with the observed values over the defined range of independent variables, with reasonably higher coefficients of determination (R^2).

Desirability plot for recovery of copper from leached solution

The desirability scale ranges from 0.0 to 1.0, corresponding to the transition from an unpleasant to a very desired state. Optimum removal of Cu 97.33 % was reached with a desirability of 0.845 at adsorbent dose of 2 g. L^{-1}, adsorbent size of 0.4 mm, and a temperature of 90°C.

Maximum copper recovery by RSM

Experiments were undertaken out using RSM findings. ANOVA, response surface plots, quadratic model equation, and CCD were also examined for experimental circumstances. The experiment was performed at the optimal conditions obtained from theortical analysis. The SEM and EDXS analysis for the corresponding sample are shown in Fig.4(c) and 4(d). The data obtained under optimal conditions reveal that copper recovery is 97.06 %.

Conclusion

The performance of two-stage chemical leaching medium (HCl & HNO_3, H_2SO_4 & HCl) for the separation of copper ions during PCB treatment has been investigated, and the findings revealed that C-A Bent adsorbents help in 97% successful copper separation for chemical leached solution. As a result, the study indicates that copper ions are efficiently recovered from leached solutions utilizing adsorption techniques under optimal circumstances in the presence of C-A Bent adsorbent. These forms of metal leaching processes are encouraged in order to alleviate the environmental issues created by heavy metals. The research illustrates the recovery rates' reliance. At an adsorbent dose of 2 gm L^{-1}, an adsorbent size of 0.4 mm, and a temperature of 90°C, a maximum removal of Cu of 97.33% with a desirability of 0.845 was reached. As a result, this type of heavy metal leaching and adsorption reclamation procedure is recommended in order to reduce environmental consequences (caused by heavy metals). It was determined that the combination of aqua regia leaching with bent adsorption is an efficient and cost-effective method of recovering copper from leached solution.

According to research, changing the surface of the clay enhances the rate of adsorption, but this boosts the overall cost and introduces extra chemicals into the environment. As a result, future efforts will be made to remedy these difficulties. Only a few field trials have been conducted, and more systematic research is required to determine the optimal conditions for employing clay minerals as adsorbents.

Acknowledgements

This research was carried out at the laboratories of Excel Engineering College, Erode Sengunthar Engineering College and Kongu Engineering College in Erode, Tamil Nadu, India. The corresponding author wishes to express gratitude to his parents and dear brother, Mr. P. Selvarasu, PG Assist (Zoology), Govt Higher Secondary School, Vellore, for their generous assistance.

Nomenclature

CCD	–	Central Composite Design
Cu	–	Copper
EEE	–	Electrical and Electronic Equipment
E – Waste	–	Electronic Waste
EDXs	–	Energy-Dispersive X-Ray Spectroscopy
HCl	–	Hydrochloric acid
Pb	–	Lead
HNO_3	–	Nitric acid
PCBs	–	Printed Circuit Boards
RSM	–	Response Surface Methodology
SEM	–	Scanning Electron Microscopy
H_2SO_4	–	Sulphuric acid
Sn	–	Tin
WEEE	–	Waste of Electrical and Electronic Equipment
Zn	–	Zinc

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