Chapter

Clay Minerals Effects for Metal Reclamation from Leached Solution

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Abstract

The recent advancements in technology play a pivotal role in mankind's life and have a significant stint in the generation of E-waste. The present investigation focuses on the recovery of heavy metals from Printed Circuit boards (PCBs) by applying two efficient techniques viz., leaching and adsorption. A combination of leaching and adsorption is a novel and productive approach to recovering heavy metals from like PCBs. After the phases of chemical leaching, the solution was recovered through adsorption and is eco-friendly. The process is carried out to increase the separation rate, reduce the time spent and reach the limits of incineration and pyrolysis methods. Adsorption provides the recovery of heavy metals with respect to the required adsorbent since it is a surface phenomenon. The optimum condition of process variables was found through response surface methodology (RSM). The maximum recovery of copper ions (97.33%) was obtained at the optimum operating conditions such as adsorbent size of 0.04 mm, adsorbent dosage of 3.5 gm L^{-1} and the temperature of 80°C with 0.845 desirability. This investigation was found to be an eco-friendly way to recover copper ions and does not cause any environmental issues.

Keywords: E-waste, leached solution, bentonite clay, EDXs, aqua regia, response surface methodology

1. Introduction

Electronic waste or e-waste is designated as the discarded electrical or electronic devices which are intended for reuse, recycle, resale, or disposal. Technological innovation, market expansion, economic growth and the short life of electrical and electronic equipment (EEE) have led to significant growth in waste of EEE (WEEE). PCBs are the main component of this equipment which generally contains 40% of metals, 30% of ceramics and 30% of plastics [1–3]. The metallic composition consists primarily of 10–30% of copper (Cu) and other metals such as Tin (Sn), Zinc (Zn), Lead (Pb), Nickel (Ni), Iron (Fe), Silver (Ag), Cadmium (Cd), Gold (Au), etc. depending on sources of printed circuit boards

(PCB) [4]. Informal processing of e-waste in developing countries can lead to adverse effects on human health and environmental pollution. In 2016, 44.7 million metric tons of e-waste were produced worldwide [5, 6]. If the e-waste was directly disposed of by filling the soil without removing metal ions from PCBs, the pollution of land and water supplies would result. These metals adopt mediums such as dust, air, water and soil to meet the human framework. 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 eye, nose, mouth, etc. are caused by exposure to Cu which is present in landfills [10, 11]. The 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 very intentional in these 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, the recovery percentage of Cu^{2+} was 97.5 percent, 65 percent and 76.5 percent respectively [12].

A novel ultrasonically assisted treatment process assisted in the reduction, recovery and higher separation from homogeneous heavy metals waste. The studies showed the complete recovery of copper and iron from PCB waste sludge by converting them into separated copper sulfate and ferric chloride solutions. The process has a high separation and recovery efficiency to extract metals. The results indicate that a metal recovery facility treating PCB waste sludge containing 3.14–4.85% copper and 3.71–4.23% iron achieved a copper recovery efficiency of 95.2–97.5% and iron recovery efficiency of 97.1–98.5%. However, because they were fully used in chemical leaching reagents, this process had some limitations in terms of waste emission and effects [13].

Many more studies were performed and reported the various operating conditions on the recovery of heavy metals such as Ag, Au, Ni, and copper found in PCBs. About 80% of the precious metals in the PCB are contained in the particle size ranges from 3.33 mm to 0.43 mm. Column leaching outcome shows that the gold dissolution rate is higher than those of the silver and copper during the first 10 days of the process. From the day 11 there is a reduction in the gold and silver recovery rate due to the copper oxide and copper hydroxide layers on the material surface. The cyanidation of PCBs provides the recovery rates such as 47.9% of Au, 51.6% of Ag, 48.1% of Ni and 77.2% of Cu in a column leaching using NaCN reagent whereas, the activated carbon adsorption process provides 97.3% of Au, 99.3% of Ag, 98.2% of Ni, and 80.7% of Cu [14]. Other useful metals remain as traces in the leaching solution. The deposition of extracted metals possesses different dendritic growth with respect to 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 μm [15, 16]. Significant recovery rates of copper through chemical leaching was reported in our previous researches [13, 14]. The hydrometallurgical method is a great concern to reseachers because it has low consumption of reagents, energy, less environmental pollution. The study deals with the extraction of copper ions Cu^{2+} from PCBs by two stage leaching technology [17] under various conditions of particle size, time, pulp density and temperature of the PCBs and find the optimum value for the maximum recovery of metals in both as well as experimental and predicted value through response surface methodology (RSM).

Researchers employed heavy metals retrieving methods such as electro wining, electro refining, cementation and the ion exchange techniques. These methods have some defects causing release of secondary pollutants, etc. So for this study, adsorption technique with natural adsorbents was introduced. Heavy metals have been acknowledged as potential health and environmentally hazardous materials. Many studies have been shown that these metals are toxic even at low concentrations. The presence of these toxic metals can cause in turn accumulative poisoning, destroy liver cancer, and brain damage when found above the tolerance level. Two locally available adsorbents namely bentonite clay and roasted date pits were collected. The date pits were roasted in an oven at 130°C for 4 hr. and ground in a mill to obtain powder for experimentation. The two adsorbents were analysedby surface area analyzer. The adsorbents are a mixture of heavy metal ions such as copper, cobalt, zinc, lead, arsenic, cadmium and chromium in the industrial waste water. The heavy metal concentration levels in the industrial waste water were above the permissible concentration levels. In addition the minimum removal efficiency of metal ions by adsorption using bentonite clay and the roasted date pits was 97% [18]. 15 g granules of the mobile PCB sample were leached in the 250 ml solution using 500 ml glass beaker which contained the pre determined amount of the ammonium thiosulfate and copper sulfate at various pH values. All leaching experiments were carried out at an agitation speed of 250 rpm and temperature. After 8 hrs leaching the solution was removed and was filtered by the Whitman 40 filter paper to separate the residual PCBs from the solution. The residue was then dried in the vacuum oven for 2 hr. at 130°C to remove all the moisture from the sample and the samples were weighed and the weight of the residue was calculated. In case PCB granules 56.7% gold could be leached under the optimized conditions viz., ammonium thiosulfate 0.1 M, stirring speed 250 rpm and at room temperature in 8 hr. time duration. In case of complete PCB unit the maximum gold leaching was 78.8% at thiosulfate 0.1 M, copper sulfate 40 mm, Ph 10–10.5, stirring speed 250 rpm at room temperature in 8 hr. time duration [19].

The residual mercury is to be treated and deposited in a geological repository with a clay barrier between the waste and the rock. In reality, hazardous chemical waste is for the most part deposited at the surface under drained conditions, while long-lived and high level nuclear waste in most programmes is intended to be deposited in geological formations below the ground water level. For sodium based bentonites, two modes of swelling exist: crystalline and osmotic. Crystalline swelling takes place only during the addition of low fractions of water. While osmotic swelling can take place for much larger additions. In the present work, the feasibility of applying nuclear long-lived waste disposal concepts to chemical hazardous substances is being tested. The elements needed for at least a simple safety analysis are identified and described in the present paper and will be tested experimentally and theoretically. In addition to mobility tests, the experiment included demonstration of a technique for compacting a mixture of spent batteries and bentonite clay. From the experiment, the sufficient extraction of chemicals was taken [20].

Based on previous researchers copper recovering techniques from various wastes and to overcome the drawbacks, the researchers opt for a new method such as adsorption by bentonite clay. Clays and clay minerals are of great importance due to the unique properties including hardness, durability, strong plasticity and plasticitythat make them ideal for industrial applications [21, 22]. Due to their complex shapes, clays have limited particle sizes and highly specific surface areas. They have been recognized as one of the most suitable low-cost adsorbents and standard components in a variety of industrial applications. Bentonite is an aluminum phyllosilicate adsorbent derived from montmorillonite. It is a sedimentary rock composed primarily of clays with a typical 2:1 layer structure and high concentrations of Na^+, Ca^{2+}, and K^+ ions found between the layers. Acid treatment on clay minerals has additional mineralogical and mineralogical impacts on a mineralogical system [23]. Due to its cation capacity, greater surface area and adsorption capacity for various organic and inorganic ions, acid-activated bentonite has been a traditional commodity for removing metal ions. In this present study, Copper recovery from PCB leached solution with treated bentonite clay has been studies thoroughly and the experimental results are optimized through RSM.

2. Materials and methods

2.1 Sample collection and preparation

The waste PCBs are obtained from e-waste disposal units in India. For experimental use, 500 g scraps of PCBs are broken into 15–20 cm particles and shredded using pliers and four blade cutting shredder into small pieces around 50 x 50 mm to 30 x 30 mm [12, 17, 24]. Metals and non-metals need to be separated [15, 16]. This separation is not as simple due to the difference in the physical characteristics of metals and non-metals. Hence, 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, 17].

The crushed PCBs obtained from the crusher are then pulverized and further exposed for milling operation 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 clearance in the same jaw crusher, yielding samples weighing 65, 53, 48, and 36 grams 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 [8], 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 in 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 grams 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 lowest sieves. The weight fractions obtained at each sieve are collected separately and subjected to leaching (**Figure 1**).

2.2 Chemical leaching experimentation with aqua regia

2.2.1 Aqua regia preparation

The metal recovery from PCBs is carried 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].

Aqua regia preparation involves mixing of strong acids. The two concentrated acids were mixed in 3:1 ratio (HCl:HNO_3), Concentrated HCl (35%) and HNO_3 (65%). The solutions should be keptaway from organic contaminants, because it leads to vigorous or violent reaction and low temperature should be maintained.

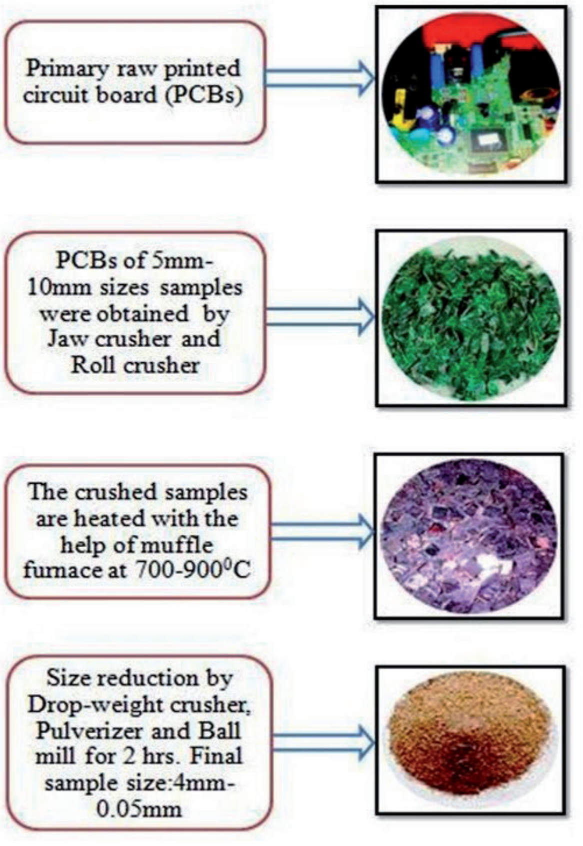


Figure 1.
Schematic diagram of primary raw PCBs; in to stepwise size reduction under the various mechanical operations (jaw crusher, roll crusher, thermal heater and pulverized mills produced small sizes between 4 and 0.05 mm).

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]

Table 1.
Recovery data of copper with different leaching agents.

2.2.2 Experimentation with various parameters on copper recovery

All the experiments were conducted in a conical flask with a temperature controlled shaker. Primary analysis was conducted by applying specific conditions to obtain a standard recovery rate. 20 g of PCB samples are allowed to mix with 0.5 liters of leaching media at 80°C and shaken in a mechanical shaker at a shaking speed of 120 rpm for 3 hours. At the end of this contact time, the shaker is stopped and solutions in the conical flask are filtered using filter paper. After complete filtration and metal composition retained is determined. The leaching rate depends on various parameters such as shaking intensity, size, contact time, pulp density and temperature. Different values for the recovery rate and the composition of heavy metals are obtained by varying these parameters. The samples are then tested and time results are analyzed over the recovery rate. The leached copper was reclaimed with the help of bentonite clay.

2.3 Copper ion reclaimation by adsorption

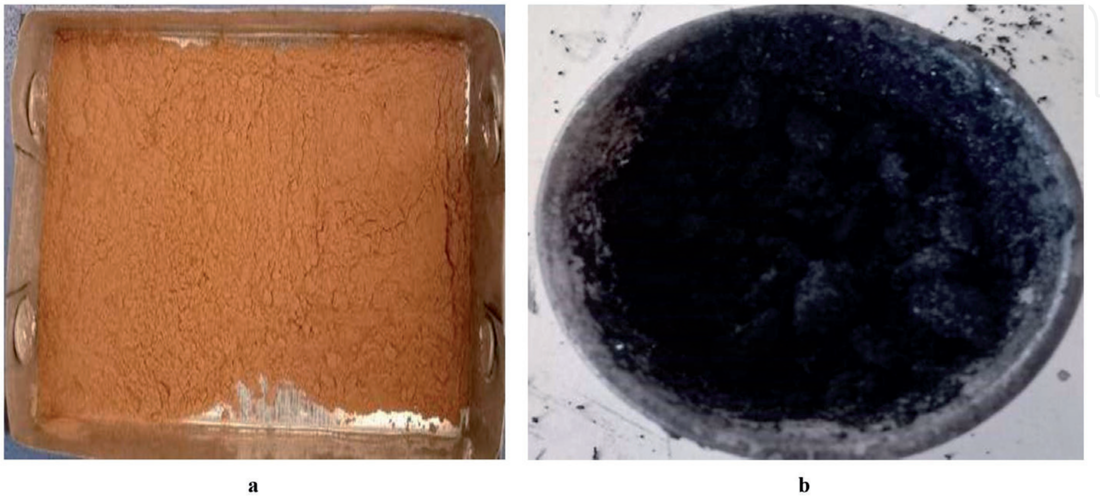
Adsorption operation is extensively applicable in chemical operations for the reclamation of copper ions from the leached solution. Some other techniques have been used in previous studies, like precipitation, cementation process, liquid membrane techniques and ion exchange process. These methods have their specific advantages and disadvantages. Some of the methods are:

- Precipitation methods were used in precipitating reagents such as carbonate, sulfide and hydroxide which are precipitated from leached solution to insoluble form [25]. The disadvantage of this precipitation method is the formation of a huge quantity of sludge that contains toxic compounds.
- To overcome this defect, the copper metal reclaiming process was done by another method called the cementation process, which involves metal displacement reactions [26]. 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 operation 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 these A-Bent adsorbent.

2.3.1 Physical activation method

250 grams of both adsorbents were taken in the thermal crucible and were dried for about 5 hr. for thermal activation at 900°C. The samples obtained are from 1 μm to 5 μm. The higher specific surface area is obtained due to the removal of unwanted gaseous molecules from the Non-Activated Adsorbents (NAA). The activated adsorbents are shown in **Figure 2a,b**.



a b

Figure 2.
(a) The schematic diagram for bentonite clay thermal activation at 600°C. (b) Chemical activation by used concentrated HCl and HNO_3.

2.3.2 Chemical activation method

The chemical activation involves the chemical reaction of the precursor with the activating agent at temperature 600°C. Initially, the Bentonite clay is washed with tap water and undergoes solar drying until the complete moisture content is removed. Once the moisture is completely removed from the adsorbent again, it will be washed with tap water and again dried under sunlight. The materials were mixed with a Nitric acid solution (85 percent of purity) at a ratio of 1:2 by mass (materials: HNO_3 solution) and they are stirred well for 2 h and then conveyed to a stainless steel plate which is placed in a muffle furnace and heated at 550°C for 2 h. By natural cooling, the temperature is brought down to room temperature. Then, the adsorbent samples (C-A Bent) were crushed to less than 1 μm size and all the adsorbent samples were weighted and washed with 0.1 mol L^{-1} HCl to remove the surface ash.

Then, the adsorbent samples were washed with de-ionized water to remove the HCl and dried for 24 h at 150°C. After drying, both samples were ground and sieved. Chemically activated samples of both adsorbents are found favorable surface properties like C-A Bent have the maximum specific surface area 817 $m^2\ g^{-1}$, less than 0.5 μm sizes and Pore volume is 0.1 $cm^3\ g^{-1}$. Then, the adsorbent samples of C-A PSC have the specific surface area of 1026 $m^2\ g^{-1}$, pore volume 0.37$cm^3\ g^{-1}$ and pore size 0.5 μm. The prepared samples were tested with the help of the Scanning Electron Microscope (SEM-FEI-Quanta FEG 200F) which is shown in **Figure 3a,b**.

2.3.3 Adsorbent characterization and studies

The feed to adsorption is copper solution recovered by leaching. Adsorption of copper ions on Bent was carried out in a batch system in both activated and NA-Adsorbents. 2 gram of adsorbent was added to 20 ml of the leached solution in a conical flask. The mixture was to be shaken at 200 rpm for 5 h at 80°C. After complete adsorption is done, the samples were filtered and copper concentration was analyzed by using EDXs 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 100Na, Schottky emitter ranges between(−200v to 30 kV), magnifications range 12X−105X, and resolution of 2 Nanometer (Gold Nano-particles suspended on carbon substrate). Then, the adsorption efficiency of an adsorbent (Bent) was determined by the following Eq. (1).

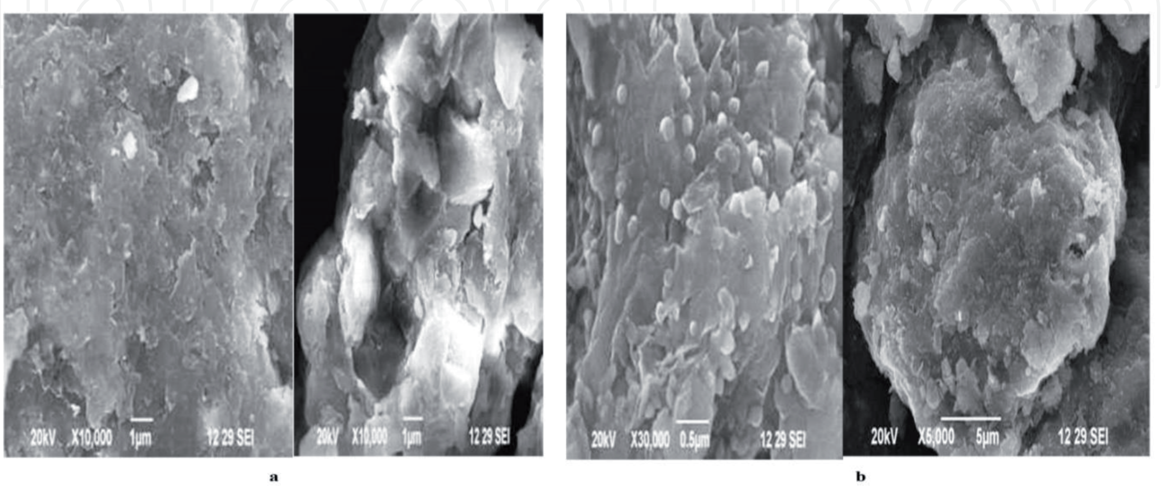


Figure 3.
(a) The SEM images of bentonite clay thermal activation at 600°C. (b) Chemical activation by used concentrated HCl and HNO_3.

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 sizeμm	0.4	2	5

Table 2.
Levels of different process variables in coded and un-coded form chemical leaching % of copper ions (box-Benhken method).

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

C_o is the initial concentration of metal ions from leached samples. C_e is the metal ions concentration after adsorption operation [27].

2.4 Response surface methodology (RSM)

Studies were conducted in order to obtain the optimum valves of various parametersfrom the recovery of copper ions from leached solution by Response surface methodology. The influence of various parameters (Size of adsorbent, adsorbent dosage and temperature) were studied for copper ions recovery. In this analysis, input parameters were taken into account aretemperature, adsorbent dosage and temperature. Based on the ideal experimental conditions for the shaking intensity and dimensions of the metals optimum recovery percentage the leaching variable input parameters were calculated (**Table 2**).

3. Results and discussion

3.1 Sample analysis of PCBs (sizes and metal elements)

The graphical representation of the size analysis reveals that, subject to size decrease sequence, the fraction of sample generated on the screens with larger mesh sizes has decreased. The total weight collected in the sieves is, however, maintained similar roughly 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 less than 0.5 mm, contributing to an elevated copper recovery rate [25]. Present findings consist of 0.05 mm of the sample held above the pan for the liquidation used particle scale. EDXs have been used to analyze the copper concentrations of preliminary samples. To ensure uniformity and to obtain results of copper by EDXs, samples were randomly mixed (**Figure 4**) and the final composition of metals by weight % (Cu 3.15%, Sn 42.4%, Zn 1.16%, Pb 27.81% and others metals 25.48%).

3.2 Maximum copper recovery of leaching by optimization study

Experiments carried out based on RSM results. In addition to that ANOVA, response surface plots, quadratic model equation and CCD were analyzed for experimental conditions. Hence, the results obtained for Optimum removal of Cu 95.33%, with a desirability of 0.761 were obtained at Time 5 hours, Temperature 90.01°C, pulp density 25 g L^{-1}.

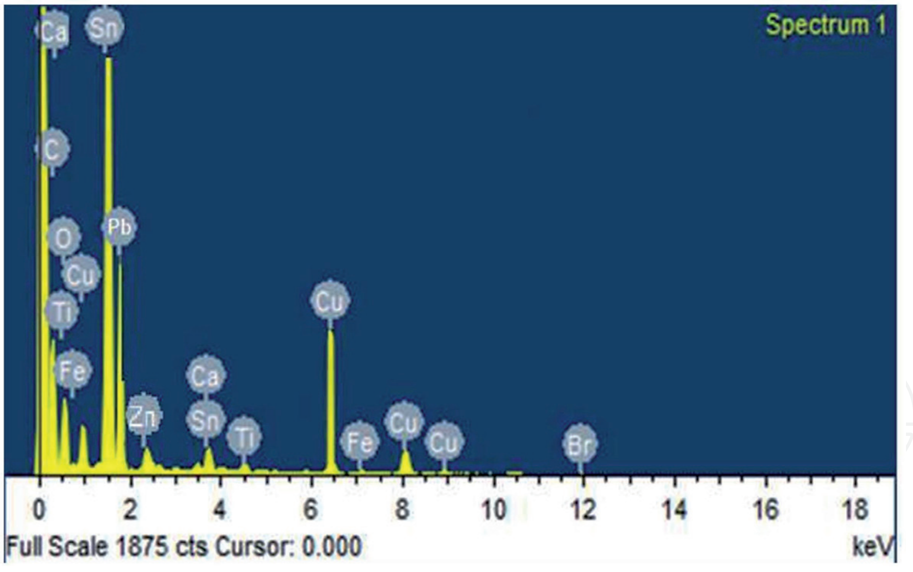


Figure 4.
Presents of copper ions from PCBs sample by EDX_S.

The optimum values were found under the studied parameters at which the maximum recovery is obtained. Therefore, The experiments done above optimized three parameters with two experimental parameters (80 rpm of speed and 0.05 mm particle size). Therefore, the optimized gives 20 grams of the sample treated with 0.5 liter of aqua regia at this optimum condition, metal compositions present in to the PCBs by after leaching (Cu 0.09 weight percentage) are shown in EDXs results (**Figure 5**). The results obtained at optimum condition shows that the recovered rate of copper is 97.06%.

3.3 Adsorption studies for copper recovery from leached solution

Adsorption studies are explained for the recovery of coppers from leached solution with the help of Bentonite clay as an adsorbent. Hence, all the adsorption results for recovery concerning various parameters are evaluated and studied as

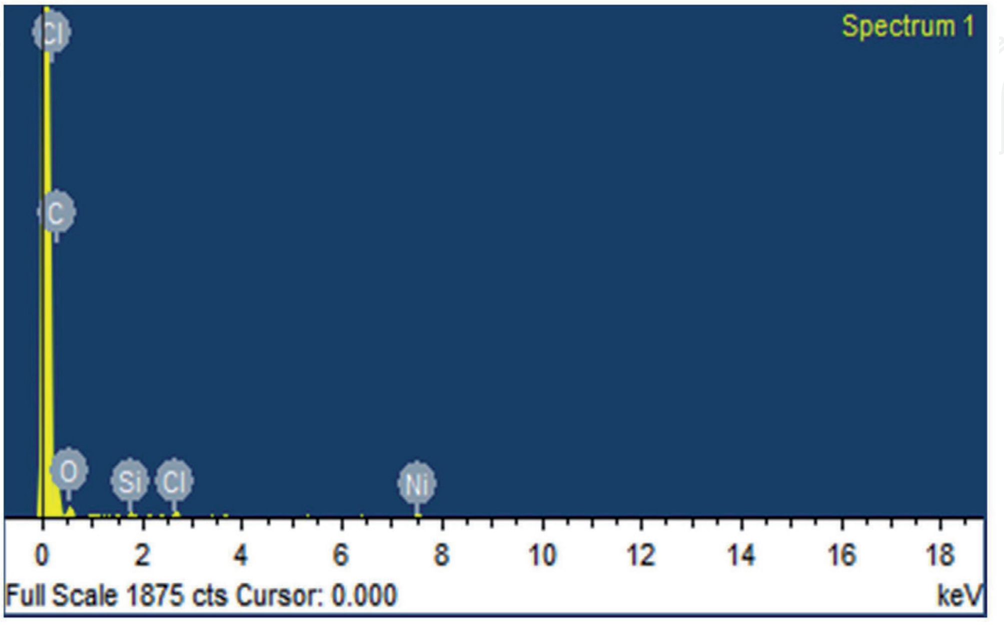


Figure 5.
EDXs spectrum analysisfor copper ions removal after leaching treatment of (PCBs).

explained based on previous research [28] and compared; we get the optimum condition to obtain maximum recovery of metals. The optimum condition is the value of concentration, size, temperature and time at which the maximum recovery is obtained. The optimum conditions are 4 g of adsorbent dosage, 0.05 μm particle size of adsorbent, 80°C of temperature and 4 hours of contacting time. Under these conditions, chemically activated bentonite clay gives maximum adsorption rate when compare to other adsorbents. Therefore, the present study was experimented to recover the copper ion (Cu^{2+}) from the leached solution with the use of optimal parameters and constructive results obtained in chemically activated adsorbents.

The results show (**Figure 6**) that at optimum condition, the recovery is 97% of Copper. This is the most favorable condition to obtain the maximum recovery of copper ions which was found initially 3.119 weight percentage of copper and after copper present in adsorbent 3 weight percent therefore copper were recoverd 97.33%. The optimal values are tested the specified parameters by response surface methodology.

3.4 Optimization parameters by design of experiments (DOE)

Optimize and evaluate individual process variables for better recovery rates by analyzing operating parameters and reducing the number of tests. The CCD (Central Composite Design) for Cu, adsorption was calculated with optimized operating parameters and RSM maximum copper recovery. The CCD results shown in **Table 3** experimental and predicted copper adsorption were analyzed.

3.4.1 RSM for copper reclaimation from leached solution

Statistical modeling methods were used to evaluate the multiple regression of the experiments designed to determine the multivariable equation (**Table 3**). RSM concept data plots collected in the final regression equation in terms of coded recovery variables for Cu recovery. The final equation in terms of the coded factors equation discussed Eq. (2). The equation in terms of 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

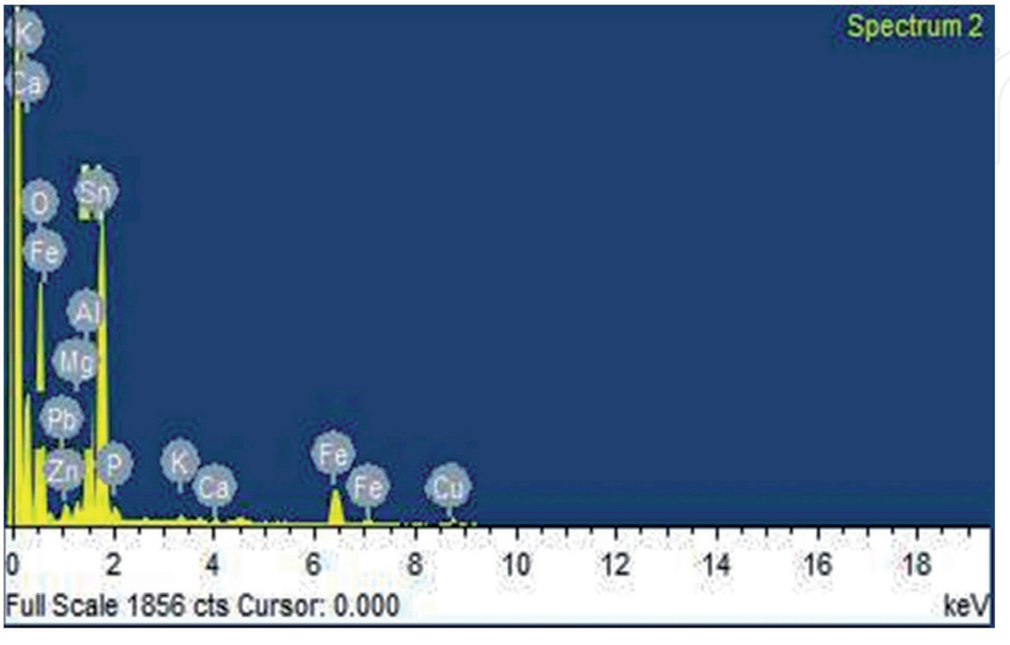


Figure 6.
EDXs images in metal compositions for after adsorption.

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

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

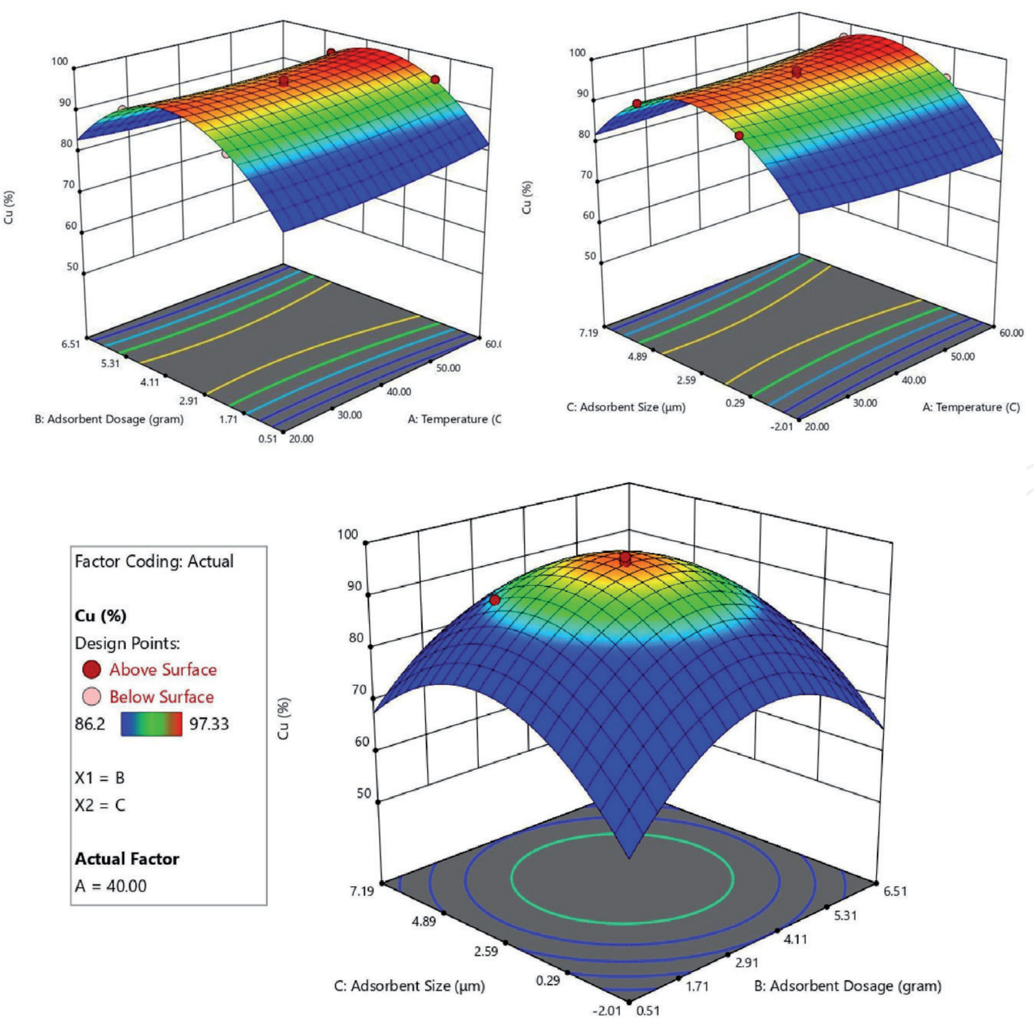


Figure 7.
RSM plots and interactions between the the temperarure, adsorbent dosage and size of adsorbent by Cu recovery.

coded equation is useful for identifying the relative impact of the factors by comparing the factor coefficients.

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

The response of each parameter was Predicted within the limits through the model in function of coded factor. Here, the maximum and minimum coded factor termed as +1 and −1. The response surface were visualized in three dimensional plots that exhibit two factors functions while keeping the other factors constant. The predicted design plots, shows the above red zones were found at 97.33% of Cu and above yellow zones confirms 93% of Cu, and above blue colors confirms 88.95% of Cu. It shown in **Figure 7** and contour plots for copper recovery (**Figure 8**).

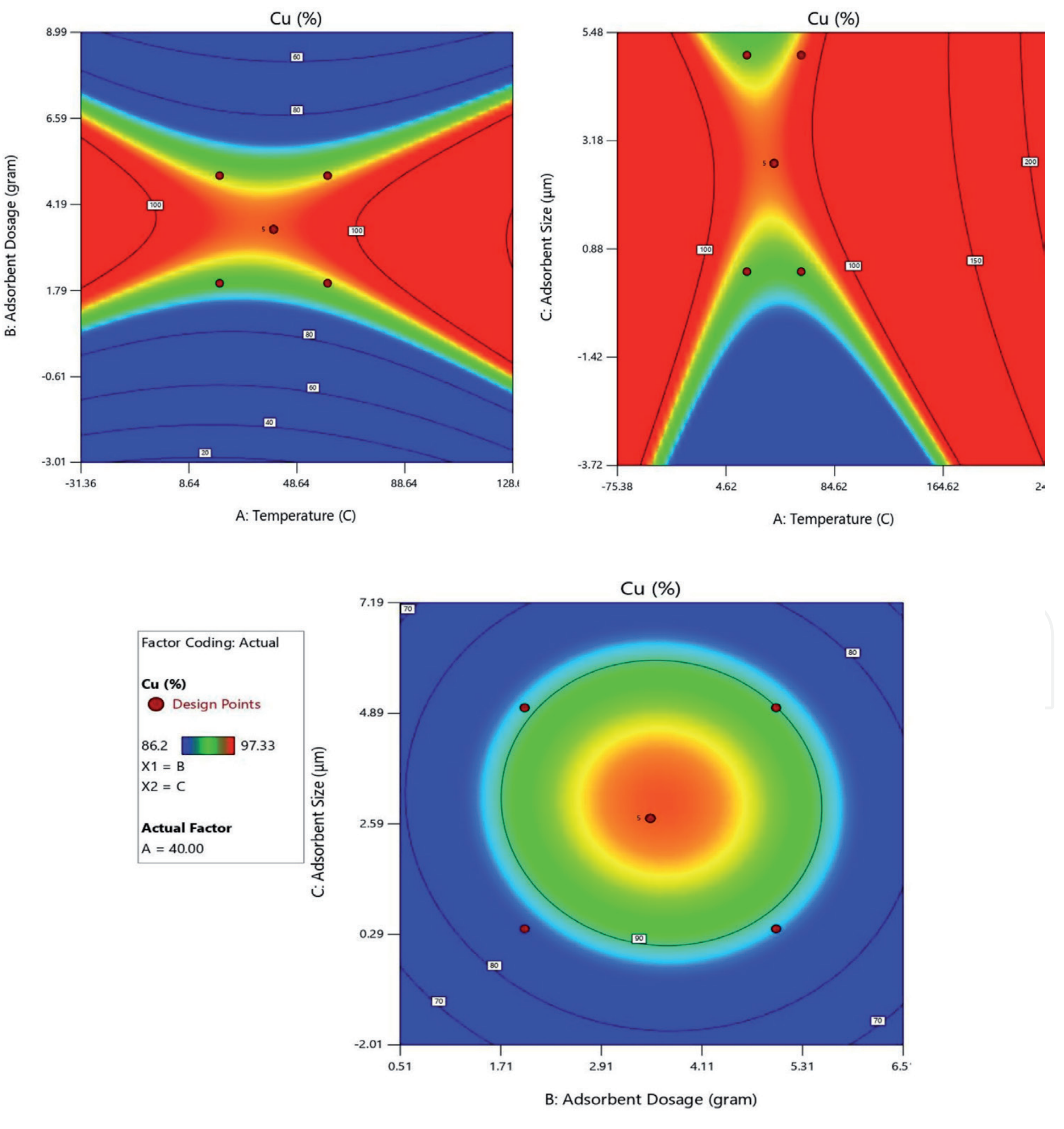


Figure 8.
Contour plots and interactions between the temperarure, adsorbent dosage and size of adsorbent by Cu recovery.

3.4.2 Evaluation of the model

P-values less than 0.0500 indicate model terms are significant. In this case A, B, C, AC, A^2, B^2, C^2 are significant model terms. Analysis of variance correspond to experimental results were presented in **Table 4**. The Model F-value of Cu 34.28 implies the model is significant. There is only a 0.01% chance that an F-value this large could occur due to noise. Also, the acceptable and reasonable value of lack of fit with F-value of Cu 0.9321, with probability (>0.05) indicates the suitability of the method for good presentation of experimental data. Implied that the model was accurate. Also, the acceptable and reasonable value of lack of fit with F-value of Cu 0.02322, with probability (>0.05) indicates the suitability of the method for good presentation of experimental data.

As presented in **Table 5**, the model presents the high R^2 value of Cu0.9778 indicates that there was a good agreement between the experimental and predicted

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 4.
ANOVA table for model to predict % of leaching 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

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

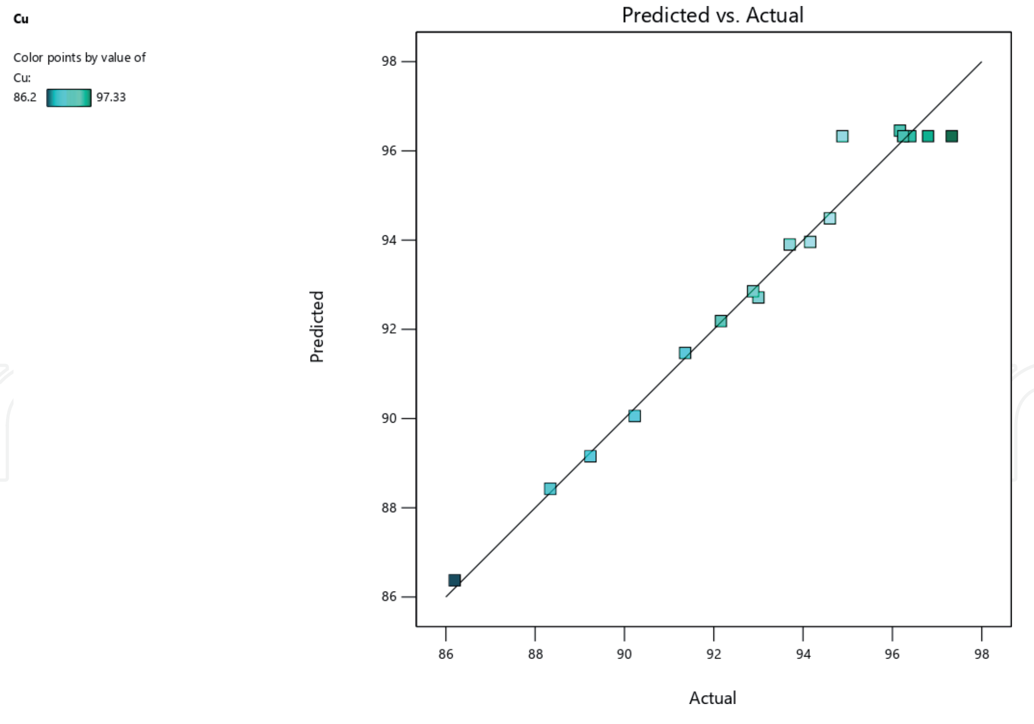


Figure 9.
Comparison plot between the experimental and predicted data.

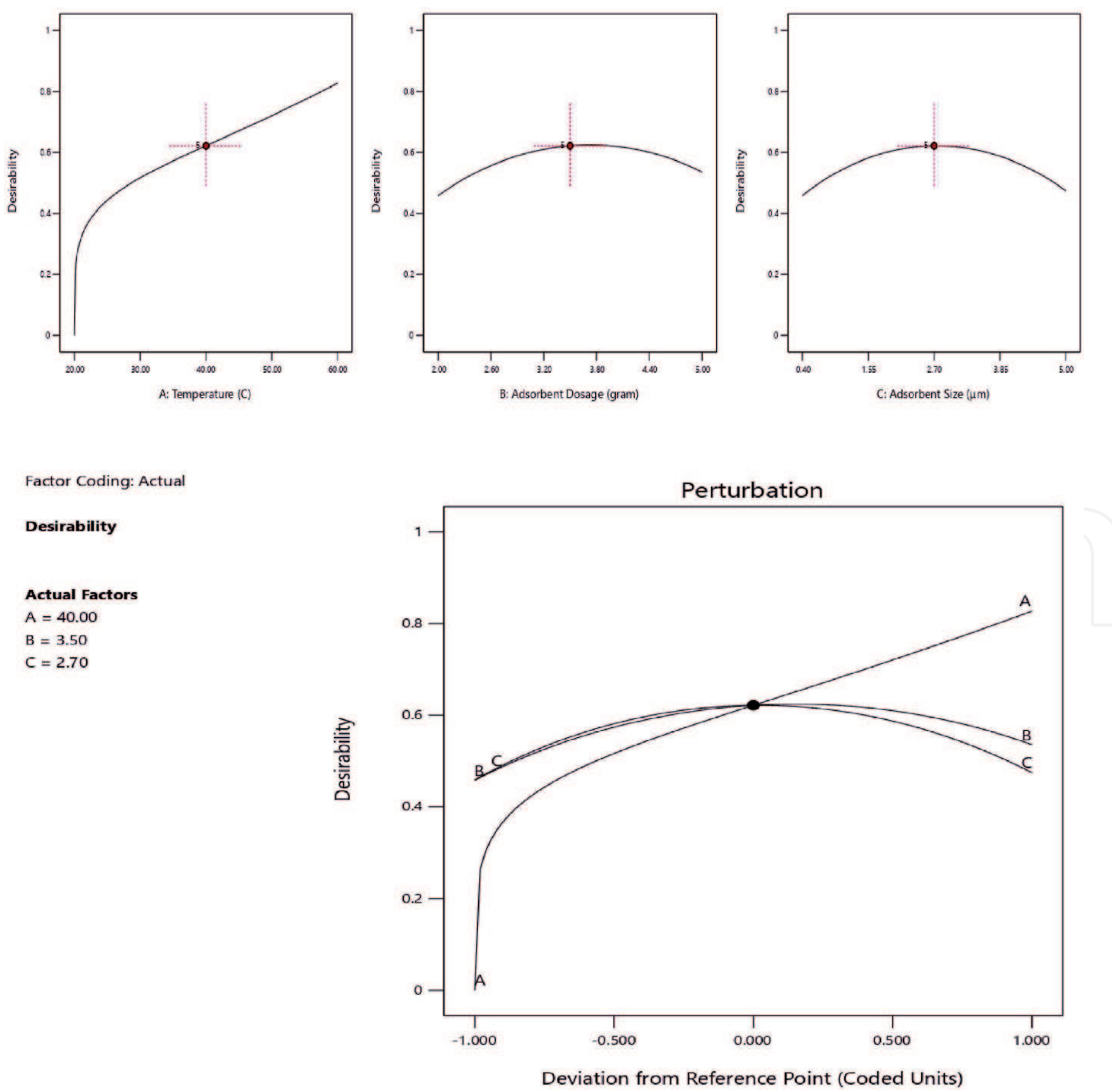


Figure 10.
Desirability plot for recovery of copper from leached solution.

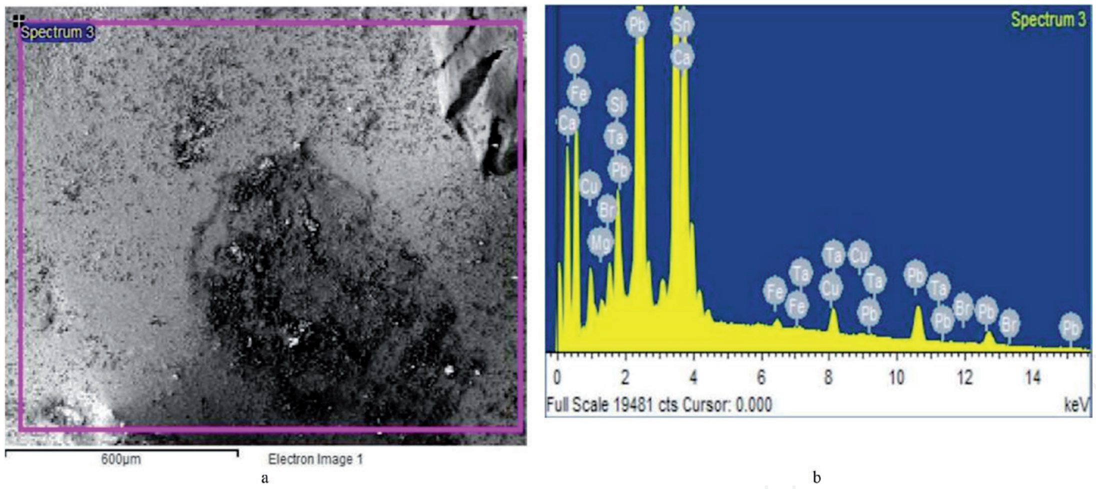


Figure 11.
EDXs spectrum analysis for metal ions obtained for after adsorption.

results. Also, the predicted R^2 value Cu 0.9353was which were in reasonable agreement with the adjusted R^2 value of Cu 0.9493. Adeq Precision measures the signal to noise ratio. A ratio greater than 4 is desirable. Your ratio of Cu 18.125 indicates an adequate signal. This model can be used to navigate the design space. Predicted value were Showed (**Figure 9**) of the responses from model was in agreement with observed values over the selected range of independent variables with reasonable higher values of coefficient of determination (R^2).

3.4.3 Desirability plot for recovery of copper from leached solution

The desirability profile for the removal percentage of copper versus the variables is shown in **Figure 10**. The desirability varies from 0.0 to 1.0 corresponds to approaching from undesirable to the very desirable condition. Optimum removal of Cu 97.33%, has been obtained with the desirability of 0.845 which was obtained at adsorbent dosage 2 gm L^{-1}, size of adsorbent 0.4 mm temperature 90°C.

Therefore, this design has been analyzed for experimental and predicted valves of metal separations' of chemical leaching, the values of desirability rate was found in the range of prediction is 0.845. Since the optimum values are predicted then optimal parameters are used to run the copper recovery process.

3.5 Maximum copper recovery by optimization study

Experiments carried out based on RSM results. In addition to that ANOVA, response surface plots, quadratic model equation and CCD were analyzed for experimental conditions. Hence, the results obtained for Optimum removal of Cu 97.33%, with a desirability of 0.845 were obtained at adsorbent dosage 2 gm L^{-1}, size of adsorbent 0.4 mm temperature 90°C.

The optimum values were found under the studied parameters at which the maximum recovery is obtained. Therefore, The experiments done above optimized three parameters with two experimental parameters (size of adsorbent 0.4 mm temperature 90°C). Therefore, the optimized gives 2 grams of the sample treated with 0.5 liter of leched solution at this optimum condition, metal compositions present in to the PCBs by after leaching (Cu 0.09 Weight percent) are shown in SEM with EDXs results (**Figure 11**). The results obtained at optimum condition shows that the recovery of copper are 97.06% of copper.

4. Conclusions and outlook

In summary, copper in waste PCBs were leached into corresponding reagents during the two-stage chemical leaching. The effectiveness of two-stage chemical leaching media (HCl and HNO_3, H_2SO_4 and HCl) was employed for the separation of copper ions during the treatment of PCBs is evaluated. The results of this study, C-A Bent adsorbents assist in the 97% of effective copper separation for Chemical leached solution Therefore, the Study concluded that, copper ions are recovered effectively from leached solutions by using adsorption techniques under optimum conditions in the presence of C-A Bent adsorbent. These types of metal leaching operations are promoted in order to reduce the environmental problems caused by these kinds of heavy metals. The analysis demonstrates the dependency of the recovery rates. Optimum removal of Cu 97.33% with a desirability of 0.845 was achieved at adsorbent dosage 2 gm L^{-1}, Size of adsorbent 0.4 mm temperature 90°C. Hence this form of heavy metal leaching and adsorption reclamation process is proposed with a view to reducing environmental impacts (caused by heavy metals). It was concluded that the combination of aqua regia leaching and bent adsorption is an effective and economic way for the recovery of copper from leached solution.

According to studies, modifying the surface of the clay increases the rate of adsorption, but this raises the total cost and results in the introduction of additional chemicals into the atmosphere. As a result, attempts will be taken in the future to resolve these issues. Only a few field trials have been performed, and more systematic studies are needed to decide the best conditions for using clay minerals as adsorbents.

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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	sulfuric acid
Sn	tin
WEEE	waste of electrical and electronic equipment
Zn	zinc

Highlights

- The heavy metals in PCBs were leached with two-stage aqua regia (first stage-HCl and HNO_3 and second stage-HCl and H_2SO_4) in order to get more recovery rate.

- A multi response optimization procedure based on the response surface methodology has been applied. The task of the optimization problems has been the maximization of the recovery of copper.

- Adsorption (Bent) clay minerals as an adsorbing media to recover copper from a chemically leached solution of PCBs.

- The overall leaching efficiency of copper (Cu) was found at 97.06%.

- Since this kind of extraction has proved to be successful in the separation and recovery of copper ions, it is not advisable to extract specific metals in a targeted manner, even though they are economically viable.

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