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Validation of Bromide Leaching through Response Surface Methodology and Separation of Gold from Waste Printed Circuit Boards

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Abstract: The daily consumption of gold is increasing worldwide; however, its availability from conventional ores is reducing. Alternatively, the presence of gold in waste electrical and electronic equipment (WEEE) is nearly 100 times higher than in natural ore. Therefore, the possibility of the recovery and separation of gold from waste printed circuit boards of obsolete mobile phones is studied in the present work. Initially, the optimization of parameters for the quantitative gold leaching from metal clads of PCBs with halide salts at acidic conditions is studied through response surface methodology. Three factors (parameters), viz. temperature, time and stirring speed are altered during the experiments based on the central composite design (CCD). Leaching parameters have been optimized with the help of the second-order empirical equations and analysis of variance (ANOVA) for maximum gold dissolution. The selective recovery and separation of gold from leach liquor have been achieved with solvent extraction with an organic amide as extractant followed by cementation with zinc powder. The separated gold powder has also been analyzed with XRD and SEM-EDS to check the purity and homogeneous elemental distribution. The statistical design of experiments and separation processes for the effective recovery of gold corroborates the economic feasibility of the proposed process.

Keywords: gold; response surface methodology; leaching; waste printed circuit boards; cementation

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1. Introduction

The widening gap in the supply-to-demand ratio for gold is steadily surmounting due to its vast usage in electrical systems, catalysts, biomedical devices, fuel cells and jewelry production in particular post pandemic [1,2]. Our technologically driven society estimates the consumption of gold at 263.3 t/year [2]. The limited resources of the metal reserves is increasing the price of gold, and the traditional recovery process makes the ecosystem toxic. In contrast, the presence of gold in electrical and electronic waste (or e-waste) is almost 100 times higher than in conventional ore, and its rapid production can substitute the traditional recovery process [3]. The annual worldwide stock of WEEE touched 53.6 million tons in 2019 and is predicted to surpass 74.7 million tons by 2030 [3]. It is increasing drastically with a CAGR of 3–5% per annum, approximately thrice that of the other urban classified wastes [4]. The printed circuit board (PCB) is an attractive secondary resource within any electronic device with an interwoven metallic fraction (40 wt%) [4] and essentially with a high tenor of base, precious and critical metals compared with primary ores [5–7].

The hydrometallurgical recovery of gold is traditionally treated with cyanide leaching. However, environmental issues have led to the development of several alternative techniques such as thiourea [8], thiosulfate [9], thiocyanate [10] and glycine solutions [11]. Moreover, the aforesaid lixiviants are less efficient when compared to cyanide

leaching and are governed by complex chemical kinetics [8]. Oxidative halide leaching is another option where chloride or bromide is generated in situ by oxidizing their halide salts in the presence of acidic medium to extract gold from obsolete PCBs as auric ions [12,13].

However, the chlorination leaching method has disadvantages such as the difficulty in controlling the chlorine gas released, and corrosion problems [14]. Similarly, in the case of iodine, a higher consumption of leaching reagent also occurs [15]. On the other hand, in bromine leaching, the generation of stabilized bromine, which has lower a vapor pressure than liquid bromine, achieved higher gold recovery from conventional ores [16]. In bromine medium, the gold is oxidized by bromine and stabilized as the gold bromide complex, as represented in Equations (1)–(3). Studies of the reduction potential (Eh) vs. pH diagram for the feasibility of gold in halide medium revealed that leaching of gold is possible [13,15,17].

$$2NaBr + 3H_2SO_4 \rightarrow 2NaHSO_4 + Br_2 + SO_2 + 2H_2O \quad (1)$$

$$Br_2 + 2e^- \rightarrow 2Br^- \quad (2)$$

$$Au + 4Br^- \rightarrow AuBr_4{}^- + 3e^- \quad (3)$$

The selective separation of Au from liquors containing a mixture of metals has been widely investigated [18–20], and among these studies, solvent extraction is a versatile process. The extraction of gold from mixed metal halide leach solution using an amide as a carrier has also been developed [21]. The back extraction of gold from organic solution is also easier with water or sodium hydroxide as a stripping reagent [12,22]. However, the separation and production of gold from the proposed bromide leaching process have rarely been studied. In the conventional gold leaching process, chemical precipitation of gold with zinc is well known as the Merrill–Crowe process [23]. In that process, initially, oxygen is removed by passing the filtered cyanide solution through deaerating columns, and zinc powder is added, which assists in the reduction and concurrent precipitation of gold as shown in Equation (4) [24].

$$Zn_{(s)} + 2Au(CN)_2{}^-{}_{(aq)} \rightarrow 2Au_{(s)} + Zn(CN)_4{}^{2-}{}_{(aq)} \quad (4)$$

In the present work, initially, the optimization and validation of gold leaching parameters such as temperature, time and stirring speed with sodium bromide and H_2SO_4 as a leaching reagent have been studied by the response surface methodology of design of experiments (DOE). WPCB metal clads residue was used for the leaching of gold post removal of base metals. The liquid–liquid extraction (solvent extraction) was carried out with 3° amides as a carrier and sodium hydroxide as a stripping reagent to selectively extract gold from the leached solution. Finally, the separation of gold from the strip solution is also studied by cementation with zinc powder and desorption of zinc with sulfuric acid. A detailed explanation of the proposed process is also shown in Figure 1.

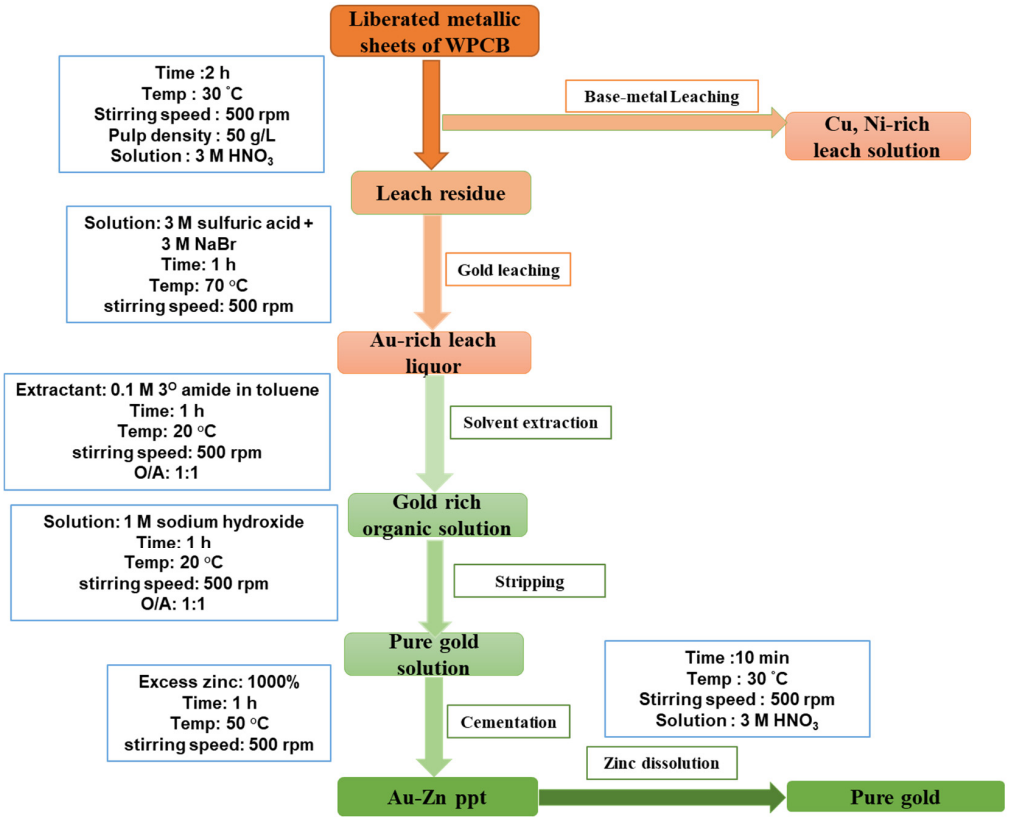

Figure 1. Flowsheet of the proposed process.

2. Experimental Section

2.1. Pre-Treatment and Removal of Base Metals

Waste printed circuit boards (WPCBs) of obsolete mobile phones are collected from e-waste retailers, in Varanasi. The removal of attached components has been achieved manually with mild heating. The cleaned PCBs are cut into 1 × 1 cm^2, and the liberation of metal clads from non-metallic fraction is observed with chemical pre-treatment, i.e., by dissolving the epoxy resin quantitatively in dimethylacetamide at 160 °C for 10 h [12]. After complete dissolution of the resin, metal clads are separated by vacuum filtration. The obtained metal clads are dissolved in aqua regia (1:3 molar ratio of HNO_3 and HCl) and analyzed with atomic absorption spectroscopy (AAS) (Elico Make, Model-SL168). The composition of metal clads is shown in Table 1. XRD pattern of the metal clad is also shown in Figure 2. The metal clads are initially used for the elimination of base metals to enhance the gold concentration [25]. The optimized parameters for the base metal leaching without any dissolution of gold are observed with 3 M dilute HNO_3 at 30 °C in 2 h [25]. Leaching experiment is carried out in a flat-bottom three-mouth reactor. A measured quantity of leach reagent (50 mL) is poured into the reactor and heated to the desired temperature using PTFE-coated hot plate attached with a magnetic stirrer and PID controller setup. Samples were withdrawn at pre-determined time intervals and then diluted in deionized water for analysis by AAS. The leach liquor is filtered after 3 h, and the obtained residue is utilized for gold leaching.

Table 1. Elemental composition of WPCB metal clad with plastic residue.

Element	Cu	Ni	Au	Ag	Sn	Zn	Cd	Pb	Ceramic and Plastic Residue
Delaminated metal clad (wt%)	84	2.3	0.05	0.03	0.4	0.4	0.2	0.1	12.4

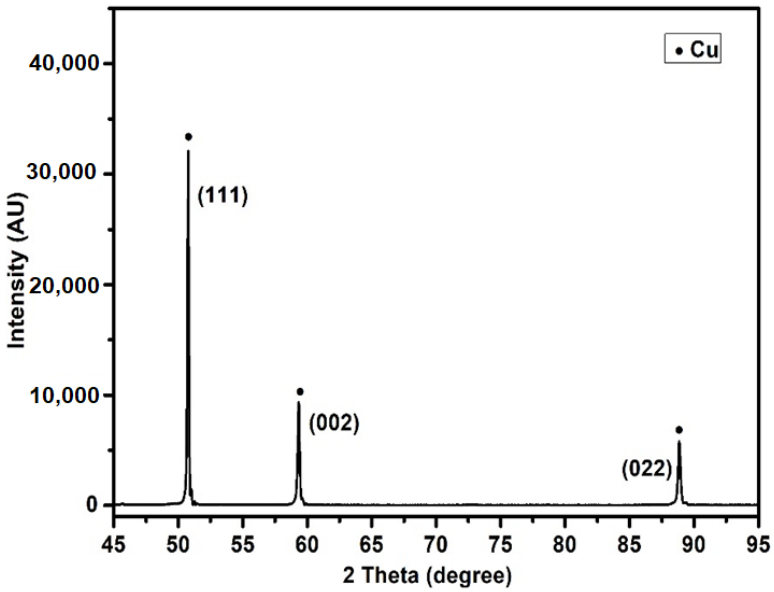

Figure 2. XRD pattern of separated metal clads of WPCBs.

2.2. Experimental Design for Gold Leaching

Design of experiments (DOE) is an appropriate method which serves the purpose of reducing the number of experiments in several experimental situations to optimize an effective response and save time [26]. To assess the relative significance of parameter deviation with the combination of the measurable data, response surface methodology (RSM) can be a valuable modelling tool which can be achieved from experimentations with mathematical and statistical equations to enable the optimization of parameters [27]. A three-factor, two-coded level central composite design (CCD)-based RSM was applied to corroborate the ideal conditions for the gold recovery. The levels are coded with -1 (low) and +1 (high). For leaching validation, time (A), temperature (B) and stirring speed (C) were considered as independent factors, with gold leaching as the response variable using a pre-optimized 3 M sodium bromide in the presence of 3 M sulfuric acid [12]. The CCD model not only renders the effect of individual and combined parameters on gold leaching, but it also assists in the analysis of statistical models for optimization of the process, and its adequacy. A representation of the independent factors (lower to higher) is displayed in Table 1 by assuming the optimized value is between them. CCD with three factors forms a cube representing axial points (or star points) (a point that lies on either of the axes in a coordinate system) (2*k), corner (or factorial) points (2*k) and center point, as shown in Figure 3, where k is number of factors (or parameters), i.e., 3.

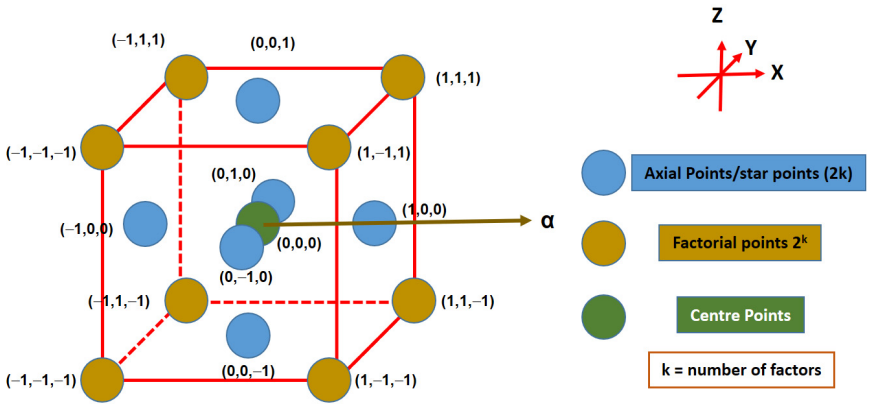

Figure 3. Central composite design (CCD) for three factors used in the present work.

The distance of every axial point with respect to the center in a CCD is termed as alpha (α). An alpha value lower than one (<1) places the axial points inside the cube, a value equivalent to one puts them on the faces of the cube, and a value higher than one (>1) places it outside the cube. In the present work, α is considered as 1. Therefore, minus α and plus α values are considered equal to the lower and higher levels of the factors, respectively, as shown in Table 2. A comprehensive interpretation of the optimization of variables can be achieved by analyzing the data by RSM.

Table 2. RSM parameters for gold leaching.

Parameter	Units	Low (−1)	High (+1)	$-\alpha$	$+\alpha$
Time (A)	min	10	60	10	60
Temperature (B)	°C	30	90	30	90
Stirring speed (C)	rpm	100	500	100	500

Gold Leaching

The filtered residue obtained from base metal leaching is utilized for gold leaching with previously optimized leaching reagent, i.e., 3 M sodium bromide in the presence of 3 M sulfuric acid [12]. Gold leaching experiments are also carried out similarly to base metal leaching. Liquid samples are being withdrawn at pre-determined time intervals and then diluted in deionized water for analysis by AAS. The leaching of gold is measured based on Equation (5).

$$\% \text{ Gold Leaching} = \frac{\text{Wt\% of gold in leach liquor}}{\text{Wt\% of gold in feed sample}} \quad (5)$$

2.3. Recovery and Separation of Gold

The recovery and separation of gold from the leach solution is achieved by solvent extraction followed by cementation processes. Initially, the selective transportation of gold is attained by solvent extraction process by using tertiary (3°) organic amide as an extracting reagent diluted in toluene and leaving the other minor elements in the raffinate. The molecular moiety of the 3° amide is displayed in Figure 4. The organic phase is subjected to stripping for back extraction of Au(III) with 1 M sodium hydroxide. The collected raffinate and strip solutions are analyzed with AAS. The cementation of gold from the strip solution is achieved by the addition of zinc dust of −200 mesh size (BSS) into the solution. An excess amount of zinc dust (1000% excess) is used for 1 h at 50 °C for cementation, along with 500 rpm stirring speed for agitation. After 1 h, the solution is filtered out and analyzed with AAS. The precipitate is separated, dried and dissolved in dilute nitric acid for 10 min to dissolve the attached zinc particles. The gold fraction is filtered, separated and analyzed with XRD (D8 Discover, Bruker AXS GmbH) and SEM (Hitachi S-3400N), attached with EDAX.

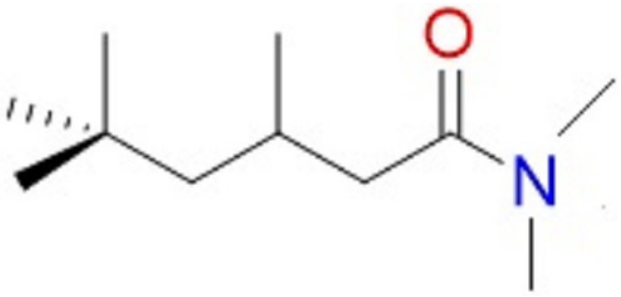

Figure 4. Molecular structure of tertiary (3°) organic amide.

AAS calibration standards were obtained from Sigma-Aldrich. Standard solutions of 1 ppm, 5 ppm and 10 ppm are used for calibration. Post calibration, samples in distilled

water were taken up by a PTFE (Teflon) capillary tube at the feed rate of 5.0 mL min^{-1} into a pneumatic nebulizer and a glass bead, High Density Poly Ethylene (HDPE) spray chamber. Air–acetylene flame conditions for the aqueous samples were: C_2H_2 as a fuel with a pressure of 15 psi and a continuous, dry air as an oxidant with an input pressure of 50 psi. The flow rate of fuel was 3 LPM, and that of oxidant was 2 LPM.

3. Results and Discussion

3.1. Validation of Bromide Leaching through Response Surface Methodology

For the validation of gold leaching, 20 parametric variations were created by considering factorial points (six), corner points (eight) and center points (with a repetition of six times) to improve the adequacy of the model. The outcome of independent and combined factors was determined by experiments performed by considering higher, centered and lower factors. Each experiment except the center point was carried out in triplicate, and the recorded values of the average response and standard error/deviation are shown in Table 3. Likewise, the center point was repeated six times, and the response value was recorded.

Table 3. Central composite design layout adopted in the present work for the leaching of gold.

	Parameter 1	Parameter 2	Parameter 3	Response 1	Error (%)
Run	**A:Time**	**B:Temp**	**C:Stirring Speed**	**Gold Leaching**	
	min	**°C**	**rpm**	**%**	
1	35	60	500	87	1
2	35	60	300	85	-
3	10	90	100	73	1
4	35	60	300	84	-
5	60	90	100	86	1
6	60	90	500	88	0
7	35	60	300	84	-
8	10	90	500	75	0
9	35	60	300	85	-
10	35	60	100	83	0
11	35	90	300	84	0
12	35	60	300	86	-
13	10	30	500	68	1
14	35	30	300	75	0
15	10	60	300	73	1
16	60	30	500	82	2
17	60	30	100	80	1
18	10	30	100	66	1
19	60	60	300	89	2
20	35	60	300	85	-

The specific individual responses of different parameters for gold leaching from Table 2 were fitted and evaluated by the higher-order and significant quadratic model. Based on this analysis, the leaching of gold was expressed in terms of the factors shown in Equation (6). The analysis of variance (ANOVA) was applied to assess the fit of the regression model (Table 4). ANOVA explains that time (<0.0001 *p*-value) and temperature (<0.0001 *p*-value) each had a greater impact than stirring speed (0.0039 *p*-value). The value of $p < 0.001$ and the higher than average F value in the regression model are thus proven to be statistically significant. Higher correlation values with predicted R^2 of 0.933 and adjusted R^2 being 0.9776 are in reasonable agreement with a difference of <0.2, as presented in Figure 5 which depicts predicted vs. actual gold leaching efficiency. The colour boxes indicates percentage of leaching. The change from blue to red colour indicates the increase in leaching of gold

$$\text{Au leaching (coded)} = 84.63 + 7.00\ A + 3.50\ B + 1.20\ C - 0.25\ AB - 3.32\ A^2 - 4.82\ B^2 + 0.6818\ C^2 \quad (6)$$

Table 4. ANOVA quadratic model for gold leaching.

Source	Sum of Squares	df	Mean Square	F-Value	*p*-Value	
Model	863.49	9	95.94	93.07	<0.0001	significant
A-Time	490.00	1	490.00	475.31	<0.0001	
B-Temp	122.50	1	122.50	118.83	<0.0001	
C-Stirring speed	14.40	1	14.40	13.97	0.0039	
AB	0.5000	1	0.5000	0.4850	0.5020	
AC	0.0000	1	0.0000	0.0000	1.0000	
BC	0.0000	1	0.0000	0.0000	1.0000	
A^2	30.28	1	30.28	29.37	0.0003	
B^2	63.84	1	63.84	61.93	<0.0001	
C^2	1.28	1	1.28	1.24	0.2915	
Residual	10.31	10	1.03			
Lack of Fit	7.48	5	1.50	2.64	0.1553	not significant
Pure Error	2.83	5	0.5667			
Cor Total	873.80	19				

The *p*-values < 0.05–0.0001 and model F-value of 93.07 imply that the model and its terms are significant. As shown in Table 4, A, B, C, A^2, B^2 are the model terms. The mean standard deviation was calculated as 1.02 with a mean variance of 80.90 and % CV being 1.26.

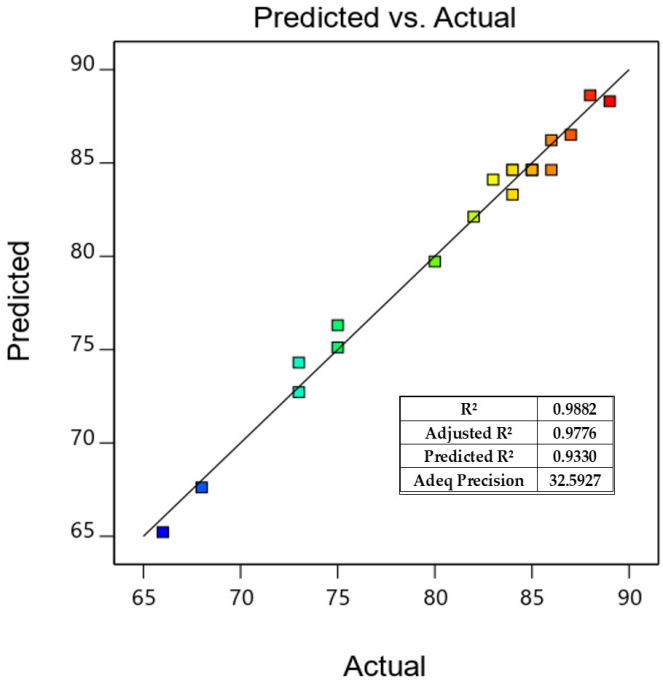

Figure 5. Predicted vs. actual plots for gold leaching.

To elucidate the correlation among the variables on the leaching of gold, 3D response surface plots and 2D contour plots were drawn as shown in Figure 6. These depict the increase in leaching efficiency of gold with longer residence time and stirring speed. However, as the temperature increases, the gold leaching efficiency increases until 70 °C and then decreases. This could be attributed to the evaporation of bromine at higher temperatures. The blue zone indicates lower leaching, and the red zone denotes the highest, as shown in Figure 7. However, the numerical optimization of parameters of RSM was also chosen for the maximum leaching efficiency.

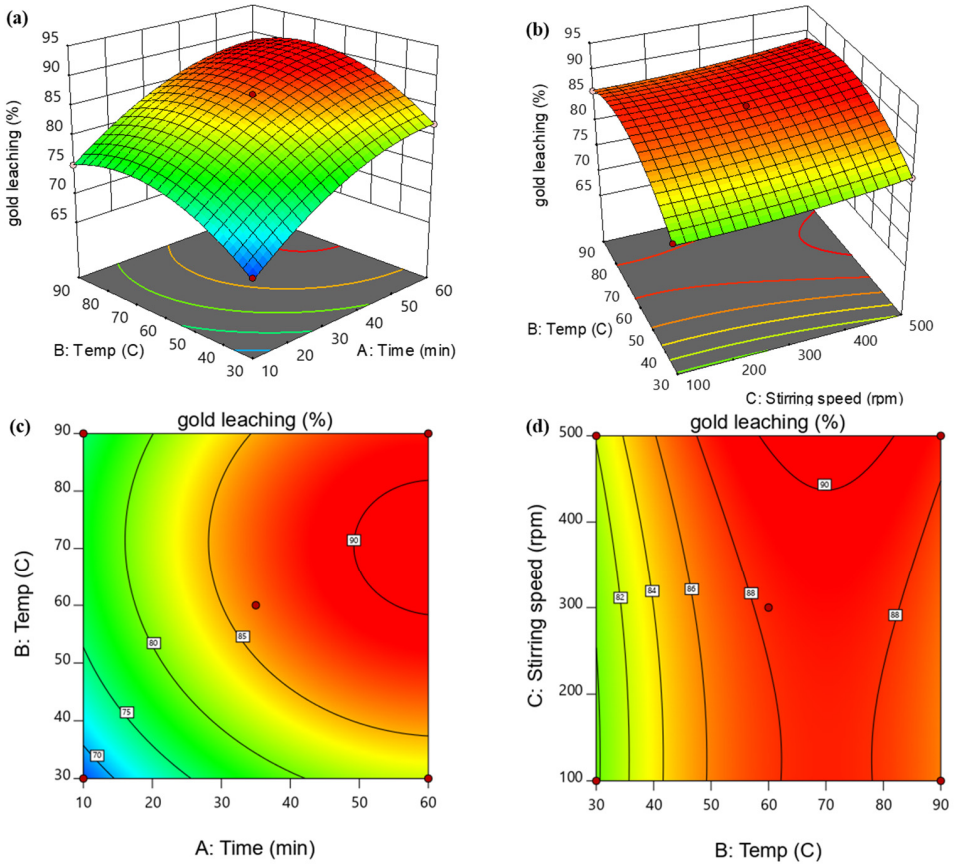

Figure 6. 3D response surface plots and 2D contour plots for the leaching of gold: (**a**,**c**) represent a constant stirring speed of 500 rpm; (**b**,**d**) represent a constant time of 1 h.

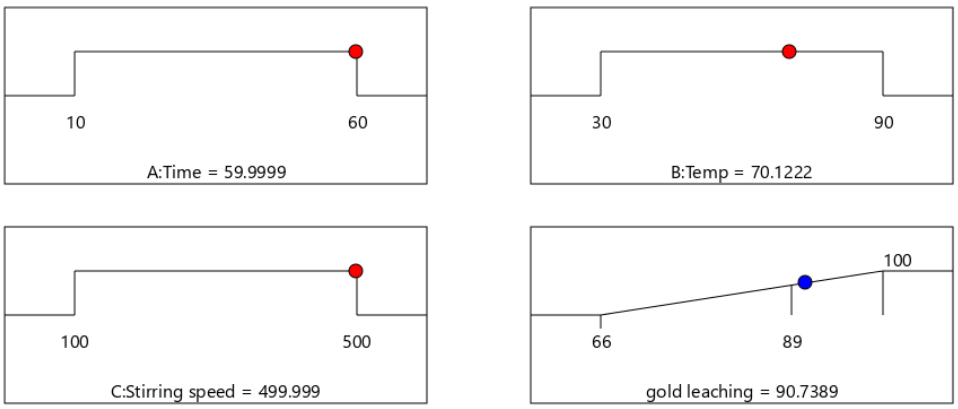

Figure 7. Optimization of parameters for the leaching of gold.

As shown in Figure 5, about 90.7% of the gold could be extracted in 1 h at 70 °C temperature and 499 rpm stirring via numerical optimization. The experimental observation under the same conditions depicted 93% gold extraction, which validates the DOE approach (Table 5), and clearly established the optimal conditions without the need for additional classical experimental optimization.

Table 5. Validation of calculated results with experimental results for gold leaching.

S. No.	Name of the Factor			% Leaching	
1.	Time (min)	Temperature (°C)	Stirring speed (rpm)	Experimental	Calculated
2.	60	70	499	93	90.73

3.2. Kinetics of Leaching

For the leaching of gold at various temperatures (30 to 70 °C), the leaching kinetics was evaluated via the shrinking core model using diffusion and surface reaction expression. However, the correlation coefficients (R^2) in these expressions were not more than 0.8, as represented in Figure 8a,b. Therefore, the kinetics of the process was evaluated with an empirical model (Equation (7)) which is governed by logarithmic rate law.

$$-(\ln(1-(1-x))^2 = k_e t \quad (7)$$

where x represents fraction of Au extracted in time t, and k_e is the apparent rate constant.

The data obtained at various temperatures (30 to 70 °C) were used to plot $-(\ln(1-x))^2$ vs. t and showed a high R^2 value, as shown in Figure 9. The best fit observed through the empirical model may be attributed to the complex nature of the leach residue used for gold leaching [28,29].

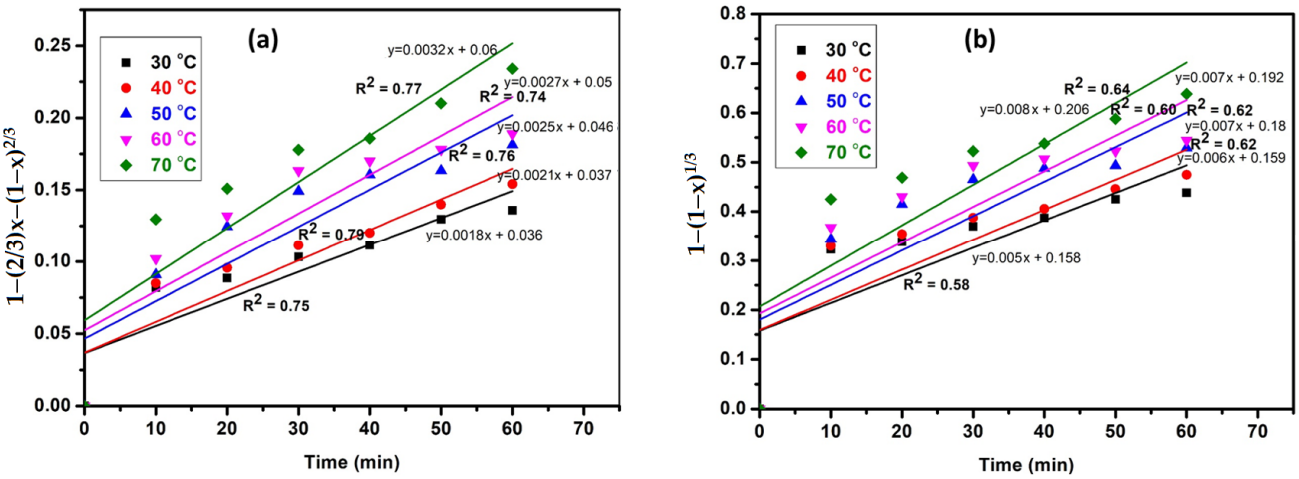

Figure 8. (**a**) Diffusion-controlled and (**b**) surface reaction-controlled models for leaching of gold (temp. 30 to 70 °C).

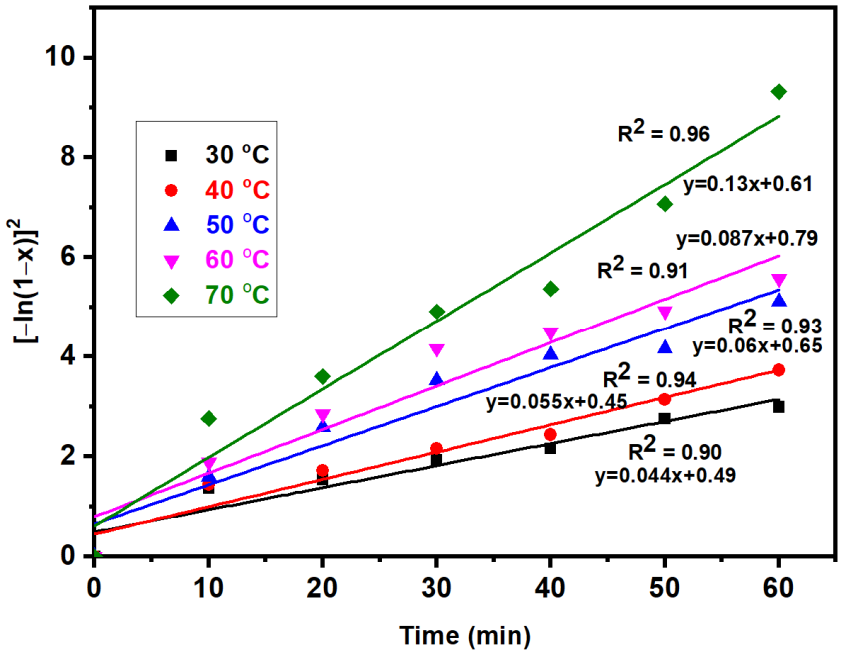

Figure 9. Empirical model for kinetics of leaching of gold (temp. 30 to 70 °C).

As shown in Figure 10, the Arrhenius plot with a high correlation coefficient (R^2 = 0.94) was used to calculate the activation energy to be 22.76 kJ/mol.

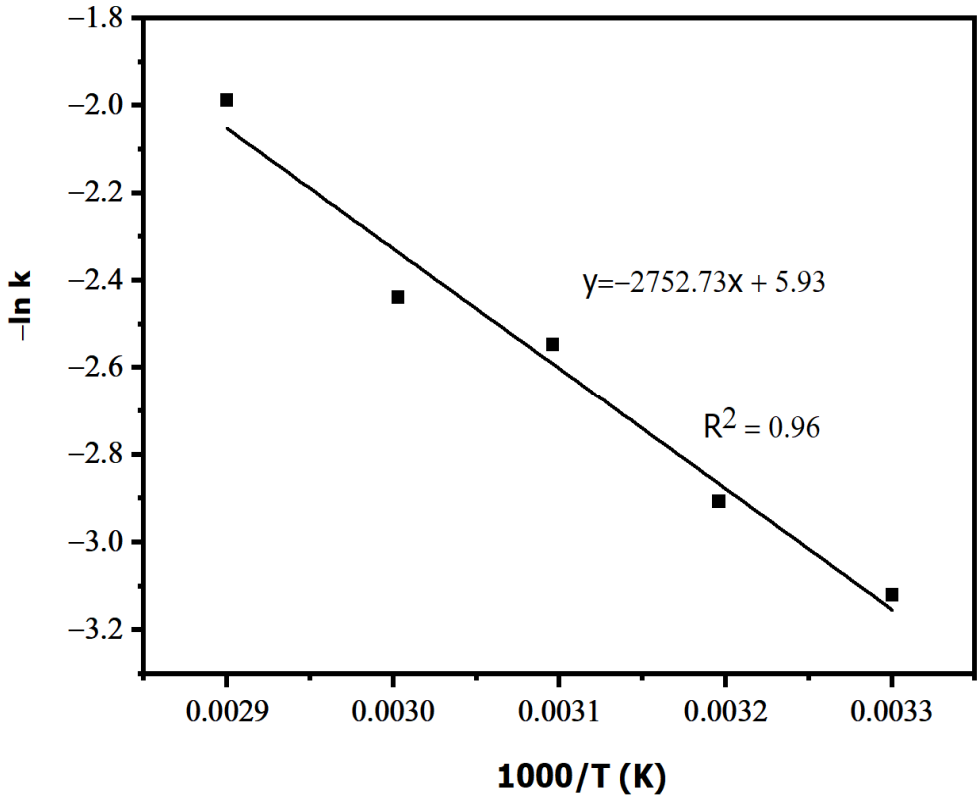

Figure 10. Arrhenius plot for the empirical kinetic model for gold.

3.3. Liquid–Liquid Extraction and Separation of Gold from Leach Liquor

Liquid–liquid extraction (solvent extraction) was carried out for extraction of gold from the leached solution using 0.1 M 3° amide as a reagent and 1 M sodium hydroxide as stripping reagent. The strip solution consists of gold with the purity of 99% after the repetition of 2 cycles. Zinc powder is used for the chemical precipitation of gold from the strip solution. From the results, it is observed that the quantitative chemical precipitation (99.8%) is achieved at the above-mentioned parameters. Table 6 shows the amount of gold (ppm) in the strip solution prior to and after cementation.

Table 6. Concentration of gold before and after cementation.

S. No.		Concentration of Gold (ppm)
1.	Strip solution (Before cementation)	409
2.	Solution after cementation	0.74

Conditions for cementation: temp. 50 °C; size −200 mesh (BSS); excess amount of zinc dust 1000%; time 1 h; stirring speed 500 rpm.

The precipitate is separated, dried and dissolved in dilute nitric acid to dissolve the zinc particles from the precipitate. The X-ray diffraction pattern for the separated gold is shown in Figure 11, and it displays the presence of dominant gold peaks. EDS (SEM) revealed that the purity of gold occupies 91.7 wt% along with minor fraction of zinc (2.9 wt%) and oxygen (2.8 wt%), as shown in Figure 12. In addition, the elemental mapping of the synthesized gold powder is also depicted in Figure 13. It reveals the presence of an homogenous arrangement of gold particles along with minor impurities of zinc, oxygen, copper and bromine. The product, when analyzed by Niton™ XL2 XRF Analyzer, showed 93.7% Au, which is in line with the XRD quantification.

Based on this, the recovery of gold from the initial WPCB metal clads was 89.8 wt%. Based on the statistical design of the experiments, we achieved > 90% recovery of gold without conducting several optimization experiments. In addition, as the gold concentration is almost 100× higher in e-waste that in natural ore, we could achieve economic benefits for gold extraction via this proposed process. The recovery of gold from raw material (WPCB metal clads) to gold powder is also calculated as follows:

(a) Gold concentration in 5 g of WPCB metal clad: 2.5 mg;
(b) After base metal leaching: 2.5 mg;
(c) After gold leaching (90.73%): 2.27 mg;
(d) After solvent extraction (99% recovery) and stripping (99.9%): 2.25 mg;
(e) After cementation with zinc powder: 2.245 mg;
(f) After zinc dissolution: 2.245 mg.

Therefore, the recovery of gold from raw material (WPCB metal clad) as gold powder is as follows: (2.245/2.5) × 100 = 89.8%.

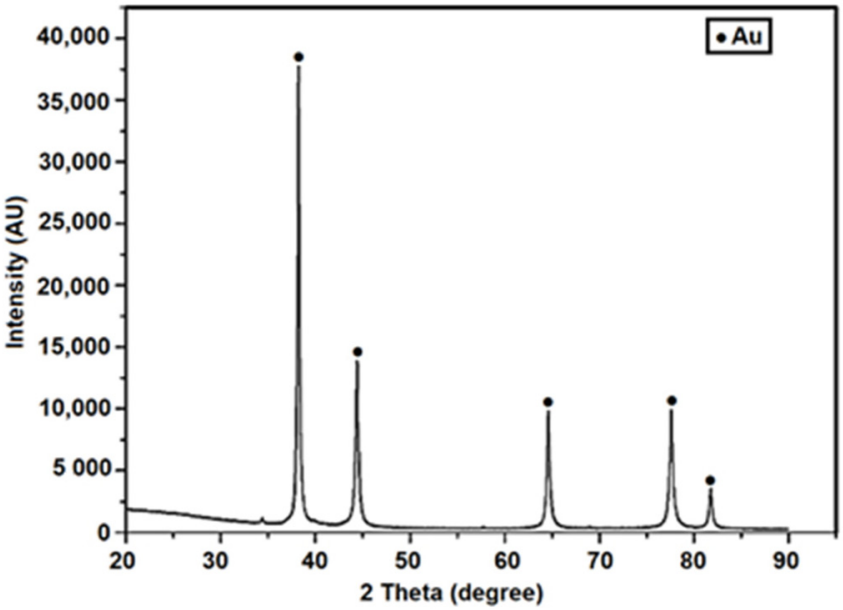

Figure 11. XRD pattern of the produced gold powder.

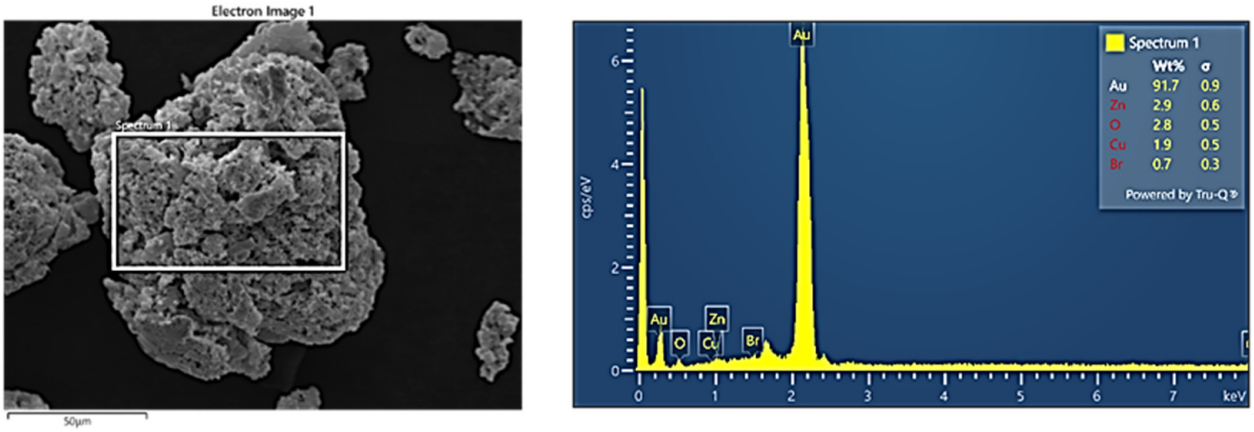

Figure 12. EDS (SEM) analysis of the produced gold powder.

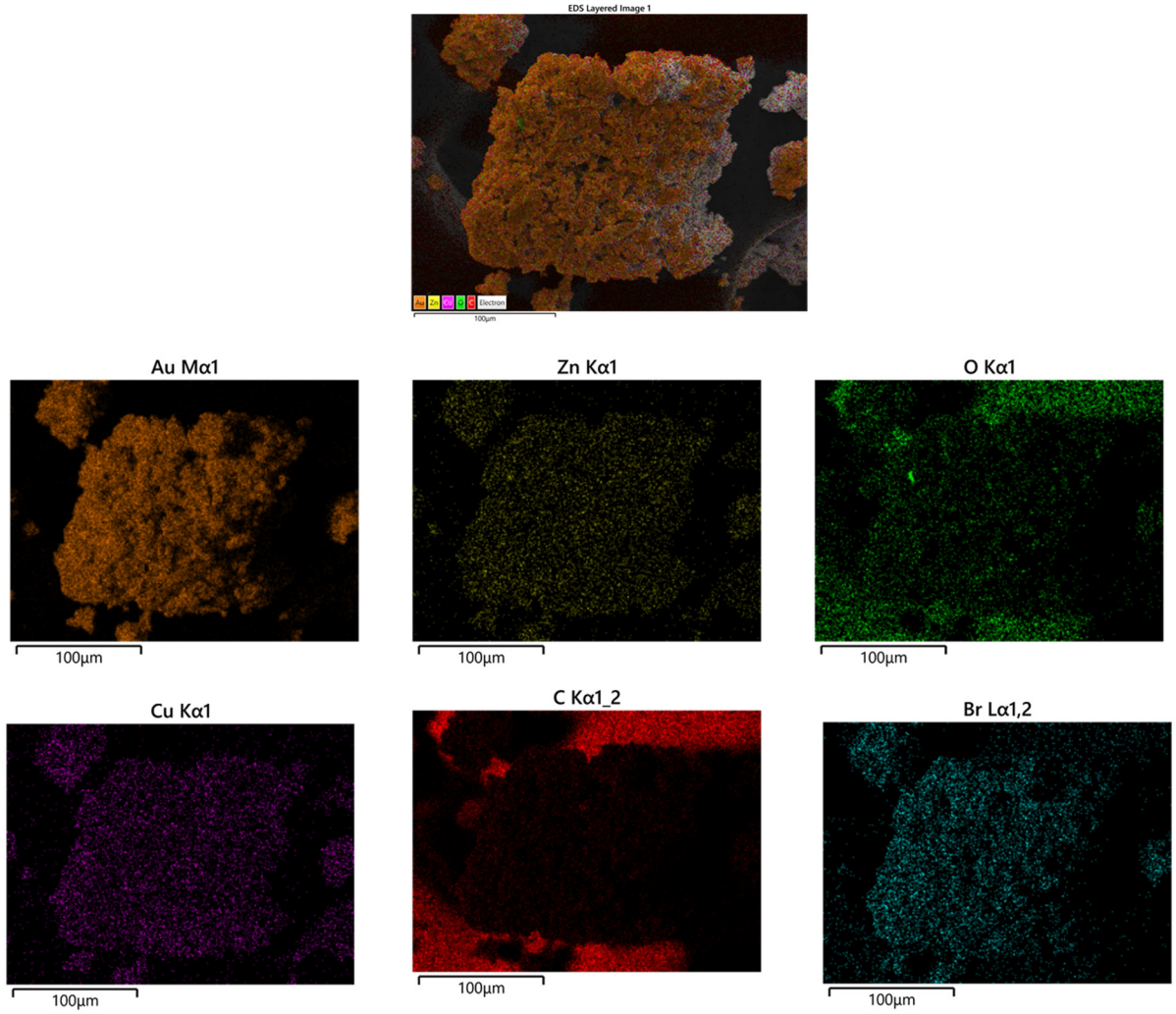

Figure 13. Elemental mapping of the synthesized gold powder.

4. Conclusions

The present investigation represents a method for selective gold extraction from metal clads of WPCBs. Using DOE and RSM methods, a clear correlation was established to validate the experimental data with 90.73% Au extraction obtained in 60 min, at 70 °C temperature and 499 rpm stirring speed, with sodium bromide (3 M) in the presence of 3 M sulfuric acid. The evaluation of the process kinetics in the extraction of gold from WPCBs shows an activation energy of 22.76 kJ/mol, and the mechanism is governed by the logarithmic dissolution model. The leach liquor generated was subjected to liquid–liquid extraction with 0.1 M 3° organic amide diluted in toluene followed by cementation with zinc dust. The quantitative chemical precipitation of gold with zinc powder was observed at 1000% excess zinc powder, at 50 °C temperature for 1 h, with 500 rpm agitation speed. The re-dissolution of zinc was also observed with dilute nitric acid for the synthesis of the gold powder. The X-ray diffraction analysis, EDS (SEM) analysis, along with the elemental mapping, showed the purity of the synthesized gold powder to be 91%. Therefore, the proposed process is technically feasible for the extraction and separation of gold from WPCBs of obsolete mobile phones.

Author Contributions: M.D.R.: conceptualization, methodology, software, validation, formal analysis, investigation; resources, writing—original draft preparation. P.M.: writing—review and editing, investigation, analysis and characterization. A.A.: writing—review and editing, investigation, analysis and characterization. K.K.S.: writing—review and editing, visualization, supervision, software,

investigation and project administration. All authors have read and agreed to the published version of the manuscript.

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