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OPTIMIZATION OF COPPER RECOVERY FROM WEEE BY BIO-METALLURGICAL PROCESS

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ABSTRACT

Bioleaching of metals from Waste Printed Circuit Board (WPCB) is an alternative path for metal mining. Metallic composition of processed PCB at different size were estimated in this study. Acidithiobacillus ferrooxidans was used as a biomining agent to recover copper. Recovery of copper using this organism can be affected with several factors like pH, temperature, pulp density, sample quantity, sample size, inoculum load etc., It is pertinent to screen and optimize the influencing variables to maximize recovery. Four factors viz. particle size, temperature, $FeSO_4$ concentration and sample quantity were screened using Plackett-Burmann design. The process variables, temperature, $FeSO_4$ concentration and sample quantity were optimized using Box-Behnken design at 30°C temperature, 40.08 g/L $FeSO_4$ concentration and sample quantity of 5 g/L with a recovery rate of 80.56%. Using the optimized values, the influence of inoculum load and treatment duration were assessed. Inoculum of 50 ml/L and treatment period of 20 days produced a recovery rate of copper of 86.9%. Redox potential curve and pH variation of the bioleaching system also supports the findings of optimization as well as enhancement.

1. INTRODUCTION

In this electronic era, waste electronic and electrical equipment (WEEE) is increasingly becoming an environmental challenge (Garcia-Valles et al., 2008). According to studies, in South Africa and China, there is a rise of 200-400% in e-waste generated from end-of-life computers in 2020, compared to 2007 (Mmereki et al., 2016). The situation is worse in the case of India with a hike of 500% (Shreyas Madhav et al., 2022). Beyond the case of land utilization, the level of toxicity it causes is very high as it contains, hazardous elements including lead, mercury, arsenic, cadmium, selenium, chromium (VI) and flame retardants more than permissible quantity (Gao et al., 2021).

Conventional methods of metal recovery, including pyrometallurgy (Jung et al., 2017), hydrometallurgy (Pant et al., 2012) and mechanical process (Meng et al., 2017) could cause secondary pollution and therefore microbiological approach of metal recovery, also known as bioleaching becomes ideal with its eco-friendly nature (Xia et al., 2017). This involves the leaching of metals from low-grade ores using suitable microbes by providing them the required growth conditions (Bosecker, 1997). This could also serve as a detoxification technique for heavy metal toxicity in industrial wastes, soil and sewage sludge (Narayan & Sahana, 2009). Besides being served as a tool against environmental contamination, this could act as a secondary source for metals, which could enhance its commercial importance (Awasthi et al., 2019).

The most common elements recovered commercially through bioleaching are copper and uranium using Acidithiobacillus ferrooxidans. This organism leaches copper through the generation of powerful oxidizing agent Fe3+ (equation 1 and 2) (Sugio et al., 1990; Nemati et al., 1998).

$$2Fe^{2+} + ½O_2 + 2H+ \text{ à } 2Fe^{3+} + H_2O \quad (1)$$
$$Cu^0 + 2Fe^{3+} \text{ à } Cu^{2+} + 2Fe^{2+} \quad (2)$$

This process is influenced by several factors like pH, temperature, pulp density, sample quantity, sample size, inoculum load etc., (Deveci et al., 2003). Among these factors, particle size, sample quantity, temperature and FeSO4 concentration were studied in addition to inoculum load and process duration for copper recovery. Copper dissolution rate is found to be highly dependent on temperature as the highest dissolution rate was previously reported as 55-60°C followed by 48°C, 42°C and 30°C (Falagán et al., 2024). In acidic environments, redox potential directly correlates with Fe speciation (Yue et al., 2016). Therefore, redox potential of the system is also analyzed along with pH throughout the bioleaching period to estimate the variation in leaching rate.

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2. METHODOLOGY

2.1 Processing of waste printed circuit board

Printed circuit boards collected from used and disposed computers and televisions has been used for the present study. These were collected in large quantity from Clean Kerala Company, Thiruvananthapuram Unit, Kerala. Additional electronic components such as capacitors, resistors, diodes, sensors, etc. were removed manually. Remaining PCBs were crushed into small powder size particles using a laboratory jaw crusher (Star Trace Private Limited, Tiruvallur, Tamil Nadu). Powdered particles categorized based on size as 300 µm and <180 µm was used for further studies.

2.2 Estimation of metallic composition of PCB

The metallic composition of the samples of different sizes were estimated quantitatively through acid digestion. One gram of PCB sample was taken in an Erlenmeyer flask and dissolved in 100 mL of aqua regia (1:3 conc. HNO_3 and conc. HCl respectively.) (Kim et al., 2018). Contents were digested inside a fume hood for 48 h. After digestion, the solution with dissolved metals was filtered first using glass wool followed by nylon membrane filter (0.45 µm). Filtered solution was diluted 10 times with milliQ water and analysed using Atomic Absorption Spectroscopy (AAS) (Chanda et al., 2014).

2.3 Preparation of bacterial culture

Industrial bacterial leaching requires an organism that could survive extreme physical and geochemical conditions. Acidophiles are such extremophiles that could multiply in higher acidic conditions with pH<3. Among different kind of acidophilic microorganisms, Acidithiobacillus groups are found to be very effective in leaching out Cu, Zn, Fe and As from different mineral sources. Acidithiobacillus ferrooxidans which is a gram-negative, chemolithoautotrophic bacteria involved in microbial leaching was selected as biomining agent (Maluckov, 2017). Acidithiobacillus ferrooxidans (NCIM No:5371, ATCC No:19859) was purchased from National Collection of Industrial Microorganisms, National Chemical Laboratory, Pune, India. The strain was activated in Medium No:54 (Four parts of Solution A: 0.5 g/L $(NH_4)_2SO_4$, 0.5 g/L $MgSO_4.7H_20$, 0.5 g/L K_2HPO_4 and 5 mL of 1N H_2SO_4. One part of Solution B: 167g/L $FeSO_4.7H_2O$, 50 mL of 1N H_2SO_4 in 1 L; static incubation at 30°C for 3-5 days). The organism was adapted to metal by repeated sub-culturing of the strain under increasing concentration of $CuSO_4.5H_2O$ (Bosecker, 1997).

2.4 Bioleaching

Bioleaching of copper from waste PCBs of WEEE was carried out in a 250ml Erlenmeyer flask. The treatments contained 100ml of modified 9K media (Four parts of Solution A: 0.5 g/L $(NH_4)_2SO_4$, 0.5 g/L $MgSO_4.7H_20$, 0.5 g/L K_2HPO_4 and 5 mL of 1N H_2SO_4. One part of Solution B: 167g/L $FeSO_4.7H_2O$, 50 mL of 1N H_2SO_4 in 1 L) inoculated with metal adapted bacterial inoculum at indicated concentrations (Gao et al., 2021). After one day powdered and sterilized PCB was added at a concentration of 10 g/l. The entire content was incubated with constant shaking (160 rpm) at 30°C for 8 to 10 days. For analysis the content was centrifuged at 8000 × g for 8 min and the copper in supernatant was quantified using AAS.

2.5 Screening Analysis

Factorial designs are currently preferred over the traditional one-factor-at-a-time approach (Alfonzetti et al., 2003). The factors responsible for copper leaching were screened using Plackett-Burmann model with four factors and two levels. The factors analyzed were temperature (25°C and 35°C), $FeSO_4$ concentration (26.72 g/L and 40.08 g/L), sample size (<180 µm and 300 µm) and sample quantity (5 g/L and 15 g/L). The treatment duration (10 days), inoculum load (10 ml/L; 8.2×10^9 cells/mL) and initial pH (2.2) were fixed constant. The data on copper content were analyzed using Minitab software (Version 21.3.1.0).

2.6 Optimization of factors

Based on the outcome of screening analysis, three factors namely temperature, $FeSO_4$ concentration and sample quantity were optimized at three levels using Box-Behnken Design(Iqbal et al., 2020).

2.7 Enhancing Recovery

The optimized factors were further explored with inoculum load (10 ml/L, 25 ml/L and 50 ml/L) and incubation time (5,10,15 and 20 days) using two-way ANOVA.

2.8 Variation in pH and Redox Potential

The variation in pH value and redox potential of the bioleaching system were recorded at a regular interval of 24 hours.

3. RESULTS AND DISCUSSIONS

3.1 Metallic Composition of PCB

Metallic composition of the processed PCB samples at different particle sizes are shown in Table 1. Samples of 180 µm size exhibited higher content of Fe (217.70 mg/g) followed by Cu (105.41 mg/g) and Mn (11.05 mg/g) with other metals such as Ni, Mg, Co, Ag in minor quantities and Cr below detection level. Increased sample size of 300 µm exhibited higher Cu content (332.056 mg/g) followed by Fe (121.559 mg/g) and Mn (9.641 mg/g). The Cu content in PCB samples of particle size 300 µm was 33.20% which is significantly higher than 180 µm particle size (10.54%). These values are found to be much higher than the previous reports, which exhibited varied range between 12.5%, 14.6%, 19% and 28% content(Creamer et al., 2006; Feldman, 1993; Hino et al., 2009; Oliveira et al., 2010; Veit et al., 2002). Similar analysis based on mobile phone PCBs, at different particle sizes has reported 15.6% and 29.13% of copper content at 150 µm and 2 mm particle size respectively (Annamalai & Gurumurthy, 2021). Many factors such as the source of PCB, sample processing, characterization methods influence the final content (Bizzo et al., 2014).

The value represents the maximum dissolution of metal when treated with acid, which could be considered as that of 100% recovery value. Dissolution value obtained af-

TABLE 1: Metal composition of PCB sample.

Sample size (μ)	Metal	Quantity (mg/g)
<180	Copper (Cu)	105.41
	Iron (Fe)	217.70
	Silver (Ag)	3.06
	Nickel (Ni)	3.44
	Magnesium (Mg)	2.39
	Manganese (Mn)	11.05
	Cobalt (Co)	0.52
	Chromium	BDL
300	Copper (Cu)	332.056
	Iron (Fe)	121.559
	Silver (Ag)	4.001
	Nickel (Ni)	8.143
	Magnesium (Mg)	2.63
	Manganese (Mn)	9.641
	Cobalt (Co)	1.166
	Chromium	BDL

** BDL - Below Detection Level*

TABLE 3: Optimization of Temperature, $FeSO_4$ concentration and Sample Quantity.

Run	Temperature (°C)	$FeSO_4$ concentration (g/l)	Sample quantity (g/l)	% Cu Recovery
1	30	40.08	15	65.96
2	30	33.4	10	68.99
3	35	26.72	10	52.03
4	35	33.4	5	74.09
5	25	40.08	10	30.34
6	30	40.08	5	80.56
7	35	40.08	10	57.67
8	25	26.72	10	16.86
9	25	33.4	15	15.83
10	35	33.4	15	60.57
11	30	26.72	15	64.22
12	30	33.4	10	68.99
13	30	26.72	5	77.31
14	25	33.4	5	41.13
15	30	33.4	10	68.99

ter microbial leaching is compared with this value to obtain the percentage recovery.

3.2 Factors influencing recovery of copper

Besides particle size, other factors such as temperature, sample quantity and concentration of electron donor play a major role. In order to find the significant factors, a screening design (Plackett-Burmann design) was employed. It is one of the most used screening designs because it needs smaller number of experimental runs and also cost-effective (Fukuda et al., 2018). The combinations of different factors provided for this design and the percentage recovery in each case are listed in Table 2. The factors which possess a probability value (P-value) below 0.05 can be considered to have a significant effect on the process. According to the statistical observations, factors including temperature, $FeSO_4$ concentration and sample quantity are significant and particle size is insignificant. It may be noted that the particle size plays a major role in chemical recovery method, but not in bioleaching. Hence, temperature, $FeSO_4$ concentration and sample quantity were further optimized.

3.3 Optimization of Copper recovery

Optimization of factors using Box-Behnken design of Response Surface Methodology considers three factors (temperature, $FeSO_4$ concentration and sample quantity). The different combinations of factors used and the obtained recovery rate are provided in Table 3. Maximum recovery rate of 80.56% is obtained at 30°C temperature,

TABLE 2: Effect of particle size, temperature, $FeSO_4$ concentration and Sample Quantity on copper recovery.

Run	Particle Ssize (μ)	Temperature (°C)**	$FeSO_4$ conc.* (g/l)	Sample quantity * (g/l)	% Cu Recovery
1	300	35	40.08	5	86.16
2	< 180	35	40.08	5	81.68
3	300	35	26.72	15	70.20
4	< 180	35	26.72	5	75.51
5	300	25	40.08	5	71.93
6	300	25	40.08	15	61.92
7	< 180	25	26.72	5	71.45
8	< 180	25	40.08	15	74.95
9	300	25	26.72	5	69.21
10	300	35	26.72	15	70.20
11	< 180	25	26.72	15	57.29
12	< 180	35	40.08	15	80.58

P values: Particle size = 0.460, Temperature = 0.007, FeSO4 conc.= 0.024, Sample Quantity=0.031

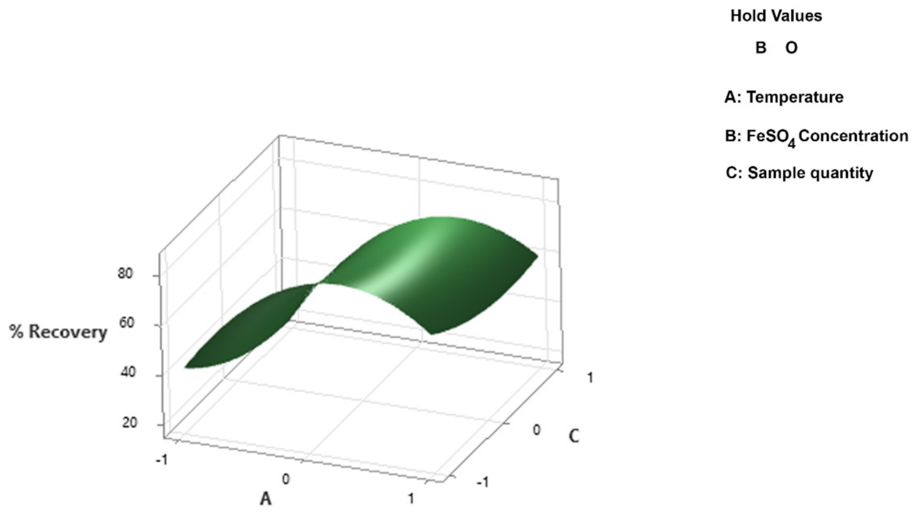

FIGURE 1: Response plot of temperature and sample quantity on % copper recovery.

40.08 g/L $FeSO_4$ concentration and sample quantity of 5 g/L. Statistical analysis of the data obtained through this model has been represented as Table 4. Here, the p-values of temperature (0.000), $FeSO_4$ concentration (0.071) and sample quantity (0.001) indicates the high significance of temperature and sample quantity along with the least significance of $FeSO_4$ concentration in altering the rate of recovery during optimization. The model exhibits a standard deviation(S) of 3.7, which represents how far the data value fall from the fitted value. The R-sq value (99%) and R-sq adjusted value (97%) of the model is found to be higher than R-sq predicted value (82%). This indicates the model is robust. The Variance Inflation Factor (VIF) value of 1.00 in all cases of interactions, shows that the factors are not correlated (Figure 1).

3.4 Enhancing the Recovery

An attempt was made to improve the recovery rate by including microbial factors such as inoculum level and treatment time. Recovery rates of copper using the optimized conditions with three different inoculum load (10, 25 and 50 ml/L) and four different treatment duration (5, 10, 15 and 20 days) are presented in Table 5. While the recovery rate improved till 10^{th} day a lag followed by improvement due to higher inoculum load was observed. This correlates well with the exponential ferrous oxidation rate of copper tolerant strains of A. ferrooxidans (ATCC 19859) between 5 and 9 days (Brahmaprakash et al., 1988). Thus, an inoculum load of 50 ml/L and treatment time of 15-20 days seems to be ideal to achieve a maximum recovery of 86.9%. The considerable elevation in the recovery rate with increased inoculum load was also reported (McSweeney et al., 2011; Wang et al., 2014).

3.5 pH and Redox Potential Curve

Variation in pH and redox potential of the bioleaching system were recorded for 10 days at a specific time interval of 24 hours and the data obtained is represented as Figure 2 a and b. The drop of pH from 2.2 to 1.5 indicates the increase in acidic nature of the system that supports the activity of Acidithiobacillus ferrooxidans. The redox poten-

TABLE 5: Effect of inoculum load and treatment period on copper recovery rate.

Run	Inoculum load (ml/L)	Treatment period (Days)	Cu Recovery rate (%)
1	10	5	25.17
2	10	10	79.89
3	10	15	79.92
4	10	20	79.94
5	25	5	54.09
6	25	10	75.73
7	25	15	76.66
8	25	20	77.41
9	50	5	66.59
10	50	10	70.68
11	50	15	84.24
12	50	20	86.94

p-values: Inoculum load = 0.016, Treatment period =0.023

TABLE 4: Statistical analysis of the optimization results.

Term	Coef	SE Coef	T-Value	P-Value	VIF
Constant	68.99	2.15	32.09	0.000	
A	17.53	1.32	13.31	0.000	1.00
B	3.01	1.32	2.29	0.071	1.00
C	-8.31	1.32	-6.32	0.001	1.00

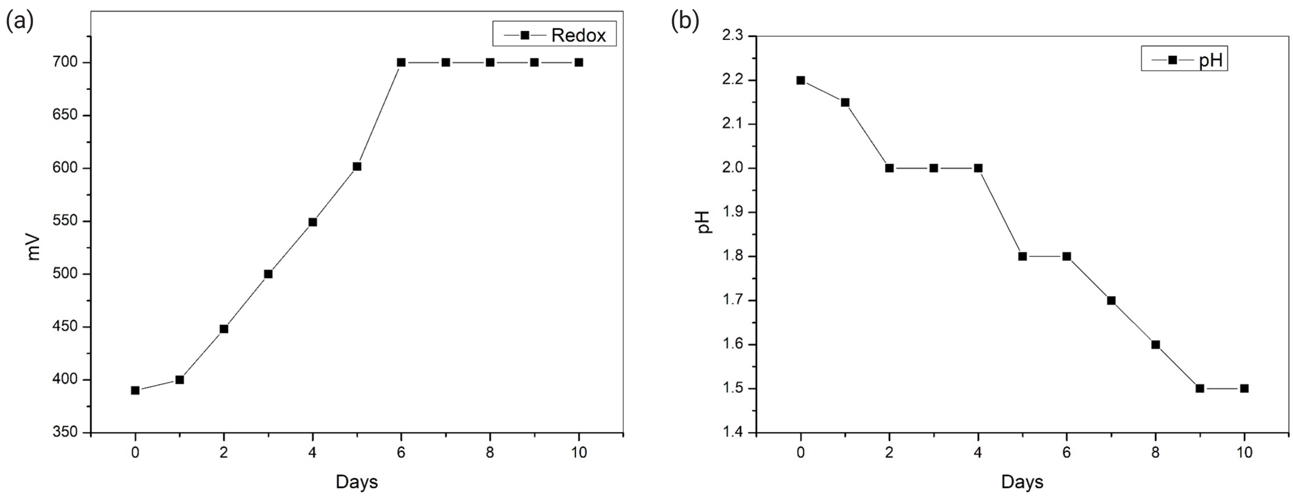

FIGURE 2: Variation in the redox potential of bioleaching system (a); Variation in the pH of bioleaching system(b).

tial data obtained indicates a gradual increase in the value from 390 to 700 mV within the first 6 days and later it is found to be maintained at 700 mV. The gradual upliftment in the redox potential is found to be the indication of a proportional upliftment in the leaching rate. The data obtained is found to be in correlation with the previously reported data (Kinnunen et al., 2020). The constant value obtained through Nernst equation (pe+pH=constant) shows an upliftment from an initial value of 8.79 to 13.63 by 6th day, thereafter a decline upto 13.33 by the 9th day and remains same thereafter.

4. CONCLUSIONS

Bioleaching of copper from used and disposed computer, television PCBs utilizing Acidithiobacillus ferrooxidans can be affected by different physio-chemical parameters among which temperature and sample quantity is found to be independent and significant. Even though $FeSO_4$ conc. is an independent variable essential for the growth of the microorganism, the recovery rate is not found to be altered due its variation during optimization. The optimum condition for bioleaching copper has been estimated as a temperature of 30°C, sample quantity of 5g/L and $FeSO_4$ concentration of 40.08 g/L. The recovery rate of copper is found to be enhanced (86.94%) at a higher inoculum load of 50 ml/L after 15-20 days of treatment under optimized conditions of temperature, $FeSO_4$ conc. and sample quantity. The positive impact of higher inoculum load and increased duration can be further supported by the persistence of redox potential at 700 mV after 6 days of bioleaching at optimized conditions.

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