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Recovery of Metals from Printed Circuit Boards (PCBs) Using a Combination of Hydrometallurgical and Biometallurgical Processes

Harikrushnan, B., Shreyass, G., Hemant, G., and Pandimadevi, M.*

Department of Biotechnology, School of Bioengineering, SRM University, Kattankulathur,
Kancheepuram, Chennai – 603203, India

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ABSTRACT: Printed circuit boards (PCBs) are one of the most important components of electronic equipment and encompass a variety of metals, including the majority of the valuable metals, and also most of the toxic components in e-waste. The objective of the present work proposes an integrated (hybrid) method for the recovery of copper (Cu), nickel (Ni) and zinc (Zn) from waste PCBs using a combination of biometallurgical (biological) and hydrometallurgical (chemical) processes. The recovery was effected using the combination of *Acidithiobacillus ferrooxidans* and 2 M HNO_3. The leaching of metals was experimented using inorganic acid, bacteria and the integration of both acid and bacteria. From the initial experiments, it was observed that the percentage of nickel, copper and zinc solubilised into leaching solution from the actual PCB basically decreased with the increase in the concentration of e-waste from 10g/L to 100g/L. The hybrid method was compared against the individual chemical and biological methods and proved to be a better method than the other two, being rapid, efficient, economical and eco-friendly.

Key words: Electronic waste (e-waste), Printed circuit boards (PCBs), Hybrid method, Integrated method, *Acidithiobacillus ferrooxidans*

INTRODUCTION

The growing use of electronic equipment and their early obsolescence due to rapid advances in technology has resulted in generation of large quantities of electronic waste (e-waste), the disposal of which is a concern, because it contains heavy metals and toxic substances which can have an adverse impact on human health and the environment. Electronic waste (e-waste) refers to electrical and electronic appliances that have reached the end of their operating life or have ceased to be useful to their owners and have been discarded. Currently, there is no one standard definition and a number of definitions are available (Widmer *et al.*, 2005). E-waste is an emerging global issue, driven by the rapidly growing use and disposal of electrical and electronic equipment due to falling prices and their early obsolescence. 1.641 million tonnes (Mt) of e-waste was generated in India and 41.8 Mt of e-waste was generated worldwide in 2014 (Baldé *et al.*, 2015). Most of the e-waste was generated in Asia, followed by the Americas, Europe, Africa and Oceania. 49.8 Mt of e-waste is expected to be generated by 2018, with an annual growth rate of 4-5% (Baldé *et al.*, 2014).

E-waste is difficult to treat and recycle because of its extremely diverse composition. Most of the components are not biodegradable. The most commonly used methods for e-waste disposal are landfills and incineration, but they are not eco-friendly (Pramila *et al.*, 2012). Several substances found in e-waste are hazardous and present a risk to human health and the environment (Gupta *et al.*, 2014).But almost 99% of the components can be recycled. By recycling, we can prevent hazardous materials from ending up in a landfill.

Recycling involves dismantling and segregation of e-waste for treatment and recovery of individual components. Recycling of e-waste is extremely important for two major reasons:

- To safeguard human health and the environment from toxic pollutants.
- For economic benefits as they are rich in many precious and rare metals and have higher concentrations of metals than even ores.

There are numerous recycling techniques, including pyrolysis, electrostatic separation, magnetic separation, hydrometallurgical separation and biometallurgical separation (Gupta *et al.*, 2014). The major metals found in PCBs are Cu, Zn, Ni, Al, Fe, Pb, Sn, Au, Ag, Pd and Pt.

Hydrometallurgical recovery of metals generally consists of three steps – leaching, solution

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*Corresponding author E-mail: pandimadevi2008@gmail.com concentration and purification, and metal recovery. The leaching step involves chemicals like acids to leach metals from e-waste. In the solution concentration and purification step, separation methods like solvent extraction, precipitation, cementation, ion exchange, filtration and distillation are used to isolate and concentrate the metals of interest from the leachate. The metals are recovered by processes like electrolysis, gaseous reduction and precipitation (Jadhav and Hocheng, 2012). The recovery method used depends upon the metal, the efficiency of the process and the costs involved. However, only a limited number of articles are currently available on the purification and separation of individual metals from the PCB leach liquor (Ghosh *et al.*, 2015).

The effect of particle size in physical and chemical processing of printed circuit boards has been studied. Shredded PCBs of various particle sizes were leached with 1 M nitric acid under different conditions. Copper dissolution increased significantly when particle size decreased, while effect on zinc leaching was less pronounced, and for Ni, Al, Sn and Pb the effects were negligible (Oliveira *et al.*, 2013).

In biometallurgical processes, solubilization of metals is based on the interactions between metals and microorganisms. This technique allows metal recycling by processes similar to that in the natural biogeochemical cycles, and is therefore environmentally friendly, with low costs and low energy requirements (Jadhav and Hocheng, 2012). Bioleaching and biosorption are broadly the two main areas of biometallurgy for recovery of metals (Gupta *et al.*, 2014). There are 4 main bioremediation techniques (Pramila *et al.*, 2012):

- ❖ Stimulation of the activity of indigenous microorganisms by the addition of nutrients, regulation of redox conditions, optimizing pH conditions, etc.
- ❖ Inoculation of the site by microorganisms with specific biotransforming abilities.
- ❖ Application of immobilized enzymes.
- ❖ Use of plants like rice, alfalfa, ryegrass, tall fescue, etc. to remove and/or transform pollutants (phytoremediation).

The major microorganisms that play a significant role in the recovery of heavy metals from e-waste belong to the acidophilic group. These acidophilic groups thrive in acidic pH ranges (pH 2.0–4.0) and help in dissolving the metals from solid phase of wastes into the aqueous phase. Bacteria like *Acidithiobacillus ferrooxidans*, *Acidithiobacillus thiooxidans*, *Leptospirillum ferrooxidans*, and *Sulfolobus* sp., are well known consortia for the bioleaching activity, while fungi like *Penicillium simplicissimum* and *Aspergillus niger* also help in metal leaching process (Mishra and Rhee, 2010; Mishra and Rhee, 2014).

Chemical methods are rapid and efficient, but are not eco-friendly. Biological methods are economical and eco-friendly, but are time-consuming and complete recovery of metals is not usually possible. A hybrid (integrated) technique, combining both chemical and biological methods so that they complement each other, would allow the rapid, efficient and economical extraction of metals while also being eco-friendly. With the help of such processes, valuable metals of high purity can be recovered from waste PCBs that are obtained from personal computers and mobile phones. Hybrid techniques have the following advantages (Pant *et al.*, 2012):

- ❖ Hybrid technique can enhance the efficiency of metal extraction from e-waste.
- ❖ This technique is comparatively less time consuming then biological leaching alone.
- ❖ Ligands like DTPA which can be used in the hybrid technique are strong, reusable, relatively biostable and can be recycled.
- ❖ It is possible to achieve metal specific extraction by using certain specific ligands and microbes.

MATERIALS & METHODS

Waste printed circuit boards (PCBs) of 25 cm x 25 cm were collected from discarded computers. The attached components were removed by desoldering with an oxyacetylene torch. A PCB was dipped in 10 M NaOH solution and left undisturbed. Every day the PCB was taken out and washed under running tap water to check if the solder mask was removed. If not, the PCB was redipped in the NaOH solution. On the 5th day, the solder mask peeled off, as can be seen in Fig. 1 (b). To remove any adhered NaOH, the PCB was further washed with distilled water until the pH of the discharge was 7 (neutral pH).

To test whether agitation would speed up the removal of the solder mask, 4 cm x 4 cm PCB chips were taken in beakers containing 10 M NaOH and agitated at 170 rpm, such that the PCB chips did not settle down at the bottom. Overnight agitation was sufficient to ensure removal of the solder mask.The treated 25 cm x 25 cm printed circuit board was filed down to obtain powder of size < 1mm. This powder was used in the experiments. The amounts of Cu, Zn and Ni present in the PCB powder, shown in Table 1, were determined using X-ray fluorescence (XRF) spectrometry.

Hydrometallurgical (chemical) treatment of PCB powder: 1 g, 5 g and 10 g of PCB powder were added in three flasks containing 100 ml of 2 M nitric acid (HNO_3), respectively (10 g/L, 50 g/L and 100 g/L). The flasks

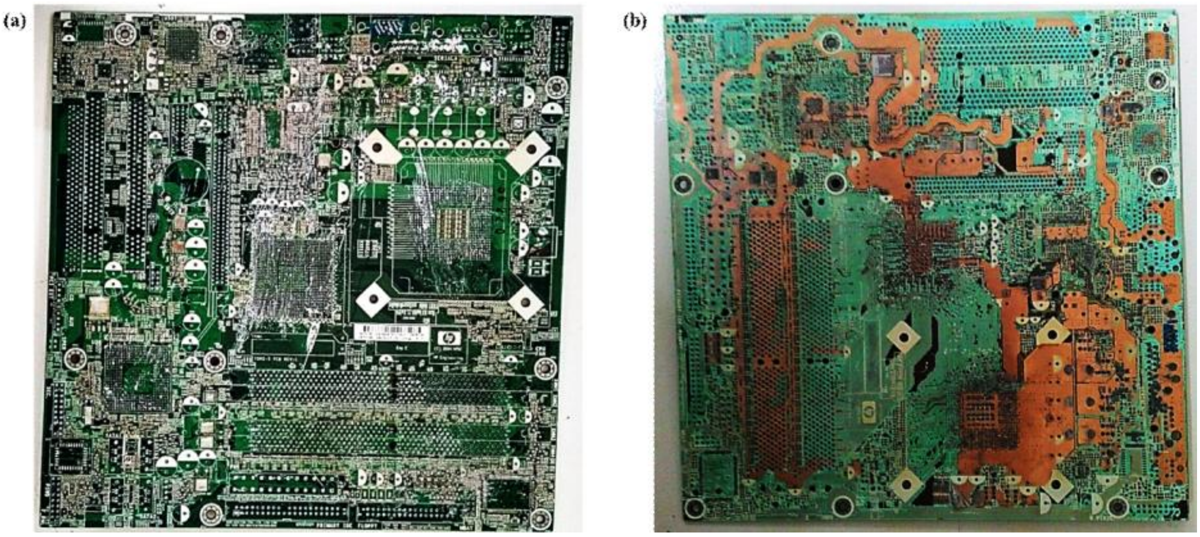


Fig. 1. (a) PCB board with intact solder mask prior to 10 M NaOH treatment, (b) Solder mask removed from PCB board after 5 days of 10 M NaOH treatment

were agitated at 100 rpm for 2 hours in a shaken water bath at 90°C. Samples were drawn at regular intervals of 30 minutes for chemical analysis. The elemental analysis of Cu, Ni and Zn present in the collected samples was done by atomic absorption spectroscopy (AAS).

Biometallurgical (biological) treatment of PCB powder: *Acidithiobacillus ferrooxidans* (NCIM No. 5370) bacteria was procured from the National Collection of Industrial Microorganisms (NCIM), National Chemical Laboratory (NCL), Pune, India. The bacteria were grown (refer Table 2 for media composition) for a week in three flasks in the absence of e-waste. After a week, 1 g, 5 g and 10 g of PCB powder were added to the three flasks, respectively (10 g/L, 50 g/L and 100 g/L). The cultures were incubated for 10 days at 30°C and 100 rpm agitation in a temperature-controlled incubator. The cultures were maintained at pH 2.5-3.Samples were collected from all the flasks every 2 days for chemical analysis. The control samples were plain media without bacteria, in which PCB powder (1 g, 5 g and 10 g) had been added. The elemental analysis of Cu, Ni and Zn present in the collected samples was done by atomic absorption spectroscopy (AAS).

Integrated (hybrid) treatment of PCB powder: Based on the results of the hydrometallurgical and biometallurgical treatments, 4 days *Acidithiobacillus ferrooxidans* leaching, followed by 30 minutes of nitric acid leaching, was used as the optimum combination. Similar to the biometallurgical treatment method, the bacteria were grown for a week in three flasks in the absence of e-waste. After a week, 1 g, 5 g and 10 g of PCB powder were added to the three flasks, respectively (10 g/L, 50 g/L and 100 g/L). The cultures were incubated for 4 days at 30°C and 100 rpm agitation in a temperature-controlled incubator. The cultures were maintained at pH 2.5-3.Samples were collected from all the flasks every 2 days for chemical analysis. The elemental analysis of Cu, Ni and Zn present in the collected samples was done by atomic absorption spectroscopy (AAS).The PCB powder was separated from the cultures to subsequently subject it to nitric acid leaching. The cultures were allowed to stand stationary to allow the insoluble PCB powder to settle down. The cultures were decanted and the PCB powder which had settled at the bottom were collected. The PCB powder was heat-dried to kill any remaining bacteria.

Table 1. Weight percentages of Cu, Ni and Zn present in the PCB powder

Metal	Weight percentage
Copper (Cu)	19.72 %
Nickel (Ni)	1.521 %
Zinc (Zn)	1.1 %

Table 2. Composition of bacterial culture media (NCIMB Medium 18). Solution I was autoclaved at 121°C for 15 minutes. Solution II was sterilized by filtration. After sterilization 4 parts of solution I was added to 1 part of solution II

Components	Amount g/L
Solution I	
$(NH_4)_2SO_4$	0.5 g
K_2HPO_4	0.5 g
$MgSO_4$	0.5 g
1 N H_2SO_4	5 ml
Distilled water	1000 ml
Solution II	
$FeSO_4.H_2O$	167 g
1N H_2SO_4	50 ml
Distilled water	1000 ml

The PCB powder was then added in three flasks containing 100 ml of 2 M HNO_3, respectively (10 g/L, 50 g/L and 100 g/L). The flasks were agitated at 100 rpm for 30 minutes in a shaken water bath at 90°C. Samples were drawn at the end of the leaching period of 30 minutes. The elemental analysis of Cu, Ni and Zn present in the collected samples was done by atomic absorption spectroscopy (AAS).

RESULTS & DISCUSSION

The PCBs were pre-treated with 10 M NaOH to remove the protective solder mask which would otherwise hinder the process by not allowing bacteria access to the metals (Adhapure *et al.*, 2014).

Hydrometallurgical (chemical) treatment results: At the end of the nitric acid leaching, the solutions were found to have turned a light bluish-green colour. This colour was mainly due to the formation of copper (II) nitrate from the reaction between copper metal present in the PCB powder and the nitric acid (Kumar *et al.*, 2014).

$$Cu + 4\,HNO_3 \rightarrow Cu(NO_3)_2 + 2\,H_2O + 2\,NO_2 \qquad (1)$$

Nitrate salts formed by the reaction of nitric acid with the other metals in the PCB powder would also have contributed to the colour but as these metals were present in much smaller quantities as compared to copper, their contribution would have been much lower. The intensity of the colour was directly proportional to the concentration of PCB powder used.

The efficiency of the hydrometallurgical treatment decreased as the concentration of PCB powder was increased. At the end of the leaching period of 2 hours, at 10 g/L concentration, 95.04% of Cu, 89.41% of Ni and 91.77% of Zn was leached [Fig. 1 (a)]. At 50 g/L concentration, 83.57% of Cu, 83.955% of Ni and 83.135% of Zn was leached [Fig. 1 (b)]. At 100 g/L concentration, 62.72% of Cu, 69.78% of Ni and 73.42% of Zn was leached [Fig. 1 (c)].

Biometallurgical (biological) treatment results:

❖***Plain media –***The colour was white and there was no change in colour over the duration of the experiment.

❖***Bacterial culture without PCB powder –***The colour was white initially but had become yellow by the end of the experiment. This was due to the oxidation of ferrous sulphate to ferric sulphate by the bacteria as part of their metabolism.

❖***Control: Plain media + PCB powder –*** The colour was initially white but had turned brown by the end of the experiment. The mobilisation of metals in the control flasks were due to leaching of the metals from the PCB powder by the sulphuric acid present in the culture media.

❖***Bacterial culture + PCB powder –***The colour was light yellow initially when the PCB powder was added and has turned dark brown by the end of the experiment. This was due to the rapid oxidation of ferrous sulphate to ferric sulphate by the bacteria in response to the stress induced by the presence of e-waste. When the flask was kept stationary and the ferric sulphate was allowed to settle down, the colour of the solution was observed to be bluish-green. This was due to the reaction of ferric sulphate with copper metal in the PCB powder, leading to the formation of copper sulphate and ferrous sulphate, which gave colour to the solution.

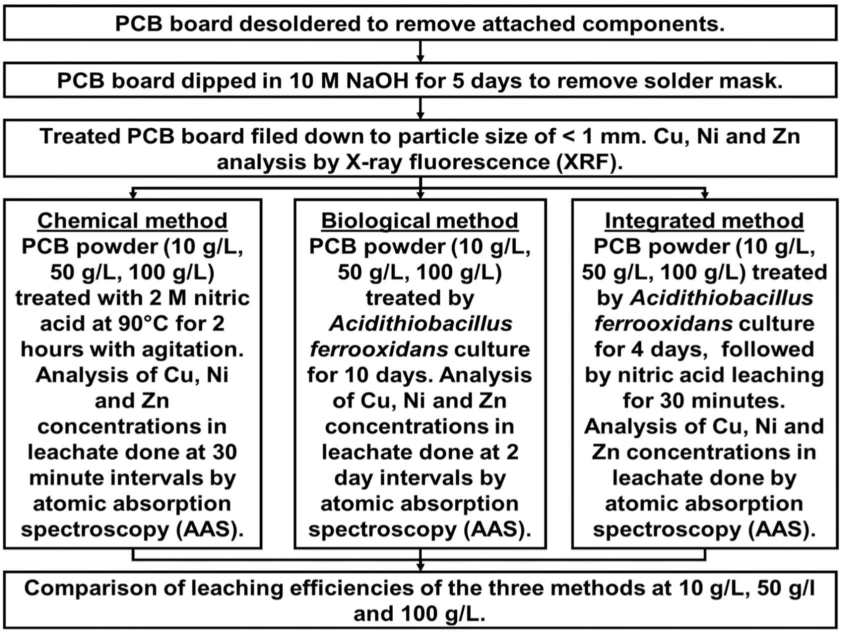


Fig. 2. Schematic diagram for the recovery of Cu, Ni and Zn from waste PCBs

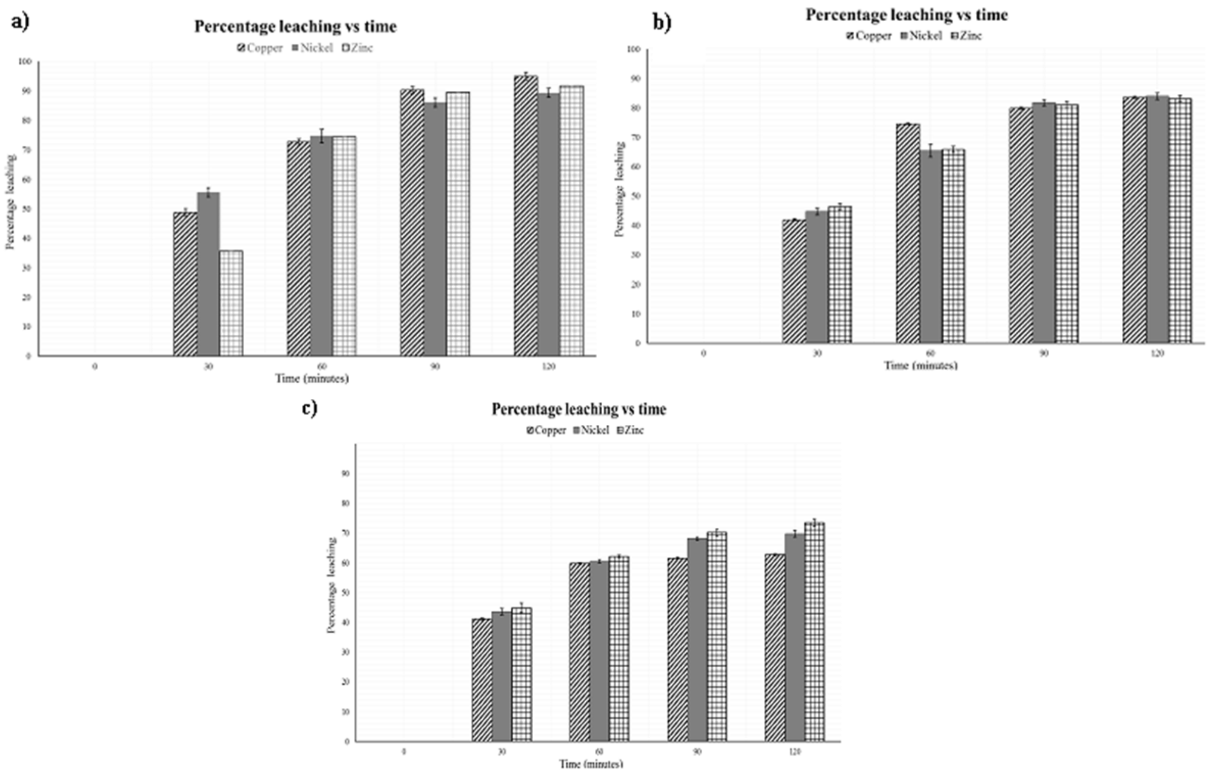


Fig. 3. Percentage leaching vs time for hydrometallurgical recovery of copper, nickel and zinc at(a) 10 g/L, (b) 50 g/L and (c) 100 g/L. Error bars in all graphs indicate ± standard errors of mean values of duplicate experiments

Metal leaching is mainly a chemical process in which ferric iron and protons carry out the leaching reactions. The role of the microorganisms is to generate the leaching chemicals and to create the space in which the leaching reactions take place. Microorganisms typically form an exopolysaccharide (EPS) layer, when they adhere to the surface of a mineral. This EPS layer serves as the reaction space (rather than the bulk solution) where the bio-oxidation reactions take place rapidly and efficiently. The microorganisms provide sulfuric acid for a proton attack and oxidise ferrous iron (Fe^{2+}) to ferric iron (Fe^{3+}) for an oxidative attack on the metals (Mishra and Rhee, 2014).

$$2\,FeSO_4 + H_2SO_4 + 0.5\,O_2 + \text{bacteria} \rightarrow Fe_2(SO_4)_3 + H_2O \tag{2}$$

$$M + Fe_2(SO_4)_3 \rightarrow M^{2+} + 2\,Fe^{2+} + 3\,SO_4^{2-} \tag{3}$$

The intensity of the colour was directly proportional to the PCB powder concentration. The efficiency of the biometallurgical treatment drastically decreased as the concentration of PCB powder was increased. At the end of the leaching period of 10 days, at 10 g/L concentration, 98.465% of Cu, 98.675% of Ni and 99.33% of Zn was leached. At 50 g/L concentration, 61.055% of Cu, 80.68% of Ni and 92.85% of Zn was leached. At 100 g/L concentration, 20.435% of Cu, 59.425% of Ni and 86.375% of Zn was leached.

Integrated (hybrid) treatment results: The integrated method removed:

- Nearly 100% Cu, Ni and Zn at 10 g/L concentration of e-waste.
- Approximately 85% of Cu and 98% of Ni and Zn at 50 g/L concentration of e-waste.
- Approximately 60% of Cu and 97-98% of Ni and Zn at 100 g/L concentration of e-waste.

It was observed that as the concentration of PCB powder was increased:

- The contribution of biological leaching to the metal recovery decreased. This was possibly due to the toxic effects of e-waste (perhaps due to high Al concentrations and the alkaline nature of the waste) on the bacteria at high concentrations (Brandl *et al.*, 2001).
- The dependence on chemical leaching for metal recovery increased.

The recoveries in all the cases were in good agreement with examples found in literature (Table 3).

CONCLUSIONS

An integrated (hybrid) model was designed by combining hydrometallurgical (chemical) and biometallurgical (biological) methods, for the recovery of copper, nickel and zinc from waste PCBs. The optimum combination used was leaching by

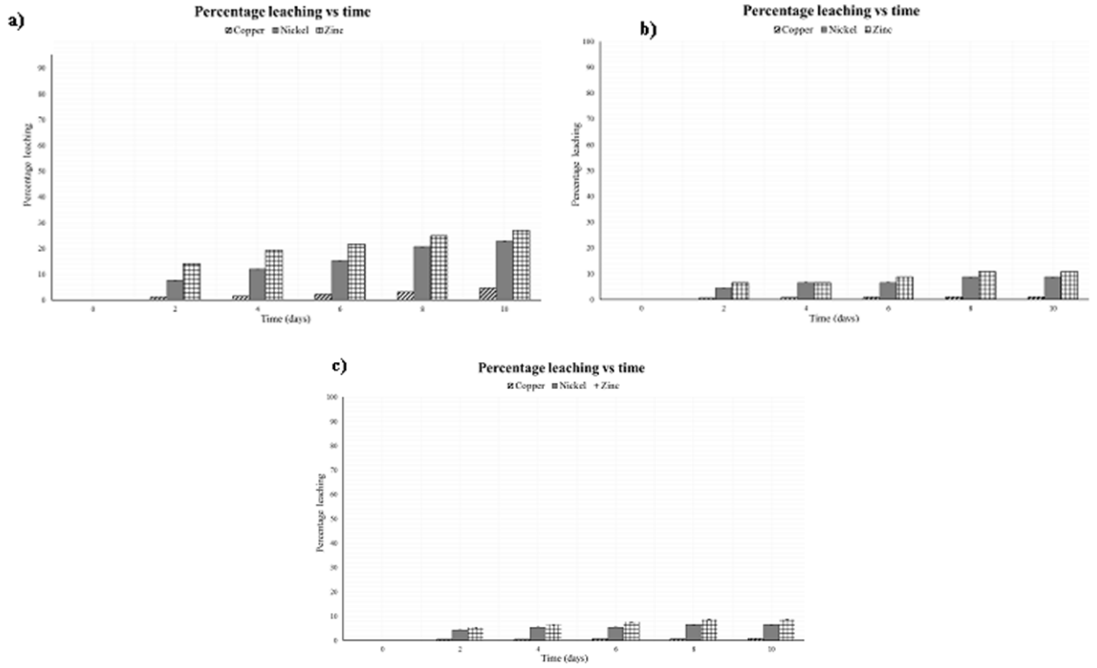


Fig.4. Percentage leaching vs time in control of copper, nickel and zinc at (a) 10 g/L, (b) 50 g/L and (c) 100 g/L. Error bars in all graphs indicate ± standard errors of mean values of duplicate experiments

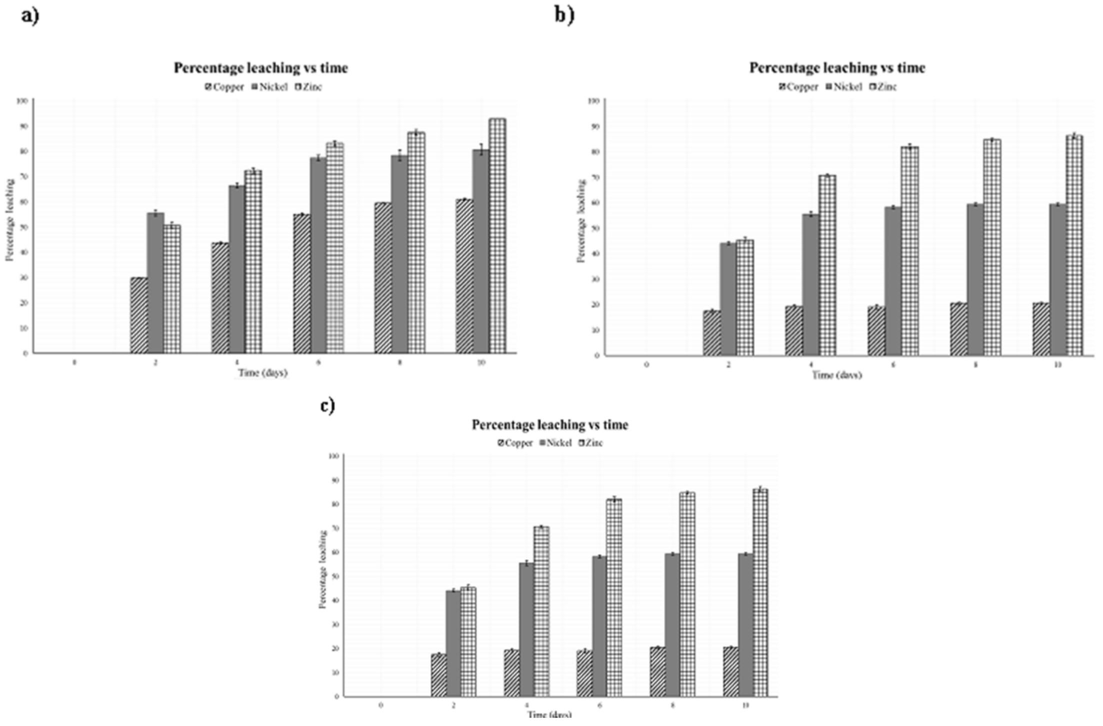
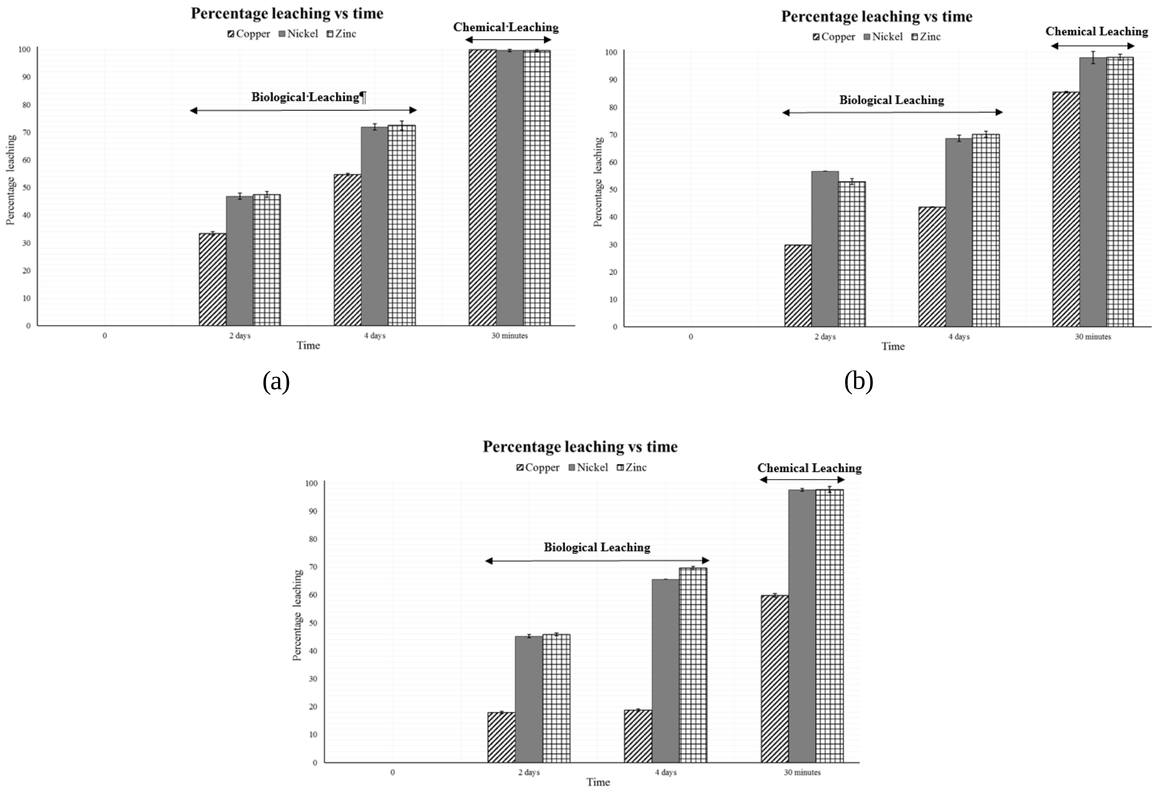


Fig. 5. Percentage leaching vs time in biometallurgical recovery of copper, nickel and zinc at (a) 10 g/L, (b) 50 g/L and (c) 100 g/L. Error bars in all graphs indicate ± standard errors of mean values of duplicate experiments

Acidithiobacillus ferrooxidans for 4 days, followed by nitric acid leaching for 30 minutes. This combination maximised the contribution of biological leaching (within a reasonable time period) and minimised the use of chemical leaching.

At 10 g/L e-waste concentration, all the three methods were found to be almost equally efficient in recovering Cu, Ni and Zn from the PCB powder. The biometallurgical and integrated treatments are suggested as the best methods for use at this concentration. The biometallurgical treatment no



(a)

(b)

(c)

Fig. 6. Percentage leaching vs time for integrated recovery of copper, nickel and zinc at (a) 10 g/L, (b) 50 g/L and (c) 100 g/L. Error bars in all graphs indicate ± standard errors of mean values of duplicate experiments

Table 3. Examples of recovery of metals from e-waste using chemicals or microorganisms in literature

Reference	Chemicals/Microorganisms	Leached metals
Kumar *et al.*, 2014	Nitric acid	96% Cu
Oliveira *et al.*, 2009a	Nitric acid	80-90% Cu, Fe, Ni, Zn, Pb, Ag
Oliveira *et al.*, 2009b	Nitric acid	90% Cu, Zn, Ni, Fe; 70% Ag
Brandl *et al.*, 2001	*Thiobacillus ferrooxidans + Thiobacillus thiooxidans*	90% Al, Cu, Zn, Ni
Wang *et al.*, 2009	*Acidithiobacillus ferrooxidans + Acidithiobacillus thiooxidans*	99.9% Cu, 88.9% Zn, Pb
Willner and Fornalzyck, 2013	*Acidithiobacillus ferrooxidans*	98-99% Cu

chemicals are required to be used and the method is economical and eco-friendly, even though the time taken for metal recovery was longer as compared to the other two methods. The hybrid method, on the other hand, reduced the time required for metal recovery as compared to the biometallurgical method. The hydometallurgical method recovered the metals in the shortest time period but it is not recommended as it is not eco-friendly.At 50 g/L e-waste concentration, the integrated method was the most efficient for the recovery of Cu, Ni and Zn, followed by the chemical and biological methods, respectively. Even though the recovery of the metals by the hybrid method was a little less as compared to its efficiency at 10 g/L e-waste concentration, the trade-off is acceptable as the recovery was on a larger scale.

At 100 g/L e-waste concentration, the efficiency of the chemical and integrated methods were approximately equal in the case of Cu recovery. In Ni and Zn recovery, the hybrid method outperformed the chemical method. The biological method exhibited poor recovery of copper. This concentration is not suggested for use in the recovery of copper by biological or hybrid methods, as the leaching of copper by *Acidithiobacillus ferrooxidans* was not as efficient as compared to lower concentrations.The integrated (hybrid) method, on comparison against both the chemical and biological methods, proved to be a better method than the other two by being rapid, efficient, economical and eco-friendly. The use of the optimised integrated method (*Acidithiobacillus ferrooxidans* leaching for 4 days, followed by nitric acid leaching for 30 minutes) at 50 g/L PCB powder concentration is recommended for the best recoveries of Cu, Ni and Zn from waste PCBs. 100 g/L concentration can be used for Ni and Zn recoveries, but is not recommended for Cu recovery. 10 g/L concentration can be used for small-scale metal recoveries, but for larger-scale recoveries, it is uneconomical to use. To conclude, the goal of this study is an initiation of resource recovery and e-waste management in our country through the integrated method.

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