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Leaching of Precious Metals from Residues of Discarded WEEE

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Abstract: E-waste recycling is majorly carried out by informal sector in India. Inefficient recycling results in residues which were disposed off in open land and drains. These residues contain unrecovered base and precious metals. Leaching of precious metals from residues (sludge and discarded printed circuit boards (PCBs) of e-waste were studied. Forty six samples were collected from waste dumping sites from four states in India. The samples were investigated for precious metals e.g., Au, Ag, Cu, Co and Ni. The elements were determined by AAS after hot plate assisted acid digestion. The concentrations of metals were ranging between Au (3.51-8324.13 mg/g), Ag (0-2211.74 mg/g), Cu (6-20865.53 mg/g), Co (0-3185.05 mg/g) and Ni (0-9822.1 mg/g). In samples, concentrations of Au, Ag, Cu and Co were found to be higher than their respective ores. The concentration of Ni in samples was found to be lower than its ore. Validation using loamy clay as CRM was carried out which showed a recovery of 67.40% for Ag, 70.87% for Cu, 81.88% for Co, 44.48% for Ni. The results show that e-waste residues can act as tertiary raw material for extracting Au, Ag, Cu, Co. Thus, proper management of these residues is imperative for recovery of precious metals.

Keywords: E-waste residues, sludge, leaching, precious metals, extraction, discarded PCBs

I. INTRODUCTION

The amount of e-waste is growing due to rapid change in technology and electronics usage all over the world. Total amount of electronic waste generated in world was 44.7 MMT (million metric ton) in 2016. It increased by 8 % from 2014 (3). Recycling e-waste acts as secondary raw material for extracting both base and precious metals. Although e-waste comprises mainly of base metals like Cu, Ni, Co, a significant amount of attention was given to precious metals like Au and Ag (17). Since PCBs contain precious metals in concentrated form, researchers have focused their attention on PCBs for precious metal recycling (17). Recovery of these precious metals resulted in energy and resource savings as shown in table 1. For example, quantity of gold extracted from one ton of e-waste specifically from personal computers was greater than that recovered from 17 ton of gold ore (9).

Table 1: Recycled materials energy savings over virgin materials (7)

No.	Materials	Energy Savings (%)
1	Aluminium	95
2	Copper	85
3	Iron and steel	74
4	Lead	65
5	Zinc	60
6	Paper	64
7	Plastics	>80

Metal recovery from electronic waste can be carried out by three processes, pyrometallurgy, hydrometallurgy and electrometallurgy. These processes were used separately or in combination for separation of target metals. Pyrometallurgy means application of heat in smelters or boilers. Various processes like incineration, combustion and pyrolysis were used in state of art smelters at very high temperature to liberate and purify metals of interest. This process works on physical and chemical properties of metals. Pyrometallurgy generated molten bath of metals and metal oxides form slag. The major drawbacks of application of pyrometallurgical process were handling process was difficult due to complex feed materials, huge investment was required to install smelters and generation of dioxins during heating process (2).

Hydrometallurgy means application of acid/chemicals to leach metals in solution. Various strong acids like HNO₃, HCl, H₂SO₄ and bases like H₂O₂ were used for dissolving metals. Cyanide, thiourea and epoxy resin were some of the chemicals used in hydrometallurgy. Hydrometallurgy is proven to be efficient, cost effective and can be conducted in controlled manner as compared

to pyrometallurgy (8). The final isolation of metals of interest were carried out by ion exchange, adsorption processes. These metals were recovered by another process called electrometallurgy. Electrometallurgy is generally the last stage of metal refining used after hydrometallurgy. Electrometallurgy uses electrodes to deposit the concerned metal. Cathode electrode of pure metal is installed against an impure anode. The pure metal from anode gets deposited on cathode and impurities get settled as anode mud (20).

E-waste was managed majorly by informal sector in India (14). There were over 3000 units engaged in informal sector for recycling. Informal units were distributed all across India. A huge cluster of small scale industries were present in outskirts of Delhi, Haryana, Uttar Pradesh, Karnataka and Tamil Nadu. Informal recyclers processed e-waste by various steps like collection of e-waste from kabadiwalas (5), manual disassembly which included sorting, segregation, dismantling to resell any useable parts which had economic value. The rest of the material was recycled by open burning and chemical leaching to recover precious metals (13). Due to unscientific method of recovery, hazardous substances and harmful gases could be released into surrounding air, soil and water. This recycling method had low efficiency and lots of residues were generated in the process. The residues were discarded on open land and in drains. Proper management of e-waste residues was required for protecting our environment. The objective of this study was to characterize and quantify precious metals in e-waste residues. This will help in closing the loop of e-waste recycling by looking at residues as a resource and not waste. Hence, circular economy can be created for management of e-waste from process of collection, dismantling, recycling and disposal. This would ultimately lead to proper management and better disposal of these residues.

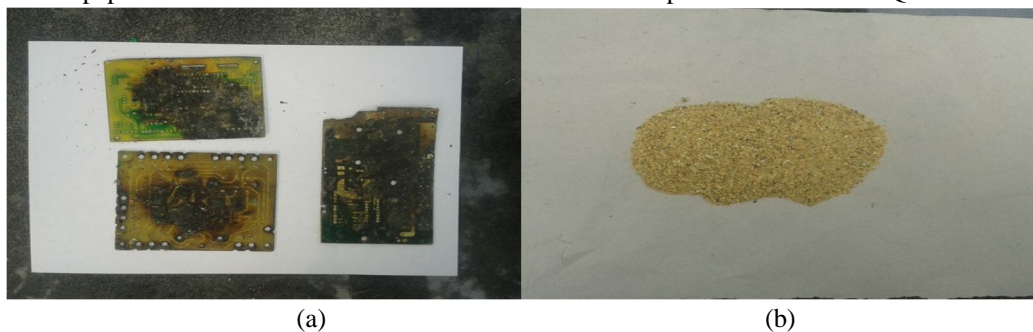
II. MATERIALS AND METHODS

A. Sample Collection

The samples were collected from five dumping sites at four states in India. Mandoli (Delhi), Moradabad (Uttar Pradesh), Panipat (Haryana) and Peenya, Goripalya (Karnataka) were selected for sample collection due to prominent informal e-waste recycling activities occurring at these sites (10). Samples were collected from e-waste dumping sites (open land, storage containers of waste acids and chemicals used by recyclers). E-waste recycling generated three types of residues like sludge, discarded PCBs and wastewater. Sludge and discarded PCBs were most important as they were highly concentrated forms of waste. Hence, sludge and discarded PCBs were collected as samples. A random sampling technique was followed to collect samples for further processing and analysis. The metals quantified were Au, Ag, Cu, Ni and Co. These metals were chosen as they were present in huge concentrations in electronics and also because of their extensive use (1).

B. Sample Processing

50 ml sludge samples were packed and transported in tarson plastic bottles. The discarded PCB samples were packed and transported in polythene zip lock bags. The samples were labeled accordingly and transported to laboratory for general characteristics. Samples were dried in an oven at 60°C for 4-6 hours. The samples before and after drying were presented in figure 1. After drying, the samples were grounded with mortar and pestle, screened through a 250 μ m sieve (BSS 60). Three replicates of each sample were prepared and 0.5 g sample weight was taken for further analysis. Samples were digested on hot plate with strong acids and bases (HNO_3 and H_2O_2) using USEPA SW 846 method 3050B. The sample digestion method was presented in figure 2. The only difference in using this method was made with sample weight which was given as 1 g in literature. 0.5 g sample weight was taken for present study as samples were highly concentrated and complete digestion of samples took place when sample weight was reduced from 1g to 0.5g. The mixture of dry sample and acid was heated carefully on a hot plate until the production of red nitrogen dioxide (NO_2) fumes had ceased. The mixture was then cooled, followed by addition of small amount (3-5 ml) of 30 % H_2O_2 . It was heated again till effervescence subsides. It was cooled and 10 ml conc HCl was added. The samples were filtered through whatmann filter paper no 42 into a 100 ml volumetric flask and made up to mark with milli Q water.





(c)

(d)

Figure 1: Collected samples (a) discarded PCBs before drying (b) discarded PCBs after drying (c) sludge before drying (d) sludge after drying in oven.

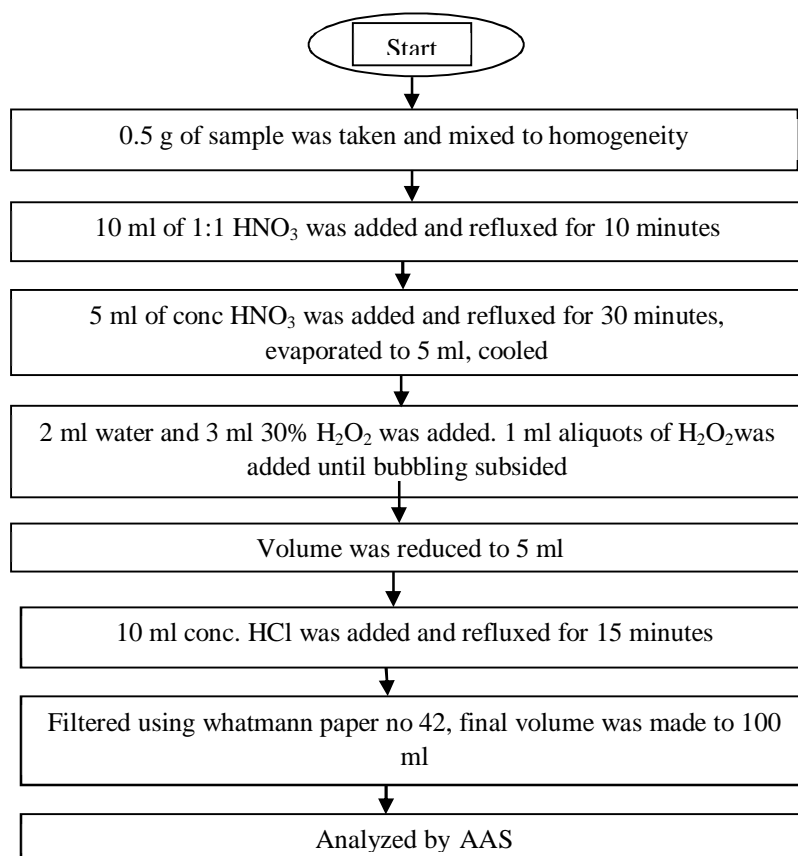


Figure 2: Process of sample digestion using method 3050 b given by USEPA SW 846.

C. Sample Analysis

After acid digestion, samples were analyzed for five metals, Ag, Au, Ni, Co, Cu using atomic absorption spectrophotometer (AAS) instrument (Model: Thermo Scientific ICE 3500). Standard solutions of samples were made for different concentrations of metals. A blank solution of distilled milli Q water and a reference sample of certified reference material (CRM) of loamy clay were run to check for quality assurance and quality control in the samples. Dilution of 5ml, 10 ml, 15 ml and 20 ml were made for standard solutions of gold, silver, copper, nickel and cobalt using the formula as given below.

$$P1V1 = P2V2$$

The concentration of these metals as present in solution were recorded in accordance with absorbance.

III. RESULTS AND DISCUSSION

The experimental results of metals tested on AAS were given in table 2. The concentrations of metals were recorded in mg/g. The economically viable limit for silver in its ore reported in literature was 100 mg/g (11). The minimum concentration of Ag was found to be 0 mg/g whereas maximum concentration measured for Ag was 2211.74 mg/g which is very much higher than the recoverable limit. More than 63 % of samples have higher value of Ag than recoverable limit. Thus these e-waste residues can be considered as a good source for recovery of silver.

Table 2: Concentration of precious metals (a) Ag (b) Au (c) Ni (d) Cu (e) Co in mg/g in samples tested on AAS.

Sample type	Sample location	Ag Conc (mg/g)	Au Conc (mg/g)	Ni Conc (mg/g)	Cu Conc (mg/g)	Co Conc (mg/g)
Sludge	Uttar Pradesh (Moradabad)	20.5-40.2	25.9-35.6	406.1-2606.7	6-44.1	8.5-246.1
	Delhi (Mandoli)	81.2-94.8	9.9-124.9	73.5-1259.7	55.5-7543.8	BDL-60.8
	Haryana (Panipat)	85.3	10.0	9822.1	8665.4	5.2
	Karnataka (Peenya)	102.2-2211.7	2493.8-2738.7	4815.6-6478.3	8311.0-8638.5	BDL-75.9
Discarded PCB	Karnataka (Goripalya)	67.1-1716.5	3.5-8324.1	BDL-9093.1	1649.4-8651.2	BDL-1386.8
	Delhi (Mandoli)	29.2-791.4	21.5-46.1	13.1-156.7	1096.1-2940.1	BDL-9.7
	Haryana (Panipat)	73.0	8.1	592.8	20865.5	8.4
	Karnataka (Komalgodu)	24.1	66.4	9206.6	8581.9	1.0
	Karnataka (Goripalya)	BDL-1343.6	21.7-184.0	586.8-8016.3	4053.8-8620.6	BDL-3185.0

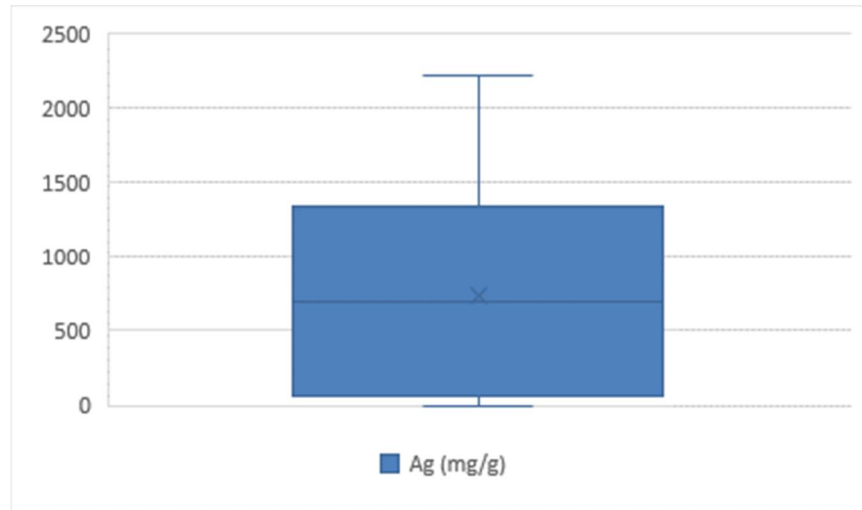
BDL-below detectable limit

For another precious metal Au, minimum and maximum values recorded were 3.51 mg/g and 8324.13 mg/g, respectively. In approximately 97% samples, concentration of Au was found to be higher than the recoverable limit reported in literature (4mg/g) (11). Therefore e-waste residues shall be looked into for recovering Au. The economically viable limit for recovery of copper from ore was found to be 7000 mg/g (22). The minimum concentration of Cu was found to be 6 mg/g and maximum concentration of Cu was found to be 20865.53 mg/g which is very much higher than the recoverable limit. More than 57 % of samples have higher value of Cu than recoverable limit. This shows that residue from e-waste were good source for recovery of copper.

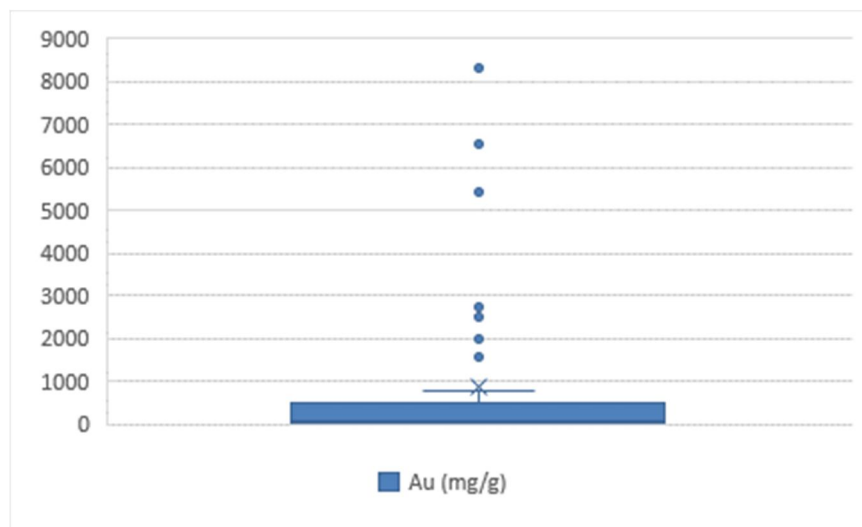
Table 3: Concentration of precious metals (a) Ag (b) Au (c) Ni (d) Cu (e) Co in mg/g in primary ores (11,22).

Metal	Concentration in Ore (mg/g)
Gold (Au)	4
Silver (Ag)	100
Copper (Cu)	7000
Cobalt (Co)	1000
Nickel (Ni)	11000

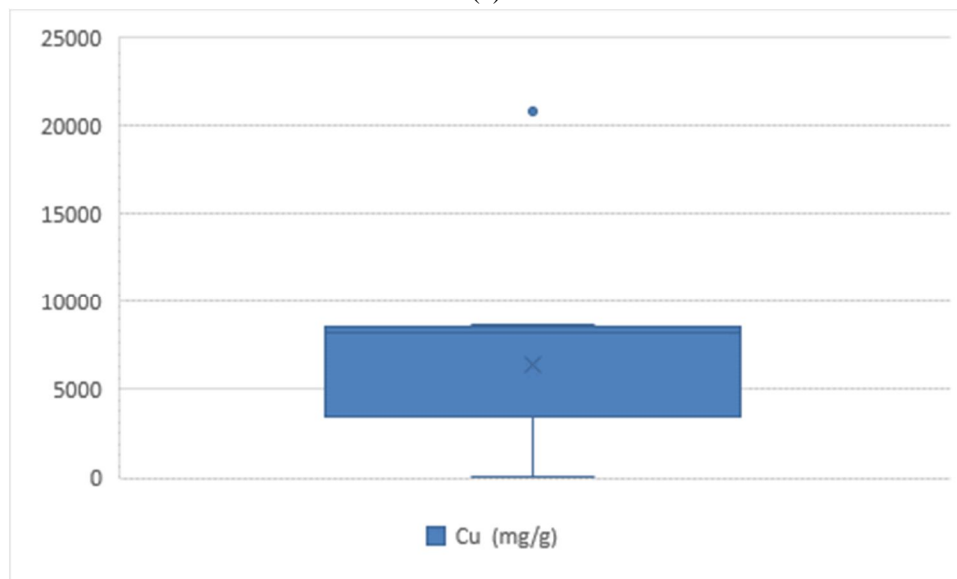
The economically viable limit for recovery of cobalt from ore was found to be 1000 mg/g (6). The minimum concentration of Co was found to be 0 mg/g and maximum concentration of Co was found to be 3185.05 mg/g which is very much higher than the recoverable limit. More than 5 % of samples have higher value of Co than recoverable limit. The results show that mostly all values of cobalt was below the economically viable limit for recovery for most samples. Hence, e-waste residues shall not be considered a good source for recovering Co. The economically viable limit for recovery of nickel from ore was found to be 11000 mg/g (6) as shown in table 3. None of the samples have higher value of Ni than recoverable limit. The results show that value of nickel remained below the economically viable limit for recovery for all samples. Nickel is a highly soluble metal and may be lost in form of salts. This shows that residue from e-waste were not good source for recovery of nickel. The concentration of Ag, Au, Cu, Co and Ni in mg/g were presented in box and whisker plot in figure 3.



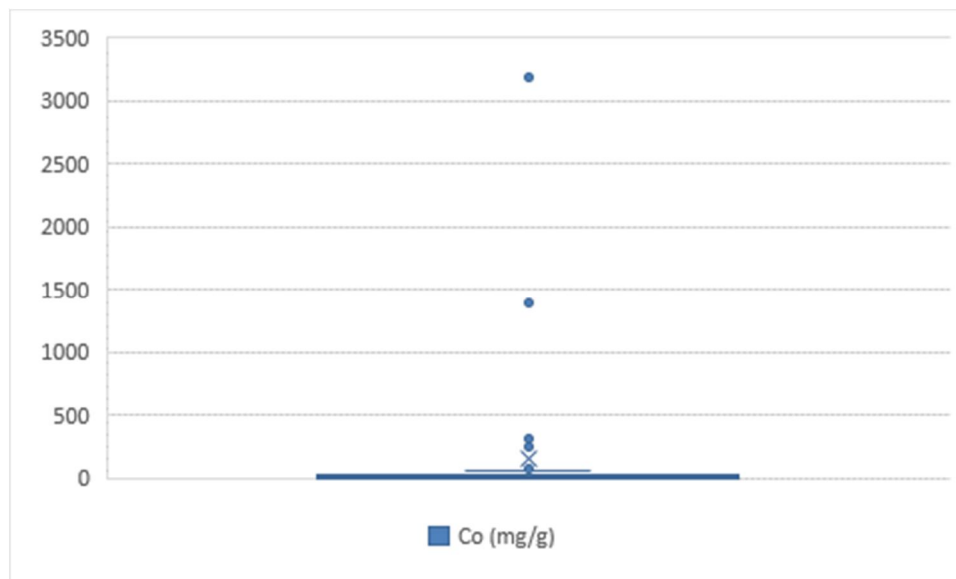
(a)



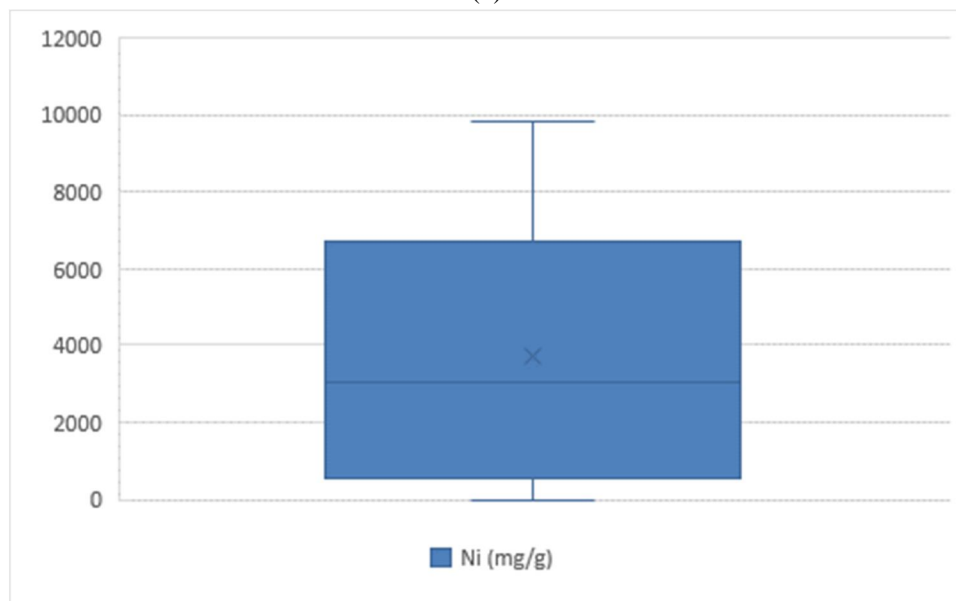
(b)



(c)



(d)



(e)

Figure: 3 Box and Whisker plot for metals (a) Ag (b) Au (c) Cu (d) Co (e) Ni in mg/g with recoverable limits.

The main focus of the study was to analyze e-waste residues for characterization of precious metals. Forty six samples were tested for five metals. Results show that residues left after e-waste recycling can be utilized for recovery of Au, Ag and Cu. The residues were not a good source for recovering Co and Ni from them. Significance of this study is that waste generated after e-waste recycling consists of precious metals in large quantities. The residues should be looked into as a resource instead of waste. They can act as tertiary raw materials for manufacturing new electronic products. Using these residues will be both environmentally friendly and economically viable. The residues should be managed in sustainable manner by scientific method of collection, recovery and disposal. Also, up gradation in recycling technology by recyclers will prevent loss of metals in the surroundings.

IV. CONCLUSION

The concentration of precious metals like Au, Ag and Cu were present above recoverable limit in sludge and discarded PCBs.

The concentrations of Co and Ni were present below recoverable limit in sludge and discarded PCBs.

The residues need to be managed properly and should be looked as a resource for recovery of metals from them.

A process for separation of individual metals from residues should be developed.

V. ACKNOWLEDGEMENTS

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