

Determination of Metal ions Concentrations in Leached Electronic Wastes Using Atomic Absorption Spectroscopy

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Abstract

Electronic wastes such as radio panel, computer adapter, digital video disc (DVD) power, television (TV) panel, half TV panel, light emitting diode (LED) board, screen board, laptop adapter, laptop batteries and mobile phone batteries, obtained from electronic shops and dumpsites in Computer Village, Ogbunabali, Port Harcourt, Nigeria were mechanically dismantled, crushed to reduce the size, incinerated using a blacksmith flame, ground in a ceramic mortar to obtain 1-2 mm size range particle size, sieved and leached with aqua regia (3HCl:1HNO₃), H₂SO₄, and H₂O₂. Thereafter, the metal ions concentrations found in the leached raw e-wastes were determined using Atomic Absorption Spectroscopy (AAS). The AAS results showed high concentration of Pb (10,875.000 ppm) in all the e-waste samples while Li had the least (0.0549 ppm). Therefore, Pb was discovered to be the most prominent metal in e-waste components. The leaching processes could be used to recover valuable resources and mitigate environmental pollution.

Keywords: *Determination, Concentrations, Metal ions, Leaching, Electronic waste*

Introduction

In the world today, there is an exponential increase in the consumer's demand for electrical and electronic equipment (EEE). This rapid growth in technology-based items in the 21st century and their consequent demand metamorphosed from Stone Age cultures to what is today known as "Electronic Age" (Agnihotri, 2011; Dave *et al.*, 2016). Meanwhile, rapid technological innovations in the electronic sector which has resulted in changes in equipment features and capabilities and escalating consumer demands for electronics in developing countries have inadvertently shortened the useful life of this EEE (Dave *et al.*, 2016; Isildar *et al.*, 2019). Decrease in prices, the growth in internet use as well as change in consumer tastes were also listed by Forti *et al.*, (2020) as factors responsible for shortening the product life of EEE. Every EEE has a predetermined life-span after which they become obsolete faster. These obsolete EEEs have hitherto, enhanced the global generation of huge quantities of wastes from electrical and electronic equipment (WEEE) otherwise called electronic waste (e-waste) (Sinha-Khetriwala, *et al.*, 2005) that needs to be properly managed.

E-waste is a term used for electronic products or discarded devices nearing the end of their “useful life” and is not economically useful to consumers anymore. E-waste includes discarded computers, televisions, refrigerators, electric lamps, cell phones, stereos, copiers, fax machines, audio equipment and batteries. It also contains precious metals (PMs) like gold (Au), silver (Ag), nickel (Ni), lithium (Li), cobalt (Co), copper (Cu), selenium (Se), mercury (Hg), platinum (Pt), Gallium (Ga), germanium (Ge), palladium (Pd), tantalum (Ta), and tellurium (Te) (Admed *et al.*, 2019). Precious metals are widely used in the manufacture of electronic appliances due to their high chemical stability and good conducting properties (Cui and Zhang, 2008). These metals are known to possess certain useful properties including ductility, malleability, lustre and good electrical conductivity. However, when applied, their tensile strength, corrosion resistance and hardness are insufficient. Therefore, these properties can be improved by mixing (alloying) metal with some other metal or non-metal.

When these e-wastes are improperly disposed, they can leach lead (Pb) and other substances into soil and groundwater (Kavitha, 2014; Nageswara, 2014; Afiukwa *et al.*, 2015). This is because e-waste contains many toxic elements like lead, mercury, cadmium, tin, etc, that can leach into the earth and even water bodies to cause severe ailments like cancer, liver damage, kidney problems, etc, for the mankind (Kavitha, 2014). Therefore, e-wastes can be prevented, recycled and reused to minimize environmental impact. Itoh (2014) recommended appropriate treatments of e-waste in order to obviate environmental pollution and to recycle the precious resources obtained.

Earlier before the statistical report on e-waste generation by Isildar *et al.*, (2019), Huisman (2010), predicted that e-waste generation will be 19.1 kg/person/year by 2012 in 27 European Union countries leading to total of 10.5 million tons of e-waste by 2014. The study also estimated a total of 41.8 million metric tons (Mt) of e-waste generation worldwide by the year 2018. In 2015, it was reported that about 54 million tons of e-waste was generated worldwide (Debnath *et al.*, 2018). On global e-waste key statistics, about 53.6 Mt of e-waste was reported to be generated at 7.3 kg per capita in 2019 (Forti *et al.*, (2020). By 2020, e-waste has increased five-fold in India (Bhat *et al.*, 2012). Forti *et al.*, (2020) estimated 74 Mt of e-waste to be generated in the year 2030. Houessionon *et al.*, (2021) also predicted that by 2030, the amount of e-waste is expected to reach 71.7 Mt.

In Dave *et al.*, (2016), countries which served as largest collectors of e-waste in the world were reported. Guiyu in Hong Kong, China is still the largest e-waste collector worldwide, and they collect e-waste that came from USA, Japan, Canada, and South Korea (Dave *et al.*, 2016). Guiyu is considered the electronic graveyard of the world (Wang *et al.*, 2020) because of its informal e-waste recycling (Abalansa *et al.*, 2021). Accra in Ghana and Lagos port in Nigeria are next to Guiyu in the importation of wastes. Daum *et al.*, (2017) described Agbogbloshie in Ghana as the most polluted e-waste site on earth. This is because it received exceptional quantities of e-waste exported to Ghana. A study carried out by Schmidt (2006) showed that an average of 5 million fairly used computers weighing 60,000 tonnes enter into Nigeria through Lagos port, out of which nearly 30,000 tonnes were non-functional or irreparable. In Adaramodu *et al.*, (2012), Basel Action Network (BAN), a Seattle-based environmental group, is quoted to have reported that an estimated 500 shipping containers with a load equal in volume to 400,000 computer monitors or 175,000 large television sets enter into Lagos, Nigeria on a monthly basis, of which 75 % of the shipments are classified as e-waste.

Although, it is on record that researchers from countries like India, Japan, Korea, Turkey, Australia and Norway have extracted, recovered and even recycled wastes and precious, base and heavy metals from electrical and electronic equipment in their countries, according to their legislation, employing mechanical processing, hydrometallurgical (Kim *et al.*, 2011), pyrometallurgical (Hall and Williams, 2017), electrometallurgical routes and biometallurgy (Ilyas and Lee, 2014), there is no report on the analysis of metal ions concentrations from electronic wastes obtained from electronic or computer villages in Nigeria. For this reason, computer village in Ogbunabali, Port Harcourt, Nigeria has been chosen to be studied.

Leaching by hydrometallurgical process using inorganic acid, organic acid and base have been employed (Cui and Zhang, 2008; Ashiq *et al.*, 2019; Urbanska 2020). The amount of metal leached by different leachants is quantified using atomic absorption spectroscopy (AAS). Among various inorganic acids, aqua regia (mixture of HNO₃ and HCl) is found to be the strongest leachant for most of the metals such as Zn (2.04 wt %), Fe (17.90 wt %), Ni (0.66 wt %), Pb (5.86 wt %) and Au (0.04 wt %) (Cui and Zhang, 2008). Leaching agents such as cyanides, ammonium sulphates, halides, thiourea, thiosulphates and *aqua regia* are used by different authors for the investigation of dissolution of precious and heavy metals such as Ag, Au, Cu, Fe, Pt and Zn from printed circuit board (PCB) of mobile phones and computers (Yunus and Sengupta, 2016).

However, Bhat *et al.* (2012) developed an integrated model to recover PMs (Au and Ag) from electronic scrap using a combination of hydrometallurgical (chemical) and biometallurgical (low cost biomass) processes. They used cyanide to leach Au/Ag from electronic scrap. The leachate containing Au/Ag complex was subjected to biosorption process using low-cost biomass. Their results showed that *Eicchornia* root biomass and waste tea powder were efficient biosorbents for leached silver-cyanide from electronic scrap. Pak and Fray (2009) employed hydrometallurgical method to recover precious metals from e-waste. They used *aqua regia* as the leaching agent and recovered Ag, Pd and Au with 98 %, 93 % and 97 %, respectively. Barragan *et al.* (2020) recovered pure Cu and Sb from e-waste by the combination of hydrometallurgical and electrochemical processes. In their study, a 96 wt % pure Cu deposit and 81 wt % pure Sb precipitate were recovered. The common hydrometallurgical method for the recovery of Cu includes chemical leaching in H₂SO₄ (Guo *et al.*, 2015), HCl (Zhang *et al.*, 2017) or HNO₃ (Choubey *et al.*, 2015; Lee and Mishra, 2018). This is in combination with different oxidants such as H₂O₂, FeSO₄, and FeCl₃ (Barragan *et al.*, 2020).

Various researchers had also recovered precious metals from e-waste at the laboratory level. Urbanska (2020) applied 1.5 M H₂SO₄ as the leaching agent, 30 % H₂O₂ solution and 5 g C₅H₈O₄ as the reducing agents to recover Co, Li and Ni from spent Li-ion batteries with hydrometallurgical technique. The results showed that it is possible to efficiently recover Co (87.85 %), Li (99.91 %) and Ni (91.46 %) from battery power using acidic leaching. Ficeriova *et al.* (2011) leached Ag and Au and accompanying metals from PCBs waste. They used H₂SO₄ and H₂O₂ to leach Zn, Cu, Fe, Al and Ni; ammonium thiosulphate {(NH₄)₂S₂O₃}, in the presence of copper sulphate (CuSO₄.5H₂O) and ammonia (NH₃) to leach Ag and Au; sodium hydroxide (NaOH) to leach Pb and Sn; and aqua regia to leach Pd. With this hydrometallurgical approach, it was possible to achieve 70 % Zn, 84 % Cu, 82 % Fe, 77 % Al, 70 % Ni, 93 % Ag, 98 % Au, 90 % Pb, 83 % Sn and 90 % Pd. Their selection was done in a way that allowed selective leaching of the metals of interest (Ficeriova *et al.*, 2011), hence, excellent recoveries of all the metals were possible. Zinc,

cadmium, lead and copper from electronic scrap had also been leached using organic acids and the *Aspergillus niger* strain (Kolencik *et al.*, 2013).

The determination of precious metals from electronic wastes has benefits economically and environmentally. Therefore, this study will determine whether the metal ions from leached e-wastes can be made available to step-up or increase the quantity of natural metals in the environment which will be beneficial to our local industries and academics. Therefore, the aim of the study is to determine metal ions concentrations in leached electronic wastes.

1. Materials and Methods

2.1 Sample Collection and Preparation

Printed circuit boards (PCBs) of discarded mobile cell phones, computers, radio and television were collected randomly from dumpsites and electronic repair workshops in Computer village, Ogbunabali, Port Harcourt, Nigeria. The components were dismantled, tested, isolated and individual components sorted out from the e-waste according to their kind as described by (Kolencik *et al.*, (2013); Sahan *et al.*, (2019)). Plate 1 is a picture of dismantled e-waste sample showing the television panel, LED board, radio panel, screen board, computer adapter, laptop adapter, laptop battery, and mobile phone battery.



Plate 1: Dismantled E-waste Samples

The different components which include; integrated circuits (IC), aluminium covers, condensers, capacitors, chopper transformers, transistors, batteries, printed circuit boards (PCB), diodes and resistors (labeled samples A to J) were subjected to incineration using the blacksmith flame. The incinerated materials were further ground in a ceramic mortar to obtain 1-2 mm size range particle size. Ground incinerated e-waste samples were leached with different leachants and analysed using AAS analysis.

2.2 Determination of Metal ions Concentrations in Leached E-wastes

2.2.1 Leaching and Analysis of Zinc

Fifty grams (50 g) of ash obtained from incineration of e-waste component was weighed into a 500 mL beaker and 120mL of aqua regia (3 HCl: 1 HNO₃) was added and placed in a fume

cupboard. The digestion was allowed to proceed for 4 hours until NO₂ fume stopped; the sample was denoxed with urea before filtering into a clean sample bottle until required for analysis. The concentration of zinc in the solution was analysed using AAS.

The concentrations of metal ions (Zn, Fe, Ni, Cd, Pb, etc) from e-wastes using AAS were calculated using equation below.

$$\text{Element (mg/g)} = \frac{\text{AAS result } \left(\frac{\text{mg}}{\text{L}}\right) \times \text{Volume of sample (L)} \times \text{DF}}{\text{Weight of sample (kg)}}$$

1

Where:

DF is Dilution factor

2.2.2 Leaching and Analysis of Iron

Aqua regia (3 HCl: 1 HNO₃) was added to 45.66 g of e-waste powder in a 500 mL beaker and digested for 4 hours. The leached liquor was denoxed using urea to remove excess HNO₃ before filtering into a clean sample bottle until required for analysis. The concentration of iron in the solution was analysed by AAS.

2.2.3 Leaching and Analysis of Nickel

The resultant ash (35.11 g) was leached with 10 mol/L H₂SO₄ for 1 hour and filtered through Whatman filter paper to give the leachate. The leaching temperature was set at 70°C, under the conditions of the reaction time of 1 hour as described by Zainuddin *et al.* (2019). The nickel concentration in the leached liquor was determined using multimeter portable colorimeter (HACH DR 900) and HACH METHOD 8009, 8506 and 8105 and AAS analysis.

2.2.4 Leaching and Analysis of Cadmium

Twenty five grams (25 g) of incinerated e-waste ash was weighed into a 500 mL beaker. Thereafter, 100 mL H₂SO₄ (5.86 vol. %) and 50 mL H₂O₂ solution were added and allowed to stand for 15 minutes at a temperature of 35°C; the mixture was allowed to cool and then filtered into a 250 mL conical flask. The concentration of cadmium in the solution was analysed using AAS.

2.2.5 Leaching and Analysis of Lead

Slightly above thirty five grams (35.11 g) of the fine ash powder was treated with 120mL 1.0M HNO₃, in fume chamber in order to avoid the liberation of toxic fumes to the environment. Stirring was by magnetic stirrer while leaching was allowed 3 hours for maximum dissolution. The leaching was followed by filtration to obtain leached liquor; the leached liquor obtained from the leaching was neutralized using NaOH pellets until pH of 10.5 at a temperature of 30°C. The leached liquor was also analyzed by AAS to determine the composition of metal present.

2.2.6 Leaching and Analysis of Cobalt

The resultant ash (50.66 g) was leached with 10 mol/L H₂SO₄ and 30mol/L H₂O₂ for 1 hour. It was filtered through Whatman filter paper to give the leachate. The leaching temperature was set at 70°C, under the conditions of the reaction time as described by Yu *et al.*, (2015). Thereafter, the

sample was sent for spectrophotometric determination of Co^{2+} ion concentration in the solution with cobalt ions spectral qualities, using AAS.

2.2.7 Leaching and Analysis of Copper

Twenty five grams (25 g) of ash from incinerated e-waste was weighed into a 500 mL beaker and copper (Cu) was removed by leaching with the mixture of 100 mL (H_2SO_4) and 50 mL (H_2O_2) for 2 hours. The concentration of Cu in the solution was analysed using AAS.

2.2.8 Leaching and Analysis of Aluminium

Slightly above fifty grams (50.16 g) of incinerated e-waste was weighed into a 250 mL beaker and placed under the fume cupboard. About 62 mL of 1.4 M KOH solution was added to the beaker. The mixture was heated on the electric hot plate for 30 minutes to dissolve. The reaction turned dark grey due to silicon impurities and it was then filtered through filter paper after cooling to remove any solids remaining. The resultant solution was transferred to a clean 250 mL beaker. The mixture was further cooled by placing on an ice water bath. On standing, a white, gelatinous aluminium hydroxide precipitate was formed. Then, 30 mL of 9 M H_2SO_4 was added slowly, carefully and stirred. The leached liquor was analysed to determine the concentration of aluminium in the solution using AAS.

2.2.9 Leaching and Analysis of Silver

Fifty grams (50 g) of e-waste ash were dissolved in 200 mL of 1 M HNO_3 . To achieve optimal dissolution, sample was allowed to stand for 3 hours at 70°C at an agitation rate of 300 rpm in a temperature controlled water bath. The experiment was carried out in fume cupboard. Nitrogen oxide gases, which formed during the leaching, expelled to the atmosphere with the help of an expeller fan. After the silver was dissolved in the nitric acid solution, the concentrations of elements present in the solutions were measured with AAS at the end of the experiment.

2.2.10 Leaching and Analysis of Lithium

One hundred (100 mL) trichloroacetic acid (TCA) and 30 mL H_2O_2 were added to a 500 mL beaker containing 25 g of the e-waste and allowed to stand for 5 hours. The leached liquor was analysed to determine the concentration of lithium in the solution using AAS.

2.2.11 Leaching and Analysis of Tin

Approximately 50 g of the sieved incinerated e-waste powder was weighed into a 500 mL borosilicate glass beaker and washed with 1.0 L of distilled water, to remove any residual non-metallic materials that may affect leaching and precipitation of metals. Washed powder was collected after removing water and characterised to identify inorganic species and to identify metal elements. The metal content in the samples was determined by AAS. Washed e-waste powder (50 g) was weighed into a 500 mL beaker and 300 mL of leached solution (3 M HCl + 1 M HNO_3) added and the vessel mounted over a heating plate (Mettler, model 752 A). A magnetic rod was used for stirring. The experiments were carried out at $50\pm 2^\circ\text{C}$, for 120 minutes and the temperature were measured with a mercury thermometer (110°C). Leached liquor samples were collected, filtered and analysed to determine tin concentration in aqueous solution through AAS.

3.0 Results and Discussion

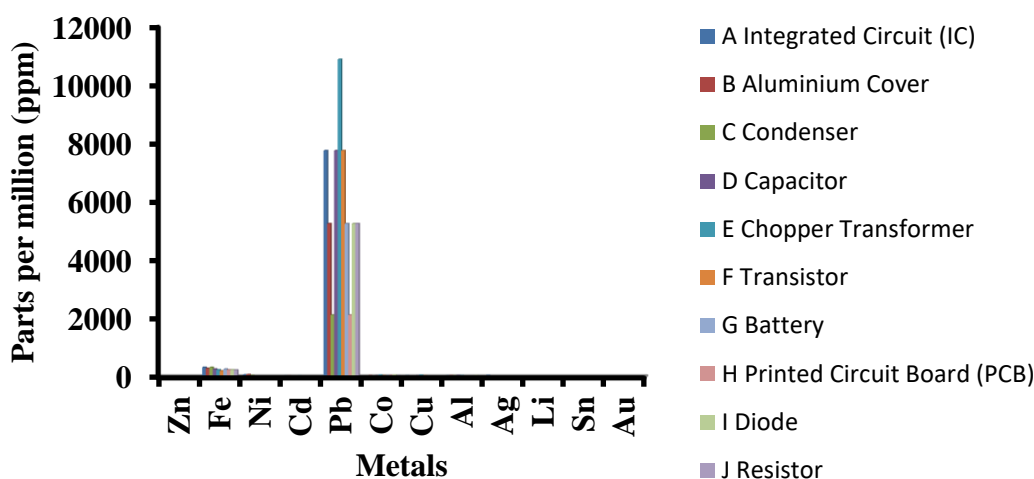


Figure 1: Concentrations of Metals in E-waste Samples in ppm (AAS)

3.1 Metal Concentrations in Leached E-waste Samples

The concentrations of various metals present in e-waste raw samples were analysed using AAS. Based on the results obtained (Figure 1), Zinc (Zn) had the highest concentrations in samples C, D, H and I with 31.639 ppm respectively and least concentration in samples A and E (16.885 ppm) respectively.

Iron (Fe) was observed to be high in samples A and C (331.125 ppm) respectively and least in sample F (211.921 ppm). Nickel (Ni) recorded the highest value in sample B (83.761 ppm) and least value in samples D, E and F (15.385 ppm) respectively. For cadmium (Cd), the highest values were observed in samples A, B and E (35.149 ppm) respectively and least values in samples D, I and J (12.871 ppm) respectively. Lead (Pb) was very high in sample E (10,875.000 ppm) and least in samples C and H (2125.000 ppm) respectively. Cobalt (Co) was prevalent in samples E and I (68.293 ppm) in each sample and least in sample A (19.512 ppm).

Copper (Cu) was high in sample E (62.212 ppm) and least in samples B, C, G and H (20.737 ppm) respectively. Aluminium (Al) was observed to have a high concentration in sample D (52.756 ppm) and least in sample F (21.260 ppm). Silver (Ag) was prevalent in sample A (49.192 ppm) followed by copper (Cu) (39.171 ppm) and least in samples G and H (15.081 ppm) respectively. This suggests the presence of sterling silver (Ag + Cu) in sample A (IC).

Lithium (Li) was observed only in sample G with a very low concentration of 0.0549 ppm. Urbanska, 2020 had earlier reported the presence of Li and Co as the prevalent metals in battery, as they occur in the form of lithium cobalt oxide (LiCoO_2). Tin (Sn) and gold (Au) were equally observed only in samples A (2.186 ppm) and H (0.2374 ppm) respectively.

Generally, the results of the AAS analysis showed that Pb had the highest concentration (10,875.000 ppm) amongst all the metals and Li, the least concentration (0.0549 ppm). This confirmed that Pb is the prevalent metal in e-waste components. Lead was dominance in all the components due to its soldering property. The lead could also be added to improve machinability. Solder connects electrical components to circuits. The samples showed high concentrations of metals in the e-waste components as follows: sample A, Pb, Fe, Ni, Ag and Cu; sample B, Pb, Fe

and Ni; sample C, Pb and Fe; sample D, Pb, Fe and Al; sample E, Pb, Fe, Cu and Co; sample F, Pb, Fe and Co; sample G, Pb, Fe and Co; sample H, Pb and Fe; sample I, Pb, Fe and Co; and sample J, Pb and Fe. On the contrary, the AAS results reported by Ajiboye *et al.*, (2019), on the composition of metals in the waste PCBs powder sample recorded Cu (41.64%) and Fe (11.118 %) as the major metals in PCB.

4.0 Conclusion

A simple and effective combination method of incineration and chemical leaching of dismantled and crushed e-waste has been applied to successfully determine metal ions concentrations from leached e-waste components. The concentration of lead (Pb) appeared to be high in all the e-waste samples while Li was the least as analysed by AAS. Therefore, Pb was discovered to be the most prominent metal in e-waste components. The leaching processes could be used to recover valuable resources and mitigate environmental pollution.

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