ESTIMATION OF THE LEVELS OF METAL OXIDES IN RECOVERED ELECTRONIC WASTES OBTAINED FROM COMPUTER VILLAGE, OGBUNABALI, PORT HARCOURT, NIGERIA

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Abstract

The levels of metal oxides in recovered electronic wastes obtained from electronic shops and dumpsites in Computer Village, Ogbunabali, Port Harcourt, Nigeria has been estimated. 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 were dismantled, crushed, incinerated, pulverized, sieved, extracted and the metal oxides found in recovered e-wastes estimated using appropriate methods. The metal oxides obtained were ZnO, Fe_2O_3, NiO, CdO, PbO, Co_3O_4, CuO, Al_2O_3, AgO, Li_2O and SnO_2 from dismantled e-wastes. The highest percentage recovered was 48.72 % (Al_2O_3) while the least was 4.75 % (AgO). The highest percentage yield was 45.20 % (Fe_2O_3) while the least was 2.16 % (AgO). The recovered metal oxides could serve as important secondary sources of critical raw materials for diverse industries. The recovery processes could be used to recover valuable resources and mitigate environmental pollution.

Keywords: *Estimation, Recovery, Electronic waste, Metal Oxides, and Leaching*

Introduction

A notable type of municipal solid waste which is growing very fast and large in the near future, that requires attention, is the electronic waste (e-waste) (Elaigwu *et al*., 2007; Ongondo *et al*., 2011; Debnath *et al*., 2016; Singh *et al*.; 2018 Isildar *et al*., 2019). Around 6 million tonnes of e-waste had been estimated in the European Union (EU) while the growth rate is expected to rise by 3-5 % per year (Dave *et al*., 2016; Sahan *et al*., 2019). Generation of e-waste has exponentially increased each year (Debnath *et al*., 2018) and in the last two decades (Ahmed *et al*., 2019) due to rapid technological advancements in the electronics sector (Singh *et al*., 2018), coupled with decreasing economic lifespan and weight of electronic gadgets and easy affordability (Isildar *et al*., 2019). The lifespan of some electronic devices is stated in the literature (Yunus and Sengupta, 2016; Isildar *et al*., 2019; Sahan *et al*., 2019).

E-wastes are discarded electronic products or devices nearing the end of their "useful lives" and are not economically useful to consumers anymore. E-waste contains precious metals (PMs) such as gold (Au), silver (Ag), lead (Pb), nickel (Ni), lithium (Li), Aluminium (Al), 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) as well as persistent organic pollutants (POPs), such as dioxin, brominated flame retardants (BFRs) (Borthakur, 2016), polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyls (PCBs), and polyvinyl chloride (PVC) (Yunus and Sengupta, 2016). The composition of e-waste is very diverse, having various sizes, shapes, chemicals and physical components (Khaliq *et al*., 2014; Yunus and Sengupta, 2016). Dave *et al*., (2016) observed that e-waste contains more than 1000 different substances which are classified as hazardous when discarded. E-waste differs chemically and physically in its composition from domestic and industrial waste. Chemically, e-waste consists of metals, metalloids, halogenated compounds and radioactive elements (Dave *et al*., 2016). Metals can be alloyed with other elements to increase a number of properties including strength, hardness, durability, and ability to conduct electricity, ability to withstand heat, corrosion resistance and machinability. Some of these alloys are used in the manufacture of electrical and electronic equipment (Debnath *et al*., 2016; Isildar *et al*., 2019).

Due to the different types of hazardous mixture of materials and toxic elements used in the production of electrical and electronic devices, disposal of e-waste is a rapidly growing global environmental problem like ozone depletion, acid rain and global warming. E-waste can be harmful to both human health and environment if not properly treated (Ahmed *et al*., 2019) because they discharge heavy metals and persistent organics (Cesaro *et al*., 2017). The presence of these metals and compounds makes e-waste to be considered as a hazardous material (Pant *et al*., 2012).

Significant amounts of Waste Electrical Electronic Waste generated in the developed countries which have state-of-the art facilities, finances and technology to handle waste (Forti *et al*., 2020) are continuously being shipped to developing or under-developed countries where there are hardly or no standard treatment methods, poor documentation of informal waste management systems, (Isildar *et al*., 2019), inadequate laws to protect the workers and the environment and moreover, these countries are probably not aware of the hazardous nature of e-waste (Dave *et al*., 2016) hence cause hazards to the environment. The Greenpeace, a non-governmental organization, reported that tonnes of e-wastes had been secretly flown or exported into countries like China, India, Malaysia, Vietnam, Pakistan, Philippines, Ghana, Nigeria and other countries where strict environmental set of laws do not exist (Dave *et al*., 2016).

The developed countries export e-waste to the developing countries in the name of free trade, for recycling and disposal owing to the lower labour costs and less stringent environmental regulations experienced in the developing countries (Singh *et al*., 2018). In an attempt to bridge the ‘digital divide’ (Ahmed *et al*., 2019), fairly used electronic equipment is exported into a developing country like Nigeria which relies on imported fairly used devices (Nnorom and Osibanjo, 2008). Meanwhile, following the Basel Convention, with about 186 parties as signatories (Awasthi *et al*., (2019), the regional Bamoko convention, with 25 countries as signatories (UNEP, 2021), and various national laws, it is illegal to export e-waste overseas until the importing country processes it in an environmentally friendly way (Khaliq *et al*., 2014; Abalansa*et al*., 2021). No country is expected to export environmental problems or e-waste or hazardous waste to another country or region (Kummer, 1992; Choksi, 2001). It is therefore expedient, that countries comply with the Basel Convention and the regional Bamoko convention by controlling the movements of hazardous waste and their disposal across boundaries. For instance, Australia, a significant producer of e-waste in the world and a signatory to the Basel Convention, exports e-waste to Singapore, India and China for processing (Khaliq *et al*., 2014). Dave *et al*., (2016) also reported that only 15-20 % of the total 50 million tonnes of e-wastes generated globally are recycled properly. Wang *et al*., (2016) posited that in 2014, only 15 % of the e-wastes generated in the developed countries were officially discarded through nationwide take-back arrangements. In fact, globally, only 10 % of the produced e-waste is recycled in developed countries while the remaining 90 % is exported to developing countries across the globe (Abalansa *et al*., 2021) for reprocessing and dumping (Adewumi *et al*., 2017). It has been critically observed that our modern cities are the focal points around which discarded electronic gadgets are concentrated, due to high population density (Hageluken, 2006; Isildar *et al*., 2019).

There are three methods widely used for recovery of metal fractions separated from e-waste components, namely, hydrometallurgy, pyrometallurgy and bio-metallurgy (Yunus and Sengupta, 2016). The three methods of recovering metals from e-waste had earlier been reviewed by Cui and Zhang (2008). Khaliq *et al*., (2014) added electrometallurgy or electrochemical process as another method. Although, these four processes have their pros and cons, hydrometallurgical process has additional benefits and is most widely used by researchers to recover valuable metals because it is environmentally friendly (Yunus and Sengupta, 2016), more exact, predictable and easily controlled (Cui and Zhang, 2008; Khaliq *et al*., 2014; Dave *et al*., 2016). Kavitha (2014) extracted precious metals from e-waste using hydrometallurgical, pyrometallurgical and bio-leaching processes. Ajiboye *et al*., (2019) conducted selective extraction of Cu and Zn, and other metals as impurities using selective lixicants such as HCl, H_2SO_4, HNO_3 and NaOH. They achieve almost 100 % Zn extraction using NaOH lixicantswithout trace of other metals. In Lee and Mishra (2018), three different acid solutions, HNO_3, H_2SO_4 and HCl were used as leaching reagents in the selective recovery and separation of Cu and Fe from fine materials of electronic waste processing. 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). Pak and Fray (2009) employed hydrometallurgical method to recover PMs 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 weight % pure Cu deposit and 81 weight% pure Sb precipitate were recovered.

Ficeriova *et al*. (2011) used H_2SO_4 and H_2O_2 to leach Zn, Cu, Fe, Al and Ni; ammonium thiosulphate $\{(NH_4)_2S_2O_3\}$, in the presence of copper sulphate ($CuSO_4.5H_2O$) and ammonia (NH_3) to leach Ag and Au; sodium hydroxide (NaOH) to leach Pb and Sn; and aqua regia to leach Pd from PCBs waste. 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.

The estimation of levels of some valuable metal oxides from e-waste and their recovery in the pure and useful forms are the focus of this research work. Therefore, the recovery and estimation of some metal oxides from e-waste will help to provide a new source of cheap and easily available metal oxides which might be relevant for uses in our industries and the world at large.

2.0 Experimental

Discarded electronic wastes such as mobile phones, computers, radio and television collected from electronic repair workshops in Port Harcourt metropolis, Nigeria were manually dismantled, de-soldered, sorted out and pulverized to reduce the sizes. The different components were separated according to their kind as described by Kolencik *et al*., (2013); Sahan *et al*., (2019). The components were identified as computer adapter, digital video disc (DVD) power, radio panel, screen board, laptop adapter, laptop batteries, mobile phone batteries, television panel and light emitting diode (LED) board, out of which different components such as integrated circuits (ICs), aluminium covers, condensers, capacitors, chopper transformers, transistors, batteries, printed circuit boards (PCBs), diodes and resistors were identified and subjected to thermal treatment using the blacksmith flame. The incinerated materials were further ground in a ceramic mortar and sieved to obtain 1-2 mm size range particle size. The powdered e-waste samples were taken to the laboratory for analysis.

2.1 Estimation of the Level of Metal Oxides in Recovered E-wastes

2.1.1 Recovery and Estimation of Zinc in E-wastes

The resulting leached liquor was cotton filtered into 250 ml conical flask. Solid NaOH was gradually added to the leached liquor, and the pH of the solution was monitored by a pH meter while stirring at 150 revolutions per minute (rpm) on a magnetic stirrer. At pH 6.8, and at a temperature of 95°C, 1.3g of zinc powder was added gradually until a greyish colour appeared. The sample was allowed to precipitate after which it was decanted. All precipitates obtained at specific pH values were collected by centrifugation, dried in an oven, and weighed as zinc oxide. The percentage recovery was calculated as shown in equation 1:

$$\%\ \text{Recovery} = \frac{\textit{mass of metal in the precipitate}}{\textit{mass of the metal dissolved in leached liquor}} \times 100 \qquad 1$$

2.1.2. Recovery and Estimation of Iron in E-wastes

The solution was heated from 25-80°C and neutralized with aqueous solution of 5-10 % sodium hydroxide (NaOH) while stirring vigorously until pH 5.8 at 25°C. Stirring was allowed for 3 hours before adding 2 g of 5 % sodium thiosulphate ($Na_2S_2O_3$). The resulting precipitate (rust-brown) was filtered; dried and weighed as iron oxide. The percentage recovery was calculated as shown in equation 1.

2.1.3 Recovery and Estimation of Nickel in E-wastes

Three hundred milliliters (300 mL) of the leached solution was filtered into 500 mL beaker and pH adjusted to 11.0 at a temperature of 25°C while stirring at 150 revolution per minute (rpm) for 3 minutes with a magnetic stirrer. Coagulant (0.4 mL for hydroxide precipitation and 0.6 ml for sodium sulphide) was added. The coagulant used in the experiment was ferric chloride ($FeCl_3$). The optimum pH and coagulant dosage for precipitation are required to enhance the coagulation process and to avoid overdose of the chemicals. Stirring was reduced to 60 rpm for 15 minutes to promote larger floc formation. The mixer was turned off between 30 to 45 minutes to allow the flocs to settle down. Then, the precipitant (either hydroxide or sulphide) was added to the sample and the mixing process was continued at similar speed for 5 minutes.

However, directly mixing the Na_2S into the sample was not done as it would produce harmful hydrogen sulphide gas. The sample would have turned black with a high turbidity value. This is associated with the acidic nature of the sample. To prevent this from happening was the reason neutralization with NaOH prior to adding the Na_2S in the sample was done. The precipitate was filtered using a Whatman no 1 filter paper and dried in an air oven at $100^{\circ}C$ for 1 hour. The resulting sky-blue powder was weighed as nickel oxide. Recovery was calculated as shown in equation 1.

2.1.4 Recovery and Estimation of Cadmium in E-wastes

A chemical substance, Na_2CO_3 (3.5g) was added at pH 11.5 to obtain dark cadmium oxide (CdO), which was analyzed. The percentage recovery was calculated as shown in equation 1.

2.1.5 Recovery and Estimation of Lead in E-wastes

The colorless filtrate obtained, believed to contain soluble lead (II) trioxonitrate (V), $Pb(NO_3)_2(aq)$, was placed in a clean combustion beaker and heated. The $Pb(NO_3)_2(aq)$ decomposed on heating to give an insoluble yellow lead (II) oxide $(PbO)_{(s)}$ with evolution of colorless gases. Coke (carbon) was added in-situ and further heated. Evolution of more colorless gas and dull grey solid mass was observed. The resulting solid was washed in deionized water. Tests for lead was carried out by shaking in dilute HNO_3, dilute H_2SO_4 and concentrated HCl. The brown sample was then dried in the oven and weighed as lead oxide. The percentage recovery was calculated as shown in equation 1.

2.1.6 Recovery and Estimation of Cobalt in E-wastes

The leached liquor was filtered into a 500 mL beaker and placed on a magnetic stirrer. The pH of the leached liquor was monitored using a pH paper. Sodium hydroxide (NaOH) solution was used to raise the pH to 7.5. At pH 7.5 and at $32^{\circ}C$, ten grams (10 g) oxalic acid powder was added to precipitate cobalt. The resultant purple precipitate was filtered and dried in the oven at $105^{\circ}C$ for 1 hour to obtain the cobalt oxide. The percentage recovery was calculated as shown in equation 1.

2.1.7 Recovery and Estimation of Copper in E-wastes

Leached liquor was filtered into another 500 mL beaker and 100 mL distilled water was added. This was followed by the addition of 30 mL of 4 M KOH at pH 5.5 and at 45°C. The solution which turned black was stirred for 10 minutes. Thereafter, 50 mL of hot water was added and it separated into two layers: the clear supernatant was decanted while 10 mL of concentrated H_2SO_4 was added to the dark sample and a sky blue colour developed.

Zinc powder (1.5 g) was added while stirring with a magnetic stirrer. More H_2SO_4 was added to remove grey or dark solids to brown colour. The sample was filtered, dried and weighed to obtain copper oxide. The percentage recovery was calculated as shown in equation 1.

2.1.8 Recovery and Estimation of Aluminium in E-wastes

Addition of H_2SO_4 to the leached liquor was done to neutralize KOH while monitoring pH with a pH meter and stirring at 150 rpm on a magnetic stirrer. At pH of 4.5 and at a temperature of 30°C stirring was stopped and mixture allowed settling for 4 hours for precipitation to take place. Addition of more H_2SO_4 evolved heat and dissolved the precipitate resulting in aluminium sulphate. The aluminium sulphate obtained was filtered, dried and weighed to obtain aluminium oxide (with a white colour). The percentage recovery was calculated as shown in equation 1.

2.1.9 Recovery and Estimation of Silver in E-wastes

In the recovery, hydroxide precipitation method was followed. In the hydroxide precipitation method, KOH was employed to precipitate silver oxide. At a pH of 7.0, precipitation was achieved, after 60 minutes, at room temperature (28°C) in a magnetic stirrer set at 200 rpm. After silver was precipitated as silver oxide, silver hydroxide was subjected to a reaction with a mixture of glucose and NaOH (Aktas *et al*., 2010). This process was by filtration and drying of residues to yield silver oxide (white). The percentage recovery was calculated as shown in equation 1.

2.1.10 Recovery and Estimation of Lithium in E-wastes

Lithium was precipitated with KOH at a pH of 6.5 at a temperature of 30°C to obtain dark-grey lithium oxide. The percentage recovery was calculated as shown in equation 1.

2.1.11 Recovery and Estimation of Tin in E-wastes

Tin was recovered by precipitation from the leached liquor. In a 500 mL beaker, solid NaOH was added to the leached liquor under vigorous stirring while pH was monitored with a pH paper. At pH 6.8 and at a temperature of 30°C, addition of solid NaOH was stopped and stirring continued for 3 hours. The neutralized liquor was allowed to stand in order to precipitate out. The precipitate (Off-white) was filtered, dried and weighed to obtain tin oxide. The results for percentage of metal oxide recovered from the leached liquor were calculated as shown in equation 1.

3.0 Results and Discussion

The percentage recovery obtained from the calculations of the extracted and recovered e-waste metal oxides from the e-wastes are presented in Table 1.

Table 1: Recovered E-waste Metal oxides (%)

S/N	Metal Oxide	Percentage (%) Recovered (Actual Yield)	Percentage (%) Yield
1	Zinc oxide (ZnO)	41.10	28.26
2	Iron (III) oxide (Fe_2O_3)	43.32	45.20
3	Nickel oxide (NiO)	14.58	10.51
4	Cadmium oxide (CdO)	28.80	13.19
5	Lead oxide (PbO)	40.22	14.00
6	Cobalt oxide (Co_3O_4)	48.00	34.55
7	Copper oxide (CuO)	20.80	11.83
8	Aluminium oxide (Al_2O_3)	48.72	12.47
9	Silver oxide (AgO)	4.75	2.16
10	Lithium oxide (Li_2O)	11.20	17.77
11	Tin oxide (SnO_2)	27.52	12.70

Table 2: Metal Oxides with their Precipitation pH and Temperature

S/N	Metal oxide	Precipitating hydroxide	pH	Temperature	Colour
1	ZnO	NaOH	6.8	95°C	Grey
2	Fe_2O_3	NaOH	5.8	25°C	Rust-brown
3	NiO	NaOH	11.0	25°C	Skyblue
4	CdO	Na_2CO_3	11.5	35°C	Dark
5	PbO	NaOH	10.5	30°C	Red or brown
6	Co_3O_4	NaOH/Oxalic acid	7.5	32°C	Purple
7	CuO	KOH	5.5	45°C	Brown
8	Al_2O_3	KOH	4.5	30°C	White
9	AgO	KOH	7.0	28°C	White
10	Li_2O	KOH	6.5	30°C	Dark grey
11	SnO_2	NaOH	6.8	30°C	Off-white

Plate 1: Recovered Zinc Oxide

Plate 2: Recovered Iron Oxide

Plate 3: Recovered Nickel Oxide

Plate 4: Recovered Cadmium Oxide

Plate 5: Recovered Lead Oxide

Plate 6: Recovered Cobalt Oxide

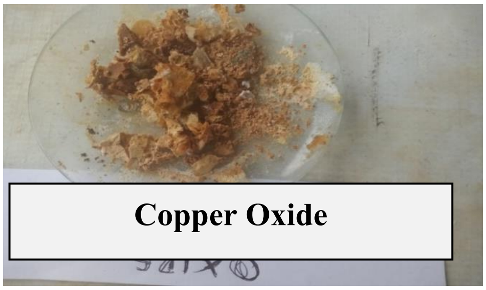

Plate 7: Recovered Copper Oxide

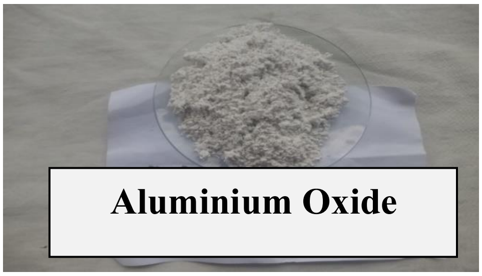

Plate 8: Recovered Aluminium

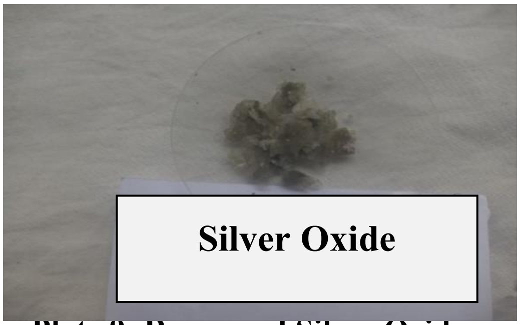

Plate 9: Recovered Silver Oxide

Plate 10: Recovered Lithium Oxide

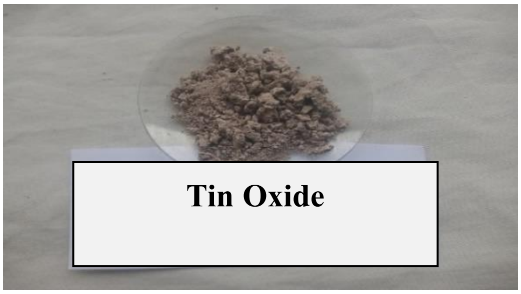

Plate 11: Recovered Tin Oxide

3.1 Levels of Metal Oxides in Recovered E-wastes

The percentages of e-waste metal oxides recovered (actual yield) as presented in Table 1 showed Al_2O_3 as the highest metal oxide recovered (48.72 %) and AgO as the least metal oxide recovered (4.75 %). Meanwhile, the percentage yield of the recovered metal oxides (Table 1) showed 45.20 % (Fe_2O_3) and 2.16 % (AgO).

Table 2 showed each dissolved metal oxide with a distinct pH and temperature value at which the optimum hydroxide precipitation occurred. The data showed that the colour of ZnO (Plate 1) was grey and precipitated at a pH of 6.8 and temperature of 95 °C. For the precipitation of Fe_2O_3, NaOH was used as the precipitating agent at pH and temperature of 5.8 and 25 °C respectively. The colour of iron oxide was rust-brown (Plate 2). The colour of NiO was skyblue (Plate 3) and precipitated at a pH of 11.0 and temperature of 25 °C. The precipitating agent used was NaOH. For the precipitation of CdO, Na_2CO_3 was used as the precipitating agent at the pH and temperature of 11.5 and 35 °C respectively. The colour of CdO was dark (Plate 4). The colour of PbO was red or brown (Plate 5) and precipitation occurred at a pH of 10.5 and temperature of 30 °C. The precipitating agent used was NaOH. The precipitating agent used for the precipitation of Co_3O_4 was NaOH/Oxalic acid at the pH and temperature of 7.5 and 32 °C respectively. The colour of Co_3O_4 was purple (Plate 6). For the precipitation of CuO, Al_2O_3, AgO and Li_2O, KOH was used as the precipitating agent at pH of 5.5, 4.5, 7.0, 6.5 and temperature of 45 °C, 30 °C, 28 °C and 30 °C respectively. The colour of CuO was brown (Plate 7), Al_2O_3 (white) (Plate 8), AgO (white) (Plate 9) and Li_2O (dark grey) (Plate 10) respectively. The colour ofSnO_2 was off-white (Plate 11) and precipitated at a pH of 6.8 and temperature of 30 °C. The precipitating agent used was NaOH.

The different colours of the metal oxides obtained with their precipitation pH and temperature as displayed in Table 2 matched with the pictures shown in Plates 1 to 11. The pictures showed that the samples are agglomerated and porous. This is an indication of their polycrystallite nature.

It should be recalled that the metal oxides recovered are as a result of the heating (drying) of the hydroxides initially formed to oxides. Drying of the hydroxides released water molecules to yield the oxides. In the process, protons are donated to the complex to form ions in solutions

4.0 Conclusion

An effective combination method of chemical leaching, precipitation, centrifugation, drying and extraction of dismantled and crushed e-waste has been applied to successfully recover metal oxides from e-waste components. Aluminium oxide (Al_2O_3) had the highest percentage recovery of 48.72 % while AgO had the least (4.75 %). Iron (II) oxide (Fe_2O_3) had the highest percentage yield of 45.20 % while AgO had the least (2.16 %).

The recovered metal oxides such as ZnO, Fe_2O_3, NiO, CdO, PbO, Co_3O_4, CuO, Al_2O_3, AgO, Li_2O and SnO_2 could serve as important secondary sources of critical raw materials for diverse industries. The recovery processes could be used to recover valuable resources and mitigate environmental pollution. Further studies should be carried out to recover metals present in e-waste components in their pure forms.

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