Evaluation of Metals in Printed Wiring Boards of Selected Discarded Mobile Phones in Nigeria

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Obsolete mobile phones form a major component of the e-waste stream. The Printed wiring boards (PWBs) of mobile phones are packed with economic and toxic metals. Regulatory bodies such as Restriction of Hazardous Substances (RoHS) directives regulate the amount of these toxic metals in electrical and electronic equipment. This study is aimed at evaluating selected critical metals present in PWBs of three popular brands of discarded mobile phones used in Nigeria to understudy the behaviour of original equipment manufacturers (OEMs) regarding toxicity, economic potentials, and level of compliance with international initiatives by regulatory bodies to reduce the level of toxic metals in electronic equipment. PWBs obtained from 60 discarded mobile phones of 3 popular mobile phone brands used in Nigeria were chopped into smaller particles, extracted according to EPA 3050B method, and analysed using the Inductive Coupled Plasma-Optical Emission Spectrometry technique. It was observed that Mn, Fe, Zn, and Cu, had the highest concentration range across all brands studied. Notable toxic metals such as Pb, Cd, Cr, and As have mean and standard deviation values of 0.43±0.38 mg/kg, 0.62±0.160 mg/kg, 5.16±1.06 mg/kg, and 84.97±13.83 mg/kg respectively. Economic metals: Cu, Ag, and Au had mean and standard deviation values of 80.37±16.89 mg/kg, 2.12±0.43 mg/kg, and 0.95±0.19 mg/kg respectively. Results from the study indicate that PWBs of mobile phones are a perfect secondary source of a large variety of metals vital for the recycling industry. Also, the low levels of toxic metals suggest that some OEMs are already adopting the 'design-for-environment' option. Therefore, PWBs studied seem eco-friendly; however, need to be handled with care as there are still some toxic metals of concern not determined in this study.

Keywords: Restriction of Hazardous Substances Directive, Environmental pollution, Recycling, Toxic metals, Eco-friendly products

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INTRODUCTION

A large percentage of municipal waste generated globally consists of e-waste. This percentage is growing at least three times faster than municipal solid waste (Genus Earth, 2022; Rimantho *et al*., 2022). The rapid growth in e-waste generated has been predicted to continue to rise since most electrical equipment is designed to have a short lifespan (Kasper *et al*., 2011; Omondi *et al*., 2022; Gómez et al., 2023), generating more obsolete products (Patel & Bina, 2016; Terena *et al*., 2017) and the craving for the advancement of communication and technology. Furthermore, the present design of electrical equipment which mixes metals (both toxic and non-toxic), plastic, and ceramic makes the separation of these materials for recycling extremely difficult and unfeasible (Osibanjo *et al*., 2016; Yken *et al*., 2021) thereby ending up in the waste stream. The 2020 Global e-waste monitor reported that 41.8 million tons of e-waste was generated by 2014. This amount of e-waste showed a growth rate of 20% in 2016 generating 44.7 million tons and a 28% rise by 2019 with a whopping sum of 53.6 million tons of e-waste which amounted to an average of 7.3 kg per capita generated globally (Forti *et al*., 2020, Ruiz, 2023). Statista (2023[a]) predicted that these values would increase to 57.4 million tons by 2021, 59.4 million tons by 2022, and 61.3 million tons by 2023. Despite the alarming amount of e-waste reported to be generated globally, only approximately 17% of this waste was reported to be recycled globally (Forti *et al*., 2020; Statista, 2023[a], Ruiz, 2023). Similarly, in 2019 Nigeria generated approximately 461,300 tons of e-waste with only an approximate amount of 0.4% recycled (Kasper *et al*., 2011; UN Environment Program (UNEP), 2019; Olu, 2023). Therefore, this shows that a greater percentage of the e-waste generated was handled in an environmentally unsustainable manner ending up in landfills.

Statista (2023[c]) stated that as at 2021, 1.43 billion mobile phone units were being sold to end users globally. This amount is on an increase due to the short lifespan of these mobile phones which makes them obsolescent (Gómez *et al.,* 2023). Nigeria, in 2012 was reported to be in the 10th position of countries with the highest mobile phone ownership globally of 90.5 million with Germany at the 9th position (107 million), the USA at the 3rd position (over 327.5 million), India at the 2nd position (over 873.6 million), and China (over 951,6 million) topping the rating (Daily Infographics, 2012). Guardian (2021) and Statista (2023[b]) reported that in 2023, Nigeria has an estimated number of 170 million mobile phone users (based on subscription). This population is expected to increase by 60% by 2025. Mobile phones are one of the smallest pieces of electrical equipment. Despite being small, it contains a large concentration of economic metals such as Au, Cu, Ag, Al, Pt, Sn, Co, and Ni. (Terena *et al*., 2017; Gómez *et al*., 2023), and according to Conocimiento (2022), most of these metals are irreplaceable when mined. An average user of a mobile phone switches phones twice a year mostly from older models to newer models thereby more of these metals are consumed on a regular basis during the process of production leading to the depletion of these metals and generating more waste. Bookhagen *et al.* (2020) reported that the PWBs of smart phones contain metal concentrations higher than current metal content in their respective ores. For instance, PWBs contain 98% of Cu, 99% of Pd, 93% of Ta 90% of Au, and 86% of In. Therefore, recycling these metals would help in reducing the depletion of the underground stock of these metals and a perfect secondary source of metals (Gabbatiss, 2019; Conocimiento, 2022).

As reported by Annamalai and Gurumurthy (2020), Kasper *et al*. (2011), and Nnorom and Osibanjo (2011), a whole mobile phone component consists of a keypad (4.05%), battery (5.304%), LCD (5.77%), metallic components (6.55%), connectors (19.5%), casing (25.89%), and PWBs (34.09%) by weight. Nnorom and Osibanjo (2011) also stated that a complete mobile phone PWB comprises 33% semiconductors, 24% capacitors, 23% bare PWB, 12% resistors, and 8% comprising of switches and other materials while the bare PWB consists of 30% ceramics, 30% plastics, and 40% metals.

The Printed Wiring Boards (PWBs) which control the functions of the phone house a large number of elements (Kasper *et al*., 2011; Omole *et al*., 2015; Terena *et al*., 2017). It constitutes about 20-30% of the weight of a mobile phone (Nnorom and Osibanjo, 2011). Studies by Nnorom and Osibanjo (2011), Hahladakis (2013), Maragkos *et al*. (2013), Ghodrat *et al*. (2018), Adie *et al*. (2019), Intrakamhaeng *et al*. (2019), Gorewoda *et al*. (2020), and Liang *et al*. (2023) showed that the metal content of PWBs varies depending on the type of mobile phone and year of manufacture. Some of the metallic components of a typical PWB of a mobile phone include economic metals (Au, Ag, Cu), toxic metals (Pb, As, Hg, Cd, Se, Cr), non-toxic metals (Fe, Pd, Zn, Ni, Ca, Mn), and non-metals such as Brominated flame retardants (Priya & Hait, 2018). Arsenic in PWBs is from Gallium Arsenide while the lead is from the lead solders used in holding the resistors, capacitors, and other components on the PWBs (Nnorom & Osibanjo, 2011; Dervišević *et al.,* 2013). In a related development, Priya and Hait (2018), Adie *et al*. (2019), Gorewoda *et al*. (2020) indicated that the toxicity potential of PWBs of mobile phone housed the largest quantity of toxic substances hence should be properly managed with caution. Improper handling can lead to pollution of the air, water, and soil (Kasper *et al*., 2011; Omole *et al*., 2015; Terena *et al.*, 2017; Liang et al., 2023). Likewise, mobile phones are packed with economic metals as reported by Annamalai & Gurumurthy (2020), a ton of waste mobile phone contains 340g of gold, 140g of palladium, 130kg of copper, and 3.2g of silver. Thus, a promising source of metals for the recycling industry.

There is a rising concern about the health and environmental impact associated with crude e-waste recycling in developing countries where there is either lack of specific e-waste regulations, regulations present in drafts, or not yet in force. Developed economies already have a system in place to curtail the menace associated with e-waste by developing some regulatory bodies like the European Union 2002/96/EC Waste Electrical and Electronic Equipment (EU WEEE) directive (EU WEEE, 2003), European Union 2002/95/EC Restriction on Hazardous Substances (RoHS) directives (RoHS Guide, 2023), United States Toxicity Threshold Limit Concentration (US TTLC) (Nnorom & Osibanjo, 2011; Adie *et al.*, 2019), European Union Waste Electrical Electronic Equipment (EU WEEE) directive 2012/19/EU (EU WEEE, 2018), and many other regulations. Monitoring this concentration of critical metals (toxic and economic metals) in electrical equipment in Nigeria to study the Original Equipment Manufacture's (OEM's) behaviour is very critical as it is perceived that most new products arriving in Nigeria are substandard with a very short life span (Adie *et al*., 2019).

The management of e-waste in Nigeria is still not appropriate and poorly managed as most of them ends up in the municipal waste stream, burnt at open dumps, surface water bodies, and background recycling leading to air pollution, discharge of toxic leachate into the environment contaminating groundwater and soil posing harm to humans, animals, and the environment (Priya & Hait, 2018; Annamalai & Gurumurthy, 2020; Liang *et al*., 2023).

Studies by Nnorom and Osibanjo (2011), Kasper *et al*. (2011), Dervišević *et al*. (2013), Priya and Hait (2018), Adie *et al*. (2019) established that the number of toxic metals such as lead exceeded RoHS Threshold limit. It is believed that the high lead content was due to the Tin-Lead solder used on the PWBs. Although recent studies done by Hahladakis (2013), Intrakamhaeng *et al*. (2019), and Gorewoda *et al*. (2020) on newer mobile phones (phones manufactured from 2006) have shown lower lead and other regulated toxic metal concentrations as related to the RoHS limits. Also, PWBs of mobile phones examined by Maragkos *et al*. (2013) manufactured between 2002-2006 had a Pb concentration of 27000 mg/kg while those manufactured between 2007-2011 had a much lesser lead concentration of 300 mg/kg indicating a decrease in the trend of lead concentration in PWBs in mobile phones. This low concentration of lead and other toxic metals might be due to the implementation of the RoHS 2006 deadline (Sargiou, 2021). Therefore, leaded solders are being replaced with lead-free solders made from alloys of non-toxic metals such as Sn, Ag, Cu, Zn, and Mn. (Ogunseitan, 2007; Nnorom & Osibanjo, 2011; Dervišević *et al*., 2013)

The main objective of this study is to evaluate the concentration of selected toxic and economic metals present in selected PWBs samples of three popular brands of discarded mobile phones manufactured after 2006 used in Nigeria. This is to understudy the behaviour of Original Equipment Manufacturers' (OEMs) regarding toxicity and economic potentials and comparing their concentration with 2006 RoHS directives.

MATERIALS AND METHODS

Sample Collection and Preparation

A total of 60 PWBs, 20 each from three (3) different models of discarded popular brands mobile phones were obtained from phone repairers' shops in Ibadan, Oyo state and Ikeja, Lagos state, both in Nigeria. The dates of manufacture of all the 60 discarded phones were captured to be from 2011 to 2017. Each discarded mobile phone was sorted according to its OEM and year of manufacture. To analyze only the PWBs, all the other components attached to the PWBs were detached using a fire gun and a screwdriver and subsequently chopped into smaller sizes of < 3 mm using stainless steel scissors according to Kulkarni (2016) and Ammamalai & Gurumurthy (2020). Then ground separately into smaller pieces using a hammer miller. To prevent cross-contamination the container and blades were cleaned with sawdust after every round as used by Adie and Onyebuenyi (2021). The smaller pieces were sorted using a 2 mm sieve as used by Nnorom and Osibanjo (2011). Each reduced PWB was properly labeled and stored in sealable plastic polyethylene bags.

Sample Analysis

To transfer elements (metals) into their liquid form, the EPA 3050B digestion protocol (U.S EPA, 1996) was used for the digestion process. EPA 3050B is used in determining the number of metals that could be leached from waste under extreme conditions. EPA 3050B method involves the treatment of waste samples with hydrogen peroxide, hydrochloric acid, and nitric acid (U.S EPA, 1996; Nnorom and Osibanjo, 2011).

One gram of the grounded samples was accurately weighed in a properly labeled digestion vessel and 10 mL of 1:1 HNO_3 was added. The solution was heated on a hot plate to 955ºC ±5ºC for 15 minutes without boiling. The sample was allowed to cool to less than 60ºC, 5 mL of Conc. HNO_3 was added, Covered, and refluxed for 30 minutes at 95ºC±5ºC without boiling. Another 5 mL of Conc. HNO_3 was added repeatedly every 30 minutes for 2 hrs until the brown fumes subsided. After cooling to less than 60ºC 2 mL of water and 3 mL of 30% H_2O_2 was added and heated continuously until effervescence was minimal. Later, 10 mL of Conc. HCl was added, heated, and fluxed at 95ºC±5ºC for 15 minutes without boiling. The digest was allowed to cool, filtered through Whatman No. 41 filter paper, and made to mark with distilled water using a 100 mL volumetric flask. An aliquot of each sample was taken and preserved with 5 drops of Conc. HNO_3. Each digest was analyzed for metals using Inductive Coupled Plasma-Optical Emission Spectroscopy (ICP-OES) using standard calibration techniques.

Quality Control

To ensure the reliability of the results, blank samples (this contained 100 mL of distilled water without adding the sample) were carried through the procedure and analyzed to check impurities in the reagents. Also, to test for precision and accuracy of the analytical process 20% of the samples were selected randomly and were passed through the analytical processes to produce replica values. These data was analyzed to determine the accuracy and precision of the analytical process.

To prevent contamination, all glassware and plastic apparatus/containers used were washed with detergent, rinsed with water, and then soaked in dilute HNO_3 overnight before rinsing with distilled water to remove any possible adsorbed metals to the walls of the containers and dried in a clean environment. Also, all reagents used for the digestion process were of Analytical grade. The analytical balance used was calibrated before use.

RESULTS AND DISCUSSION

Analysis of Metal Concentration in Examined Selected Mobile Phones PWBs

An analytical summary of the mean ± standard deviation and ranges of metal concentrations (mg/kg) in the various categories of mobile phone PWBs examined are presented in Table 1. The metal concentrations of Mn, Fe, Zn, As, and Cu had the highest values in the order Mn > Fe>Zn>As>Cu, while the concentration of Cr, Ag, Co, Au, Al, Cd, Ni, and Pb had relatively lower values. The results of the ICP-OES analysis of replica samples, and metal concentrations of the bulk samples to determine the degree of precision and accuracy of the analytical method were analysed using interclass correlation (F-test). Perfect reliability (Cronbach's Alpha=1.0) was gotten. The comparison test between the mean concentration of metals within each OEM using Tukey HSD and one-way ANOVA at a 95% confidence interval shows no significant difference ($p>0.05$) and normal distribution of 0.977 was determined which denoted an almost perfect distribution. This implies that these OEMs use a standard template in manufacturing their products which has been maintained over the years. The standard deviation of Mn among examined PWBs of OEM's was high. This high standard deviation values might be due to the disparity of colours among mobile phone brands and manufacturers as it is known that transition metals are inorganic pigments for materials. This property is due to the transfer of electrons from lower d-orbital to a higher energy d-orbital (Adie *et al.,* 2019).

Table 1: Average metal concentrations (mg/kg) in PWBs of all mobile phones

Metal	OEM 1 N=20		OEM 2 N=20		OEM 3 N=20	
(mg/kg)	Mean±SD	Range	Mean±SD	Range	Mean±SD	Range
Al	1.25±0.18	1.46-2.97	1.32±0.16	1.17-1.64	0.80±0.18	1.23-2.00
Fe	1357.1±256	1887.38-1794.45	1248.70±148.41	1105.68-1550.54	1254.56±201	1243.21-2057.21
Ni	0.49±0.07	0.62-1.26	0.52±0.06	0.46-0.64	0.31±0.07	0.43-0.75
Zn	691.6±82.90	640.89-1295.99	763.09±90.69	675.69-947.56	457.1±34.37	240.98-606.27
Mn	1486±213	532.43-1076.67	1577.30±187.46	1396.65-1958.58	951±217	685.43-1603.41
Co	2.35±0.34	0.18-0.37	2.49±0.30	2.21-3.10	1.50±0.34	0.07-0.17
Cu	89.30±12.80	87.51-170.48	94.73±11.26	83.88-117.63	57.1±13.0	75.20-150.53
Au	1.06±0.15	1.66-3.36	1.12±0.13	1.00-1.40	0.68±0.16	1.74-2.52
Ag	2.36±0.34	12.50-25.27	2.50±0.30	2.22-3.11	1.51±0.35	8.76-10.79
Pb	0.48±0.07	0.47-0.97	0.5±0.06	0.45-0.64	0.31±0.07	0.18-0.45
Cd	0.69±0.10	2.41-4.87	0.73±0.09	0.65-0.91	0.44±0.10	0.91-2.28
Cr	5.73±0.82	5.61-11.36	6.08±0.72	5.39-7.55	3.67±0.84	1.75-4.40
As	99.39±18.81	64.99-131.42	89.20±17.52	0.79-1.10	66.31±3.51	24.44-61.48

Number of total sample=60, Unit= (mg/kg)

Table 2: Summary of toxic and economic metal levels in PWBs

	Mean±StDev mg/kg	Median mg/kg	Range mg/kg	TTLC RoHsGuide.(2023)	RoHS
Cu	80.37±16.89	67.53	75.20-170.48	2500	NA
Au	0.95±0.19	1.83	1.00-3.36	NA	NA
Ag	2.12±0.43	10.65	2.22-25.27	500	NA
Pb	0.43±0.38	0.64	0.18-0.97	1000	1000
Cd	0.62±0.16	2.23	0.65-4.87	100	100
Cr	5.16±1.06	6.46	1.75-11.36	2500	1000
As	84.97±13.83	58.36	0.79-131.42	500	NA

NA= Not available, Number of total sample=60, TTLC= Toxicity Threshold Limit Concentration , RoHS= Restriction on Hazardous Substance

Comparison with RoHs Directive Maximum Limits and Total Threshold Limit Concentration (TTLC)

All the selected toxic metals i.e. As, Pb, Cd, and Cr incidentally had concentration values relatively below the permissive threshold value limits TTLC and RoHS Directive maximum limits as shown in Table 2. This low concentration of toxic metals is a strong indication that the OEMs studied might have considered the option of 'design-for-environment' by possibly reverting to the use of more environmentally friendly solders like Sn/Ag/Cu blend other than Pb/Sn solder which is popularly known to be used as a conductive glue that sticks components on the board (Cadence, 2022). The RoHS Directive mandated the use of lead-free solders with an implementation deadline of 2006 (RoHS Guide, 2018), and all examined PWBs were manufactured between 2011 and 2017. It is also obvious that Cu is still much in use in the circuitry works as demonstrated by the significant concentration in all the examined samples (75.20-170.48 mg/kg). The presence of metals such as Zn, Al, Fe, Ni, Mn, and Co in high concentration as shown in Table 1 might be an indication that Lead-free solders were used rather than Pb/Sn solders; though, Sn was not measured in this study. Most Lead-free solders are made from an alloy of Sn, Ag, and Cu while some contain a fourth metal such as Zn, or Mn (Ogunseitan, 2007). Posch (2020) also reported that elements such as Al, Ni, Mn, Co, Bi, and Ge are added to Lead-free solder alloys to improve their properties such as thermal reliability, vibration resistance, and electro migration resistance. It is very important to take note that Hg, a toxic metal regulated by RoHS Directive was not within the scope of this study. Therefore, examined selected PWBs of mobile phones should still be classified as hazardous substances and be handled with caution.

Comparison with Literature Data

The comparison data of some previous work done on metal levels of PWBs of end-of-life mobile phones are shown in Table 3. Adie *et al.* (2019) using the EPA 3050B leaching method reported a mean value concentration of Cd of 0.28 mg/kg falling within the range of the concentration of this study. Also, the concentration value of Au reported by Ernst *et al.* (2003); Huisman *et al.* (2007); Hageluken and Buchert (2008), and Kasper and Veit (2018) are similar to the mean concentration value of the present study. The mean concentration of selected toxic metals such as Pb, Cd, As, and Cr are lower than the values reported by Nnorom and Osibanjo (2011); Ghodrat *et al.* (2018); Sahan *et al.* (2019); and Annamalai and Gurumurthy (2020). However, recent studies on PWBs of mobile phones manufactured after 2006 done by Intrakamhaeng *et al.* (2019) and Gorewoda *et al.* (2020) as shown in Table 3, has a mean concentration of Pb comparable to the mean values of this study. Maragkos *et al.* (2013) reported mean concentrations of Pb (300 mg/kg), Cd (4.5 mg/kg), and As (110 mg/kg) for PWBs manufactured between 2007-2011; though higher than mean value concentrations gotten from this study but much lower than the RoHS limit. This relatively low concentration of toxic metals in the examined PWB gives a strong indication that the 2006 RoHS recommendation of toxic metals limits in electrical equipment may have been implemented since all examined PWBs of mobile phones were all manufactured after 2006.

Table 3: Comparison with Literature Data

References	Year of Manufacture	Leaching agent	METALS (mg/kg)						
			Pb	Cd	As	Cr	Cu	Au	Ag
Ernst *et al.*, 2003	-	Aqua Rega	-	-	-	-	-	0.37	3.57
Huisman *et al.*, 2007	-	-	-	-	-	-	-	1.3	5.7
Hageluken & Buchert, 2008	-		-	-	-	-	-	0.98	5.54
Nnorom & Osibanjo, 2011	EPA 3050B		20100	2.1			250000		227
Maragkos *et al.*, 2013	EPA 3052	2002-2006 2007-2011	27000 300	3.7 4.5	74 110	540 4400	12700 21900		
Ghodrat *et al.*, 2018	Before 2005	-	11700	-	-	-	395600	600	600
	After 2005		12600	-	-	-	383300	1000	600
Kasper & Veit, 2018	2001-2005	$S_2O_3^{2-}$	-	-	-	-	-	0.49	-
Sahan *et al.*, 2019	-	EPA 3051A	12000	-	-	17000	335000	1400	3600
Intrakamhaeng et al. (2019)	2006-2007	-	0.31	-	-	-	-	-	-
Adie *et al.*, 2019	2000-2015	EPA 3050B	2507	0.28	-	1002	-	-	-
Gorewoda *et al.*, 2020	After 2014		0.75	-	-	-	7645	0.41	3.19
Annamalai & Gurumurthy,2020	-	Aqua rega	118300	-	-	-	431900	3100	100
Present study	**2011-2017**	**EPA 3050B**	**0.18-0.97**	**0.65-4.87**	**0.79-131.42**	**1.75-11.36**	**75.20-170.48**	**1.00-3.36**	**2.22-25.27**

CONCLUSION

Quantities of end-of-life mobile phones are on the increase of which a large percentage ends up in landfills or dumpsites resulting in the loss of valuable resources and creating environmental pollution. This study provides insight into the metal contents (both toxic and economic) of PWBs' of three popular brands of mobile phones in Nigeria and their level of compliance with the RoHS 2003 directives. The concentrations of toxic metals regulated by RoHS Directives were below the permissible level indicating a possible implementation of RoHS Directive by some Original Equipment Manufacturers which had an implementation deadline of 2006. The metal concentration of the examined PWBs was also below the TTLC limits. Though, since Hg a toxic metal regulated by RoHS was not within the scope of this study, it is recommended that mobile phones should be handled with caution.

Results from the study indicate that PWBs of mobile phones are a perfect secondary source of a variety of economic metals such as Cu, Ag, and Au which are vital for the recycling industry thereby reducing the amount of waste that ends up in landfills and incinerators with a huge economic advantage.

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