Introduction

Management of e-waste (also referred to as end-of-life (EoL) electrical electronic equipment (EEE) and waste electrical electronic equipment (WEEE)), is one of the biggest environmental challenges of our times. E-waste is the fastest growing waste stream in the world, with current annual quantities in the order of 40 million tons.\(^1\) This critical fact raises unprecedented challenges, as on the one hand, e-waste contains hazardous substances such as copper (Cu), lead (Pb), tin, nickel (Ni), iron, aluminum, cadmium (Cd).\(^2\) Waste from printed wiring boards (PWB) alone contains up to 6% of Pb/tin solder, representing up to 3% of the gross weight of the original PWB. Cathode ray tubes from televisions and computer monitors contain up to 400 g/kg Pb.\(^3\) On the other hand, e-waste also contains locked up resources, such as precious and/or critical metals which have great economic value and is of rising strategic importance, especially in light of growing global resource scarcity.\(^4\)

Background. Unsound recycling of e-waste releases toxic metals into environmental media and has deleterious health consequences to humans as the metals transfer to humans through the food chain, direct contact and inhalation.

Objectives. This study assessed soil contamination with lead (Pb), copper (Cu), chromium (Cr), nickel (Ni) and cadmium (Cd) arising from crude e-waste recycling.

Methods. Forty-eight soil samples were collected from the vicinity of high-, medium- and low-activity recycling operations in Ogunpa in Ibadan, Nigeria as well as from the botanical garden of the University of Ibadan for background samples. Total extractable metals were leached with aqua regia and the leachates were analyzed using flame atomic absorption spectrometry. Speciation analysis was also conducted on soil samples that showed high concentrations of metals to determine the distributions in various phases.

Results. All soil samples were determined to be sandy loam in composition with pH and organic matter ranging from 7.1–7.9 and 1.56–1.81%, respectively. Metal concentrations (mg/kg) for soils from the study area ranged as follows: Pb, 269 – 5650; Cu, 203 – 3483; Cr, 3.30 – 42.4; Ni, 0.14 – 24.0; and Cd, below detection limit – 2.50. The results indicated enrichment in soil by all metals, especially Pb and Cu, which were many times higher compared with background concentrations. Additionally, average Pb and Cu concentrations were higher than regulatory limits for soil set by selected countries across the globe. Speciation studies indicated that about 65% and 88% of Pb and Cu, respectively, were liable to potential mobility with slight changes in natural conditions. Other metal concentrations, although with higher concentrations compared with background levels, were within the permissible limits in soils accepted by many countries across the globe. There were significant correlations between all metals, suggesting that they may have been released from a common source.

Conclusions. Soils from the study area require urgent clean-up, especially for Pb and Cu, to safeguard human health and the environment.

Competing Interests. The authors declare no competing financial interests.

Keywords. E-waste, recycling, soil, toxic metals, speciation

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monthly tons of e-waste and EEE near end-of-life from abroad.\textsuperscript{8, 9} Most of the e-waste in Nigeria is normally dumped with the municipal solid waste stream and subsequently scavenged by the informal sector for recycling using environmentally unsound methods like unprotected acid leaching of PWBs, unprotected manual dismantling and burning to recover valuable components and precious metals. Pollution of soil, water and air by toxic metals, flame retardant, dioxins and furans resulting from these crude activities are common. These pose great risks to both human health and the environment. Humans, especially children and women, have been reported to be exposed to toxic metals and organic pollutants arising from crude e-waste management through dermal contact, inhalation of burning smoke/dust and dietary intake through contaminated water and food.\textsuperscript{10} Many deleterious health consequences have been associated with either humans living or working near crude e-waste recycling areas. An increase in spontaneous abortions, stillbirths, premature births, reduced birth weights and infant lengths, changes in thyroid function, cellular dysfunction, adverse neonatal outcomes and DNA damage in human peripheral blood lymphocytes as a result of exposure to e-waste pollutants have been documented.\textsuperscript{10,11,12} There is a paucity of data in the literature on soil pollution around the vicinities of crude e-waste recycling activities in Ibadan, Nigeria. This study was therefore carried out to:

(i) determine the concentrations of environmentally available Pb, Cu, Cr, Ni and Cd in soils collected from high-, medium- and low-activity crude recycling activities in the Ogunpa area of Ibadan, Nigeria;

(ii) conduct speciation studies on selected soil samples that show high concentrations of metals to determine concentrations in the various phases; and

(iii) compare the concentrations of metals determined with background concentrations and regulatory limits to show the extent of pollution.

It is hoped that the data obtained from this study may provide/add to the database of metal pollution statistics from e-waste crude recycling sites in Nigeria and illustrate the danger of adverse effects to humans interacting with this site if proper cleanup/checks are not put in place.

### Abbreviations

| Symbol | Description               |
|--------|---------------------------|
| Cd     | Cadmium                   |
| Cr     | Chromium                  |
| Cu     | Copper                    |
| EEE    | Electrical electronic equipment |
| Fe-Mn  | Iron-manganese           |
| Pb     | Lead                      |
| Ni     | Nickel                    |
| PWB    | Printed wiring board      |

### Methods

#### Description of Study Area

Ogunpa Market is one of the biggest markets within Ibadan Metropolis where used EEE and automobile spare parts are sold. The site is normally littered with informal e-waste recyclers. Activities such as manual dismantling of e-waste components without protection using simple tools like pliers, hammer, screw driver and open burning of plastics and PWBs to recover valuable metals like Cu characterize the site (Figure 1).
Sampling Design and Sample Collection

The site under consideration was divided into three sections: Section A - high activity (> 10 recyclers), Section B - medium activity (10 – 5 recyclers) and Section C - low activity (< 5 recyclers). Soils were collected from 3 points each for high, medium and low activity areas. At each of the points, soil samples were obtained with a stainless steel auger and a hand trowel at 0 – 15 cm, 15 – 30 cm, 30 – 45 cm and 45 – 60 cm depths. Soil samples from a background location were collected in the same manner from the botanical garden of the University of Ibadan which is located within the same geographical region as the sampling site, but is influenced by little or no anthropogenic activities. A total of 48 soil samples were gathered from each of the sections (A, B, C) and background. Samples were packed in clean polythene bags and taken to the laboratory for further preparation and analyses.

Sample Preparation and Analyses

All soil samples were air dried, pulverized and sieved through a 2 mm mesh size sieve and all soil properties except for particle size analysis were performed on < 2 mm soil samples. Soil pH was performed with a Jenway glass electrode, organic matter was determined using the Walkley-Black method, and soil particle size analysis was determined by Bouyoucos hydrometer method. Metals were leached from soil samples by modifying the method by Nieuwenhuize et al. as follows: 2 g of soil were placed in a previously acid washed 100 mL Pyrex beaker, then 12 mL of HCl + 4 mL of HNO3 were added to the beaker in the hood and immediately covered with a watch glass. This mixture gave rise to aqua regia, a strong oxidizing solution that produces nascent chlorine that converts all organic components (carbon and hydrogen) to carbon dioxide and water. This was placed in a water bath (95 – 100°C) and refluxed for 2 hours with intermittent shaking every 20 minutes. The leachate was filtered using Whatman No.1 filter paper and made up in an appropriate standard flask. The leachate was stored in an acid washed plastic bottle and kept in the fridge at 4°C prior to analysis. This was repeated for all other soil samples. Following the sequential extraction procedure of Tessier et al., Pb and Cu were partitioned into five fractions representing the exchangeable fraction, bound to organic matter fraction, bound to iron-manganese (Fe–Mn) oxides fraction, bound to organic matter fraction and the residual fraction in 10 soil samples. The procedure was carried out with an initial weight of 1 g. Deionized water was used to prepare all reagents for each step. Detailed extraction procedures for each step can be found in a report by Tessier et al.

Results

Soil Properties

The summary of soil properties from the study and background areas is presented in Figure 2. The pH and organic matter for all soils ranged from 7.1 – 7.9 and 1.14 – 2.10%, respectively. The sand and fine
particles (silt and clay) in all soil samples ranged from 47.2 – 81.2% and 22.8 – 50.8%, respectively. The nature of the soils from both the study and background areas was calculated to be sandy-loam using the US Department of Agriculture Soil Texture Calculator. There is usually high leachability of metals from the topsoil level to the lower horizons in this type of soil. The soil properties of the study area compared with the background area indicate comparable properties. This demonstrates that the two sites are from the same geographical zone. The small standard deviations for all soil properties suggest that the samples were prepared in a uniform manner and were also well homogenized before analyses.

**Metal Concentrations in Soil**

The summary of metal concentrations in all soils is presented in Table 1. The average metal concentrations generally followed the sequence Pb > Cu >> Cr > Ni >> Cd.

All metals decreased in concentrations from the topsoil level (0-15 cm) towards the lower horizon soils (Table 1). Steady decreases of all studied metals were observed in soil samples collected from the medium activity zone.
Metal Speciation Studies

The summary of metal speciation of Pb and Cu on ten (10) selected soil samples is presented in Table 2.

The order of % Pb abundance in the phases was residual > bound to organic matter > exchangeable > bound to Fe-Mn oxides > bound to carbonates, while the order of % Cu abundance was bound to Fe-Mn oxides > bound to carbonates > bound to organic matter > residual > exchangeable.

Correlation Studies

Table 3 presents Spearman correlations at a 95% significant level between all studied soil properties. All metals showed a significant correlation of varying degrees ranging from 0.685-0.920, an indication that they came from a common source. In addition, pH showed an inverse significant correlation with all metals. This indicates that the metal concentrations decreased as pH increased within the very narrow pH range of 7.1-7.9 in the studied soils.

Discussion

It has been reported that many metals within the pH range of 6.0-9.0 are not easily available, but exist either as complexes or precipitates. For instance, the predominant insoluble Pb compounds at pH > 6 are lead sulfides, lead phosphates, lead carbonates and lead (hydr) oxides. The low organic matter content exhibited by all soils in the present study could encourage high leachability of metals to lower horizons as retention of metals at the topsoil may be reduced due to fewer adsorption sites on humic materials in organic matter.

The average metal concentrations in soil samples generally followed the sequence Pb > Cu >> Cr > Ni >> Cd. The high enrichment by several folds for all metals in the soil samples from the study area compared with background soil samples could certainly be associated with releases during the crushing and burning of printed wiring boards as well as crushing and/or dumping of cathode ray tubes on the soils. E-waste has been reported to contain more than 1000 different substances performing various functions.

Some of these substances, such as Pb, Cd, Cr, Cu, Ni, polybrominated biphenyls (PBBs), polybrominated diphenyl ethers (PBDEs) and mercury...
Research

Soil Pollution by Toxic Metals near E-waste Recycling Operations in Ibadan, Nigeria

| pH | OM  | Sand | Fines | Pb   | Cu   | Cr   | Ni   | Cd   |
|----|-----|------|-------|------|------|------|------|------|
| 1  | 0.555\(^{\delta}\) | -0.215 | -0.034 | -0.548\(^{\delta}\) | -0.557\(^{\delta}\) | -0.601\(^{\delta}\) | -0.687\(^{\delta}\) | -0.778\(^{\delta}\) |
| OM | 1   | -0.272 | -0.250 | -0.252 | -0.420\(^{\delta}\) | -0.217 | -0.476\(^{\delta}\) | -0.588\(^{\delta}\) |
| Sand | 1 | -0.637\(^{\delta}\) | -0.120 | -0.141 | -0.071 | -0.127 | -0.060 | 0.194 | 0.185 |
| Fines | 1 | -0.099 | 0.071 | -0.127 | -0.687\(^{\delta}\) | -0.778\(^{\delta}\) | 0.799\(^{\delta}\) |
| Pb | 1 | 0.790\(^{\delta}\) | 1 | 0.734\(^{\delta}\) | 0.685\(^{\delta}\) | 0.710\(^{\delta}\) |
| Cu | 1 | 0.734\(^{\delta}\) | 0.685\(^{\delta}\) | 0.710\(^{\delta}\) |
| Cr | 1 | 0.685\(^{\delta}\) | 0.799\(^{\delta}\) | 0.685\(^{\delta}\) |
| Ni | 1 | 0.710\(^{\delta}\) | 0.799\(^{\delta}\) | 0.685\(^{\delta}\) |

Table 3 — Spearman Correlation Studies on Soil Properties from Study Area

1 significant at p < 0.05
Abbreviations: OM, organic matter

are present in varying amounts and pose varying degrees of toxicity to human health and the environment when the waste is improperly managed. The cathode ray tubes from television and computer monitors contain high amounts of Pb and Cu cables/coils. The PWBs of all e-waste also contain high content of tin and Pb used to produce solder for joining components on the board as well as Cu, which is used for circuitry works. Other investigated metals (Cr, Cd and Ni) in this study have also been reported to be contained in various e-waste components.

The concentrations of the toxic metals investigated in this study are in agreement with similar studies reported in the literature. Lead and copper concentrations in soils from the study area were many times higher than their regulatory limits in soils across many countries, but Cr, Ni and Cd were within the acceptable limits. This could mean that the content of the latter metals in EEE are too small to have an appreciable content in soil when soils are exposed to these metals.

Humans are exposed to pollutants arising from crude e-waste management through dermal contact, inhalation of burning smoke/dust and dietary intake through contaminated water and food. Children and adults (especially lactating mothers) working or living near crude e-waste recycling sites are at risk of health problems related to e-waste. Children are more vulnerable to the toxic effects of these contaminants due to their high gastrointestinal uptake of heavy metals.

Changes in thyroid function, cellular expression and function, adverse neonatal outcomes, temperament and behavior, and decreased lung function have also been linked with exposure to pollutants arising from improper management of e-waste. DNA damage in human peripheral blood lymphocytes due to exposure to e-waste pollutants in Nigeria and China has also been reported. This study presents useful data that could serve as an indicator of the adverse effects of exposure to heavy metals from the study area.

Steady decreases of all studied metals were observed in soil samples collected from the medium activity zone. The decrease in metal concentrations seemed to be influenced by the percentage fines for the medium activity zone as this could be an indication of the retention of metals within fine particles due to multifunctional group sites where metals may undergo ion exchange, adsorption or complexation. Major factors that could also influence the mobility of metals in soils include soil pH, which influences higher metal mobility in acidic solution as many of them are usually in the ionic form and as a result can easily move. Another factor is soil organic matter. Soil organic matter, just like fine particles (silt + clay), are usually rich in humic materials with functional groups with the ability to complex metals, thereby retaining them in soil. Another factor is the nature of soil. Soil can be sandy, clayey, etc. A more sandy soil supports higher metal mobility than clayey soil.

The retention of metal ions at depth witnessed in this study in all activity zones within the topsoil (0 – 15 cm) could be caused by various factors described above. First, the soil pH for all soils was within the neutral to alkaline range. As previously reported, within this pH range, many...
metals are not easily available because they exist either as complexes or precipitates. This could have resulted in a higher retention of metal ions in the topsoil. Secondly, considering the speciation studies of Pb and Cu presented in Table 2, metal ions were present more in the organic matter and residual phases. This suggests that more metals were retained in the humic materials in soil organic matter (Pb – 25%, Cu – 21%) and silicon dioxide (SiO2) lattice of the residual phase (Pb – 35%, Cu – 12%), making them mostly unavailable. Lead and Cu were not exchangeable to any significant degree, affirming their high unavailability.

The relatively high sand content (47.2 – 81.2 %) of the studied soils and high metal content in the residual phase suggests that an appreciable percentage of metals could be associated with parent materials that form the soils. However, it is important to note in Table 2 that the average potential mobility of Pb was 64.8% and 87.9% for Cu, implying that any slight change in the natural conditions could release these metals into the environment.

About 65% of Pb and 88% of Cu distributions have the potential to be mobile with slight changes in natural conditions. The forms in which metals exist in soil matrix is greatly influenced by the soil pH, ionic strength, electron activity, solid and solution component, soil texture, amount of organic matter, etc. It has been reported that Cu and Pb concentrations in solution increase at a pH range of 6 and 7, because soluble metal-organic complexes are formed in this pH range, but in soil samples with low organic matter content (< 2.8 %), the concentrations of Cu and Pb in solution may show only slight increases, especially at neutral to alkaline soil pH. In fact, at a pH of 7, almost 99% of dissolved Cu is complexed by soluble organic matter. This may explain why Cu is very low in the exchangeable phase in the present study because of the low organic matter content. In the pH range of this study (7.1 – 7.9), Pb and Cu exhibited appreciable levels of 25.4% for Pb and 17.6% for Cu, respectively, in the organic phase. Copper showed the highest average percentage (38.7%) in the oxides phase, because Fe-Mn oxides, which are abundant in many soils, exist as nodules and cement between particles. These oxides retain trace metals in neutral conditions and can be mobilized under reducing or acidic conditions. The high percentage of Pb in the residual phase has also been reported in the literature. This could be because Pb is ubiquitous in the environment and probably undergoes ion exchange on layer silicate materials like clays, and therefore may be a part of the parent material of most soils.

**Conclusion**

The physico-chemical properties of soils collected near crude e-waste recycling activities in the Ogungpa area of Ibadan, Nigeria were determined. The present study revealed gross soil pollution by all metals investigated, especially Pb and Cu, by several orders of enrichment compared with background concentrations. The concentrations of Pb and Cu were also higher than the permissible limits set by many countries around the world. Other metals such as Cr, Ni and Cd were within the range of permissible limits set by selected countries. Metal speciation studies on some soil samples revealed that 65% and 88% of Pb and Cu, respectively, had the potential to be mobile with slight changes in natural conditions. Regulations guiding e-waste recycling activities in Nigeria are urgently needed, along with cleanup of soils around Ogungpa which are polluted by Pb and Cu.

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