Preliminary assessment of soil contamination by trace metals in peri-urban municipal landfills in Ibadan, Nigeria [version 1; referees: 2 approved with reservations]

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Abstract
Background: Soil contamination by trace metals as a result of improper waste management and disposal in Ibadan, Nigeria has been evaluated in this study. Several studies have shown the link between trace metal soil contamination and improper solid waste disposal.
Methods: Soil samples were taken from two major landfills in Ibadan, in the south-west of Nigeria, and subjected to laboratory analysis using inductively coupled plasma-optical emission spectrometry (ICP-OES) as part of a wider study to evaluate the waste management practices in the city.
Results: The results of this investigation, without taking into account the background values of the trace metals at both landfill sites, showed that the quantified levels of lead at both sites exceeded threshold levels. The quantified values of zinc and copper metals exceeded the threshold levels specified in the Finnish government decree on the assessment of soil contamination and remediation needs, at 1098 mg/kg and 233.20 mg/kg in the Aba-Eku landfill site, and 1205 mg/kg and 476.55 mg/kg in the Lapite landfill site, respectively. This calls for a comprehensive risk assessment.
Conclusions: It is hoped that the results of this study will serve as a basis for a wider risk assessment of all landfill sites within the city.

Corresponding author: Benjamin Oyegbile (hollander196@yahoo.com)
Competing interests: No competing interests were disclosed.
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Introduction

Landfills offer an inexpensive disposal option for different streams of solid waste and are widely used around the world\(^1\). They are especially popular in developing and low-income countries, where waste management resources or the expertise necessary to operate other capital-intensive and more sophisticated waste disposal options, such as incinerators or waste-to-energy facilities, are limited. However, it is well-known that there exists a significant risk of environmental contamination from untreated landfill leachate which may infiltrate into the surrounding soil and groundwater\(^2-4\). Therefore, the design, construction, operation and de-commissioning of a modern landfill facility must ensure that the leachate from the landfill is collected and properly treated in order to prevent release of toxic substances into the environment.

Solid waste as a type of waste that emanates from human activities is a potential source of environmental pollutants. Hence, the release of contaminants upon exposure and contact with the environment should be anticipated. Leaching is the major route through which soluble contaminants are released into the environment upon contact with water. This results in potential risk to human and environmental health\(^5-11\).

The environmental, ecological, and human health impacts of elevated levels of trace metals from soil samples and untreated leachate from open landfills have been well-documented in numerous scientific publications. The health risks associated with the presence of trace metals in environmental samples are mainly attributed to their carcinogenic nature when in contact with living tissues via different exposure routes such as dermal contact, inhalation or consumption of contaminated fruits and vegetables\(^12-17\). Therefore, soil has been identified as one of the routes through which trace metals are released into the environment\(^18,19\).

The disposal of solid waste in open landfills not only increases the risk of soil and groundwater contamination but also increases the risk of fire outbreak as there is no mechanism for the control and collection of landfill gaseous emissions. Open and uncontrolled landfilling of waste therefore constitutes a real threat to public and environmental health. However, open landfills will continue to serve as the preferred disposal option in some parts of the developing world, and to some extent in other emerging economies, at least into the foreseeable future. Consequently, public health and safety must be given top priority in the operation of these landfills.

In order to minimize the environmental burden arising from the operation of these landfills, it is very important to carry out environmental risk assessments on a regular basis. The assessment will serve as an environmental monitoring tool and help to establish if there is any need for a comprehensive risk assessment or remediation strategy, especially when the quantified values of trace metals exceed regulatory values. In addition, previous research has shown that the levels of trace metals in any soil sample is a combination of natural and anthropogenic factors\(^20-22\). Anthropogenic sources of trace metals in soils are mainly from hazardous solid wastes, combustion processes in industries and transportation, mining activities, as well as long-term and extensive use of pesticides on agricultural land\(^23\). The concentration of trace metals from anthropogenic inputs may exceed those from natural sources\(^25\).

The aim of this study is to determine the levels of trace metals in soil samples taken from open landfill sites using inductively coupled plasma-optical emission spectrometry (ICP-OES). While there are many studies on waste management practices within urban centres in Ibadan\(^25,26\), there are very few studies on the risk assessment of the city’s waste management system with respect to trace metals and other soil contaminants. The present study aims to fill this gap in knowledge. The results presented here are only a preliminary assessment, and they are site-specific. They do not reflect the general quality of soils around all the sampling sites as this work does not take into account the background values of the trace metals at the sampling locations and the effect of contaminant migration. However, it is hoped that this study will serve as a guide for a more comprehensive risk-assessment of all landfill sites in Ibadan for heavy metal contamination.

Spectrometry for metal analysis

In carrying out a laboratory analysis of environmental samples for the presence of trace metals, there are two commonly used methods, namely: atomic absorption spectrometry (AA), and the inductively coupled plasma optical emission spectrometry (ICP-OES), both of which are based on emission spectroscopy\(^27,28\). ICP-OES is a special type of atomic emission technique which employs inductively coupled plasma to generate excited atoms that emit electromagnetic radiation when heated to a very high temperature, with wavelengths that correspond to a particular element. The sample atoms are subjected to a temperature of around 6727 °C\(^29\). ICP-OES is based on the principle of atomic light emission properties of samples; when they are excited at high enough temperature, the concentration of the sample is directly proportional to the intensity of the emitted light.

To carry out spectroscopic analysis for the detection of trace metals in environmental samples, acid digestion is commonly performed on the collected sample. For example, in soil and sediment analysis, and in the determination of total phosphorus, and total nitrogen under heated conditions\(^30\). The digestion procedure breaks down an organically bound substance and converts it to the analysable form by using oxidising agents such as sulphuric acid (H\(_2\)SO\(_4\)), nitric acid (HNO\(_3\)), perchloric acid (HClO\(_4\)), or hydrochloric acid (HCl), or using oxidising mixtures such as aqua regia. Sometimes, addition of bromide (Br\(_2\)) or hydrogen peroxide (H\(_2\)O\(_2\)) to mineral acids will increase their solvent action and speed up the release of bound organic materials in the sample.

Materials and methods

Sample collection

A detailed description of the study area as well as the geographical location of the sampling sites is available elsewhere\(^31,32\). Soil sampling, pre-treatment and preservation was carried out according to\(^33\). Several grab samples of the top soil were collected using hand-held augers and tube samplers from a depth of about 15 cm at two sub-urban landfill sites, namely: Aba-Eku and Lapite (Table 1, Figure 1), located on the outskirts of the Ibadan metropolis. The samples were immediately transferred into 50 ml airtight plastic containers (Burkle GmbH, Rheinauen, Germany) with screwed cap and labelled appropriately as samples A and B depending on the landfill sites. The soil was in dry condition at the time of sampling, with an ambient temperature of about 29 °C.
Table 1. Location and capacities of peri-urban landfills (Soil samples were collected from Aba-Eku and Lapite landfills).

<table>
<thead>
<tr>
<th>Landfills</th>
<th>Capacity (Ha)</th>
<th>Coordinates</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aba-Eku, Olunloyo Road, Akanran, Ona Ara LG</td>
<td>9.419</td>
<td>7.2631, 4.0262</td>
</tr>
<tr>
<td>Lapite, Oyo Road, Moniya, Akinyele LG</td>
<td>20.669</td>
<td>7.5212, 3.9112</td>
</tr>
<tr>
<td>Awotan, Apete-Akufo Road, Ido LG</td>
<td>20.259</td>
<td>7.4876, 3.8298</td>
</tr>
<tr>
<td>Ajakanga, Old Ijebu Road, Oluyole LG</td>
<td>10.034</td>
<td>7.2839, 3.8333</td>
</tr>
</tbody>
</table>

Figure 1. Map of the study area, showing the locations of the landfill sites.

The sample containers were stored in the laboratory at room temperature without any further pre-treatment, before carrying out the ICP-OES analysis.

Preparation of test solutions

Test solutions were prepared according to the standard method\(^a\). A weighed portion of the soil sample (1.0 g) was placed in three separate glass beakers that had been thoroughly rinsed with dilute nitric acid and thereafter deionized water. 20 ml of nitric acid (7 mol/l) was added to each beaker and thereafter transferred into an autoclave for acid digestion. The beakers were kept in the autoclave at 121°C for about 30 minutes. The content of the beakers was carefully filtered into three 100 ml volumetric flasks after cooling to room temperature.

Preparation of standard solution

The mixed standard solution for the calibration step was prepared following Rezic & Stefan’s method\(^b\). Six 100 ml volumetric
flasks were carefully rinsed with dilute nitric acid followed by deionized water. A calculated volume of the prepared test solution with 0.1 % nitric acid solution (Table 2) was added to each flask using a pipette, and filled to the 100 ml mark with distilled water. The test solution and the calibration solutions were subsequently transferred for ICP-OES analysis.

**Heavy metal analysis**

The samples of the test solutions were analysed for trace metals according to 29. A test sample (100 ml) was placed in the measurement chamber of the iCAP™ 7000 spectrometer (ThermoFisher Scientific GmbH, Germany) after allowing sufficient time for initial start-up of the machine. The relevant test parameter values were given via a PC connected to the machine. The entire process is fully automated and the measured quantities of trace metals were generated as printed output. A total of two different measurements were performed on the samples from each landfill site and the result was given as the average of all measurements (Figure 2).

**Results and discussion**

The ICP-OES spectrophotometer generated data on the concentration of trace metals in the analysed soil samples. The reported concentration is the concentration in the liquid digest samples in milligrams per litre (mg/l). This data was thereafter converted to obtain the concentration in milligrams per kilogram dry weight of the dry soil samples (See Equation 1 and Table 4, Table 5).

\[
C_d \text{ (mgkg}^{-1}\text{)} = C_w \text{ (g}^{-1}\text{)} \times \frac{\nu}{w}
\]

The equation above is used to calculate the concentration of trace metals in milligrams per kilogram dry weight of the soil samples, using the reported soil concentration from the liquid digest samples. \(C_d\) is the heavy metal concentration of the dry soil sample,

**Table 3. Standard regulatory values for heavy metal contamination in soil.**

<table>
<thead>
<tr>
<th>Metals</th>
<th>Natural conc. (mgkg(^{-1}))</th>
<th>Threshold value (mgkg(^{-1}))</th>
<th>Lower guideline (mgkg(^{-1}))</th>
<th>Higher guideline (mgkg(^{-1}))</th>
</tr>
</thead>
<tbody>
<tr>
<td>Arsenic</td>
<td>0.1–25</td>
<td>5</td>
<td>50</td>
<td>100</td>
</tr>
<tr>
<td>Chromium</td>
<td>6–170</td>
<td>100</td>
<td>200</td>
<td>300</td>
</tr>
<tr>
<td>Copper</td>
<td>5–110</td>
<td>100</td>
<td>150</td>
<td>200</td>
</tr>
<tr>
<td>Nickel</td>
<td>3–100</td>
<td>50</td>
<td>100</td>
<td>150</td>
</tr>
<tr>
<td>Lead</td>
<td>1–5</td>
<td>60</td>
<td>200</td>
<td>750</td>
</tr>
<tr>
<td>Zinc</td>
<td>8–110</td>
<td>200</td>
<td>250</td>
<td>400</td>
</tr>
<tr>
<td>Cadmium</td>
<td>0.01–0.15</td>
<td>1</td>
<td>10</td>
<td>20</td>
</tr>
</tbody>
</table>

**Table 2. Preparation of the mixed standard (calibration) solutions in 1 % nitric acid solution.**

<table>
<thead>
<tr>
<th>Metals</th>
<th>S(_{0}) (µl)</th>
<th>S(_{0.25}) (µl)</th>
<th>S(_{0.5}) (µl)</th>
<th>S(_{1.0}) (µl)</th>
<th>S(_{2.5}) (µl)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lead</td>
<td>-</td>
<td>25</td>
<td>50</td>
<td>100</td>
<td>500</td>
</tr>
<tr>
<td>Zinc</td>
<td>-</td>
<td>25</td>
<td>50</td>
<td>100</td>
<td>500</td>
</tr>
<tr>
<td>Chromium</td>
<td>-</td>
<td>25</td>
<td>50</td>
<td>100</td>
<td>500</td>
</tr>
<tr>
<td>Copper</td>
<td>-</td>
<td>25</td>
<td>50</td>
<td>100</td>
<td>500</td>
</tr>
<tr>
<td>Iron</td>
<td>10</td>
<td>200</td>
<td>500</td>
<td>1000</td>
<td>3000</td>
</tr>
<tr>
<td>Nitric acid</td>
<td>10</td>
<td>10</td>
<td>10</td>
<td>10</td>
<td>10</td>
</tr>
</tbody>
</table>

**Table 4. Quantified levels of trace metals in soil samples from Aba-Eku landfill.**

<table>
<thead>
<tr>
<th>Metals</th>
<th>Sample A1 (g/l)</th>
<th>Sample A2 (g/l)</th>
<th>Sample A3 (g/l)</th>
<th>Sample A4 (g/l)</th>
<th>Mean (mgkg(^{-1}))</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chromium</td>
<td>0.42</td>
<td>0.42</td>
<td>0.66</td>
<td>0.66</td>
<td>54.06</td>
</tr>
<tr>
<td>Copper</td>
<td>2.17</td>
<td>2.16</td>
<td>2.45</td>
<td>2.51</td>
<td>233.20</td>
</tr>
<tr>
<td>Nickel</td>
<td>0.24</td>
<td>0.24</td>
<td>0.28</td>
<td>0.28</td>
<td>26.00</td>
</tr>
<tr>
<td>Lead</td>
<td>1.43</td>
<td>1.43</td>
<td>1.72</td>
<td>1.72</td>
<td>157.15</td>
</tr>
<tr>
<td>Zinc</td>
<td>11.16</td>
<td>11.59</td>
<td>10.38</td>
<td>10.79</td>
<td>1098</td>
</tr>
</tbody>
</table>

**Table 5. Quantified levels of trace metals in soil samples from Lapite landfill.**

<table>
<thead>
<tr>
<th>Metals</th>
<th>Sample A1 (g/l)</th>
<th>Sample A2 (g/l)</th>
<th>Sample A3 (g/l)</th>
<th>Sample A4 (g/l)</th>
<th>Mean (mgkg(^{-1}))</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chromium</td>
<td>0.52</td>
<td>0.52</td>
<td>0.52</td>
<td>0.52</td>
<td>51.88</td>
</tr>
<tr>
<td>Copper</td>
<td>4.68</td>
<td>4.85</td>
<td>4.68</td>
<td>4.85</td>
<td>476.55</td>
</tr>
<tr>
<td>Nickel</td>
<td>0.37</td>
<td>0.37</td>
<td>0.37</td>
<td>0.37</td>
<td>37.17</td>
</tr>
<tr>
<td>Lead</td>
<td>1.76</td>
<td>1.76</td>
<td>1.76</td>
<td>1.76</td>
<td>175.80</td>
</tr>
<tr>
<td>Zinc</td>
<td>11.82</td>
<td>12.28</td>
<td>11.82</td>
<td>12.28</td>
<td>1205</td>
</tr>
</tbody>
</table>
\( C \) is the heavy metal concentration of digested soil sample as obtained from ICP-OES analysis, \( v \) is the volume of digested solution (ml), and \( w \) is the dry weight of the soil samples.

**Bio-availability of trace metals in plant species**

Trace metal uptake in plants has been widely reported in several studies, and many bio-remediation techniques are based on this principle. The danger to public health arises from the fact that trace metals can end up in the food chain as a result of bio-accumulation in plant species, especially those grown for human consumption\(^{26,31}\). The key to breaking this chain is to ensure that fruits and vegetables are not grown in areas prone to trace metal contamination such as landfill sites. A more detailed discussion on the bioavailability of trace metals in the environment can be found elsewhere\(^{26,37}\).

**Risk assessment and remediation need**

The results of the quantified levels of trace metals and their comparison with the regulatory limits are shown graphically in Figure 3–Figure 6. The implication of these results can better be understood by making a direct comparison to specified values in the appropriate regulatory standard, or by comparing with quantified values from an uncontaminated sample. The test results show that the quantified levels of copper and zinc exceeded both the threshold and the lower guideline values specified by the Finnish government decree on the assessment of soil contamination and remediation needs (Table 3), while the values for lead exceeded only the threshold values at both landfill sites. The lower and higher guideline values is an indication of soil contamination under different land use conditions\(^{38}\). The reference document, ‘Government Decree on the Assessment of Soil Contamination and Remediation Need’ was adopted as a source for threshold and lower guideline values for levels of copper and zinc in the tested soil samples as there was no applicable regulatory standard in Nigeria with respect to soil contamination and remediation.

These results also confirms findings from previous communications that reported poor leachate management at the Aba-Eku landfill site\(^{26}\). Based on these findings and owing to increasing risk of human exposure, there is an urgent need for comprehensive risk assessment at the landfill sites, and if possible a remediation measure needs to be put in place in order to protect the rapidly expanding residential areas around these sites as well as the groundwater source. However, it may be worthwhile to point out that this study does not take into account the background values of the trace metals at the sampling sites. Therefore, a direct comparison with a regulatory limit only serves as a guide, and a more comprehensive study of the landfill sites is needed in order to establish the full ecological and health risks, including the risk of groundwater contamination.

![Figure 3](image.png)

**Figure 3.** Quantified values of trace metals in soils from Aba-Eku landfill.
Figure 4. Quantified values of trace metals in soils from Lapite landfill.

Figure 5. Comparison of quantified values with the regulatory standards for Aba-Eku landfill.
Conclusions
This paper reports the findings of a study that assessed soil contamination by trace metals as a result of uncontrolled disposal of solid waste in two municipal landfills in the Ibadan metropolis. The investigation was part of an environmental risk assessment of the current waste management and disposal practices in Ibadan, Nigeria. Several samples of soil collected at the landfill sites were analysed for trace metals using ICP-OES. The results of this analysis, without taking into account the background values of the trace metals in the area, show elevated levels of lead, zinc and copper for the two landfill sites, above the specified limits in the applicable regulatory standard. There is a need for a more comprehensive risk assessment of the landfill sites with respect to soil and groundwater contamination, and this study will serve as a basis for a more comprehensive evaluation of all landfill sites in the city.

Data availability
Dataset 1: Trace metal assessment results from the Aba-Eku landfill site using the ICP-OES technique. DOI, 10.5256/f1000research.9673.d163188
Dataset 2: Trace metal assessment results from the Lapite landfill site using the ICP-OES technique. DOI, 10.5256/f1000research.9673.d163189

Author contributions
A. B. and O. B. designed the study, A. B. performed the experiments, A. B. and O. B. carried out the data analysis. All authors contributed towards the preparation and submission of the manuscript.

Competing interests
No competing interests were disclosed.

Grant information
The author(s) declared that no grants were involved in supporting this work.
References

Your Introduction suggests that you are mistaking large urban open solid waste dumps for sanitary landfills. Best waste management practices require that material and energy recovery processes be applied to solid waste streams before disposal in landfills. What you have described fits open dumps and not landfills. I suggest you change the term landfill to open dump.

2. Why was the Finnish government decree for soil remediation used? You argued that Nigeria has no such standards. If that is so, you should have used USEPA guideline values. However, it may interest you to know that the Department of Petroleum Resources has published guideline values for heavy metals Nigerian soils. Please see the article published by Wuana and Okeimen (2011) which can be assessed with the following doi: http://dx.doi.org/10.5402/2011/402647. It is suggested that you also use the DPR guideline values even if you would like to retain the Finnish guideline values.

3. Your experimental months contain too much detail which can easily be found in standard methods or equipment manufacturers’ manuals.

4. The major weakness of this paper is that very few samples were analysed. Taking four random samples from a 21 Ha waste dump is inadequate and the results can hardly be generalized. One would expect that there would be a spatial variation of these heavy metals in the soil which should have necessitated a systematically staggered/spaced sampling points in order to obtain representative results.

5. Both table and graphs were presented for the same dataset. You should either use tables or graphs for your data depending on which you find more suitable, but you cannot use both for the same data.

6. Discussion of results is shallow.

**Is the work clearly and accurately presented and does it cite the current literature?**
Yes

Is the study design appropriate and is the work technically sound?
Partly

Are sufficient details of methods and analysis provided to allow replication by others?
Yes

If applicable, is the statistical analysis and its interpretation appropriate?
Not applicable

Are all the source data underlying the results available to ensure full reproducibility?
Yes

Are the conclusions drawn adequately supported by the results?
Partly

Competing Interests: No competing interests were disclosed.

Referee Expertise: Water and wastewater treatment, water supply, solid waste management, soil and water pollution.

I have read this submission. I believe that I have an appropriate level of expertise to confirm that it is of an acceptable scientific standard, however I have significant reservations, as outlined above.

Mynepalli Sridhar
Department of Environmental Health Sciences, Faculty of Public Health, University of Ibadan, Ibadan, Nigeria

The paper is interesting as it focuses on some of the heavy metals which find their way into food via wastes disposed in the city. The authors have taken pains in bringing out the salient features of the heavy metal contamination. The analytical methods used are appropriate. They can be reproduced by other scientists. To strengthen the outcome, the study should have looked into the composition of the wastes being dumped at the landfill and also compared with age of the wastes and their heavy metal composition and leachability. The type of soils and the composition is not adequately described. The sample size of the soils should have been more than what the authors have collected, sieved through a specific particulate size before processing. This should have given a better and homogenous sample. Also, finding the levels of heavy metals at various depths of soil may have been useful. Heavy metal levels of control soil samples should have added the value to the work.

Is the work clearly and accurately presented and does it cite the current literature?
Yes
Is the study design appropriate and is the work technically sound?
Yes

Are sufficient details of methods and analysis provided to allow replication by others?
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Are all the source data underlying the results available to ensure full reproducibility?
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Partly

**Competing Interests:** No competing interests were disclosed.

I have read this submission. I believe that I have an appropriate level of expertise to confirm that it is of an acceptable scientific standard, however I have significant reservations, as outlined above.

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