Determination of the Level of Heavy Metals in Some Selected Vegetables from an Irrigated Farmland of Kudenda in Kaduna Metropolis, Nigeria

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Authors’ contributions

This work was carried out in collaboration between all authors. Author GYS designed the study, performed the statistical analysis, wrote the protocol, wrote the first draft of the manuscript. Authors AE and JEE managed the analyses of the study. Authors IJ and MSG managed the literature searches and edited the manuscript. All authors read and approved the final manuscript.

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ABSTRACT

Heavy metal contamination of soil resulting from wastewater irrigation is a cause of serious concern due to the potential health risk of consuming contaminated produce. The use of wastewater for irrigation increases the contamination of heavy metals (Cd, Cu, Pb, Mn, Ni, Zn etc) in the plants. In this study, an assessment is made on the impacts of wastewater irrigation on heavy metals (Cd, Cu, Pb, Mn, Ni and Zn) contamination on vegetables cultivated in an irrigated farmland of Kudenda in Kaduna Metropolis, Nigeria. Samples of water, soil and some selected vegetables were collected and analysed for Cd, Cu, Pb, Mn, Ni and Zn using Buck scientific VGP 210 Atomic Absorption Spectroscopy (AAS). The wastewater used for irrigation had the following concentration 0.006±0.003 µg/mL Cd, 0.002±0.001 µg/mL Cu, 0.002±0.001 µg/mL Mn, 0.002±0.001 µg/mL Zn.
while Pb and Ni were below detectable limit (BDL). The level of heavy metals in the vegetables under study differs with vegetables species. Cd level in tomato, cabbage and garden egg is the same 0.01±0.00 μg/g, and the level in one of the soil sampling site is found to be 0.03±0.01. The level of heavy metal was higher in soil than in vegetables studied. Accumulation of heavy metals varied from vegetable to vegetable. The mean concentrations of all the heavy metals studied were below the internationally permissible limits set by FAO/WHO and USEPA.

Keywords: Heavy metals; vegetables; soil; wastewater.

1. INTRODUCTION

Environmental pollution is a major problem in developing countries like Nigeria due to both domestic and industrial anthropogenic activities including municipal wastes that end up in rivers, lakes and sea. Anthropogenic activities and natural source are two main sources of heavy metals contamination in the soil [1]. Some of these metals after accumulating in the soil are transferred to food chain which can cause serious health hazards to human beings and animals [2,3]. Heavy metal such as Hg, Cd, Cr, Ti, and Pb are elements that exhibit metallic properties and are defined based on high density, atomic number or atomic weight, chemical properties or toxicity [4]. Heavy metals are particularly dangerous because they tend to bio-accumulate in the body tissues and organs. When a large amount of heavy metal like chromium, cadmium and lead are ingested, it causes reduction in the size, weight and causes severe damage to the liver and kidney [5,6]. Heavy metals like Fe, Cu, Ni and other trace elements are important for proper functional of biological systems and their deficiency or excess could lead to a number of disorders. Cadmium adversely affects several important enzymes. It can cause painful osteomalacia (bone disease), destruction of red blood cells. Lead (Pb) has been found to be bio-accumulated by benthic bacteria, plants and other aquatic biota [7,8]. Heavy metals are considered to be one of the main sources of the environmental pollution, since they have a significant effect on soil quality. They may be toxic at very low concentration and their toxicity increases with the accumulation of water in the soils [9-11].

The use of wastewater for irrigation for a long time may transport dissolved heavy metals to agricultural field and may lead to gradual accumulation of some potentially toxic heavy metals in agricultural soil and in return have an adverse effects on the growth of the irrigated plants [12]. Studies have shown that sewage water irrigation has elevated the levels of heavy metals such as Ni, Zn, Co, Mn, and Fe in receiving soil [13-15]. Similarly, Sharma et al. [16] reported that plants grown on wastewater-irrigated soils are contaminated with heavy metals and pose a health concern. Absorption and accumulation of heavy metals in plant tissues depend upon the nature of the vegetable and many other factors such as temperature, moisture, organic matter, pH and nutrient availability. Heavy metals after accumulating in the soil are transferred to food chain which can cause a serious health hazard to both human being and animal. Food chain contamination by heavy metals has become a burning issue in recent years because of their potential accumulation in bio-systems through contaminated water, soil and air [17].

The research work aim to assess the impacts of wastewater irrigation on heavy metals (Cd, Cu, Pb, Mn, Ni and Zn) contamination on vegetables cultivated in farmland of Kudenda in Kaduna Metropolis, Nigeria and to ascertain the safety or otherwise of the vegetables consumed by the people of Kaduna Metropolis.

2. MATERIALS AND METHODS

2.1 Equipment/Apparatus


2.2 Chemicals/Reagents

Sulphuric Acid (Sg. 1.84), Hydrogen Peroxide (Sg. 1.13), Distilled water, Hydrochloric acid (Sg. 1.18), Perchloric acid (70%), Nitric acid (70%).
2.3 Study Area

Kaduna State is located on the southern end of the high plains of northern Nigeria, bounded by parallels of latitude 9°02′N and 11°32′N [18]. The state is divided into three senatorial zones, namely; Kaduna North, Central and South and it comprises twenty three (23) Local Government Areas, 46 Local Development Areas (LDAs) [19]. Kaduna State shares its boundary with Katsina State to the North, Niger State and Abuja to the west, Plateau State to the South and Kano State to the east. The State occupies an area of approximately 45,711.2 km² and had a population of 6,113,503 people with an annual growth rate of 3% during the 2006 census [20].

2.4 Sample Collection

The vegetables used in this study include Spinach (*Spinacia oleracea*), Tomatoes (*Solanum lycopersicum*), Cabbage (*Brassica oleracea*) and Garden Egg (*Solanum melongena*) were collected from farmland of Kudenda in Kaduna Metropolis, Nigeria. A total of sixteen (16) samples, (four (4) samples each of Spinach, Tomatoes, Cabbage and Garden Egg) were randomly collected from four (4) different locations around river mashi during their harvesting period. The harvested vegetables were washed with the river water to remove soil particles and then oven-dried at 70°C until stable weights were obtained. Samples were then grounded in a mortar, passed through a 2 mm sieve and stored at room temperature before the analysis.

A total of ten (10) samples of water used for irrigation from River Mashi in Kudenda farmland was collected along the water pathway in a pre-acid-washed plastic bottle and 1 cm³ concentrated HNO₃ were added to the sample to avoid microbial activity.

Soil samples from the study area were collected at a depth of 0-30 cm. Kudenda is a commercial vegetable farm and is considered one of the most important sites to supply vegetable markets within the town and beyond. Thirty two (32) soil samples were randomly collected from eight (8) different location during November, 2016 to April, 2017 (months of peak irrigation activities). The soil samples were placed in polythene bags, ensuring that the natural soil moisture content was preserved and taken to the laboratory for analysis.

![Fig. 1. Map of Nigeria showing the study area](image-url)
2.5 Water Digestion for Metal Analysis

One liter of the wastewater used for irrigating the farmland was collected and treated with 1.5 ml of concentrated HNO₃. Fifty milliliters of the water sample was transferred to an evaporating dish and evaporated on a steam bath to about 20 ml. Ten milliliters of 8 M HNO₃ of 98% purity was added and evaporated on a hot plate to near dryness. The residue was quantitatively transferred using two aliquot of 10 and 15 ml of concentrated HNO₃ into a 250 ml flask. Twenty milliliters of concentrated HClO₄ was added and boiled until the solution became clear and white fumes of HClO₄ appeared. It was then cooled and de-ionised distilled water of 50 ml was added and the solution filtered. The filtrate was quantitatively transferred to a 100 ml volumetric flask with two portions of 5 ml of de-ionised distilled water. The solution was diluted to mark and mixed thoroughly by shaking. The level of heavy metals under study was determined using Buck scientific VGP 210 atomic absorption spectrophotometer (AAS) [21].

2.6 Plant Digestion for Metal Analysis

One gram each of prepared vegetable samples was weighed into 125 cm³ conical flasks using the United States Environmental Protection Agency (USEPA 3050 method) by Miller and McFee, (1983). 10 cm³ of 8M HNO₃ was added and the mixture was heated for 30 minutes in a water bath at 100°C. The digest was allowed to cool and another 5 cm³ of concentrated HNO₃ was added and heating continued for 1 hour at 100°C. The volume of the digest was reduced by boiling on the water bath and this was allowed to cool. 5 cm³ of de-ionised water was added when effervescence subsided, 10 cm³ of H₂O₂ (60%) was added and heating continued for another 30 minutes. The final digest was allowed to cool and then filtered. The final volume of digest was made up to 50 cm³ with de-ionised distilled water and was analysed for the required heavy metals by Buck scientific VGP 210 Atomic Absorption spectrophotometer (AAS).

2.7 Soil Digestion for Metal Analysis

The method developed by the United States Environmental Protection Agency (USEPA) for (total sobbed) heavy metals in soil, sediments and sludge (USEPA SW-846, method 3050) (USEPA 1986), was used in the preparation of the soil samples for the determination of total metal content in this study [21]. One gram of the soil sample was weighed into a beaker for acid digestion. Analar grade Nitric acid, Hydrogen peroxide (about 30%) and concentrated Hydrochloric acid were used for the digestion. The digest was filtered through whatman filter paper. Each filtrate was collected in 100 ml volumetric flask and deionised water was used to rinse the filter paper into volumetric flask. Each filtrate was later made up to 100 ml with deionised water. Standards were prepared with serial dilution techniques within the range of each metal determined. The standards used were Analar grade; the instrument was first calibrated with stock solutions of the prepared standards before analysed using Atomic Absorption Spectrophotometer.

3. RESULTS AND DISCUSSION

3.1 Soil Characteristics of the Study Area

The pH values of the soil samples from the irrigated farmland under study ranged from 6.5±0.2-7.2±0.2. All the soils studied from the farmland were either slightly acid or neutral. The soil organic carbon concentrations ranged from 0.65±0.02-1.30±1.02. The soils from the study area were generally low in organic carbon contents. The clay contents ranged from 13±1-16±1%. The cation exchange capacity ranged from 10.3±1.0-19.2±1.2 meq/100 g, (Table 1).

Table 1. Soil characteristics of the study site

<table>
<thead>
<tr>
<th>Sample sites</th>
<th>pH</th>
<th>O.C</th>
<th>Clay (%)</th>
<th>Sand (%)</th>
<th>Silt (%)</th>
<th>C.E.C meq/100 g</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>6.5±0.2</td>
<td>0.87±0.20</td>
<td>14±1</td>
<td>74.9±1.1</td>
<td>11.1±1.0</td>
<td>16.6±1.2</td>
</tr>
<tr>
<td>B</td>
<td>6.8±0.2</td>
<td>1.30±1.02</td>
<td>14±1</td>
<td>72.5±1.0</td>
<td>13.9±0.7</td>
<td>15.4±1.2</td>
</tr>
<tr>
<td>C</td>
<td>7.1±0.2</td>
<td>0.92±0.50</td>
<td>16±1</td>
<td>71.0±1.0</td>
<td>13.1±0.7</td>
<td>18.2±1.0</td>
</tr>
<tr>
<td>D</td>
<td>6.9±0.2</td>
<td>0.68±0.02</td>
<td>13±1</td>
<td>76.1±1.2</td>
<td>11.2±0.8</td>
<td>17.6±1.3</td>
</tr>
<tr>
<td>E</td>
<td>7.2±0.2</td>
<td>0.98±0.60</td>
<td>14±1</td>
<td>73.6±1.1</td>
<td>12.7±0.8</td>
<td>16.8±1.0</td>
</tr>
<tr>
<td>F</td>
<td>6.8±0.2</td>
<td>0.98±0.60</td>
<td>14±1</td>
<td>72.6±1.0</td>
<td>13.1±0.7</td>
<td>10.3±1.0</td>
</tr>
<tr>
<td>G</td>
<td>6.5±0.2</td>
<td>0.72±0.60</td>
<td>13±1</td>
<td>73.8±1.1</td>
<td>13.0±0.9</td>
<td>17.6±1.3</td>
</tr>
<tr>
<td>H</td>
<td>7.0±0.2</td>
<td>0.65±0.02</td>
<td>16±1</td>
<td>70.9±1.0</td>
<td>13.4±0.6</td>
<td>19.2±1.2</td>
</tr>
</tbody>
</table>

Values are presented as mean ± SD of triplicate measurement
3.2 Levels of Heavy Metals in Water

The mean concentration of heavy metals in the water used for irrigation is presented in Table 2 with Cd>Zn>Mn>Cu. Pb and Ni mean concentrations were below detectable limit. Even though the concentrations 0.0055±0.003 µg/mL Cd, 0.00013±0.0004 µg/mL Cu, 0.0022±0.001 µg/mL Mn, 0.0023±0.001 µg/mL Zn were found to be lower than the maximum permissible limits set by FAO/WHO [22] and USEPA [23], the simple reason for the detection of the heavy metals in the irrigation water could be attributed to the presence of small scale industries like International Beer & Beverage Industries (IBBI), Ideal Flour Mill Ltd, Sunglass Ltd and some Fuel Filling Stations located in the area that discharge their effluents into the river. Household waste products are also discharged into the river which might also be a reason.

The water sample from the study area shows low concentration in almost all the estimated heavy metals when compared with the FAO/WHO standard limit as shown in Fig. 2, except for Cd which is little above the maximum permissible limits allowed in the irrigation water set by FAO/WHO [22].

3.3 Level of Heavy Metal Concentrations in Vegetables

The mean concentration (µg/g) of heavy metals in vegetable samples from the study area is presented in Table 3. From the mean concentrations of heavy metals in vegetables, the concentration of Cd is found to be the same 0.01±0.00 in tomato, cabbage and garden egg, while in spinach, it is below detectable limit. Copper is one of the most important essential elements for plants and animals [21]. Copper was extensively translocated, as it is essential to the plant metalloenzymes diamine oxidase, ascorbate oxidase, cytochrome C oxidase, superoxide dismutase and plastocyanin oxidase [24] and photosynthesis [25]. Cu accumulation appears to be highest (0.3±0.1 µg/g) in tomato compared to the other plants. The uptake of manganese (Mn) by plants varies with plant species. Tomato had the highest concentration of 0.05±0.01 µg/g and spinach and garden egg had the least concentration of Mn (0.01±0.00 µg/g).

Plants uptake of heavy metals partly correlates with the total content of heavy metals in the soils. Plant grown on soils possessing enhanced metal concentrations have increased heavy metal ion content. The uptake of metal ions has been shown to be influenced by the metal species and plant parts [26]. Muhammad et al. [12] reported that the accumulation of heavy metals such as Fe, Mn, Cu, Zn and Pb in plant significantly increased by sewage water irrigation.

Babandi et al. [27] investigated the level of heavy metals like, Pb, Cr, Cd, Cu, Co, Zn and Ni in soil and plants irrigated with

<table>
<thead>
<tr>
<th>Sample</th>
<th>Cd</th>
<th>Cu</th>
<th>Pb</th>
<th>Mn</th>
<th>Ni</th>
<th>Zn</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>0.005</td>
<td>0.002</td>
<td>BDL</td>
<td>0.003</td>
<td>BDL</td>
<td>0.003</td>
</tr>
<tr>
<td>B</td>
<td>0.004</td>
<td>0.001</td>
<td>BDL</td>
<td>0.002</td>
<td>BDL</td>
<td>0.003</td>
</tr>
<tr>
<td>C</td>
<td>0.005</td>
<td>0.002</td>
<td>BDL</td>
<td>0.002</td>
<td>BDL</td>
<td>0.002</td>
</tr>
<tr>
<td>D</td>
<td>0.002</td>
<td>0.002</td>
<td>BDL</td>
<td>0.004</td>
<td>BDL</td>
<td>0.002</td>
</tr>
<tr>
<td>E</td>
<td>0.010</td>
<td>0.001</td>
<td>BDL</td>
<td>0.003</td>
<td>BDL</td>
<td>0.001</td>
</tr>
<tr>
<td>F</td>
<td>0.005</td>
<td>0.002</td>
<td>BDL</td>
<td>0.002</td>
<td>BDL</td>
<td>0.001</td>
</tr>
<tr>
<td>G</td>
<td>0.002</td>
<td>0.002</td>
<td>BDL</td>
<td>0.001</td>
<td>BDL</td>
<td>0.003</td>
</tr>
<tr>
<td>H</td>
<td>0.010</td>
<td>0.001</td>
<td>BDL</td>
<td>0.001</td>
<td>BDL</td>
<td>0.004</td>
</tr>
<tr>
<td>I</td>
<td>0.002</td>
<td>0.001</td>
<td>BDL</td>
<td>0.002</td>
<td>BDL</td>
<td>0.001</td>
</tr>
<tr>
<td>J</td>
<td>0.010</td>
<td>0.001</td>
<td>BDL</td>
<td>0.002</td>
<td>BDL</td>
<td>0.003</td>
</tr>
<tr>
<td>Mean</td>
<td>0.006</td>
<td>0.002</td>
<td>-</td>
<td>0.002</td>
<td>-</td>
<td>0.002</td>
</tr>
<tr>
<td>S.D</td>
<td>0.003</td>
<td>0.001</td>
<td>0.000</td>
<td>0.001</td>
<td>0.000</td>
<td>0.001</td>
</tr>
<tr>
<td>FAO/WHO</td>
<td>0.003</td>
<td>2.000</td>
<td>0.050</td>
<td>50.000</td>
<td>0.020</td>
<td>0.030</td>
</tr>
</tbody>
</table>

Values are presented as mean ± SD of triplicate measurement
SD = Standard Deviation
BDL = Below Detectable Limit, WHO = World Health Organization
waste water of Sharada industrial site and found out that the level of heavy metals in soil and plant samples is higher than the WHO permissible limit. This study also revealed that these plants accumulate metals though below the internationally permissible limits set by FAO/WHO and USEPA but with time could be dangerous to body metabolic systems.

Even though, the concentration of Zn and Cu were found to be slightly high in all the vegetables, but it is within the recommended (daily) dietary allowances of food and nutrition set by FAO/WHO [22] and USEPA [23]. Zinc (Zn) is essential for growth and has important role in cellular immunity, sexual maturation, promotion of wound healing, etc. Elevated intake of Zinc over an extended period of time can actually harm the immune system instead of assisting it. However, zinc deficiency can cause liver and kidney failure and anaemia [28,29].

3.4 Level of Heavy Metal Concentrations in Soil

The mean concentration (µg/g) of heavy metals in the soil from the irrigated farmland of Kudenda is presented in Table 4. The highest mean concentration (µg/g) is seen in Cu 0.9±0.2, followed by Mn 0.50±0.08, while Pb mean concentration was below detectable limit in all the soil samples.

![Graph](Image)

**Fig. 2. Mean concentration (µg/mL) of heavy metal in water and FAO/WHO standard**

**Table 3. Heavy metal concentration (µg/g) in vegetables from the study area**

<table>
<thead>
<tr>
<th>Vegetable</th>
<th>Cd</th>
<th>Cu</th>
<th>Pb</th>
<th>Mn</th>
<th>Ni</th>
<th>Zn</th>
</tr>
</thead>
<tbody>
<tr>
<td>Spinach</td>
<td>BDL</td>
<td>0.2±0.0</td>
<td>BDL</td>
<td>0.01±0.00</td>
<td>BDL</td>
<td>0.4±0.1</td>
</tr>
<tr>
<td>Tomato</td>
<td>0.01±0.00</td>
<td>0.3±0.1</td>
<td>0.001±0.000</td>
<td>0.05±0.01</td>
<td>BDL</td>
<td>0.5±0.1</td>
</tr>
<tr>
<td>Cabbage</td>
<td>0.01±0.00</td>
<td>0.2±0.1</td>
<td>BDL</td>
<td>0.02±0.00</td>
<td>BDL</td>
<td>0.4±0.1</td>
</tr>
<tr>
<td>Garden Egg</td>
<td>0.01±0.00</td>
<td>0.2±0.0</td>
<td>BDL</td>
<td>0.01±0.00</td>
<td>0.001±0.000</td>
<td>0.3±0.1</td>
</tr>
</tbody>
</table>

*Values are presented as mean ± SD of triplicate measurement  
BDL = Below Detectable Limit*

**Table 4. Heavy metal concentrations (µg/g) in soil from the study area**

<table>
<thead>
<tr>
<th>Sample site</th>
<th>Cd</th>
<th>Cu</th>
<th>Pb</th>
<th>Mn</th>
<th>Ni</th>
<th>Zn</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>BDL</td>
<td>0.2±0.1</td>
<td>BDL</td>
<td>0.01±0.00</td>
<td>0.02±0.01</td>
<td>0.03±0.02</td>
</tr>
<tr>
<td>B</td>
<td>BDL</td>
<td>BDL</td>
<td>BDL</td>
<td>BDL</td>
<td>0.01±0.00</td>
<td>BDL</td>
</tr>
<tr>
<td>C</td>
<td>0.02±0.01</td>
<td>0.5±0.3</td>
<td>BDL</td>
<td>BDL</td>
<td>BDL</td>
<td>0.05±0.02</td>
</tr>
<tr>
<td>D</td>
<td>0.02±0.00</td>
<td>0.3±0.1</td>
<td>BDL</td>
<td>BDL</td>
<td>0.01±0.00</td>
<td>BDL</td>
</tr>
<tr>
<td>E</td>
<td>0.03±0.01</td>
<td>BDL</td>
<td>BDL</td>
<td>BDL</td>
<td>0.02±0.01</td>
<td>0.01±0.00</td>
</tr>
<tr>
<td>F</td>
<td>0.02±0.01</td>
<td>BDL</td>
<td>BDL</td>
<td>BDL</td>
<td>0.08±0.01</td>
<td>BDL</td>
</tr>
<tr>
<td>G</td>
<td>BDL</td>
<td>0.2±0.1</td>
<td>BDL</td>
<td>0.50±0.08</td>
<td>BDL</td>
<td>0.05±0.02</td>
</tr>
<tr>
<td>H</td>
<td>BDL</td>
<td>0.9±0.2</td>
<td>BDL</td>
<td>0.02±0.01</td>
<td>BDL</td>
<td></td>
</tr>
</tbody>
</table>

*Values are presented as mean ± SD of triplicate measurement, BDL = Below Detectable Limit*
3.5 Statistical Analysis

Pearson product-moment correlation coefficient analysis between the mean concentration of heavy metals in water, vegetable and soil sample was carried out. “There is no statistically significant correlation at 0.05 level of confidence.

4. CONCLUSION

The study has assessed the impacts of wastewater irrigation on heavy metals (Cd, Cu, Pb, Mn, Ni and Zn) contamination of soil and vegetables (edible parts) cultivated in an irrigated farmland of Kudenda in Kaduna, Nigeria. All the samples investigated contained detectable concentrations of the heavy metals (Cd, Cu, Mn, Ni and Zn) but within the permissible limits set by FAO/WHO [22] and USEPA [23] with the exception of Pb that is below detectable limit in soil. Therefore, the result of this study did not show any abnormal accumulation of heavy metals by the vegetables due to irrigation and other industrial activities in the area. Hence, the vegetables cultivated in the irrigated farmland of Kudenda in Kaduna Metropolis may be considered safe for consumption.

COMPETING INTERESTS

Authors have declared that no competing interests exist

REFERENCES


