Assessment of heavy metals contamination and pollution around mining sites of Anka, north-west Nigeria

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Abstract. Artisanal mining is a common practice in Anka communities, North-West Nigeria, and this has brought about serious environmental and health concerns in the area. This study assessed the concentration of heavy metals in the rocks, soils, sediments and plants in the mining environment of Anka. Thirty two samples were collected and analysed for K, V, Cr, Mn, Fe, Ni, Cu, Zn and Pb using Energy Dispersive X-ray Fluorescence. The results obtained showed the heavy metal concentration in rock > soil > sediment > plant. The evaluation of the contamination factor in soil and sediment revealed that all the samples were severely contaminated. The results of geo-accumulation index revealed that the contamination with heavy metals was generally higher in rock and soil; followed by sediment while plant samples accounted for the least contamination. The result of the contamination factor and pollution index of soil and sediment indicated severe contamination and pollution respectively. The contamination assessment was complemented with the use of contamination degree and the modified contamination degree indices. The assessment of contamination factor and potential ecological risk from heavy metal in both soil and sediment was shown to come majorly from Pb, Ni and Cu. Active mining sites found in Dareta, Daki Takwas, Dan Kamfani and the processing areas of Abare, Dan Kamfani, Dateta and Abare were identified as areas with high heavy metal pollution risk. These results generally show that artisanal mining and processing in the study area pose significant environmental risk to the local population and the environment.

Keywords: Anka; Artisanal mining, Contamination, Concentration, Geo-accumulation index, Heavy metal

Abbreviations: EDXRF - Energy Dispersive X-ray Fluorescence; QXAS - Quantitative Analysis of X-ray Spectroscopy; MTL – Maximum Tolerable Limit; ND – Not Detected; RK – Rock; SL – Soil; SD – Sediment; PT – Plant/Vegetable

1. INTRODUCTION

The Earth environment serves as a reservoir for several resources, which also comes in the form of solid, liquid or gas. The presence of heavy metals in the environment comes from both natural and anthropogenic sources; the natural sources include weathering of metal-containing rocks and volcanic eruptions while anthropogenic sources include agricultural activities, mining, industrial emissions and smelting (Ali et al., 2019; Udiba et al., 2019). The concentration of mineral deposits in the earth varies according to the geological formation, altitude and the anthropogenic activities; this variation is largely responsible for the uneven distribution of elements and other natural resources in the Earth environment (Joshua et al., 2009).

Heavy metal contamination of soil has been identified as a primary origin of metals in plants/vegetables, which finally finds its way into the food chain (Gebeyehu and Bayissa, 2020). This could be as a result of migration and release into the surface and subsequent incorporation into the food chain. Vegetable plants take up these heavy metals by absorbing them from the air, polluted environments and contaminated soils (Kachenko & Singh, 2006). According to Kapile and Matundi (2016), plant uptake of Cu and Zn as essential micronutrients is higher than that of toxic elements such as Cd and Pb. Studies have shown that heavy metals such as Co, Cr, Cu, Fe, Mg, Mn, Mo,
Se and Zn have nutritional value while Cd, Pb, Hg and Ni are said to have no known nutritional value (Kacholi and Sahu, 2018; USEPA, 2009).

In Anka, gold is mined from gold-bearing deposits containing an unusually problematic concentration of lead and other heavy metals. The crushing of rocks in dry running flour mills produces enormous amounts of dust which in some places is highly contaminated with lead and other heavy metals. Hence, mining and ore processing is accompanied by major negative physical, biological, hydrological as well as environmental impacts. Environmental and health problems have been reported in the study area and there are speculations that this could be as a result of local and crude mining activities. According to Udiba et al. (2019), mining generally generates volumes of mineral waste such as waste rocks or tailings, as the materials of interest are often surrounded by other ores and rocks during mining.

Moreover, toxic contamination from the mined ores and the waste arising from the processing mills find their way into the natural environment thereby affecting air, arable land (soil), habitat and water (Merem et al., 2017). As a result of this heavy metal contamination, there have been reported cases of deaths, mostly among children of ages 0 - 5 years (Innocent et al., 2013; Dooyema et al., 2012). The number of fatalities was put at over 400 children (IGF, 2017). These fatalities and other illnesses reported were attributed to heavy metal pollution (Tirimba et al., 2016; Kaufman et al., 2012). Acute lead toxicity is also said to be destroying the biodiversity13 (Uduma and Jimoh, 2014).

The study of heavy metal contamination is aimed at identifying the particular heavy metals responsible and potential areas of contamination. The study area is an agricultural region of Anka in the present Zamfara State, North-West Nigeria. Over the past years, Anka has been greatly exploited and degraded due to artisanal mining activities. Traditionally, most villagers rely on farming to earn a living, however, some areas are also rich in mineral resources, such as gold hosted in lead-zinc ores. Hence, artisanal mining and crude value addition (processing) is also one of the thriving enterprises in the area.

Therefore, the prime objective of this work to determine the concentration of heavy metal (K, V, Cr, Mn, Fe, Ni, Cu, Zn and Pb) in rock, soil, sediment and plant samples in Anka, North-West Nigeria. This study is also aimed at evaluating the risk of heavy metals in soil and sediments using different contamination, pollution and ecological risk indices. The study will help in understanding the effect of artisanal mining on the environment.

2. MATERIALS AND METHOD

2.1 Study Area

The study was carried out in artisanal mining and mineral processing communities of Anka, North-West Nigeria. Anka lies between latitude 11°51’N and 12°08’N, and longitude 5°51’E and 6°08’E and covers an area of 2,940 km² (Akpanowo et al., 2019). The map of Anka showing the study area is shown in Figure 1. The geology of Anka is characterized by the Anka schist belt that hosts the lead mineralization, and the lead-copper-silver-gold poly-metallic association (Buba, 2016). Anka, like some other similar geological belts in Nigeria, has been the focus of artisanal gold exploitation for several decades. Gold found in Anka is hosted by schists, phyllites and quartzites associated with sub-regional structural elements subsidiary to the Anka fault (Waziri and Andrews, 2013).

2.2 Sampling and Sample Preparation

A total of Thirty Two samples were collected from the study area; the Thirty two samples consisted of 6 rocks, 15 soils, 8 sediments and 3 plants/vegetables. The edible parts of plant leaves were considered and the samples were collected in October 2018 and November 2019, when most of plant/vegetable had reached maturity. Majority of the samples were collected within 200 m of the mining and mineral processing sites in Dareta, Abare, Dan Kamfani and Daki Takwas communities (Figure 1). After collection, all the samples were conveyed to the laboratories at the Centre for Energy Research and Training (CERT) for sample preparation.

Initial sample preparation was carried out at the Material Science laboratory at the Centre for Energy Research and Training, Zaria, Nigeria. The rock, soil and sediment samples were oven-dried between 120 and 150°C, while the plant samples were dried at room temperature of about 22 to 25°C to avoid ashing. The samples were ground into fine powder using agate mortar and pestle, made of 99% silica to avoid contamination. The ground samples were sieved through a Test Sieve through an aperture of 250 μm to achieve the greatest possible homogeneity and to prevent low concentration associated with large samples (Zhu and Shaw, 2000). About 200 g of the dried sample was weighed using the Sartorius (CP622) weighing balance and packaged for onward transfer for laboratory analysis.
2.3 X-Ray Fluorescence (XRF) Elemental Analysis

Thirty-two samples (6 rocks, 15 soils, 8 sediments and 3 plants/vegetables) were analyzed for elemental composition at the Centre for Energy Research and Development (CERD), Ile-Ife, Nigeria. The AMPTEK(R) Energy Dispersive X-ray Fluorescence (EDXRF) system was employed for the analysis.

Before the analysis, certified Soil-7 reference standards were irradiated for calibration purposes using the following parameters: For potassium (K), the certified and experimental values were the same, 12100 ppm; for iron (Fe), the certified and experimental values were also the same, 25700 ppm; however the certified and experimental values for Zirconium (Zr) were 185 ppm and 184.96 ppm respectively. Sample analysis was done by exposing the samples in the sample holder of the XRF system and bombarded by X-ray fluorescence spectrometer with a silver (Ag) anode at a voltage of 25 kV and current of 50 µA for 1000 seconds or 16.67 minutes in an external chamber setup (Oyedotun, 2018).

The instrumentation of the EDXRF spectrometry system, the x-ray released from the sample strikes directly on the solid-state lithium floated silicon detector. The detector is capable of discriminating the x-ray spectra tens to thirty elements simultaneously (Oyedotun, 2018); hence, the simultaneous analysis of the elements in the periodic table can be achieved.

Characteristic X-ray of the sample was detected by the solid-state Si-pin detector system and spectrum acquisition was done using an Amptek model multi-channel analyzer while elemental analysis was done using the thick target mode of the International Atomic Energy Agency (IAEA) Quantitative Analysis of X-ray Spectroscopy (QXAS) software. The resolution of the detector system of the EDXRF was 220 eV FWHM for the 5.9 keV peak of 55Fe. The detector was attached to a Multichannel Analyzer with model number MCA8000A, which was used to acquire data and process signals.
2.4. Environmental Risk Assessment

This work employed some well-known single and multiple contamination/pollution indices to analyze the concentration of heavy metals in the study area. These include contamination factor (Cf), Contamination degree (Cd), geo-accumulation index (Igeo) and pollution index (PI).

2.4.1. Contamination Factor (Cf)

According to Brady et al. (2015) the application contamination factor (Cf) is the most direct method of determining the sediment or soil quality. It is the ratio of an element in a sampling site to the background value of the element and it is calculated as:

$$Cf = \frac{C_s}{C_b}$$  \hspace{1cm} (1)

where $C_s$ is the concentration of the element of interest at a site and $C_b$ is the reference concentration of the same element at a control site.

2.4.2. Geo-accumulation Index (Igeo)

The geo-accumulation index (Igeo) is commonly applied in the evaluation of the extent of contamination or pollution in the terrestrial and marine environment by heavy metals (Okedeyi et al., 2014). The Igeo was calculated by Equation 1 following the studies of Santos-Francés et al. (2017); Fosu-Mensah et al. (2017); Okedeyi et al. (2014) and Nowrouzi and Pourkhabbaz, (2014):

$$I_{geo} = \log_2 \left( \frac{C_n}{B_n \times 1.5} \right)$$  \hspace{1cm} (2)

where $C_n$ is the measured concentration of the n metal examined in the soil, and $B_n$ is the background level of the n metal. The factor 1.5 was used to correct possible variations in the background values of a particular metal in the environment. The quantity Igeo was calculated using the global average shale data reported by Nowrouzi and Pourkhabbaz (2014).

2.4.3. Contamination Degree Index (Cd)

The contamination degree index was proposed by Hakanson (1980) for the quantifying the degree of contamination of elements due to the limitation of single element pollution indices. It is given as the sum of the Cf for each sample:

$$Cd = \sum_{i=1}^{n} Cf_i$$  \hspace{1cm} (3)

where $Cf_i$ is the contamination factor of the ith element.

2.4.4. Modified Contamination Degree Index (mCd)

The Hakanson’s contamination degree (Cd) index was modified by Brady et al. (2016) to assess the overall contamination degree at particular site by heavy metal. The modified contamination degree (mCd) is given as the sum of the contamination factors (Cf) for a particular set of heavy metal divided by the number of heavy metals analyzed. This is given by:

$$mCd = \frac{\sum_{i=1}^{n} Cf_i}{n}$$  \hspace{1cm} (4)

where $n$ is the number of analyzed heavy metals and $i$ is the ith heavy metal.

2.4.5. Pollution Index (PI)

The pollution index indicates the quality of sediment (soil) and was developed by Nemerow to take into consideration the effect of a single element contamination (Brady et al., 2016; Nemerow, 1991). This is similar to the modified contamination degree index but it is calculated based on the average values of the contamination factors ($Cf_{average}$ and the maximum contamination factor ($Cf_{max}$).

$$PI = \sqrt{\frac{(Cf_{average})^2 + (Cf_{max})^2}{2}}$$  \hspace{1cm} (5)

The corresponding threshold values of contamination factor (Cf), Contamination degree (Cd), modified Contamination degree (Cd), geo-accumulation index (Igeo) and pollution index (PI) are presented in Table 1.
The Ecological Risk Index (RI) to evaluate the hazard of heavy metal in soil and sediments was used. For Pb, the minimum concentration of 16 mgkg⁻¹ was measured in Dan Kamfani while the maximum of 262910 mgkg⁻¹ was obtained from Dareta 4. The concentration of Ni ranged from 223 to 394 mgkg⁻¹. The concentration of Mn ranged from 38 to 111 mgkg⁻¹. The concentration of lead (Pb) ranged from 7 to 6675 mgkg⁻¹.

Hakanson proposed Potential Ecological Risk Index (RI) to evaluate the hazard of heavy metal in soil and sediments (Guo et al., 2010; Hakanson, 1980). The primary function of the index is to point out the heavy responsibility for the contamination of the environment to allow for streamlined studies (Sivakumar et al., 2016).

Ecological risk factor (Eᵣ) is used to assess the toxicity of individual heavy metals in soils and sediments (Santocs-Francés et al., 2017). The Ecological risk factor (Eᵣ) is expressed as:

\[ Eᵣ^i = Tᵣ^i \times Cf \]  

where Cf is the contamination factor and Tᵣ^i is the toxic response factor. Hakanson had given a set of values for Cr, Mn, Ni, Cu, Zn and Pb as 2, 1, 5, 5, 1 and 5 respectively (Kolawole et al., 2018; Hakanson, 1980). Therefore, the potential ecological risk index (RI) is expressed:

\[ RI = \sum_{i=1}^{n} Eᵣ^i \]  

The classification of potential ecological risk index and ecological risk factor is presented in Table 2.

### Table 2. Ecological risk Factor and Potential ecological Risk Index for Pollution Level Assessment

<table>
<thead>
<tr>
<th>Level of Ecological Risk</th>
<th>Ecological Risk Factor (Eᵣ)</th>
<th>General level of Potential Ecological Risk</th>
<th>Potential Ecological Risk Index (RI)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Low</td>
<td>Eᵣ ≤ 40</td>
<td>Low</td>
<td>RI ≤ 150</td>
</tr>
<tr>
<td>Moderate</td>
<td>40 &lt; Eᵣ &lt; 80</td>
<td>Moderate</td>
<td>150 ≤ RI &lt; 300</td>
</tr>
<tr>
<td>Considerable</td>
<td>80 &lt; Eᵣ &lt; 160</td>
<td>High</td>
<td>300 ≤ RI &lt; 600</td>
</tr>
<tr>
<td>High</td>
<td>160 &lt; Eᵣ &lt; 320</td>
<td>Very high</td>
<td>RI ≥ 600</td>
</tr>
<tr>
<td>Very High</td>
<td>Eᵣ ≥ 320</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

(Adapted from Kolawole et al., 2018; Guo et al., 2010)

### 3. RESULTS AND DISCUSSION

#### 3.1. Analysis of Elemental Concentration

The results obtained for the various samples are presented in Table 3 for rocks, Table 4 for Soils, Table 5 for Sediments and Table 6 for plant/vegetables.

The samples were collected from Dareta 3 and Dareta 1 respectively. Dareta 1 seemed to present the highest concentrations for most of the elements while Dareta 3 presented the least concentrations as indicated in Table 3. The elemental Fe concentration for rock samples ranged from 750 to 296090 mgkg⁻¹, K ranged from 33290 to 256910 mgkg⁻¹, V ranged from 472 to 3125 mgkg⁻¹, Cr ranged from 343 to 2548 mgkg⁻¹ and Mn ranged from 80 to 111 mgkg⁻¹. The concentration of Ni ranged from 223 to 394 mgkg⁻¹, Cu ranged from 126 to 8574 mgkg⁻¹ and Zn ranged from 59 to 357 mgkg⁻¹. The concentration of lead (Pb) ranged from 7 to 6675 mgkg⁻¹.

The summary of the concentrations for the 15 soil samples is presented in Table 4. From the result of elemental concentration, Fe ranged from 360 to 390090 mgkg⁻¹, K ranged from 20180 to 359810 mgkg⁻¹, V ranged from 201 to 4678 mgkg⁻¹ and Cr ranged from 473 to 4955 mgkg⁻¹. The concentration of Mn ranged from 380 to 19380 mgkg⁻¹, Ni ranged from 124 to 255000 mgkg⁻¹, Cu ranged from 130 to 3567 mgkg⁻¹ and Zn ranged from 43 to 665. For Pb, the minimum concentration of 16 mgkg⁻¹ was measured in Dan Kamfani while the maximum of 2629 mgkg⁻¹ was obtained from Dareta 4.

Table 5 summarizes the concentration of elements determined for sediment samples. The Eight (8) sampling sites including Abare (3 locations), Anka town, Anka River, Dan Kamfani and Dareta (2 locations). The values of

### Table 1. Threshold values for Contamination/Pollution Indices in Soil and Sediment

<table>
<thead>
<tr>
<th>Class</th>
<th>Contamination/Pollution level</th>
<th>Igeo</th>
<th>Cf</th>
<th>Ca</th>
<th>mC₄</th>
<th>PI</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>Unpolluted</td>
<td>≤ 0</td>
<td>Cf &lt; 1</td>
<td>mC₄ &lt; 6</td>
<td>C₄ &lt; 1.5</td>
<td>P₁ &lt; 0.7</td>
</tr>
<tr>
<td>1</td>
<td>Slightly</td>
<td>0 &lt; Igeo &lt; 1</td>
<td>1 &lt; Cf</td>
<td>6 ≤ mC₄ &lt; 12</td>
<td>1 &lt; P₁ &lt; 2</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>Moderately</td>
<td>2 &lt; Igeo &lt; 3</td>
<td>1 &lt; Cf</td>
<td>6 ≤ mC₄ &lt; 12</td>
<td>1 &lt; P₁ &lt; 2</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>Moderately – Strongly</td>
<td>3 &lt; Igeo &lt; 4</td>
<td>1 &lt; Cf</td>
<td>6 ≤ mC₄ &lt; 12</td>
<td>1 &lt; P₁ &lt; 2</td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>Strongly</td>
<td>4 &lt; Igeo &lt; 5</td>
<td>1 &lt; Cf</td>
<td>6 ≤ mC₄ &lt; 12</td>
<td>1 &lt; P₁ &lt; 2</td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>Severely</td>
<td>4 &lt; Igeo &lt; 5</td>
<td>1 &lt; Cf</td>
<td>6 ≤ mC₄ &lt; 12</td>
<td>1 &lt; P₁ &lt; 2</td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>Extremely</td>
<td>Igeo ≥ 5</td>
<td>1 &lt; Cf</td>
<td>6 ≤ mC₄ &lt; 12</td>
<td>1 &lt; P₁ &lt; 2</td>
<td></td>
</tr>
</tbody>
</table>

Abbreviation: C₄ – Contamination degree; P₁ – Nemerow pollution index; Igeo – Geo-accumulation index; Cf – Contamination factor (Adapted from Kolawole et al., 2018; Brady et al., 2015)
elemental concentration for K ranged from 19200 to 223700 mg kg\(^{-1}\), V ranged from 455 to 5311 mg kg\(^{-1}\), Cr ranged from 381 to 3282 mg kg\(^{-1}\) and Mn ranged between 700 and 18900 mg kg\(^{-1}\). Additionally, the concentration of Fe ranged 26800 to 547900 mg kg\(^{-1}\), Ni ranged from 138 to 302 mg kg\(^{-1}\), Cu from 138 to 970 mg kg\(^{-1}\) and Zn ranged from 55 to 469 mg kg\(^{-1}\).

Table 3. XRF Elemental Concentration in Rock samples (mg kg\(^{-1}\))

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>K</th>
<th>V</th>
<th>Cr</th>
<th>Mn</th>
<th>Fe</th>
<th>Ni</th>
<th>Cu</th>
<th>Zn</th>
<th>Pb</th>
</tr>
</thead>
<tbody>
<tr>
<td>RK 1</td>
<td>39440</td>
<td>564</td>
<td>721</td>
<td>1510</td>
<td>38200</td>
<td>374</td>
<td>126</td>
<td>118</td>
<td>43</td>
</tr>
<tr>
<td>RK 2</td>
<td>49990</td>
<td>598</td>
<td>538</td>
<td>4060</td>
<td>57570</td>
<td>253</td>
<td>289</td>
<td>124</td>
<td>64</td>
</tr>
<tr>
<td>RK 3</td>
<td>256910</td>
<td>1893</td>
<td>2169</td>
<td>11140</td>
<td>296090</td>
<td>394</td>
<td>8574</td>
<td>248</td>
<td>6675</td>
</tr>
<tr>
<td>RK 4</td>
<td>34200</td>
<td>572</td>
<td>343</td>
<td>650</td>
<td>22490</td>
<td>311</td>
<td>132</td>
<td>59</td>
<td>7</td>
</tr>
<tr>
<td>RK 5</td>
<td>33290</td>
<td>472</td>
<td>472</td>
<td>80</td>
<td>750</td>
<td>278</td>
<td>136</td>
<td>74</td>
<td>11</td>
</tr>
<tr>
<td>RK 6</td>
<td>219510</td>
<td>3125</td>
<td>2548</td>
<td>9640</td>
<td>252410</td>
<td>223</td>
<td>5864</td>
<td>357</td>
<td>4719</td>
</tr>
</tbody>
</table>

Mean 105556.7

Max 256910

Min 33290

SD 103601.7

Table 4. XRF Elemental Concentration in Soil Samples (mg kg\(^{-1}\))

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>K</th>
<th>V</th>
<th>Cr</th>
<th>Mn</th>
<th>Fe</th>
<th>Ni</th>
<th>Cu</th>
<th>Zn</th>
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<tbody>
<tr>
<td>SL 1</td>
<td>38390</td>
<td>3301</td>
<td>902</td>
<td>8500</td>
<td>223400</td>
<td>338</td>
<td>2563</td>
<td>463</td>
<td>1922</td>
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<tr>
<td>SL 2</td>
<td>31910</td>
<td>539</td>
<td>1278</td>
<td>500</td>
<td>360</td>
<td>130</td>
<td>490</td>
<td>98</td>
<td>147</td>
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<td>SL 3</td>
<td>124230</td>
<td>5290</td>
<td>4955</td>
<td>11900</td>
<td>367500</td>
<td>130</td>
<td>490</td>
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<td>147</td>
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<tr>
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<td>114650</td>
<td>3849</td>
<td>964</td>
<td>6380</td>
<td>291600</td>
<td>202</td>
<td>1419</td>
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<td>990</td>
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<tr>
<td>SL 5</td>
<td>151600</td>
<td>309</td>
<td>591</td>
<td>12400</td>
<td>340600</td>
<td>282</td>
<td>1627</td>
<td>665</td>
<td>912</td>
</tr>
<tr>
<td>SL 6</td>
<td>129580</td>
<td>3583</td>
<td>516</td>
<td>1800</td>
<td>90000</td>
<td>3495</td>
<td>1175</td>
<td>479</td>
<td>141</td>
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<td>29920</td>
<td>201</td>
<td>530</td>
<td>380</td>
<td>23500</td>
<td>23480</td>
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<td>18</td>
</tr>
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<td>32650</td>
<td>478</td>
<td>540</td>
<td>740</td>
<td>44800</td>
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<td>152</td>
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<td>37150</td>
<td>526</td>
<td>473</td>
<td>1040</td>
<td>35900</td>
<td>212</td>
<td>239</td>
<td>200</td>
<td>73</td>
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<td>SL 10</td>
<td>136230</td>
<td>2822</td>
<td>837</td>
<td>11330</td>
<td>338290</td>
<td>318</td>
<td>615</td>
<td>485</td>
<td>36</td>
</tr>
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<td>SL 11</td>
<td>20180</td>
<td>638</td>
<td>584</td>
<td>1040</td>
<td>44820</td>
<td>367</td>
<td>188</td>
<td>108</td>
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<td>SL 12</td>
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<td>4029</td>
<td>789</td>
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<td>250990</td>
<td>328</td>
<td>1056</td>
<td>567</td>
<td>155</td>
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<td>SL 13</td>
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<td>3543</td>
<td>309</td>
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<td>390090</td>
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<td>255000</td>
<td>12480</td>
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<td>2599</td>
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<td>1421</td>
<td>5840</td>
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</table>

Mean 107307.3

Min 20180

Max 359810

SD 89552.6

Pb recorded the minimum concentration of 7 mg kg\(^{-1}\) at Anka Town while the maximum was recorded at Dan Kamfani 2 with a value of 187 mg kg\(^{-1}\). Generally, the least concentrations per site was recorded against Anka.
Town while the highest was recorded Dan Kamfani 2. The elemental concentration for samples collected from Abare 2 and Anka River was low.

The elemental concentrations measured from plant samples are summarized in Table 5. Samples were collected from 3 locations, including Abare, Daki Takwas and Dareta. From the data presented, the minimum concentration of K was 15470 mgkg$^{-1}$ while the maximum was 87430 mgkg$^{-1}$. The Concentration of V ranged from 38 to 648 mgkg$^{-1}$, Cr ranged from 311 to 1240 mgkg$^{-1}$, Mn ranged from 380 to 10780 mgkg$^{-1}$ and Fe ranged from 24450 to 172670 mgkg$^{-1}$.

Additionally, the concentration of Ni ranged from 226 to 291 mgkg$^{-1}$, Cu ranged from 95 to 979 mgkg$^{-1}$ and Zn ranged from 38 to 648 mgkg$^{-1}$. The range of Pb concentration was recorded as 12 mgkg$^{-1}$ (Abare) to 97 mgkg$^{-1}$ (Daki Takwas). Among the sampled location, plant/vegetables from Abare recorded the least elemental concentration while Daki Takwas recorded the highest concentrations (Daki Takwas).

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>K</th>
<th>V</th>
<th>Cr</th>
<th>Mn</th>
<th>Fe</th>
<th>Ni</th>
<th>Cu</th>
<th>Zn</th>
<th>Pb</th>
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<td>12</td>
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<tr>
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<td>3672</td>
<td>1240</td>
<td>2800</td>
<td>172670</td>
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<td>97</td>
</tr>
<tr>
<td>PT 3</td>
<td>15470</td>
<td>571</td>
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<td>10780</td>
<td>74930</td>
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<th>Sample ID</th>
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<th>Cr</th>
<th>Mn</th>
<th>Fe</th>
<th>Ni</th>
<th>Cu</th>
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<td>172670</td>
<td>291</td>
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<tr>
<td>Min</td>
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<td>134</td>
<td>311</td>
<td>380</td>
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<td>226</td>
<td>95</td>
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<td>75355.3</td>
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<td>351.9</td>
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<tr>
<th>Sample ID</th>
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<th>Cr</th>
<th>Mn</th>
<th>Fe</th>
<th>Ni</th>
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<td>34.0</td>
<td>508.1</td>
<td>351.9</td>
<td>48.2</td>
</tr>
</tbody>
</table>

As shown in Table 8, the maximum mean concentrations in Cu and Pb with 2520 mgkg$^{-1}$ and 1920 mgkg$^{-1}$, were recorded against rock samples while soil samples accounted for the maximum mean concentration in Cr, Mn, Fe, Ni, Zn. On the other hand, the minimum mean concentrations were recorded against plant samples, except for Mn which value was marginally lower in rock samples.

From the mean concentrations of elements for all the samples, there seems to a trend exists; while K and Fe have higher concentrations, the concentrations of Cr, Ni, Cu, Zn, and Pb were observed to be lower. The variation in the concentration of elements in the samples could be due to the distribution of elements in the various media, the geology of the study area, which is dominantly schist, hosting the lead mineralization, and the lead-copper-silver-gold poly-metallic association (Buba, 2016). The high concentration of K could be attributed to the application of fertilizers for agricultural purposes. The elemental concentration could be linked to the disparity in the parent materials at the various sampling sites and other factors such as hydrology and the actual mining and
processing locations, which is in agreement with the argument put forward by Towett et al. (2015) as shown in Table 7.

Table 7. Mean elemental concentration in rock, soil, sediment and plant measured in mg/kg and those of other studies in the world

<table>
<thead>
<tr>
<th>Description</th>
<th>K</th>
<th>V</th>
<th>Cr</th>
<th>Mn</th>
<th>Fe</th>
<th>Ni</th>
<th>Cu</th>
<th>Zn</th>
<th>Pb</th>
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<td>1204</td>
<td>1132</td>
<td>4513</td>
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<td>Soil</td>
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<td>198514</td>
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<td>185125</td>
<td>240</td>
<td>452</td>
<td>204</td>
<td>84</td>
</tr>
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<td>Plant</td>
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<td>1459</td>
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<td>4653</td>
<td>90683</td>
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<td>242</td>
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<tr>
<td>Okpoko (Nig)b</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>17.1</td>
<td>1437.1</td>
<td>1.52</td>
<td>9</td>
<td>24.55</td>
</tr>
<tr>
<td>Soil (Africa)b</td>
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<td>1400</td>
<td>45900</td>
<td>20</td>
<td>30</td>
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<td>Soil (Africa)c</td>
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<td>64</td>
<td>466</td>
<td>27954</td>
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<td>-</td>
<td>-</td>
<td>-</td>
<td>30</td>
<td>60</td>
<td>100</td>
</tr>
</tbody>
</table>

a Nwankwoala and Emenu, 2018
b Towett et al., 2013
c Towett et al., 2015
d Gebeyehu and Bayissa, 2020
e Kacholi and Sahu, 2018
f Rahman et al., 2015
g Reported by Towett et al., 2015
h Reported by Kapile and Makundi, 2016

The concentration of elements in this study for soil was compared with other studies carried out in the world and is presented in Table 7. The trend of elemental concentrations in soil were obtained as Fe > K > Ni > Mn > Cr > V > Cu > Pb > Zn. This does not translate to the fact the concentration of the elements such as Cu, Pb and Zn were low. Kapile and Makundi, (2016) reported the tolerable maximum limits of some chemically toxic elements in soil measured in mg/kg as follows: Cu (30), Zn (60), Pb (100) and Cr (400) as shown in Table 6. The results of the study exceed these limits, which suggest very high toxicity levels despite the seemingly low concentration compared to Fe, K and Ni. Exposure to metal/metalloid mixtures of Pb, Cr and Cd have been reported to produce more severe effects at both relatively high dose and low dose levels (Tchounwou et al., 2012). K and a couple of elements are regarded as nutrients to the plants/vegetables and much less toxic.

Furthermore, the results obtained were generally higher than those of other studies. Hence, the results for K, Cr, V, Ni, Zn, and Pb were highest among elements determined and well above the world mean values given in Table 6. The mean concentration of Mn was 6321 mg/kg\(^{-1}\) obtained in this study was higher than the world mean of 418 mg/kg\(^{-1}\) and the results reported by Towett et al. (2015; 2013) but lower than that reported by Rahman et al. (2015) for Bangladesh. For Cr, the mean concentration in soil was 1401 mg/kg\(^{-1}\); the value was many times higher than the MTL of 400 mg/kg\(^{-1}\) (Kapile and Makundi, 2016) as well as results reported in Africa by Towett et al. (2015) and in Bangladesh by Rahman et al. (2015).

Pb recorded a mean concentration of 685 mg/kg\(^{-1}\) for soil, which is greater than the World mean of 25 mg/kg\(^{-1}\) and the maximum tolerable limit of 100 mg/kg\(^{-1}\). The mean concentration of Pb presented by Udiba et al. (2019) in Dareta was higher than those obtained in this study; 1404.57±141.00 mg/kg\(^{-1}\) and 6724.68±84.00 mg/kg\(^{-1}\) obtained from abandoned and active processing mills, respectively. The concentration of Pb obtained from rock in Dareta 1 and Dareta 4 was 6675 and 4719 respectively, which is much similar to the result by Udiba et al. (2019). This is a proof that the mined rock ores contribute significantly to the high concentrations of Pb in Dareta and Anka in general.

Similarly, the mean concentration of Cu obtained in this study (1231 mg/kg\(^{-1}\)) was higher than the world mean and the maximum tolerable limit (Kapile and Makundi, 2016) as well as those reported by Towett et al. (2015, 2013) as presented in Table 7.
The data obtained from studies conducted by Rahman et al. (2015) in Bangladesh showed that the mean elemental concentrations in soil for Fe (957533 mgkg\(^{-1}\)) and Zn (11640 mgkg\(^{-1}\)), were higher than other studies, including the present study with 198514 mgkg\(^{-1}\) and 362 mgkg\(^{-1}\) respectively. Conversely, Ni recorded 19014 mgkg\(^{-1}\) as the mean concentration in soil; the world mean and the results presented by Towett et al. (2015, 2013) all ranged between 18 and 20 mgkg\(^{-1}\).

<table>
<thead>
<tr>
<th>Sample</th>
<th>Cr</th>
<th>Mn</th>
<th>Fe</th>
<th>Ni</th>
<th>Cu</th>
<th>Zn</th>
<th>Pb</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rock</td>
<td>1132</td>
<td>4513</td>
<td>111252</td>
<td>306</td>
<td>2520</td>
<td>163</td>
<td>1920</td>
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<tr>
<td>Soil</td>
<td>1226</td>
<td>6321</td>
<td>198514</td>
<td>2846</td>
<td>1231</td>
<td>362</td>
<td>685</td>
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<tr>
<td>Sediment</td>
<td>970</td>
<td>6486</td>
<td>185125</td>
<td>240</td>
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<td>Plants</td>
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<td>90683</td>
<td>253</td>
<td>392</td>
<td>242</td>
<td>41</td>
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</tbody>
</table>

The result of Nwankwoala and Ememnu, (2018) on Okpoko, Southeastern Nigeria shows the least concentrations of heavy metals in soil when compared to the present study. This is commonly as a result of the difference in geological formation in the study areas. The southeastern region of Nigeria is known to have a sedimentary formation, where sandstone and shale can be found compared to the basement complex formation in North-West Nigeria (Joshua et al., 2009). Moreover, the mining and mining processing activities contribute significantly to the high heavy metal concentration in the study area. This position is supported by Udiba et al. (2019).

The concentrations obtained in this study are well beyond the maximum tolerable limits. Hence, appropriate measures should be taken to create awareness on the hazards of the toxic elements in the mining and processing areas. Measures should be taken in protecting the local population (especially children) from undue contamination from these heavy metals.

### 3.2. Assessment of Single Element Indices

For single element analysis, the contamination factor (Cf) and the geo-accumulation index (\(I_{geo}\)) were employed. The computed contamination factor of different heavy metals analyzed for soil and sediment in the study area is presented in Table 9 and Table 10 respectively. Figure 2 shows that the soil is severely contaminated with Ni (41.86), Pb (34.32), Cu (24.61), Cr (12.26) and Mn (7.43) in their respective order. Only Zn (3.81) is shown to be strongly contamination.

The sediments show severe contamination from Cr, Cu and Mn with Cf of 9.70, 9.05 and 7.63 respectively. Pb (4.21) and Ni (3.53) fell within the category of strongly contaminated and moderately contaminated with Zn.

Generally, the Cf of computed in soil is higher than that of sediments as shown in Figure 3; this is because the enrichment of heavy metal in soil is higher than in sediments. Hence, the enrichment of heavy metals higher in soil is than in sediments.

The geo-accumulation indices of some heavy metals were calculated to assess the level of contamination of the samples and by extension, the artisanal mining environment, the threshold values are presented on Table 1. The extent of metal contamination is assessed in terms of geo-accumulation index; this has been divided into seven classes based on the increasing numerical value of the index according to Kolawole et al. (2018), Brady et al. (2015) and Al-Haidarey (2010). The classification of the geo-accumulation index ranges from uncontaminated to very strongly contaminated.

The highest index of 7 was recorded for Ni from soil sample while the least index of 0.2 was recorded for Zn from rock sample. For Cr, the \(I_{geo}\) was 3 for rock, soil and sediment samples indicating moderately to strongly contaminated while plant/vegetable returned an index of 2, meaning moderately contaminated. The value of \(I_{geo}\) for Mn was 2 for all the samples, it is moderately contaminated. Rock and Plant samples recorded an index of 4 for Fe.
signifying strongly to severely contaminated. The $I_{geo}$ for soil and sediment was 5, which means severely contaminated.

![Figure 2. Contamination factor of soil and sediment in Anka](image)

For Ni, the $I_{geo}$ was 2 for rock (moderately contaminated), 7 for soil (extremely contaminated), 1 for plant/vegetables and sediment (slightly contaminated). The geo-accumulation index for Cu decreased according to the samples; rock (5), soil (4), sediment (3) and plant/vegetable (2). This implies that the level of contamination decrease from extremely contaminated in rock to moderately contaminated in plant/vegetable.

![Figure 3. Relationship between contamination degree and the modified contamination degree in soil](image)
Comparatively, the $I_{geo}$ for Zn was low; ranging from slightly contaminated for rock, sediment and plant samples to moderately contaminated in soil. On the other hand, the $I_{geo}$ of Pb was above 5 for rock and soil, signifying extremely contaminated. Sediment was moderately contaminated (1) while plant/vegetable was slightly contaminated with 0.5.

Geo-accumulation index values of Cr, Mn, Fe, Ni, Cu and Pb were all ranged between 2 and 6 for rock samples, indicating that moderately contaminated to very extremely contaminated with these metals. Pb, which is a poisonous contaminant, ranked the highest with 6 while Cu was second with 5; both, however, belong to the most risky group, extremely contaminated. The $I_{geo}$ of Fe (4) > Cr (3) > Mn (2) and Ni (2). The values were generally
higher than those obtained at the Matla power plant by Okedeyi et al., (2014) with Pb (2.32), Fe (0.005) Mn (0.03) and Ni (0.65).

![Figure 4. Relationship between contamination degree and the modified contamination degree in Sediment](image)

**Table 5.** Pollution index in soil sample of Anka

For soil, the $I_{geo}$ for Ni was 7, Fe was 5 and Pb was 5, all belonging to Class 7. The trend from high to low shows that the $I_{geo}$ of Cu > Cr > Mn > Mn > Zn; the level of contamination ranged from moderately contaminated to extremely contaminated. In comparison with the studies by Santos-Francés et al. (2014) in Andes Mountain Range, Peru, soils were not included in Classes 3, 4, 5, and 6 (from moderately contaminated to very strongly contaminated). Hence, the risk of heavy metal contamination in the study area is significantly high.

The geo-accumulation index for heavy metals in the sediment samples varied according to the elements. Fe was the highest with $I_{geo}$ of 5 while Zn was the least with $I_{geo}$ of 0.5 according to the classification by Kolawole et al. (2018); Brady et al. (2015) and Al-Haidarey (2010). The trend of $I_{geo}$ was Fe > Cr and Cu > Mn > Ni and Pb > Zn. The contamination risk of heavy metals in the sediment samples can be said to be high and as such sediments in the study area can be said to be highly contaminated with heavy metals.

Plant samples were not left out in the contamination by heavy metals; while the $I_{geo}$ of Zn and Pb were classified as uncontaminated to moderately contaminated, Fe recorded the highest $I_{geo}$ for plants with 4 - strongly
contaminated to very strongly contaminated. The level of contamination was however lower than those of rock, soil and sediments.

The single element assessment indices show that heavy metals, which originate from the artisanal mining; the heavy metals present in the ores also find their way into the environment through indiscriminate and unregulated disposal of mine tailing and other waste materials. The implication of this is that the active mining and mineral processing enhance the spread of harmful heavy metal contaminants in the environment to a very high degree.

### Table 11. Ecological risk factor (Er) and potential ecological risk index (RI) of soils in Anka

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Cr</th>
<th>Mn</th>
<th>Ni</th>
<th>Cu</th>
<th>Zn</th>
<th>Pb</th>
<th>Mean</th>
<th>RI</th>
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<td>7.50</td>
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<td>247.50</td>
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<td>20.74</td>
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<td>SL 13</td>
<td>61.98</td>
<td>22.80</td>
<td>9.12</td>
<td>169.40</td>
<td>3.20</td>
<td>146.75</td>
<td>68.87</td>
<td>413.25</td>
</tr>
<tr>
<td>SL 14</td>
<td>18.26</td>
<td>7.58</td>
<td>917.65</td>
<td>356.70</td>
<td>5.78</td>
<td>649.75</td>
<td>325.95</td>
<td>1955.71</td>
</tr>
<tr>
<td>SL 15</td>
<td>28.42</td>
<td>6.87</td>
<td>18.46</td>
<td>325.90</td>
<td>4.51</td>
<td>657.25</td>
<td>173.57</td>
<td>1041.40</td>
</tr>
</tbody>
</table>

### Table 12. Ecological risk factor (Er) and potential ecological risk index (RI) of Sediment in Anka

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Cr</th>
<th>Mn</th>
<th>Ni</th>
<th>Cu</th>
<th>Zn</th>
<th>Pb</th>
<th>Mean</th>
<th>RI</th>
</tr>
</thead>
<tbody>
<tr>
<td>SD 1</td>
<td>20.20</td>
<td>21.18</td>
<td>15.96</td>
<td>57.90</td>
<td>3.11</td>
<td>25.50</td>
<td>23.97</td>
<td>143.84</td>
</tr>
<tr>
<td>SD 2</td>
<td>7.62</td>
<td>1.76</td>
<td>18.46</td>
<td>22.50</td>
<td>0.58</td>
<td>24.75</td>
<td>12.61</td>
<td>75.67</td>
</tr>
<tr>
<td>SD 3</td>
<td>18.32</td>
<td>10.24</td>
<td>10.15</td>
<td>60.00</td>
<td>2.94</td>
<td>21.25</td>
<td>20.48</td>
<td>122.89</td>
</tr>
<tr>
<td>SD 4</td>
<td>7.78</td>
<td>0.82</td>
<td>22.21</td>
<td>16.10</td>
<td>0.77</td>
<td>1.75</td>
<td>8.24</td>
<td>49.43</td>
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<tr>
<td>SD 5</td>
<td>8.70</td>
<td>1.88</td>
<td>16.25</td>
<td>13.80</td>
<td>0.68</td>
<td>3.00</td>
<td>7.39</td>
<td>44.32</td>
</tr>
<tr>
<td>SD 6</td>
<td>19.28</td>
<td>1.76</td>
<td>19.12</td>
<td>75.90</td>
<td>3.49</td>
<td>41.00</td>
<td>26.76</td>
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<tr>
<td>SD 7</td>
<td>65.64</td>
<td>22.24</td>
<td>17.57</td>
<td>97.00</td>
<td>4.94</td>
<td>46.75</td>
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<td>7.72</td>
<td>1.16</td>
<td>21.54</td>
<td>18.60</td>
<td>0.71</td>
<td>4.25</td>
<td>9.00</td>
<td>53.98</td>
</tr>
</tbody>
</table>

### 3.3. Assessment of Multi-element Indices

The assessment of multiple elements was carried out by evaluating the contamination degree (Ca), the modified contamination degree (Cm), and the pollution index (PI). The contamination degree of ranged from 15.6 to 407.30. About 80% of the sites fell within the category of extremely contaminated while the remaining 20% were strongly contaminated. In applying the modified contamination degree index, the values ranged from 2.61 to 67.88. 20% of
the sites are extremely contaminated, 27% were severely contaminated, 20% were strongly contaminated. 13% were moderately to strongly contaminated and the remaining 20% were moderately contaminated. The relationship between the contamination degree and the modified contamination degree in soil is presented in Figure 3.

The Pollution index of soil in the study area indicated values from 4.09 to 247.72. Although all the sites fell under the category of severely polluted, with all the sites exceeding Anka Town (SL 7), Dareta 3 (SL 14) and Dareta (SL 15) were ranked highest in that order.

Table 6. Pollution index in soil sample of Anka

As presented in Table 10 for sediments, contamination degree of ranged 13.49 to 92 with 50% of the site indicating severely contaminated and the remaining 50% falling within the category of strongly contaminated. On the other hand, the modified contamination degree index ranged from 2.25 to 15.37. 25% of the site was within the strongly contaminated category. 25% were also moderately to strongly contaminated while 50% were moderately contaminated. Figure 4 shows the variation of $C_d$ and $mC_d$ in sediment samples of the study sites.

The Pollution index for sediment ranged from 3.46 to 25.62 as shown in table 10; this indicates that all the sites are severely contaminated just like in soil. The highest values were found in Dan Kamfani 2 (SD 7), Dan Kamfani 1(SD 6) and Abare BF (SD 3) in corresponding order, as shown in Figure 4.

This results obtained in this study is similar to the earlier studies conducted in industrial area of Southwest Nigeria by Kolawole et al. (2018), where the soil and sediments were described as severely polluted.

3.4. Assessment of Ecological Risk Index

Table 11 and Table 12 summarize the ecological risk factor ($E_r$) and the potential ecological risk index (RI) of Cr, Mn, Ni, Cu, Zn and Pb in soil and sediment of the study area. For soil, of the 15 sampling sites, the highest mean ecological factor and the highest potential ecological risk index of 325.95 and 1955.71 were recorded at the site of SL14 respectively. These are followed by SL7 and SL 15 with mean $E_r$ of 292.58 and 173.57 respectively, and RI of 1755.47 and 1041.40. The least mean $E_r$ and RI values were obtained in SL8 (9.35 and 56.11), SL11 (9.35 and 63.83) and SL9 (10.63 and 21.51).

Considering the soil pollution level assessment of Cr, Mn, Ni, Cu, Zn and Pb, based on the ecological risk factor presented in Table 2, 47% of the sites can be considered as low, 26.7% is moderate, and 20% is considerable and 6.7% is high. On the other hand, 33.3% of the sites recorded low level of potential ecological risk with heavy metals, 13.3% was moderate, 26.7% was high and 26.7% was very high. The $E_r$ and the RI pollution assessment indices are however not exactly the same based ecological risk. The $E_r$ here seems to be skewed towards low ecological pollution risk while RI gives a wider spread, that is, from low to very high. Hence, the application of only one of the indices might lead to a wrong conclusion. The two indices can best be complementary to each other.

In sediment, the maximum mean of $E_r$ in the 8 sampling sites was 42.36 in Dan Kamfani 2 (SD 7) while the minimum was 7.39 from Anka River (SD5). The ecological risk factor was low for 87.5% of the sites while 12.5% was moderate. On the other hand, the RI ranged from 44.32 to 160.56. Of the 8 sampling sites, 75% had low
potential ecological risk while 25% has moderate potential ecological risk. The results for Er and RI in sediment agree better than in soil. However, the result further confirms higher potential ecological risk in soil than in sediment.

![Graph of heavy metal contributions in soil](image1)

![Graph of heavy metal contributions in sediment](image2)

**Figure 7.** Average contribution of Heavy Metals to the Ecological Risk of Anka

### 4. CONCLUSION

This study examined the concentration of heavy metals in rocks, soils, sediments and plants in artisanal mining area of Anka. The estimated mean elemental concentrations of the heavy metals indicate that the values obtained in this study are well beyond the maximum tolerable limits and the world average reported by the World Health Organization. The concentration of Pb was particularly noted to be higher with the analysis of rock and soil samples.

Different contamination, pollution and ecological risk indices have been employed to assess the environmental risk of heavy metals in the study area. The results of $I_{geo}$ revealed that severe heavy metals contamination in rock and soil. This is associated with the geology and the anthropogenic activities in the study area. The contamination of the soil originates from the parent ores (rocks) that are excavated and brought to the surface, which finds its way into the environment and are dispersed in air, absorbed in the soil, deposited in sediments and absorbed by plants. The absorption in plants was found to be least among the sample types. Using the pollution index, the soil and sediments was categorized as severely polluted but the enrichment of heavy metal was observed to be higher in soil than in sediment. The assessment of contamination factor and potential ecological risk from heavy metal in Anka majorly comes from Pb, Ni and Cu. Zn presented the least values. The potential ecological risk is higher soil than in sediments.

The use of single and multiple element indices allowed the assessment of individual heavy metal and the sampling locations. Among the study areas, the active mining sites found in Dareta, Daki Takwas, Dan Kamfani and the processing areas of Abare, Dan Kamfani, Dateta and Abare were identified as areas with high heavy metal pollution risk.
5. RECOMMENDATIONS

With the level of contamination of the various sample types with heavy metals, there is a need to ensure adequate protection of the local populace and the worker as ingestion of contaminated materials or inhalation of dust particles containing these heavy metals poses a significant health risk. The Ministry of Mines and Steel Development and Ministry of Environment should collaborate to educate and create the needed awareness on the risk associated with artisanal mining and processing in the area using crude methods.

Agricultural activities such as crop planting and animal grazing should be restricted, especially in the mining areas where the concentration of heavy metals in soil and plants are beyond recommended levels. This will reduce the risk of consuming vegetables and meat contaminated with heavy metals. It is also recommended further studies should be conducted in the study area to assess the health risk associated with the consumption of water and vegetables in the study area.

Conflict of Interest Statement
The authors declare no conflict of interest.

REFERENCES


