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ORIGINAL ARTICLE

Physico-chemical Properties and Heavy metal Contamination Levels of Soils from Riruwai Mining Area, North-western Nigeria

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Abstract

Mining activities, despite their economic importance, are well documented as one of the most significant sources of heavy metal pollution in the soil. In the present study, the physico-chemical properties and heavy metal contamination levels of the soils around the Riruwai mining area, in north-western Nigeria, were investigated. A total of 60 surface soil samples from four (4) sampling sites, which include 18 from active mine sites, 12 from abandoned mine sites, 21 from farmlands and 9 from control sites, were collected during the dry and rainy seasons. The physico-chemical properties such as pH, electrical conductivity (EC), soil organic matter (SOM), and cation exchange capacity (CEC) were determined using various analytical techniques. The concentrations of heavy metals were analyzed using a Microwave Plasma Atomic Emission Spectrometer (MP-AES), and the measured concentrations were used to evaluate the pollution load index (*PLI*). The findings of the study revealed that mining activities had a significant impact on the physicochemical characteristics of the soils, with lower pH, CEC, and SOM values in particular in active and abandoned mining sites. The physico-chemical characteristics of soil vary seasonally, with higher values typically reported during the rainy season, except for pH. The concentrations of arsenic, cadmium, lead, and zinc in the soils of the Riruwai mining area exceeded the threshold value recommended by WHO/FAO (2001) in all the sampling locations and seasons, with the exception of the control sites. The *PLI* values indicated that the soils in active and abandoned mining sites are strongly polluted by heavy metals, while farmland soils are moderately polluted and control site soils are unpolluted. The soils in the study area are significantly contaminated by heavy metals, particularly in active and abandoned mining sites, and the contamination has spread to farmland, suggesting a significant need for proper containment of heavy metal pollution levels in the area, particularly arsenic, cadmium, lead, and zinc.

Keywords: Heavy metals, mining activities, physico-chemical properties, Riruwai mining area, pollution load index

Introduction

Mining activities, despite their economic importance, are well documented as one of the most significant sources of heavy metal pollution in the soil (Sun et al., 2018; Sihotang et al., 2021). Heavy metals are released into the environment due to mining operations through erosion, weathering, and leaching (Rodríguez et al., 2009). Once absorbed by the body, heavy metals continue to accumulate in key organs such as the kidney, liver, brain, liver, and bones for many years, causing serious health challenges (Kabata-Pendias, 2011; Yuan et al., 2014). For example, acute exposure to arsenic can cause nausea, abdominal pain, muscle spasms and diarrhea, whereas chronic exposure can cause diabetes. Lead, on the other hand, causes renal cancers and interferes with the normal functioning of the reproductive and nervous systems (Ebrahimi et al., 2020). According to Kim et al. (2016), mercury can harm the endocrine system, cause kidney damage, and cause respiratory failure. Cadmium has been linked to breast, hepatic, pancreatic, lung, and skin cancers (Buha et al., 2017). Asthma and shortness of breath are caused by inhaling high levels of chromium (IV) compounds. Similarly, nickel is known to cause both oral and intestinal cancer. It is also linked to depression, heart attacks, haemorrhages, and kidney problems. Even though zinc is essential for human life, excessive consumption may have non-carcinogenic effects on human health. It has the potential to impair growth and reproduction (Cao et al., 2010). The routes of exposure to heavy metals of the people living near the mining area are reported to be direct ingestion, dermal contact, inhalation, and consumption of vegetables, water, and animals (Halatek et al., 2014).

Soil, as an essential component of the ecological system, functions as a carrier of heavy metals as well as a medium for heavy metals to spread to the atmosphere, organisms, and water bodies (Adedeji et al., 2019). Heavy metals in soils are potential environmental threats that can harm human health via a variety of absorption pathways, including direct ingestion, dermal contact, and inhalation (Wu et al., 2017). Soils near mining sites are most likely contaminated by effluents and tailings in mining areas (Wang et al., 2019). Several studies have underscored the importance of soil physico-chemical properties on soil quality and the behaviour of heavy metals in soils (Keskin & Makineci, 2009; Fashola et al., 2020). The concentrations of heavy metals in the soils depend on the soil's physico-chemical properties like pH, electrical conductivity, organic matter, and cation exchange (Bhatti et al., 2016). High organic matter content, for example, can enhance metal adsorption, reducing mobility in the soil. While acidic soil conditions (lower pH) reduce soil cation exchange capacities and increase metal solubility in the soil environment, metals become more mobile and toxic (Ayangbenro & Babalola 2017).

There are numerous studies on the degree of heavy metals contamination in the environment caused by mining activities in Nigeria and various parts of the world (Kamunda et al., 2016; Chukwu & Oji, 2018; Prematuri et al., 2020; Fagbenro et al. 2021; Yao et al., 2021). However, to the best of our knowledge, no scientific research has been conducted to assess the physicochemical properties and heavy metal contamination levels in the soils of the study area. For this reason, this study was aimed at investigating the physico-chemical properties and heavy metal contamination levels of the soils around the Riruwai mining area, north-western Nigeria. The study will provide baseline data on the degree of heavy metal contamination in the soils of the study area and other comparable locations for use by policy-makers, mining companies, and the general public.

Materials and Methods

Description of the study area

Riruwai, the Doguwa Local Government Area headquarters, is located in Kano state, northwerstern Nigeria. It is situated between latitude 10°43"97"N - 10°45'01"N and longitude 8°43"3"E - 8°47'39" E. (Rilwanu, 2017). Riruwai is one of Nigeria's younger granite complexes. The complex is an example of eroded roots of an alkaline volcano that formed as part of an early Jurassic chain of anorogenic centers (Ogunleye et al., 2006). According to the 2006 census, Riruwai has a population of 150,645 people (NPC, 2006). Riruwai is predominantly a mining community. Large scale mining began in 1979 and was expanding rapidly with close to 900 tons of Zinc and tin ore production per day. The mine was closed after five years of continued operation. Artisanal and small-scale mining are, however, still taking place in the area. The research carried out by Nigerian Mining Cooperation revealed that close to five million tons of mineral ore containing tin and zinc were found in the Riruwai community. An ore mineralogy study also confirmed the presence of large deposits of columbite, granite, copper, zinc, lead, and uranium (Amosu, 2021). Figure 1 depicts the geographical map of Riruwai.

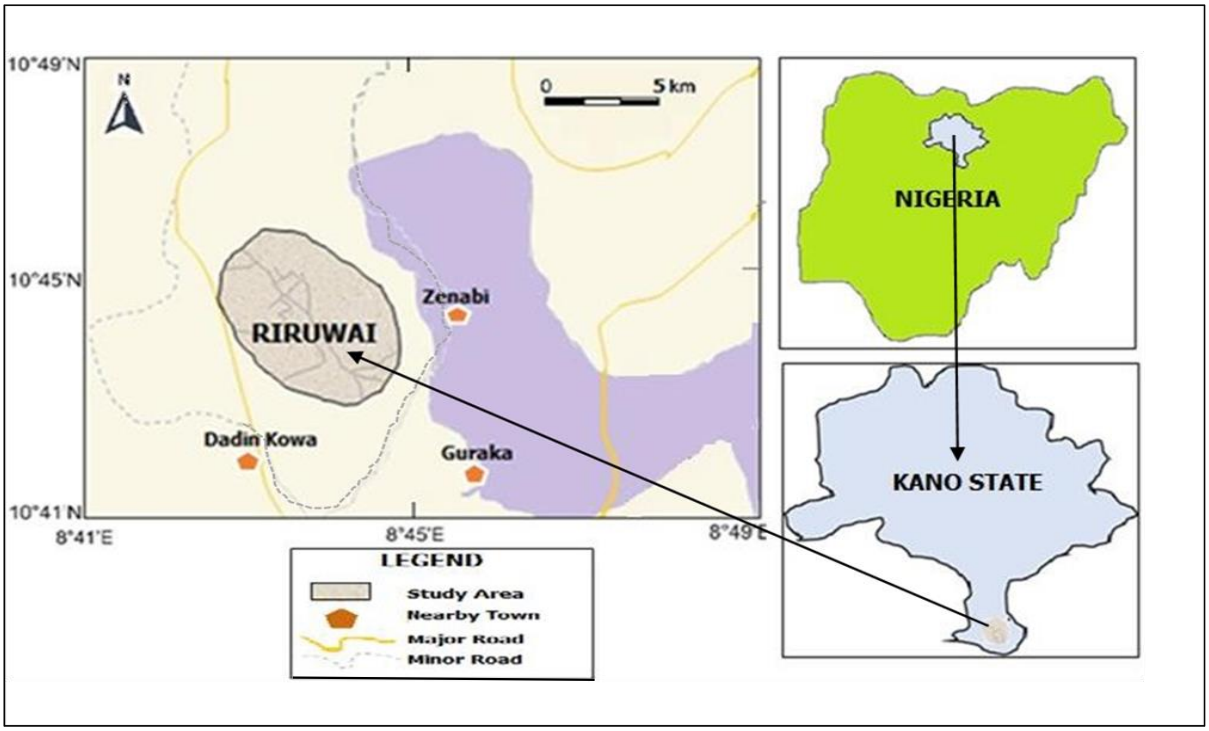

Figure 1. Map showing the study area

Soil Sampling and Treatment

A total of 60 surface soil samples (0-20 cm) were collected from four (4) sampling sites, including 18 from active mine sites, 12 from abandoned mine sites, 21 from farmlands, and 9 from control sites, using a steel soil auger in two different seasons, namely the rainy season (August, 2020) and the dry season (February, 2020). In order to obtain representative and uniform samples,

each soil sample was obtained by mixing 3 sub-samples at each sampling point to form a composite sample. A 1.00 kg sample from the mixed samples was selected by quartering, stored in a polyethylene bag, labelled and transported to the laboratory. In the laboratory, soil samples were air-dried at room temperature and the large fragments, plant roots and leaf residues, stones, and gravel were removed. The soil samples were pulverized, passed through a 0.15 mm nylon screen, sieved and homogenized (Fan & Wang, 2017).

Determination of Physico-chemical Parameters of the Soil

The pH of the soil was measured using pH meter (Hanna HI9921) as described by Beane et al. (2016): a 4.00 g of each sample was agitated in 10.00 cm^3 of deionized water using a 25 cm^3 centrifuge tube and left overnight. The pH of the supernatant was measured and calibrated at pH 4.00 and 7.00. Electrical conductivity was measured based on the procedure outlined by Sani et al. (2012) as follows: 25.00 g of air-dried soils sample was placed into a 250 cm^3 beaker and 50.00 cm^3 of deionized water was added slowly drop by drop evenly over the entire soils surface until the soils appeared wetted. A stainless steel spatula was used to form a homogeneous soils saturated paste. The beaker was then covered with a petri-dish; 50.00 cm^3 deionized water was added and agitated for one hour. The diluted extract (40.00 cm^3) was placed into a 100 cm^3 beaker and the conductivity meter (Hanna HI198331) was inserted and the electrical conductivity of the soils was recorded in µS/cm. For cation exchange capacity (CEC) measurement, a 10.00 g of soil sample was weighed into a 100 cm^3 plastic beaker. A 40.00 cm^3 of 1.00 mol/dm^3 ammonium ethanoate solution pH 7.00 buffers was added and the suspension was stirred with a glass rod and left overnight. It was suction-filtered with 55 mm Buchner funnels. The residue from filtration was leached with 25.00 cm^3 portions of 1.00 mol/dm^3 NH4Cl solutions. The solution was discarded and the electrolyte washed out of the sample with 150.00 cm^3 concentrated ethanol. The sample was allowed to drain completely and leached gradually with acidified NaCl (acidified with dilute HNO_3) to a 250.00 cm^3. A 50.00 cm^3 of 1.00 % boric acid was measured into a 250 cm^3 conical flask and 3 drops of mixed indicator were added. The acidified NaCl leachate was transferred into a 500 cm^3 Kjeldahl flask and 10.00 cm^3 of 1.00 mol/dm^3 NaOH and anti-bumping granules were added. The leachate was distilled over boric acid solution. Ammonium borate distillate (1.50 cm^3) was titrated with standard 0.10 mol/dm^3 HCl and the CEC was determined according to the following equation (Todorovi et al., 2001):

$$CEC(cmol|kg) = \frac{(Titre-Blank)\times M\times 100}{Weight\ of\ the\ sample} \quad (1)$$

A loss-on-ignition method adopted by Bakr & El-Ashry (2018) was used to determine the organic matter of the soil. In this method, 3.00 g of the air-dried soils were heated in the oven at 105 ºC for 24 hours, cooled in a desiccator, and weighed to obtain the oven-dried weight. The samples were combusted at 550 ºC for 2 hours in a muffle furnace. After combustion, the samples were cooled in a desiccator and weighed. The soil organic matter (*SOM*) was computed based on the Schulte & Hopkins (1996) equation:

$$SOM = \frac{SW_{OD}-SW_{AC}}{SW_{OD}} \times 100\ \% \quad (2)$$

Where *SOM* is the soil organic matter, SW_{OD} is the oven-dry weight of the soil sample, and SW_{AC} is the weight after combustion at 550 ºC.

Determination of heavy metals in the soil

The soil sample was digested with a mixture of three concentrated acids (HNO_3, HF, and $HClO_4$) as reported by Fan & Wang (2017), briefly as follows: A soil sample (0.050 g) was placed in a 200 cm^3 Teflon beaker and 10.00 cm^3 of HNO_3, 10.00 cm^3 of HF, and 10.00 cm^3 of $HClO_4$ were added. The mixture was heated on an electric hot plate at 180 °C until the solution was almost dried. The beaker was removed and cooled to room temperature. A 5.00 cm^3 of concentrated HNO_3, 5.00 cm^3 of HF, and 5.00 cm^3 of $HClO_4$ were added, and the mixture was boiled until dense white fumes appeared. The beaker was removed, cooled, and 15.00 cm^3 of deionized water was added and boiled for 5 minutes. The digest was filtered using Whatman filter paper No. 42 into a 50 cm^3 volumetric flask. The wall of the beaker was washed three times with 10.00 % dilute HNO_3. The solution was made up to the mark with HNO_3 for heavy metal analysis. A blank solution was treated and prepared in a similar way. The concentrations of As, Cd, Cr, Hg, Mn, Ni, Pb, and Zn were analyzed at their respective wavelengths using a Microwave Plasma Atomic Emission Spectrometer (MP-AES, Model: Agilent 4200). The concentrations of heavy metals were reported in mg/kg.

Pollution Load Index (PLI)

The pollution load index (*PLI*) was used to identify multi-element contamination. It was calculated for every sampling site using the equation adopted by Khalifa and Gad (2018):

$$PLI = \left(C_f^1 \times C_f^1 \times C_f^1 \times \ldots C_f^n\right)^{\frac{1}{n}} \quad (3)$$

Where: n = Number of metals investigated and C_f^n = Contamination factor for n^{th} element. The value of contamination factor (C_f) was computed as follows:

$$C_f = \frac{C_{sample}}{C_{background}} \quad (4)$$

Where, C_{sample} = concentration of heavy metal in sample, $C_{background}$ = concentration of the heavy metal in the background.

Quality control and *statistical analysis*

Analytical grade reagents were used throughout the study. All glassware and plastic containers were soaked overnight in 10% (v/v) nitric acid, washed three times with deionized water, and oven dried. The containers were further dried in a desiccator for 20 minutes before being used for analysis (Ashraf et al., 2011). All samples were analysed in triplicate. The standard solution of each heavy metal was prepared by successive dilution of certified standards (1000

mg/dm^3) procured from Sigma Aldrich and a calibration curve for each heavy metal was constructed. The calibration curves with $R^2 > 0.998$ were accepted for concentration calculation. Blank determinations were run to correct any background contamination from reagents, filter papers, or any other systemic source of error. All data were analysed using the mean standard deviation method. One-way Analysis of Variance (ANOVA) was carried out using SPSS 23.0 (SPSS Inc., Chicago, USA). One-way Analysis of Variance (ANOVA) and correlation analysis were used to compare heavy metal and physico-chemical parameter contents from the different sampling sites. A statistically significant difference was established using the Tukey HSD post-hoc test ($p \leq 0.05$). Microsoft Excel 2013 was used in plotting graph.

Results and Discussion

Physico-chemical Parameters of Soils

The physicochemical properties of the soils at the active mining site, abandoned mining site, farm, and control site during the dry and rainy seasons are presented Tables 1 and 2. The pH values of the soil during the dry season ranged from 6.05 (active mining site) to 6.78 (control site), with the highest value recorded at the control site while the lowest was found at the active mining sites. The pH values of 6.26 (abandoned mining site) and 6.48 (farm) fell between the lowest and highest observed values. During the rainy season, the pH values varied from 5.72 (active mining sites) to 6.52 (control sites). The pH values of 5.96 (an abandoned mining site) and 6.03 (farms) lied between the highest and lowest experimental values. The pH values observed in this study were closer to the published literature values of 5.91 (mining sites) and 6.85 (control sites) determined in the soils of the Ilesha gold mining site in Osun state, Nigeria (Abiya et al., 2019). The pH values of the soils in all the sampling locations were generally higher during the dry season and decreased in the following order: active mining sites > abandoned mining sites > farms > control sites. The formation of sulfuric acid from the oxidation of pyrite in the ore may be responsible for the low pH values in the active and abandoned mining sites compared to farms and control sites (Boularbah et al., 2006). The acidic nature of mining site soils, particularly during the rainy season, increases the mobility of potentially toxic heavy metals and hence their potential to harm the environment (Ngole-Jeme & Fantke, 2017).

The electrical conductivity (EC) values ranged from 700.68 (control site) to 4400.76 µS/cm (active mining site), with 2300.02 (farm) and 2500.31 µS/cm (abandoned mining site) falling between the lowest and highest values during the dry season. During the rainy season, the EC values spread from 1000.52 µS/cm (control site) to 4600.11 µS/cm (active mining site). The EC values of 2520.02 (farm) and 2607.93 µS/cm (abandoned mining site) fell between the extreme values. The findings of this research revealed that EC values decreased during the dry season and increased during the rainy season. This was consistent with the findings of Salem et al. (2020), who reported similar trends when they studied the physiochemical properties and concentration of heavy metals in agricultural soils fertilized with chemical fertilizers.

The soil organic matter (SOM) ranged from 1.11 (active mining site) to 11.13 (control) with 2.04 (abandoned mining site) and 4.08 (farm) falling between the highest and lowest observed values determined during the dry season. During the rainy season, the SOM varied from 1.62 (active mining site) to 13.93 % (control site). The percentage organic matter values of 2.27 % (abandoned mining site) and 4.62 % (farm) fell between the maximum and minimum experimental values. The observed values of SOM were slightly lower than reported literature values of 4.7–6.7 % determined in the soils of Draa Lasfar Mine in Marrakech, Morocco (Yassir & Alain, 2016). The highest percentage of SOM is generally observed during the rainy season, with the maximum

value recorded at the control site and the minimum at the active mining sites. For tropical soils, organic matter levels of less than 3.44 % are considered very low (Landon, 1991). The soil samples analysed in active and abandoned mining sites were low in organic matter based on this rating. Prematuri et al. (2020) discovered comparable findings when they investigated the impact of nickel mining on soil properties and the growth of two fast-growing tropical tree species.

The cation exchange capacity (CEC) of the soil varied from 12.59 to 37.45 cmol/kg and 13.87 to 42.35 cmol/kg in the dry and rainy seasons, respectively. In the dry season, the highest cation exchange capacity value was observed at the control site while the lowest was recorded at the active mining site. A similar pattern was also observed during the rainy season. According to the Landon (1991) classification, a soil with a CEC value of 5–15 cmol/kg is regarded as low for agricultural soils. Based on this rating, the soils in the active mining sites were low in CEC in both seasons. The values decreased in the following order: control site > farm > abandoned mining site > active mining site. Lower CEC values in the active and abandoned mining sites might be attributed to the low content of soil organic matter in the sites (Ngole-Jeme & Fantke, 2017). The present findings corroborate the work of Abah et al. (2014). The CEC represents the soil's ability to absorb or release cations and, as such, is an important parameter in heavy metal-contaminated sites. It measures the soil's ability to bind or hold exchangeable cations (Salem et al., 2020).

Table 1. Physico-chemical parameters of the soil around Riruwai mining area during the dry season

Sampling Location	pH (H_2O)	EC (µs/cm)	SOM (%)	CEC (cmol/kg)
RSS1	6.05 ± 0.02	4400.76 ± 5.17	1.11 ± 0.02	12.59 ± 0.11
RSS2	6.26 ± 0.03	2500.31 ± 2.83	2.04 ± 0.01	17.61 ± 0.17
RSS3	6.48 ± 0.02	2300.02 ± 2.88	4.08 ± 0.02	22.52 ± 0.12
RSS4	6.78 ± 0.04	700.68 ± 1.281	11.13 ± 0.03	37.45 ± 0.45

Values are mean ± standard deviation (n = 3), **RSS1** = Active mining sites, **RSS2** = Abandoned mining sites, **RSS3** = farm, **RSS4** = Control site. **EC** = Electrical conductivity, **SOM** = Soil organic matter, **CEC** = Cation exchange capacity

Table 2. Physico-chemical parameters of the soil around Riruwai mining area during the rainy season

Sampling Location	pH (H_2O)	EC (µs/cm)	SOM (%)	CEC (cmol/kg)
RSS1	5.72 ± 0.01	4600.11 ± 6.51	1.62 ± 0.02	13.87 ± 0.07
RSS2	5.96 ± 0.04	2607.93 ± 2.65	2.27 ± 0.03	20.01 ± 0.14
RSS3	6.03 ± 0.03	2520.02 ± 4.05	4.62 ± 0.03	24.22 ± 0.11
RSS4	6.52 ± 0.02	1000.52 ± 2.33	13.93 ± 0.06	42.35 ± 0.26

Values are mean ± standard deviation (n = 3), **RSS1** = Active mining sites, **RSS2** = Abandoned mining sites, **RSS3** = farm, **RSS4** = Control site. **EC** = Electrical conductivity, **SOM** = Soil organic matter, **CEC** = Cation exchange capacity

Heavy Metal Concentrations in the Soils

The levels of heavy metals in four sampling locations (active mining site, abandoned mining site, farmland, and control site) during dry and rainy seasons are shown in Tables 3 and 4. The concentrations of arsenic during the dry season ranged from 13.40 (control site) to 35.23

mg/kg (active mining site), with 21.54 mg/kg (farm area) and 33.72 mg/kg (abandoned mining site) falling between the lowest and highest experimental values.

The highest level of arsenic was found in the active mining area, while the lowest value was observed at the control site. The concentration of arsenic was above the threshold value of 20.00 mg/kg recommended by WHO/FAO (2001) except in the control site, which implied that there was considerable arsenic contamination. During the rainy season, the concentrations of arsenic ranged from 7.62 mg/kg (control site) to 29.12 mg/kg (active mining site). The levels of 13.09 mg/kg (farm) and 18.50 mg/kg (abandoned) fell between the highest and lowest values of arsenic determined. Except at the active mining site, the level of arsenic in all the sampling locations was below the WHO/FAO (2001) permissible limit. Ameh et al. (2020) observed a similar pattern of higher HM concentrations during the dry season than during the rainy season when they studied the seasonal variations of toxic metals in the soils of Okaba coal mining area, in Kogi, Nigeria. Eludoyin et al. (2017) also observed similar trends when they studied the effects of artisanal mining activities on soil properties in Itagunmodi, southwestern Nigeria.

The levels of cadmium (mg/kg) determined varied from 0.91 (control site) to 13.50 (active mining site). The concentrations (mg/kg) of 1.51 (farm) and 5.01 (abandoned mining site) lied between the lowest and highest cadmium values obtained during the dry season. The concentration of observed cadmium is above the recommended value of 3.00 mg/kg set by WHO/FAO (2001) in agricultural soils for active and abandoned mining sites and below the recommended value for farm and control areas. During the rainy season, the concentrations of cadmium (mg/kg) spread from 0.07 (control site) to 11.61 (active mining site), with 0.09 mg/kg (farm area) and 2.15 mg/kg (abandoned mining site) falling between the extreme experimental values. The concentrations of cadmium in all the sampling sites are below the recommended values, except in the active mining sites where the observed cadmium concentration was more than three times higher than the threshold values. The observed values of cadmium in the present study are slightly lower than reported literature values of 12.62-20.70 mg/kg determined in soils around abandoned Pb-Zn mines in Yelu in Bauchi state, northern Nigeria (Sanusi et al., 2017).

The concentration of chromium spread from 59.52 to 116.05 mg/kg during the dry season. The highest concentration (59.52 mg/kg) was observed at the active mining site, while the lowest concentration (59.52 mg/kg) was found at the control site. The experimental values of 96.44 mg/kg (farm site) and 103.37 mg/kg (abandoned mining site) fell between the highest and lowest obtained concentrations. The observed concentrations of chromium in the active and mining sites were slightly higher than the WHO/FAO (2001) threshold concentration of 100.00 mg/kg, whereas the chromium levels in the farm and control sites were lower than the threshold value. During the rainy season, the levels of chromium ranged from 38.50 to 97.24 mg/kg, with 71.36 mg/kg (farm) and 84.73 mg/kg (abandoned mining) values falling between the observed extremes. The levels of chromium in all the samples were below the threshold value of 100.00 mg/kg established by WHO/FAO (2001). The concentrations of chromium during the rainy season were generally lower than during the dry season. The experimental chromium values were relatively close to the literature values of 46.00 to 123.00 mg/kg reported by Fazekašová & Fazekaš (2020) when they studied the soil quality and heavy metal pollution assessment of iron ore mines in Nizna Slana, Slovakia. Olarinoye et al. (2021) also found a chromium content of 151.14 mg/kg in the soils of the Jos mining area, which was relatively similar to the experimental values of 116.05 mg/kg and 103.37 mg/kg found in active and abandoned mining sites, respectively, during the dry season.

The levels of mercury in the soils varied from 0.01 (control site) to 1.01 mg/kg (active mining site), with 0.50 mg/kg (farm) and 0.84 (abandoned mining site) falling between the lowest and highest observed values during the dry season. During the rainy season, the mercury concentrations ranged from 0.01 (control site) to 0.35 mg/kg (active mining site). The values of 0.36 mg/kg (farm) and 0.67 mg/kg (abandoned mining site) fell between the lowest and highest concentrations of mercury determined. Focus et al. (2021) reported similar results of 0.05 mg/kg mercury in soil in the mining area of the Rwamagasa gold mine in Tanzania's Geita region. The concentrations of mercury in all the sampling locations are below the threshold value of 2.00 mg/kg set by WHO/FAO (2001) in both dry and rainy seasons, denoting less or no apparent contamination of soil by mercury. Mercury and its compounds affect the nervous system. Increased mercury exposure can alter brain functions and cause tremors, irritability, memory problems, and changes in hearing or vision (Obasi et al., 2020).

The observed concentrations of manganese (mg/kg) during the dry season ranged from 115.19 to 314.49. The active mining site had the highest concentration (314.49), while the control site had the lowest concentration (115.19). The experimental values of 148.11 and 203.04 are between the lowest and highest values of manganese determined in the soils. Manganese concentrations observed during the rainy season are lower than those observed during the dry season, but the difference is not statistically significant. The values are 256.00, 155.03, 96.00, and 114.15 for active mining sites, abandoned mining sites, farms, and control sites, respectively. The observed manganese concentrations were much lower than the WHO/FAO (2001) background concentrations of 2000.00 mg/kg in both seasons as well as all sampling locations. This could imply that the soils in the study area were not contaminated with manganese. The observed values of manganese in the present study are much lower than reported literature values of 189.00-2007.00 mg/kg determined in soils around the pegmatite mining sites at Olode area, Ibadan, southwestern Nigeria (Okonkwo et al., 2021).

The levels of nickel in the soil investigated spread from 11.04 (control site) to 26.04 mg/kg (active mining site), with values of 18.31 mg/kg (farm) and 24.50 mg/kg (abandoned mining site) lying between the extreme experimental values determined during the dry season. During the rainy season, the levels of nickel ranged from 7.34 mg/kg (control site) to 24.43 mg/kg (active mining site). The observed values of 11.45 mg/kg (farm) and 13.09 mg/kg (abandoned mining site) fell between the lowest and highest concentrations determined. Okonkwo et al. (2021) reported a similar literature value of nickel (22.33 mg/kg) found in pegmatite mining sites in Olode and its environs in Ibadan, Southwestern Nigeria. In all the seasons and sampling locations, the experimental values of nickel are below the threshold value of 50.00 mg/kg recommended by international standards (WHO/FAO, 2001). This indicates that the soils of the Riruwai mining area are not polluted by nickel.

The concentrations of lead ranged from 43.11 to 245.00 mg/kg. The highest concentration of 245.00 mg/kg was observed at the active mining site, while the lowest concentration of 43.11 mg/kg was recorded at the control site. The concentrations of 87.21 mg/kg and 89.30 mg/kg were between the lowest and highest concentrations. The observed values of lead in the present study are substantially lower than reported literature values of 46.00 to 6100.00 mg/kg determined in the soil Pb/Zn mining area of north-western Spain (Monterroso et al., 2014). All the sampling locations and seasons, with the exception of the control site, had higher lead concentrations than the WHO/FAO (2001) threshold value of 50.00 mg/kg, suggesting soil lead contamination in the study area.

The concentrations of zinc during the dry season (mg/kg) spread from 135.09 (control site) to 1653.40 (active mining site), with the concentrations of 426.51 and 1248.90 falling within the

observed range. During the rainy season, the levels of zinc (mg/kg) ranged from 115.30 to 1394.56. The concentrations of 367.05 and 731.18 fell between the highest and lowest observed concentrations. Similar results of 1035.2 mg/kg of zinc in the surface soil of Irankouh zinc–lead mine, Isfahan, Iran, were reported by Jahromi et al. (2020). Ezeh & Chukwu (2011) found a comparable concentration of 1460.5 mg/kg in the soils of the Ishiagu mining district in South Eastern Nigeria. The levels of zinc in all the sampling sites and seasons exceeded the WHO/FAO (2001) recommended value of 300.00 mg/kg, with the active and abandoned mining sites having significantly higher concentrations, while the farm sites had moderately higher concentrations. An exception was found in the control sites.

Generally, the concentrations of arsenic, cadmium, chromium, mercury, manganese, nickel, lead, and zinc in active and abandoned mining sites were significantly higher ($p \leq 0.05$) than in farm and control sites, with the control sites having the lowest values. The heavy metal concentrations in soils decreased in the following order: active mining site > abandoned mining site > farm > control site. This study confirmed previous research that heavy concentrations of active and abandoned mining sites are significantly higher than the rest of the locations (Obasi et al., 2020). High concentrations of Pb, Zn, and As observed in the farm area beyond their respective safe limits indicate the spread of heavy metal pollution from the mining sites. Acidic drainage and dust transport by wind may be the primary mechanisms causing pollution dispersion (Rodr guez et al., 2009). Heavy metal concentrations in the soils were found to be higher in the dry season than in the rainy season. The high levels of heavy metals in the dry season could be due to dilution, dispersion, high mobility, adsorption, oxidation, hydrolysis, and rapid precipitation (Ameh et al., 2021).

Table 3. Heavy metal concentrations (mg/kg) in the soils of Riruwai mining area during the dry season

Heavy Metals	Sampling Location				WHO/FAO (2001)
(mg/kg)	RSS1	RSS2	RSS3	RSS4	(mg/kg)
As	$35.23^{a} \pm 9.01$	$33.72^{a} \pm 7.43$	$21.54^{b} \pm 8.81$	$13.40^{c} \pm 4.02$	20.00
Cd	$13.50^{b} \pm 3.42$	$5.01^{b} \pm 1.11$	$1.51^{b} \pm 0.06$	$0.91^{b} \pm 0.14$	3.00
Cr	$116.05^{c} \pm 20.13$	$103.37^{c} \pm 17.52$	$96.44^{c} \pm 30.75$	$59.52^{c} \pm 10.71$	100.00
Hg	$1.01^{d} \pm 0.003$	$0.84^{d} \pm 0.09$	$0.50^{e} \pm 0.09$	$0.01^{f} \pm 0.00$	2.00
Mn	$314.49^{e} \pm 16.36$	$203.04^{e} \pm 28.63$	$148.11^{e} \pm 47.56$	$115.19^{e} \pm 27.11$	2000.00
Ni	$26.04^{f} \pm 6.05$	$24.50^{f} \pm 9.05$	$18.31^{f} \pm 5.32$	$11.04^{f} \pm 3.84$	50.00
Pb	$245.00^{g} \pm 31.87$	$89.30^{g} \pm 11.98$	$87.21^{g} \pm 20.15$	$43.11^{g} \pm 13.06$	50.00
Zn	$1653.40^{h} \pm 53.25$	$1248.90^{h} \pm 77.22$	$426.51^{h} \pm 35.69$	$135.09^{h} \pm 19.67$	300.00

Values are mean ± standard deviation (n = 3). The values on the same raw with similar superscript letters are statistically the same ($p < 0.05$), whereas values on the same raw with different superscript letters are statistically different ($p < 0.05$), as revealed by one-way ANOVA and the Tukey HSD post-hoc test. RSS1 = Active mining sites, RSS2 = Abandoned mining sites, RSS3 = farm, RSS4 = Control site.

Table 4. Heavy metal concentrations (mg/kg) in the soils of Riruwai mining area during the rainy season

Heavy Metals (mg/kg)	Sampling Location				WHO/FAO (2001) (mg/kg)
	RSS1	RSS2	RSS3	RSS4	
As	$29.12^{a} \pm 8.92$	$18.50^{a} \pm 7.79$	$13.09^{a} \pm 6.02$	$7.62^{a} \pm 0.51$	20.00
Cd	$11.61^{b} \pm 5.01$	$2.15^{b} \pm 0.52$	$0.09^{c} \pm 0.02$	$0.07^{c} \pm 0.02$	3.00

Cr	$97.24^{d} \pm 31.44$	$84.73^{d} \pm 25.81$	$71.36^{d} \pm 17.13$	$38.50^{d} \pm 11.45$	100.00
Hg	$0.35^{e} \pm 0.11$	$0.67^{f} \pm 0.10$	$0.36^{e} \pm 0.01$	$0.01^{e} \pm 0.00$	2.00
Mn	$256.00^{g} \pm 37.53$	$155.03^{g} \pm 33.97$	$96.00^{g} \pm 23.04$	$114.15^{g} \pm 18.19$	2000.00
Ni	$24.43^{h} \pm 8.31$	$13.09^{h} \pm 5.04$	$11.45^{h} \pm 4.18$	$7.34^{h} \pm 1.05$	50.00
Pb	$237.00^{i} \pm 30.09$	$82.33^{i} \pm 19.56$	$66.01^{i} \pm 16.14$	$39.07^{i} \pm 11.28$	50.00
Zn	$1394.56^{j} \pm 93.30$	$731.18^{j} \pm 41.15$	$367.05^{j} \pm 55.11$	$115.30^{j} \pm 28.38$	300.00

Values are mean ± standard deviation (n = 3). The values on the same raw with similar superscript letters are statistically the same ($p < 0.05$), whereas values on the same raw with different superscript letters are statistically different ($p < 0.05$), as revealed by one-way ANOVA and the Tukey HSD post-hoc test. RSS1 = Active mining sites, RSS2 = Abandoned mining sites, RSS3 = farm, RSS4 = Control site.

Pollution Load Index (PLI) of Heavy Metals

Figure 2 shows the values of the *PLI* of studied heavy metals in the active mining sites, abandoned mining sites, farms and control sites in the Riruwai mining area during the dry and rainy seasons. Yu et al. (2014) classified the *PLI* values of heavy metals in the soils. According to their classification, *PLI* of 1 indicates unpolluted soil. A *PLI* of 1 to 2 signifies moderately polluted soil. A *PLI* of 2 to 10 denotes strongly polluted soil, while a *PLI* of 10 indicates extremely polluted soil. In this study, the *PLI* values of heavy metals ranged from 0.96 (control site) to 9.71 (active mining site) during the dry season. *PLI* values of 1.97 (farm) and 5.09 (abandoned mining site) fell between the lowest and highest values observed. *The PLI* values of heavy metals in the active and abandoned mining sites are in the range of 2–10, which indicates that the soils in these locations are strongly polluted by heavy metals. The *PLI* value of the soil in the farm area is within the range of 1-2. This demonstrates that the soil in the studied farm area is moderately polluted with heavy metals. The *PLI* value of the soil in the control site was, however, not polluted by heavy metals (*PLI* > 1). During the rainy season, the PLI values of the studied heavy metals ranged from 0.58 (control site) to 8.65 (active mining site), with values of 1.19 (farm) and 2.72 (abandoned mining site) falling between the lowest and highest extremes. The *PLI* value of the soil at the abandoned mining site is greater than 10. This indicates that the soil at the site is extremely polluted with heavy metals. At the active mining site, the *PLI* value of soil is in the range of 2–10, which indicates strong pollution. The *PLI* value of soil from a farming area fell within the range of 1-2, signifying moderate pollution. At the control site, the *PLI* value of the soil is less than one, which signifies that the soil in that location was not polluted with heavy metals. Active mining sites are the locations with the largest pollution, accounting for 66.00 % of the total load. This is followed by abandoned mining sites (21.00 %), farms (9.00%), and the control site had the lowest pollution load (4.00 %). The results corroborate with the findings of Chukwu & Oji (2018) who assessed the level, distribution and contamination status of Pb, Zn, As, Ni, Cu, Cr, and Cd in agricultural soils around the settlements of the abandoned Lead-Zinc Mine in Nkpuma Ekwoku, Southeastern, Nigeria. Stephen & Maryann (2020) also made similar observations when they evaluated heavy metal pollution status and risk assessment due to artisanal gold mining activities in Bagega Community, Zamfara state, Northern Nigeria.

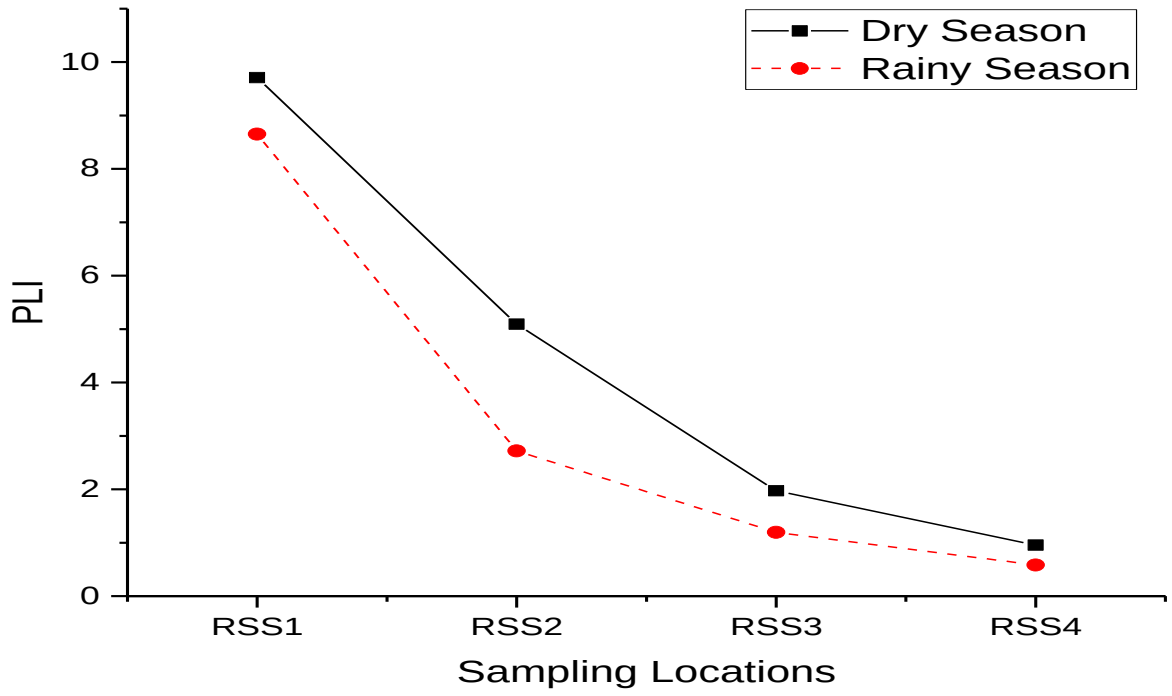

Figure 2. Pollution Load Index (*PLI*) of heavy metals in the soils of Riruwai mining area

Conclusion

The pH, CEC, and SOM, particularly in active and abandoned mining sites, are generally low, with higher values obtained during the rainy season, with the exception of pH. While EC values are higher at active and abandoned mining sites. The concentrations of arsenic, cadmium, lead, and zinc in the soils of the Riruwai mining area exceeded the threshold value recommended by WHO/FAO (2001) in all the sampling locations and seasons, with the exception of the control sites. This indicates that the soils, particularly in the active and abandoned mining sites, are contaminated by these metals. The contamination had spread to farmland. The pollution load index (*PLI*) results confirmed heavy metal contamination of soils in the study area.

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