Indonesian Journal of Environmental Management and Sustainability
e-ISSN:2598-6279 p-ISSN:2598-6260

IJEMS

Research Paper

Source Distribution and Ecological Risk Assessments of Heavy Metals from the Soils of Riruwai Mining Area, North-Western Nigeria

Hamza Badamasi[1*], Aminu Dauda[1], Abdullahi Khalil Suleiman[1], Sa'adatu Eri Muhammad[1], Naseer Inuwa Durumin-Iya[1], Hadiza Usman Abdullahi[1]

[1] *Department of Chemistry, Faculty of Science, Federal University Dutse, Dutse, Jigawa State, 7156, Nigeria*
***Corresponding author e-mail**: hamza.badamasi@fud.edu.ng

Abstract
Mining is an important global economic driver, contributing to human civilization's advancement and foreign exchange revenues. However, it causes significant environmental contamination, particularly in developing nations with inadequate waste management and ineffective mining legislation. In the present study, the source distribution and ecological risk of heavy metals (HMs) from the soils of the mining area around Riruwai town, northern Nigeria, were assessed. Soil samples were obtained from active and abandoned mining sites, farmlands, and control sites and were analyzed for physicochemical characteristics and HM contents. The measured concentrations of HMs were used to evaluate the source distribution and ecological risks. The study's findings indicated that, with the exception of Mn, Hg, and Ni, all HM concentrations were above threshold levels in all sample locations aside from the control sites. The results of the correlation analysis showed that there was a significant correlation between the HMs, indicating that they came from comparable origins. Principal component analysis and hierarchical cluster analysis results indicated that As, Cr, Hg, Ni, and Zn might originate from similar anthropogenic sources, possibly mining activities, while Cd, Mn, and Pb might come from natural sources (parent material). Results of an ecological risk assessment revealed that the soils from active and abandoned mining sites were severely polluted by HMs. The degree of pollution has begun to extend onto farmland, signifying a critical need for adequate HM pollution control in the study area.

Keywords
Ecological Risk Assessment, Heavy Metals, Mining Activities, Riruwai Mining Area, Source Distribution

Received: 12 December 2023, Accepted: 7 March 2024
https://doi.org/10.26554/ijems.2024.8.1.25-33

1. INTRODUCTION

Mining is a major vehicle of the national and global economies that has made significant contributions to the advancement of human civilization and the provision of foreign exchange earnings, both of which are considered necessary for national economic development (Jerie and Sibanda, 2010; Jain, 2015; Chen et al., 2022). However, mining operations are significant sources of environmental pollution, particularly in developing countries where mine waste management is inadequate and mining regulations are weak (Jiménez-Oyola et al., 2021). Even after it has been closed, mining can still harm the environment by contaminating the air, water, soils, and sediments (Aliu et al., 2016; Fazekašová and Fazekaš, 2020; R et al., 2021).

Metals are natural elements that have been present in the ecosystem since the dawn of civilization. The Bronze and Iron Ages are named after the usage of metals, and their significance is still important to industry and urbanization (Rwiza et al., 2016). Among the 35 naturally occurring metals, 23 have a density higher than 5.00 g/cm^3 and are named heavy metals (HMs) (Li et al., 2017). HMs are non-biodegradable elements that accumulate in the environment and food chain over time, producing substantial public health issues and environmental degradation (Liu et al., 2022). HMs enter the environment via geogenic and human processes such as weathering and mining (Owsianiak et al., 2015). The impact of mining activities on higher HM concentrations is well documented in the literature (Abdul-Wahab and Marikar, 2012; Armah et al., 2014). As an essential component of the terrestrial ecosystem, soil serves as a medium and route for HMs to transfer to water, air, and animal bodies (Adedeji et al., 2019; Zeng et al., 2023). HMs in soils can impair human health through a number of pathways, including direct ingestion, cutaneous contact, diet via the soil-food chain, inhalation, and oral consumption

(Cao et al., 2010; Wu et al., 2017).

Ecological risk assessment is frequently used to determine the degree of a negative impact on the environment and to recommend appropriate management actions. It is a controlling pollution evaluation instrument that integrates HM content with ecological effect, environmental effect, and toxicity (Wang et al., 2018; Bali and Sidhu, 2021). Various ecological risk assessment indices have been widely described in the literature (Saha et al., 2017; Bali and Sidhu, 2021). For example, the potential ecological risk factor (ER) and contamination degree (CD) are two indices that are commonly used and combined to form an integrated potential ecological risk index (IPERI), which is used to provide comprehensive information on the ecological risks of multiple contaminants in a given medium (Baran et al., 2018).

In order to identify possible contamination sources and evaluate the relationships between HMs and physicochemical parameters of the soil, multivariate techniques like Pearson correlation analysis, hierarchical clustering, and principal component analysis have been successfully used in previous studies and are well documented (Hossain et al., 2015; Kumar et al., 2018; Liu et al., 2022; Cao et al., 2023). The multivariate analysis involves the characterization of the distribution, sources, and risks of HMs in the soil (Zhu et al., 2017; Jianfei et al., 2020; Keshavarzi et al., 2021).

There is mounting evidence worldwide that residents of mining areas are experiencing health issues as a result of heavy metal contamination (Aluko et al., 2018; Irzon et al., 2018). For instance, mining activities have resulted in methylmercury and cadmium poisoning in Japan (Aoshima, 2016). In Zamfara State, Northern Nigeria, roughly ten thousand individuals were impacted by lead poisoning, resulting in hospitalizations and over four hundred infant fatalities (Lo et al., 2012). Furthermore, exposure to waste from previous lead and zinc mining caused acute lead exposure for many thousands of people living in Kabwe, Zambia (Yamada et al., 2020). HM contamination has also been confirmed in Enugu, Ebonyi State, Ogun State, and Abeokuta due to coal and gold mining (Merem et al., 2017). If mining activities are not properly monitored and best practices are not put into place, the consequences could be disturbing (Omotehinse and Ako, 2019). Therefore, to protect public health and the environment, it is absolutely essential to evaluate the source distribution and ecological risks of HMs in the soils of the Riruwai mining area. To the best of our knowledge, this was the first study that evaluated the sources and probable ecological risks of HM exposure to soils in the study area. The present study will provide a basis for sustainable management and reclamation of the HM-contaminated sites in the Riruwai mining area and similar places.

2. EXPERIMENTAL SECTION

2.1 Description of the Study Area

Riruwai is situated within latitudes 10°43”97”N and 10°45’01 ”N and longitudes 8°43”3”E and 8°47’39”E. It is located at the far end of southern Kano, in North-western Nigeria (Figure 1). It shares a border with Kaduna and Bauchi (Rilwanu, 2017). The Riruwai is within the ring complex, which is characterized by rough terrain. The complex originated as a component of an earlier Jurassic series of anorogenic centers and is a manifestation of the degraded base of an alkaline volcano (Olasehinde and Ashano, 2014). Riruwai is predominantly a mining community. Mining on a large scale commenced in 1979 and was steadily developing, with over nine hundred tons of zinc and tin ore processed every day. After five years of operation, the mine was shut down. However, small-scale and artisanal mineral extraction continues in the area (Amosu, 2021; Mukhtar et al., 2023). According to a study conducted by the Nigerian Mining Cooperation, close to six million tons of ores comprising zinc and tin were discovered in the Riruwai hamlet.

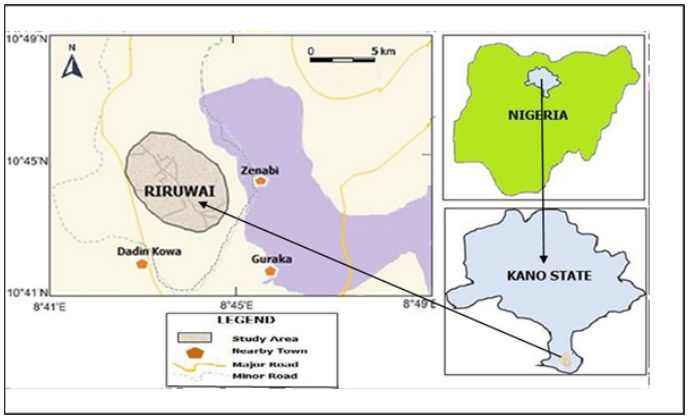

Figure 1. Map of the Study Area

2.2 Chemicals and Instrumentation

2.2.1 Chemicals

In preparing the solutions, analytical-grade reagents (supplied by Sigma-Aldrich) were used throughout the study without further purification. Deionized water was used in the preparation of the reagents throughout the study.

2.2.2 Equipment/Instrument

The following equipment or instruments were used in this study: pH Meter (Hanna HI9921), Conductivity Meter (Hanna HI198331), Hydrometer (152H), Steel Soil Auger (Model 05.08, Royal Eijkelkamp, Netherlands), Deluxe Portable Water and Soil Analysis Kit (Model No. 1024 G), and Microwave Plasma Atomic Emission Spectrometer (Model No. 4200, Agilent USA).

2.3 Soil Sampling and Treatment

Sixty (60) soil samples were obtained at a depth of 0-20 cm from active mine sites (18 samples), abandoned mine sites (12 samples), farmlands (21 samples), and control sites (9 samples) using a steel soil auger. In order to produce a composite sample, each soil sample was obtained by merging three sub-samples at each sampling point. One kilogram sample was chosen from the combined samples, quartered, packed in a plastic bag, labeled, and brought to the laboratory. Soil samples were air dried at room temperature in the laboratory before being crushed, sieved through a 0.15 mm nylon sieve, and homogenized (Fan and Wang, 2017).

2.4 Determination of Physico-chemical Parameters of the Soil

The soil pH was determined at a soil water ration of 1:25 using the method outlined by Beane et al. (2016). Electrical conductivity (EC) was determined using the method described by Sani et al. (2012). The cation exchange capacity (CEC) was measured using the ammonium acetate technique. The loss-on-ignition method was employed for the determination of soil organic matter (SOM) based on the method of Bakr and El-Ashry (2018).

2.5 Determination of Heavy Metals in the Soil

Three acid mixtures (HF, $HClO_4$, and HNO_3) in a ratio of 1:1:1 were employed in the soil sample digestion (Fan and Wang, 2017). A 0.050 g of the soil sample 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 solution was heated, cooled, and boiled with concentrated HNO_3, HF, and $HClO_4$. The digest was filtered and washed three times with 10% HNO_3. The solution was made up to the mark with 10% HNO_3, and the concentrations of HMs were measured using a microwave plasma atomic emission spectrometer. The blank was prepared and treated using a similar procedure.

2.6 Ecological Risk Assessment

To assess the potential ecological risks of heavy metals, this study used Hakanson (1980) Integrated Potential Ecological Risk Index (IPERI). The IPERI method is used to quantify the probable ecological risk of a specific pollutant (Douay et al., 2013). The IPERI consists of three major components: contamination degree (CD), toxic-response factor (TR), and anticipated ecological risk factor (ER). Using this method, the following equations can be used to determine the potential environmental risk index of a single element and the comprehensive potential ecological risk index (RI):

$$C_i^f = \frac{C_i^d}{C_i^n} \tag{1}$$

$$E_R^i = T_R^i \times C_i^f \tag{2}$$

$$IPERI = \sum_{i=1}^{n} E_R^i = [T_R^{As} \times C_f^{As} + T_R^{Cd} \times C_f^{Cd} + T_R^{Cr} \times C_f^{Cr} + ...n] \tag{3}$$

Where C_f^i is the contamination factor, is the analyzed concentration of HM in each sampling point (mg/kg); is HMs geochemical background concentration; is the ecological risk index of a particular HM; $IPERI$ is an integrated potential ecological risk index; and is the biological toxic reference factor of a single HM (Hakanson, 1980). The grading standards for the potential risk of HMs in soils are summarized in Table 1. The values for each metal are arsenic = 13, cadmium = 0.3, chromium = 90, mercury = 80, manganese = 850, nickel = 68, lead = 20, and zinc = 95 (Turekian and Wedepohl, 1961), and those for each are arsenic = 10, cadmium = 30, chromium = 2, mercury = 40, manganese = 1, nickel = 5, lead = 5, and zinc = 1 (Douay et al., 2013).

Table 1. Classification of Potential Ecological Risk of Heavy Metals (Douay et al., 2013 and Mandeng et al., 2019)

$IPERI$ Category	Classification
< 150	Low risk
150 to 300	Moderate risk
300 to 600	Considerable
600 to 1200	High risk
≥ 1200	Very high risk

2.7 Quality Control

Appropriate quality control and assurance strategies were employed to guarantee the reliability of the analytical methods. For this purpose, analytical-grade chemicals were employed, and glassware and plastic containers were soaked in 10% HNO_3 for 24-hours, washed three times with deionized water, and dried (Ashraf et al., 2011). All samples were analyzed in triplicate. Calibration curves with R^2 values greater than 0.999 were created using standard HM solutions, with blank determinations carried out to account for background contamination.

2.8 Statistical Analysis

All analyzed data were treated as mean ± standard deviation. Pearson correlation, principal component, and hierarchical cluster analyses were performed using SPSS 23 (Chicago, United States of America). A one-factor analysis of variance (ANOVA) was employed to compare HM and physico-chemical parameter levels from all the sampling sites. Microsoft Excel 2013 was utilized in graph plotting.

3. RESULT AND DISCUSSION

3.1 Physico-chemical Properties of the Soils

Table 2 shows the results of some physical and chemical characteristics of soil samples. The pH values of the soil varied from 6.05 to 6.78, as shown in Table 2, with the control site having the highest value and the active mining site having the lowest. The pH values obtained in the present study were comparable to the values of 5.91 and 6.85 reported in the soils of the Ilesha gold

Table 2. Physical and Chemical Characteristics of the Soils

Sampling Location	pH (H_2O)	EC (μS/cm)	SOM (%)	CEC (cmol/kg)
Active mining site	$6.05^{a} \pm 0.02$	$4400.76^{a} \pm 5.17$	$1.11^{a} \pm 0.02$	$12.59^{a} \pm 0.11$
Abandoned mining site	$6.26^{b} \pm 0.03$	$2500.31^{b} \pm 2.83$	$2.04^{b} \pm 0.01$	$17.61^{d} \pm 0.17$
Farmland	$6.48^{c} \pm 0.02$	$2300.02^{c} \pm 2.88$	$4.08^{c} \pm 0.02$	$22.52^{b} \pm 0.12$
Control	$6.78^{d} \pm 0.04$	$700.68^{d} \pm 1.281$	$11.13^{d} \pm 0.03$	$37.45^{c} \pm 0.45$

mining site in Osun State, southern Nigeria (Abiya et al., 2018). The observed values were lower than the 7.00–8.30 reported by Paramasivam and Anbazhagan (2020). The production of acidic sulfuric acid during the process of oxidation of pyrite sulfur ore might be the reason for the lower pH values observed at active and abandoned mining sites when in comparison with farmland and the control site (Boularbah et al., 2006; Fashola et al., 2020). The acidic characteristics of mining site soil enhance the mobility of HMs, thereby increasing their effects on the environment (Ngole-Jeme and Fantke, 2017). The pH of soil is important in regulating the solubility and mobility of heavy metals. The majority of HMs were available for plant uptake in an acidic pH condition (Wang et al., 2018). This, therefore, shows that the nutrients in the soil will be available for plant uptake.

The observed electrical conductivity (EC) values varied from 700.68 to 4400.76 μS/cm. The highest value was reported at the active mining site, while the lowest value was found in the control. The findings agreed with those of Salem et al. (2020), who discovered comparable patterns in the physicochemical characteristics and levels of HMs in agricultural soils supplied with synthetic fertilizers. EC is commonly used to detect the presence of dissolved salts in soil and is closely connected to this characteristic since it produces electrical charges from the dissolved ions (Karn et al., 2021).

Soil organic matter (SOM) spread from 1.11 (active mining site) to 11.13% (control), with the highest value obtained in active mining and the lowest in the control site. The SOM values found in the soils of the Draa Lasfar Mine in Marrakech, Morocco, were found to be between 4.70 and 6.70 percent, which is slightly less than the levels described in the literature (Barkouch and Pineau, 2016). Similar results were obtained by Prematuri et al. (2020) in their investigation of the effects of nickel mining on soil characteristics and the growth of two species of rapidly developing tropical trees. For tropical soils, SOM levels less than 3.44% were considered extremely low (Landon, 1991). Based on this ranking, the soil samples examined at active and abandoned mining sites had low SOM content. Low SOM content in soils near active and abandoned mining areas could be due to the absence of plant nutrients in the environment and the acidic nature of tailings, which inhibit vegetation growth and limit the amount of SOM in the study area (Ideriah and Abere, 2017). SOM plays an important role in regulating the mobility of HMs in the soil. It may reduce available HM concentrations in soils through precipitation, adsorption, and complexation processes (Salem et al., 2020).

The cation exchange capacity (CEC) of the studied soil ranged from 12.59 to 37.45 cmol/kg. The highest CEC value was found at the control site, whereas the lowest was observed at the active mining site. A comparable trend was also reported in the rainy season. The CEC of the soil can be classified based on the rating proposed by Landon (1991), and according to his categorization, a soil with a CEC value of 5.00–15.00 cmol/kg is considered low (Ideriah and Abere, 2017). The soil at the active mining site was low in CEC in all the seasons, according to this rating. Lower CEC values at the active and abandoned mining sites could be attributed to the low levels of SOM in the mining locations (Ngole-Jeme and Fantke, 2017). These results corroborate the findings of Abah et al. (2014), Yakubu et al. (2019), and Yinga et al. (2022).

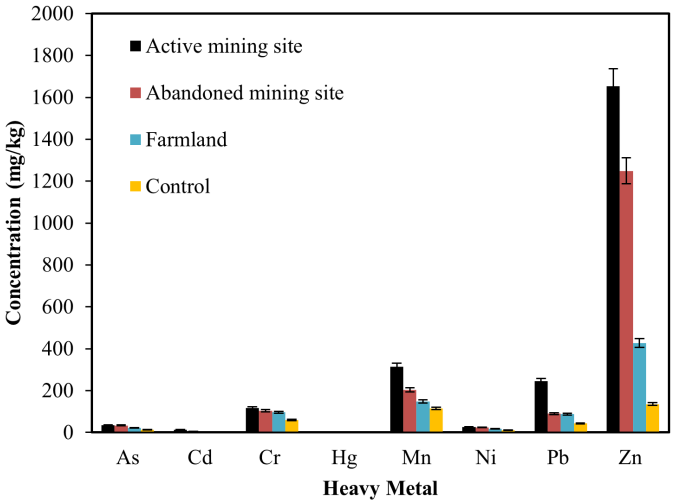

Figure 2. Concentrations of HMs in the Riruwai Mining Area

3.2 Concentrations of HMs in the Soil

The concentrations of HMs in the soil samples from the mining areas of Riruwai are shown in Figure 2. As presented in Figure 2, the concentrations of As, Cd, Cr, Hg, Ni, Mn, Pb, and Zn in soil were significantly varied ($p \leq 0.05$) with the sampling sites, with higher concentrations observed in active mining sites while the lower concentrations were found in control sites. The concentrations of the examined HMs decreased in order: active mining site > abandoned mining site > farmland > control site. The present study corroborated with previous studies that HM levels at active and abandoned mining sites were considerably higher than at other locations (Krishna et al., 2013; Ahmad and Al-Mahaqeri, 2014; Obasi et al., 2020). The levels of Hg, Ni, and Mn were found to be lower than their respective threshold limits of 2.00 and 2000.00 mg/kg, respectively, in both locations and seasons, which demonstrated that the soils in the studied locations were not contaminated with Hg and Mn. Higher levels of As, Pb, and Zn recorded in the farmlands above their threshold

Table 3. Correlation between HM Levels and Physico-Chemical Properties of Soil

	As	Cd	Cr	Hg	Mn	Ni	Pb	Zn	pH	EC	SOM	CEC
As	1											
Cd	0.81	1										
Cr	0.93	0.76	1									
Hg	0.98*	0.82	0.98*	1								
Mn	0.87	0.99**	0.84	0.89	1							
Ni	0.99**	0.79	0.97*	0.99**	0.87	1						
Pb	0.74	0.97*	0.78	0.79	0.96*	0.75	1					
Zn	0.97*	0.91	0.88	0.95*	0.95*	0.95*	0.84	1				
pH	-0.97*	-0.88	-0.96*	-0.99**	-0.94	-0.98*	-0.86	-.97*	1			
EC	0.87	0.92	0.94	0.93	0.96*	0.90	0.95	0.90	-0.96*	1		
SOM	-0.93	-0.71	-0.99**	-0.97*	-0.81	-0.97*	-0.73	-0.86	0.95*	-0.91	1	
CEC	-.95*	-0.780	-0.99**	-0.99*	-0.86	-0.98*	-0.79	-0.91	0.98*	-0.94	0.99**	1

*Correlation is significant at the 0.05 level (two tailed); **Correlation is significant at the 0.01 level (two tailed)

limits could signify the spread of HM pollution from the mining sites. The dispersion of HMs into the farmlands could be caused by wind and acid mine drainage (Rodríguez et al., 2009; Adeniyi et al., 2022).

3.3 Correlation Analysis

As presented in Table 3, pH shows a strong negative correlation with all the studied HMs and EC. However, a strong positive correlation was observed between pH, SOM, and CEC. The negative correlation between pH and the studied HMs implies that the levels of the HMs increase with decreasing pH. This study collaborates with earlier research conducted by Hu et al. (2021), who studied the sources and distributions of HMs in the Nanjing district of China. EC shows a strong positive relationship with all the HMs. This signifies that electrical conductivity influences the availability of all metals. SOM and CEC are found to be positively correlated with each other but negatively correlated with other parameters. A positive relationship between SOM and CEC indicates that when SOM increases, the CEC will increase as well. All the studied HMs were found to be strongly and positively correlated with one another. The present results corroborate the earlier results reported by Yahaya et al. (2021). A strong association between HM levels demonstrates that HMs share similar contamination sources (Li et al., 2017).

3.4 Principal Component Analysis

Principal component analysis (PCA) was applied to evaluate the contribution of different sources and, as a result, to determine the dominant component(s) responsible for pollution depending on their factor loadings (Egbueri et al., 2022). The factor loadings after rotations are classified as follows: strong (greater than 0.70), moderate (0.50-0.70), and weak (less than 0.50) (Elhadi et al., 2017). Two components were extracted from the variable set of data for soil samples during the dry and rainy seasons (Table 4). The first component, which has strong positive loadings on As (0.848), Cr (0.877), Hg (0.864), Ni (0.882), and Zn (0.706), as well as strong negative loadings on pH (-0.791), SOM (-0.911), and CEC (-0.880), accounted for 56.60% of the total variance. The second component shows very strong positive loadings on Cd (0.907), Mn (0.842), Pb (0.903), and EC (0.728), which contribute to 41.32% of the total variance. This demonstrates that As, Cr, Hg, Ni, and Zn might originate from similar anthropogenic sources, possibly mining activities, while Cd, Mn, and Pb might come from natural sources (parent material).

Table 4. PCA Extraction and Component Matrix of HMs

Parameters	Component	
	1	2
As	0.848	0.484
Cd	0.414	0.907
Cr	0.877	0.449
Mn	0.537	0.842
Ni	0.882	0.461
Pb	0.397	0.903
Zn	0.706	0.665
pH	-0.791	-0.611
EC	0.660	0.728
SOM	-0.911	-0.385
CEC	-0.880	-0.466
Eigen values (%)	6.79	4.96
Variance (%)	56.60	41.32
Cumulative (%)	91.51	97.92

3.5 Hierarchical Cluster Analysis

The results of the hierarchical cluster analysis of physical and chemical parameters and HM levels are presented in Figure 3. Three clusters are identified. Cluster one contains As, Cr, Hg, Ni, and Zn. Cluster two contains Cd, Mn, Pb, and EC, while cluster three contains pH, SOM, and CEC. It could therefore be established that As, Cr, Hg, Ni, and Zn may have originated from the same anthropogenic source, namely mining and smelting processes. On the other hand, Cd, Mn, and Pb might have come from natural sources. The hierarchical cluster analysis confirms the results of the principal components analysis.

3.6 Ecological Risk Assessment

Ecological risk assessment quantitatively evaluates the potential detrimental effects of single and multiple HMs on the external environment (Fodoué et al., 2022). To assess the accumulative

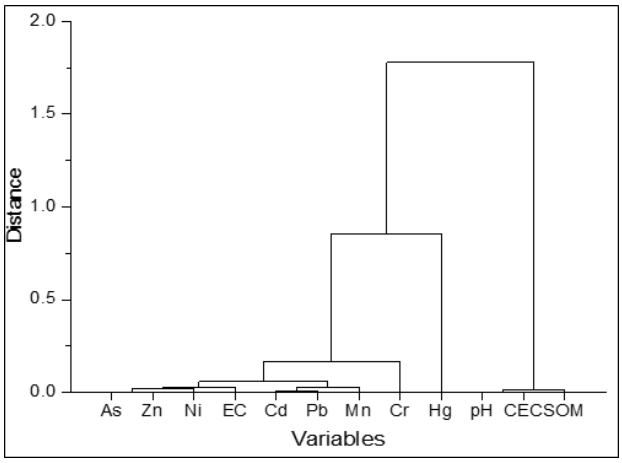

Figure 3. Hierarchical Cluster of Physico-chemical Parameters and HMs in Soils

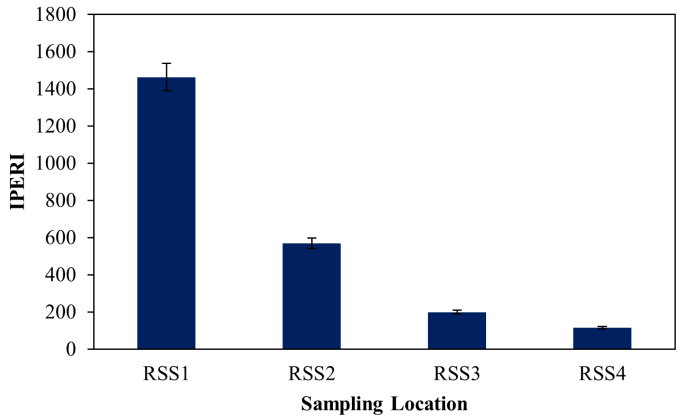

Figure 4. Integrated Potential Ecological Risk Index (*IPERI*) of Heavy Metals in the Soil

effects of HMs, the integrated potential ecological risk index (*IPERI*) is usually employed (Pan et al., 2022; Chen et al., 2023). In the present study, the *IPERI* values of HMs varied from 115.90 to 1462.64, with the highest value recorded in the active mining site, whereas the lowest value was obtained in the control site (Figure 4). The active mining site contributes to 62% of the ecological risk. This was followed by an abandoned mining site (24%), farmland (9%), and a control site (5%) (Figure 5). According to Douay et al. (2013) and Mandeng et al. (2019), *IPERI* values less than 150 indicate low risk, those between 150 and 300 indicate moderate risk, and those between 300 and 600 indicate substantial risk. The *IPERI* between 600 and 120 indicates high risk, while the *IPERI* greater than or equal to 1200 signifies very high ecological risk. In this study, the soils in RSS1 were at very high ecological risk, while the soils in the abandoned mining site were at considerable ecological risk. The soils from farmland were found to have moderate ecological risk, whereas the soil from the control site had low ecological risk. According to the results of the ecological risk assessment, the soils of the study area, particularly those from active and abandoned mining

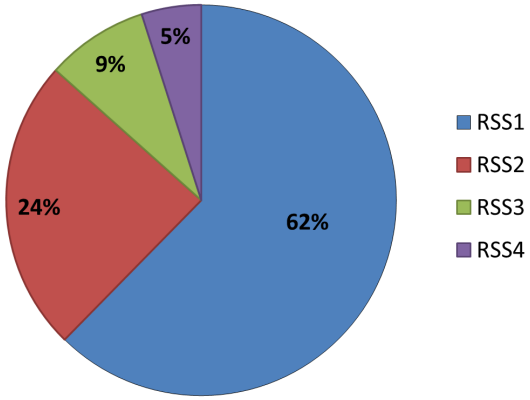

Figure 5. Percentage Contributions of the Sampling Locations to *IPERI* of the Soil

sites, are contaminated by HMs and have the potential to cause ecological risks. The contamination has begun to extend into farmland, which is extremely concerning.

4. CONCLUSIONS

In this study, the source distribution and ecological risk of HMs in the soils of the Riruwai mining area of northern Nigeria were assessed. The results of the study indicated that the soils of the Riruwai mining area, particularly those from active and abandoned mining sites, were polluted by HMs, and the contamination has begun to extend into farmland. Ecological risk assessment revealed that the soils from all the sampling locations except the control sites have the potential to cause significant ecological risk. Multivariable analysis results revealed that HMs may have come from anthropogenic and geogenic sources. The findings of the study would serve as reference criteria for government policy interventions aimed at controlling heavy metal contamination in the area.

5. ACKNOWLEDGEMENT

The authors are grateful to the Tertiary Education Trust Fund (TETfund) of Nigeria for sponsoring this research.

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