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SOURCE APPORTIONMENT AND TOXICOLOGICAL RISK OF POTENTIALLY TOXIC ELEMENTS IN RIVER OSUN ECOSYSTEM WITHIN ILESHA GOLD BELT

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Abstract

The study examined the physico-chemical parameters, ecological and potential human risk associated with metals in sediment and water in Ilesha gold belt Osun state Nigeria. The metal analysis was carried out using inductively Coupled Plasma-Optical emission Spectroscopy (ICP-OES) method. The primary source of contamination was carried out using integrated method such as principal component analysis (PCA). The ecological risk index was used to review the potential ecological risk associated with the elements. The potential health risk assessment was calculated for a lifetime exposure (ingestion) based on the United State Environmental Protection Agency (USEPA, 2011) models to verify the carcinogenic and non-carcinogenic risks for adults. The study established the heavy metal concentrations in water and sediment samples, revealing varying levels of contamination across different metals. Metals such as Fe, Pb and Cd exhibited significant contamination particularly in sediment. The Ecological Risk Index (ERI) values demonstrated that metals like Cd and Pb posed significant ecological risks, particularly in sediment. The Hazard Quotient (HQ) values revealed that Cd, Pb, and Ni posed significant risks, particularly through long-term exposure. The finding suggests that regulatory measures are necessary in region where mining activities takes place in order to protect public health.

Keywords: Source apportionment, toxicological risk, Heavy metals, Contaminants, Potential toxic elements

1.0 Introduction

Toxic elements are naturally occurring or anthropogenic chemical substances that, even in small concentrations, can pose serious risks to human health and the environment. Common toxic elements include heavy metals such as arsenic (As), lead (Pb), mercury (Hg), and cadmium (Cd), which are often released into the environment through industrial activities such as mining, smelting, and improper waste disposal (Twerefou, 2019).

Gold occurs in little concentrations, amounting to 0.0005 ppm, across the globe, largely taking place in its native state and seldom in a combined state with any other minerals, except selenium, bismuth, and tellurium (Obaje *et al*.,2023). It usually occurs in alluvial, eluvial placers, and primary veins from several parts of supracrustal (shale) belts in many parts of the world including Nigeria (Ministry of Mines and Steel Development, 2020). Alluvial gold deposits occur at different times along a river channel, while eluvial gold deposits form the flanks of actively rising antiformal mountain ranges (Youngson *et al.*, 2020). Both alluvial and eluvial deposits are the most common type of placer gold. They are often the richest, as they hold pieces of gold that were washed away from the set down by the force of water and deposited in sediments in or near watercourses or former watercourses (Olajide-Kayode *et al*., 2020). In addition, Consumption of unsafe vegetables can potentially expose humans to metal toxicities (Azeez *et al*., 2020); Aishafran *et al*., 2021). The occurrence of heavy metal pollution from mining activities has grown to be predominantly in places where artisanal gold mining is prevalent (Gnonsoro *et al*., 2022; Marrugo-Madrid *etal*., 2022). Some earlier study has highlighted risks related with the consumption of vegetables planted in/around gold mining activities (Orimoloye & Ololade, 2020; Hao *et al*., 2020; Marrugo-Madrid *et al*., 2022).

2. Materials and Methods

2.1. Materials

Sediment and water samples were collected from four (4) gold mining sites around Ilesha Osun state namely Ora ($7^0$42’30.5”N $4^0$39’59.0”E) near Idominase/Ilase, Ora (7^0 42’15.5”N 4° 40’30.5”E) near Osogbo- Ilesha road, Oke-Ibode (7^0 42’00.0” N $4^0$38’ 00.0”E) and Osogbo ($7^0$45’30.0” N $4^0$33’30.0”E) Southwest, Nigeria

at a depth between 0 and 25cm in the month of September 2024 and transported to the laboratory for analysis. Physicochemical parameters were measured in-situ using the multi parameter probe (total hardness, electrical conductivity, total dissolved solids, pH and dissolved oxygen) and recorded. Using the Ekman grab, sediment samples collected were kept in a Ziplock bag using the procedures outlined by Olayinka-Olagunju *et al.,* (2021) and Huang *et al.,* (2020). Samples were tr

ansported to the laboratory for analysis. The collected sediment samples were air-dried for two weeks at room temperature and sieved with a mesh size of 2mm



Plate 1:Ora sampling location 1 (near idominase/ilase) (ORS1)



Plate 2: Ora sampling location 2 (near Osogbo(ORS2)



Plate 3:Oke-Ibode sampling location(OKS)



Plate 4; Osun river sampling location(ORS)

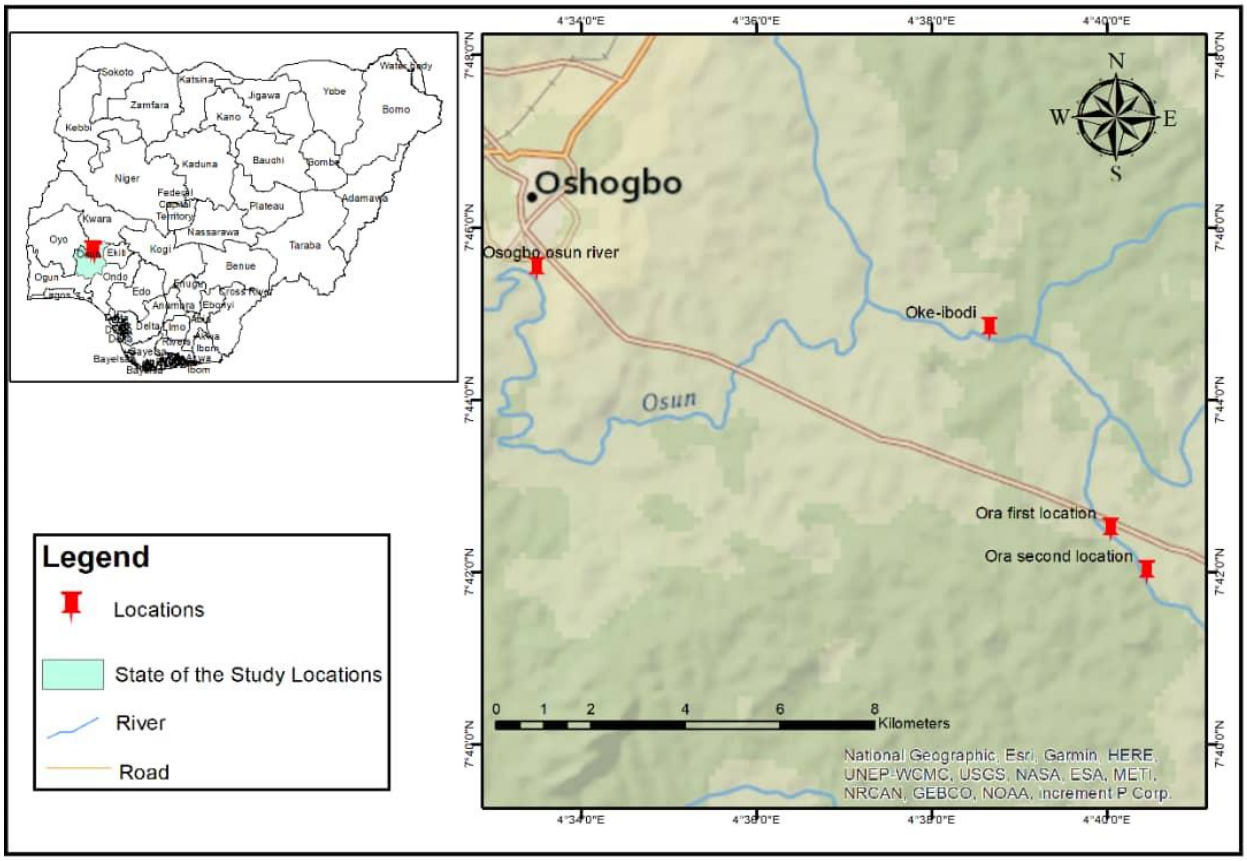

Plate 5: Geographical locations of the sampling sites

2.2. Digestion and sample analysis

After transferring 20 mL of each water sample to a beaker, 400 µL (4 ml) of concentrated HNO3 and 1 mL of concentrated HCl were added, and the beaker was heated to 90ºC for an hour (do not boil). The beaker was then taken out and allowed to cool, and the digested and particulate-free water was filtered to get the final volume down to 20 mL with de-ionized water.

Before being ground into fine particles using a mortar and pestle and sieved through a 2mm sieve, sediment samples were gathered and allowed to air dry for two weeks. Using a 2:1 (v/v) ratio of HCL and HNO3, followed by the addition of concentrated H3PO4 (20 ml), an extraction method was taken from Binning and Baird (2001) and filtered through Whatman 42 filter paper into a 50 ml volumetric flask. Using distilled water, the volume was increased to the 50 ml mark.

2.3. Analysis of heavy metals in gold mine water and sediment

The concentration of heavy metals in different samples was ascertained using inductively coupled plasma optical emission spectroscopy. While the three autosamplers were used to introduce the sample, the Agilent 720-ES with megapixel CCD detector was utilized to provide simultaneous measurement. The instrument was controlled and data was collected using Agilent Expert II software. The following sources of information were used to prepare calibration and quality control (QC) solutions.

- Accustandard QCSTD-27 Quality Control Standard
- Deionized water from Elga B114 Wall Mounted Deionizer system

2.4. Determination of primary source of contamination

2.4.1. Principal Component Analysis (PCA)

To further investigate patterns and relationships in the information, Principal Component Analysis (PCA), a multivariate analysis and dimensionality reduction technique, was employed. A smaller group of uncorrelated principal components (PCs), which are produced using principal component analysis (PCA) from a larger set of connected variables, account for the greatest variation in the data. This method helped identify groups of elements with similar geochemical trends and potential sources of variation. Interpretability is improved via varimax rotation, a PCA extraction method that maximizes the variation of squared loadings inside each component. By examining the Component Matrix, the elements with high factor loadings (≥ 0.7) were found to be significant contributors to each major component. Eigen values greater than one were the selection criterion for maintaining principal components.

The first principal component (PC1), which combined things with similar geochemical sources or processes, accounted for the highest percentage of the overall variation. Subsequent components (PC2 and PC3) reported additional sources of variability that were likely influenced by human inputs or different environmental circumstances. The combination of PCA and DMRT comparative analysis allowed for a robust framework for distinguishing elemental trends, identifying sources of variation, and understanding the underlying geochemical processes influencing the dataset.

2.5. Ecological Risk Assessments

The potential ecological risk index is used to assess potential ecological risk related to metals/any other environmental contaminants in the sediment and water. The potential ecological risk index is calculated:

$$ERI = \sum(CF_i \, x \, TR_i)$$

Where:

$$CF_i = Contamination\ Factor\ for\ element\ i$$

$$CF_i = \frac{Measured\ Concentration}{Reference\ Concentration}$$

$$TR_i = Toxic\ Response\ Factor\ for\ element\ i$$

When ERI*r* <40 indicates low potential ecological risk; 40 <ERI*r* <80 indicates moderate ecological risk; 80 <ERI*r* <160 indicates considerable ecological risk; 160 <Ei *r* <320 indicates high ecological risk; and Ei *r* >320 indicates very high ecological risk (Hakanson, 1980).

2.6. Human Health Risk Assessment

The human health risk assessment method used in this study was for non-carcinogenic as described by Muhammed *et al* (2011). The Chronic Daily Intake (CDI) of heavy metals in water samples was evaluated by the equation (USEPA, 2011):

The Health Risk Assessment (HRA) method which involve using formula for chronic daily intake (CDI) and hazard quotient (HQ):

$$CDI = \frac{C\ x\ IR\ x\ EF\ x\ ED}{BW\ x\ AT}$$

where CDI represents the potential daily exposure of consumers to heavy metals (mg/l). The concentrations of heavy metals in the water and sediment are denoted by CW (mg/l), IR (ingestion rate), EF (frequency of exposure), ED (duration of exposure), BW (body weight), and AT (averaging time). The following lists the input parameters that are utilized to evaluate CDI values.

3.0 Results and Discussion

Four sample groups (OSW, OKW, ORW1, and ORW2) have varying concentrations of various components, according to the descriptive statistics for gold mine water presented in Appendix A. sodium (Na) levels vary from 0.4 mg/L in OSW to 0.59 mg/L in ORW1.

Magnesium (Mg) concentrations in OSW were 0.63 mg/L, whereas ORW1 had the highest concentration (1.59 mg/L). In contrast to the other groups, where potassium (K) was below 0.25 mg/L, there are notable differences, particularly in ORW1, where it increases to 2.59 mg/L. Calcium (Ca) concentrations also vary greatly; OSW and ORW2 had values between 1.57 and 1.58 mg/L, while ORW1 had the highest value at 3.59 mg/L. Compared to other groups, ORW has a significantly greater concentration of barium (Ba), 4.59 mg/L. Most groups lack vanadium (V) and chromium (Cr), with the exception of ORW1, where V reaches 5.59 mg/L and Cr is not found in any sample.

The result of metal Concentration in goldmine water (mg/L) is presented in table 3.1

Table 3.1: Metal Concentration in goldmine water (mean± standard error)

Groups	Na	Mg	K	Ca	Ba	V	Cr
OSW	0.4 ± 0.006^{a}	6.30 ± 0.006^{a}	$0.18\pm0.035^{a,b}$	1.57 ± 0.034^{b}	0.10 ± 0.024^{a}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}
OKW	0.56 ± 0.006^{a}	1.26 ± 0.121^{d}	$0.25\pm0.029^{a,b}$	2.27 ± 0.006^{c}	0.08 ± 0.006^{a}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}
ORW1	0.59 ± 0.006^{a}	1.60 ± 0.006^{e}	2.59 ± 0.006^{e}	3.59 ± 0.006^{d}	4.59 ± 0.006^{d}	5.59 ± 0.006^{d}	0.00 ± 0.00^{a}
ORW2	0.4 ± 0.006^{a}	7.90 ± 0.029^{b}	0.17 ± 0.040^{a}	1.58 ± 0.029^{b}	0.10 ± 0.012^{a}	0.04 ± 0.015^{a}	0.00 ± 0.00^{a}

Mean with the different superscript are significantly different at $p<0.05$

	Mn	Fe	Cu	Zn	Al	Ag	Cd	Th
OSW	0.02±0.006[a]	0.19±0.023[a]	0.08±0.006[a]	0.70±0.029[b]	2.36±0.003[a]	0.13±0.064[a]	ND	ND
OKW	0.05±0.029[a]	0.00±0.00[a]	0.06±0.025[a]	0.88±0.046[c]	0.59±0.017[a]	0.07±0.023[a]	ND	ND
ORW1	0.07±0.006[a]	1.30±0.052[b]	0.05±0.017[a]	0.75±0.040[b]	22.26±0.035[c]	0.51±0.023[c]	ND	0.12±0.040[a]
ORW2	0.05±0.006[a]	0.00±0.00[a]	0.07±0.012[a]	1.07±0.040[d]	9.94±0.023[a]	0.36±0.023[d]	0.07±0.017[b]	ND

Mean with the different superscript are significantly different at $p<0.05$

Groups	Ag	Cd	Th
OSW	0.13±0.064[a]	ND	ND
OKW	0.07±0.023[a]	ND	ND
ORW1	0.51±0.023[c]	ND	0.12±0.040[a]
ORW2	0.36±0.023[d]	0.07±0.017[b]	ND

Mean with the different superscript are significantly different at $p<0.05$

ND: Not Detectable

Table 3.2: **Metal Concentration in Goldmine sediment (Mean ± standard error)**

Significant variations in elemental concentrations, standard deviations, and ranges were found in the descriptive statistical analysis of gold mine sediment samples (OSS, OKS, ORS1, and ORS2), as indicated in Table 3.2. With ORS1 having the greatest mean value (4.92 mg/kg) and ORS2 having the lowest (0.69 mg/kg), sodium (Na) concentrations varied throughout the sample groups. According to standard deviation analysis, OKS has the most variability (1.78), whereas ORS2 has the lowest (0.01). Significant differences were also observed in the amounts of magnesium (Mg), with ORS exhibiting the highest mean concentration (7.48 mg/kg) and OKS the lowest (1.14 mg/kg). The highest standard deviation (0.06) in ORS suggests greater dispersion in Mg values.

Groups	Na	Mg	K	Ca	Ba	V	Cr
OSS	2.25±0.03[c]	2.97±0.023[f]	0.43±0.006[c]	5.89±0.468[e]	1.48±.0.854[b]	1.80±1.016[b,c]	4.74±0.047[c]
OKS	1.78±1.03[b,c]	1.14±0.006[c,d]	0.26±0.040[b]	8.10±0.023[a]	1.02± 0.012[b]	0.98±0.006[a,b]	2.09±0.017[b]
ORS1	4.92±0.03[d]	7.48±0.035[g]	1.87±0.013[d]	3.38±0.040[d]	3.06±0.017[c]	2.74±0.012[c]	7.56±0.040[d]

ORS2	0.69±0.006[a,b]	1.04±0.006[c]	0.21±0.006[a,b]	9.40±0.012[a]	0.35±0.191[a]	0.57±0.035[a]	4.72±0.023[c]

Mean with the different superscript are significantly different at $p < 0.05$

Groups	Mn	Fe	Co	Ni	Cu	Zn
OSS	12.05±0.104[e]	40.09±0.462[e]	0.34±0.035[c]	0.18±0.012[c]	0.31±0.006[c]	1.79±0.006[e]
OKS	9.76±0.063[d]	28.49±0.248[d]	0.11±0.046[b]	0.09±0.012[b]	0.40±0.006[d]	3.90±0.046[a]
ORS1	7.55±0.038[c]	45.29±0.121[f]	0.28±0.040[c]	0.78±0.040[d]	1.06±0.006[f]	2.00±0.052[f]
ORS2	2.82±0.006[b]	13.53±0.087[c]	0.00±0.00[a]	0.00±0.00[a]	0.13±0.006[b]	3.30±0.052[a]

Mean with the different superscript are significantly different at $p<0.05$

Groups	Al	Se	Pb	Ag	Cd	Th	U
OSS	7.17±3.140[a]	0.023±0.009[b]	0.72±0.012[c]	0.14±0.006[a]	0.03±0.012[a]	0.99±0.087[c]	0.2±0.017[c]
OKS	270.50±2.887[d]	0.00±0.00[a]	0.21±0.012[b]	0.077±0.032[a]	0.017±0.007[a]	0.63±0.179[b]	0.09±0.012[b]
ORS1	1077.82±8.747[e]	0.00±0.00[a]	0.00±0.00[a]	0.28±0.012[b]	0.037±0.015[a]	1.55±0.196[d]	0.3±0.006[d]
ORS2	108.62±0.191[c]	0.00±0.00[a]	0.00±0.00[a]	8.88±0.035[d]	0.38±0.017[c]	0.00±0.00[a]	0.00±0.00[a]

Mean with the different superscript are significantly different at $p<0.05$

3.3. Communalities of Elemental Composition in Gold Mine Sediments

	Initial	Extraction
Na	1.000	0.991
Mg	1.000	0.979
K	1.000	0.990
Ca	1.000	0.899
Ba	1.000	0.994
V	1.000	0.998
Cr	1.000	0.959
Mn	1.000	0.863
Fe	1.000	0.967

Co	1.000	0.995
Ni	1.000	0.998
Cu	1.000	0.975
Zn	1.000	0.895
Al	1.000	0.999
Se	1.000	0.969
Pb	1.000	0.964
Ag	1.000	0.788
Cd	1.000	0.904
Th	1.000	0.988
U	1.000	0.992

Three extracted components were identified by the Principal Component Analysis (PCA) Component Matrix in Table 3.4. These components each represented distinct elemental groups according to their factor loadings. For elements like Na, Mg, K, Ba, V, Fe, Ni, Cu, Al, Se, Th, and U, Component 1 has substantial positive loadings, suggesting that these elements have a similar geochemical or mineralogical property. This implies that Component 1 mostly explains the dataset's overall mineral composition or a particular geochemical signature.

Component 2, on the other hand, showed high positive loadings for Ca, Mn, Fe, Co, Pb, and Cd, while elements like K, Ni, Cu, Al, and Mg exhibit negative associations. This pattern suggests that Component 2 is linked to variations in transition metal content and calcium-rich compounds, which may be indicative of certain mineralogical processes or environmental factors influencing metal distribution.

Lastly, Component 3 is characterized by strong positive loadings for Cr, Ag, Cd, and Th, showing that this component captures differences linked to heavy metal content or specific trace elements. Component 3 seems to depict the distribution of trace and heavy metals, which may have different industrial or environmental ramifications than the first two components, which concentrate on key mineral elements and transition metals.

Table 3.4: Component Matrix

	Component		
	1	2	3
Na	.971	-.216	-.029

Mg	.940	-.285	-.118
K	.871	-.460	-.142
Ca	.670	.594	-.312
Ba	.985	-.153	.015
V	.992	-.014	.113
Cr	.880	-.073	.423
Mn	.766	.449	.274
Fe	.943	.164	.225
Co	.924	.374	-.051
Ni	.925	-.360	-.114
Cu	.924	-.348	.026
Zn	.812	.144	-.462
Al	.960	-.278	.006
Se	.962	-.140	-.154
Pb	.702	.681	-.087
Ag	-.171	-.136	.860
Cd	.751	.394	.431
Th	.983	-.027	.146
U	.994	.036	-.061

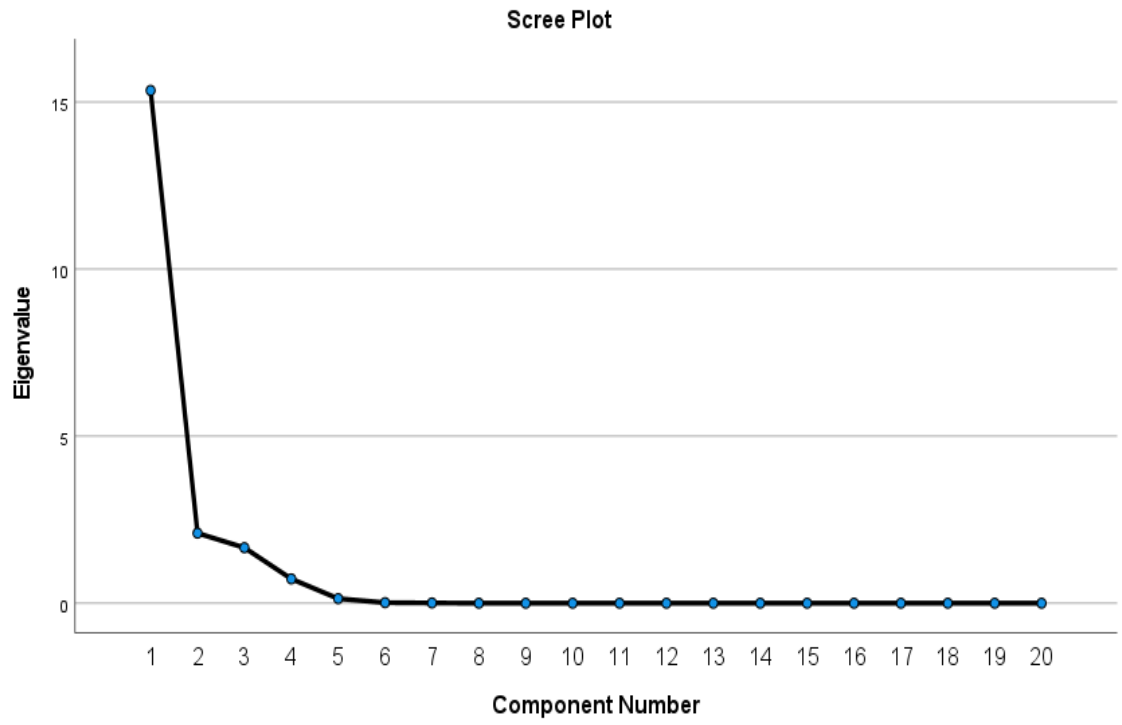

Figure 3.1: Screen Plot of PCA

A considerable amount of the variance in the elemental components can be explained by the first principal component, as seen by the plot in figure 3.1, which displays a sharp drop in eigen values from the first to

the second component. The eigenvalues level out, creating a "elbow" point, after a smaller fall for the second, third, and fourth components. This elbow point, which showed up around the third or fourth component, indicated that keeping three or four principal components would be suitable in order to capture the most significant variance in the data. After this, the eigenvalues stay comparatively small and stable, suggesting that other factors contributed little variance and were probably ignored.. Therefore, the selection of three or four components would provide an effective balance between reduced dimensionality and preserving critical information in the dataset.

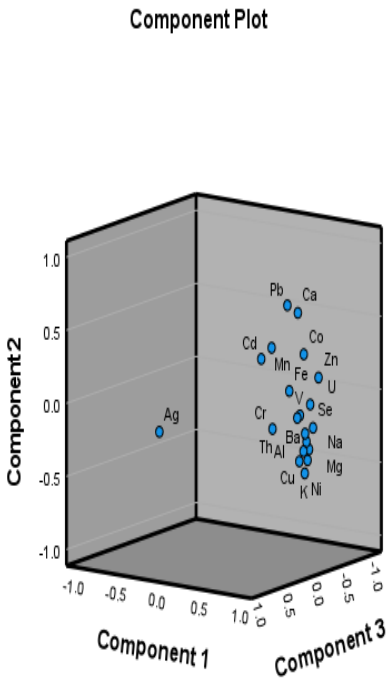

Fig.3.2: Component plot

The component plot, which shows the relationships between variables in the transformed component space seen in figure 3.2, is a three-dimensional representation of factor loadings from a Principal Component Analysis (PCA) or Factor Analysis. According to Ma et al. (2016), the plot shows that the majority of the variables cluster together close to the center, indicating that they may have been associated in the original dataset since they have similar loading patterns across the three components. The fact that Ag (silver) is an

outlier and is located farther from the cluster suggests that it behaves differently from the other elements. Potential links between the variables are suggested by their middle grouping, which suggests that they may heavily load onto the same principal components.

Table 3.5: Potential Human Health Risk Results for Water and Sediment Sample

Element	Ingestion Rate (IR) (L/day)	Exposure Frequency (EF) (days/year)	Exposure Duration (ED) (day)	Body Weight (BW) (kg)	Averaging Time (AT) (days)	Chronic Daily Intake (CDI) (mg/kg/day)	Reference Dose (RfD) (mg/kg/day)	Hazard Quotient (HQ) Water	Hazard Quotient (HQ) Sediment
Be	2	365	30	70	10950	0	0.002 mg/kg/day (EPA)	0	1.0004
Na	2	365	30	70	10950	0.5379	1.5g/day (US Institute of Medicine)	0	0
Mg	2	365	30	70	10950	0.194731	310-420 mg/day (US Institute of Medicine)	0	0
K	2	365	30	70	10950	0.179054	4.7 g/day (US Institute of Medicine)	0	0
Ca	2	365	30	70	10950	0.533269	1000-1300 mg/day (US Institute of Medicine)	0	0
V	2	365	30	70	10950	0	0.009 mg/kg/day (EPA)	0	0
Cr	2	365	30	70	10950	0.0067	0.0003 mg/kg/day (EPA)	0.017143	0.00789
Mn	2	365	30	70	10950	0.02404	0.14 mg/kg/day (EPA)	0	0.0057
Fe	2	365	30	70	10950	0.064546	8-18 mg/day (US Institute of Medicine)	0	1.0078
Co	2	365	30	70	10950	0	0.03 mg/kg/day (EPA)	0	1.7890
Ni	2	365	30	70	10950	0.02	0.02 mg/kg/day (EPA)	0.022857	1.8970
Cu	2	365	30	70	10950	0.04	0.04 mg/kg/day (EPA)	0.232714	1.7654
Zn	2	365	30	70	10950	0.30	0.3 mg/kg/day (EPA)	0	1.8765
Al	2	365	30	70	10950		1.0 mg/kg/day (EPA)	0	0.875
Th	2	365	30	70	10950	0.0006	0.04mg/kg (EPA)	0	0.754
U	2	365	30	70	10950	0.003	0.003 mg/kg/day (EPA)	0	1.548.
Se	2	365	30	70	10950	0	0.005 mg/kg/day (EPA)	0	1.006
Ag	2	365	30	70	10950	0.023	0.005 mg/kg/day	0	0.007
Cd	2	365	30	70	10950	0.017	0.0005 mg/kg/day (EPA)	0.00234	1.0876
Pb	2	365	30	70	10950	0.018	3.5 µg/dL (CDC)	0.611429	1.0045

Table 3.5 presents an assessment of potential human health risks due to exposure to heavy metals in both water and sediment samples. It calculates the Chronic Daily Intake (CDI) based on ingestion rate (IR), exposure frequency (EF), exposure duration (ED), body weight (BW), and averaging time (AT). The Hazard Quotient (HQ) is calculated by dividing the CDI by the Reference Dose (RfD), which is the daily intake of a substance that is considered safe over a lifetime of exposure. The HQ gives an indication of the potential risk to human health, where a value greater than 1 indicates a potential health risk.

4.0 Conclusion

Significant regional differences were found when the total metal concentrations in the water and sediment from four gold mining sites in Ilesha, Osun State, were assessed in this study. The highest concentrations of important elements such as calcium, magnesium, sodium, potassium, and barium were found in water samples from ORW, suggesting potential mineral pollution or enrichment. As a result, they are somewhat polluted. Sediment study revealed clear geochemical variations, with the greatest metal concentrations found in ORS1. They are therefore extremely contaminated. There may be health and environmental hazards Changes in the constituent composition point to the impact of both mining operations and natural geological processes. Localized pollution and mineral leaching are highlighted by the iron, aluminum, and zinc enrichment in OSS. Heavy metal concentrations must be continuously monitored in order to reduce environmental hazards and preserve water quality. To reduce contamination, sustainable mining methods and appropriate waste management are required.associated with ORS2's heavy metal content, which includes uranium, thorium, cadmium, and lead.

Author contributions

ROA: Conceptualization, Writing - Review & Editing, Validation, Supervision, Data curation, Resources. **ZOL:** Writing - Original Draft, Methodology, Formal Analysis, Validation, Resources. **LAA:** Investigation, Validation, Supervision, Visualization. **TOK:** Data curation, Visualization, Validation

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Data availability

All data generated or analysed during this study are included in this submitted manuscript and its supplementary information document.

Declarations

Ethics approval and consent to participate

Not applicable. This study did not involve human participants.

Consent for publication

Not applicable.

Competing interests

The authors declare no competing interests.

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