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The Use of Instrumental Neutron Activation Technique to quantify Na, K, Fe, Co, and Zn in Ajiwa Dam Sediments Employing a Miniature Neutron Source Reactor

***Haruna Halilu and Emmanuel Joseph**

Department of Physics, Federal University Dutsin-Ma, Katsina State, Nigeria

*Corresponding author's email: 020harunahalilu@gmail.com

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ABSTRACT

Identifying and quantifying trace amounts of chemical elements in a sample particularly in sediments has helped researchers to understand their characteristics. This study investigates the elemental composition of five essential elements for plants growth and radiological significance of Ajiwa Dam sediments using Instrumental Neutron Activation Analysis (INAA) at the NIRR-1 Miniature Neutron Source Reactor (MNSR), Ahmadu Bello University, Zaria. Concentrations of Na, K, Fe, Co, and Zn were quantified in ten sediment samples collected from different locations around the dam randomly selected. The results show dominance of Fe and K, with mean concentrations of 19,811 mg/kg and 10,622 mg/kg respectively, followed by Na (1,400 mg/kg), Zn (43.3 mg/kg), and Co (4.9 mg/kg). Statistical analysis revealed moderate variation among elements (CV = 16–37%) and strong correlations between Fe–Co (r = 0.82) and Fe–Zn (r = 0.74), suggesting common lithogenic origins. Radiological evaluation based on potassium-40 (K-40) activity yielded an average absorbed dose rate of 4.2 nGy/h and an annual effective dose of 0.0051 mSv/year, well below the global average limit (0.07 mSv/year). The findings indicate that Ajiwa Dam sediments are geochemically natural and radiologically safe for aquatic life and human use.

INTRODUCTION

Sediments serve as key indicators of environmental quality, reflecting both natural geochemical processes and anthropogenic inputs. Earlier report indicated that sediments represent significant sources of heavy metal pollution in the aquatic environment as a result of changes in pH, redox potential or diagenesis or physical perturbation within their primary sedimentary sinks (Akan et al., 2010). The occurrences of enhanced concentrations of heavy metals especially in sediments may be an indication of human induced perturbation rather than natural enrichment through geological weathering (Daves et al., 2006, Binning and Baird, 2001, Eja et al, 2003). The significance of heavy metals in the environment is that they are non-biodegradable, persist in the environment and may become concentrated up the food chain (Eja, 2003). It is usually observed that sediments near urban areas contain high level of contaminants (Cooks and Wells, 1996; Lamberson et al., 1992), which constitute a major problem to the environment being faced by many anthropogenically impacted aquatic environment (Magalhaes et al., 2007). Sediments act as both carrier and sources of contaminants in aquatic environment (Shuhaimi, 2008), they considered as sink for heavy metals (Atta et al, 1997, Adeniyi and Yusuf, 2007, Chyne-Eng et al., 1987, Olowu et al., 2010). Pollutants released to surface water from industrial and municipal discharges, atmospheric deposition and run off from agricultural, urban and mining areas can accumulate to harmful levels in sediments (Chukwujindu et al., 2007). Additional report has also stated that sediments are an important sink of a variety of pollutants, especially in estuarine ecosystems (Veettil et al., 2019). Metals such as zinc, copper, and chromium are crucial for the functioning of living organisms. However, over-consumption of these metals causes a variety of undesirable effects. Furthermore, due to the bioaccumulation and biomagnification of toxic metals, toxicity increases from low to high trophic levels in aquatic and terrestrial ecosystems will be most severely affected (Talukder et al., 2021; Yunus et al., 2020). Therefore, it is essential to monitor trace elements in sediment to increase knowledge of environmental processes. The elemental composition of nearby pollutant sources and other factors all influence the concentration of elements in sediment (Tiwari et al., 2020).

A number of studies of elements which are beneficial to plant and along major sediments have been carried out in different locations in Nigeria using various analytical techniques: Owalla Reservoir, Osun State, SW Nigeria (Aduwo & Adeniyi, 2018); Stream sediments, Jos Plateau, North-central Nigeria (Odewumi, 2024); Stream sediments, about 400 km² area (lat ~7°N, long ~5°E) – Nigeria (Awosusi & Adisa, 2020); Coastal sediments at Araromi, Southwestern Nigeria (Samuel, Adebayo & Olajide, 2022); Sediments of Warri River, Niger Delta region, Nigeria (Akinwole et al., 2024); among others. Many studies however are localized; hence broader spatial coverage across Nigeria is still needed. The high-precision, nuclear quantitative analytical technique of instrumental neutron activation analysis (INAA) is currently been used by many Researchers in the investigation of various environmental samples such as sediments or soils in particular due to its very high sensitivity. Instrumental Neutron Activation Analysis (INAA) is a precise, multi-elemental, and non-destructive nuclear technique that utilizes neutron irradiation to determine trace elements in environmental matrices (Iyengar and Kasperek (1977). The NIRR-1 Miniature Neutron Source Reactor (MNSR) at Ahmadu Bello University, Zaria, provides an ideal setup for this analysis due to its stability, reproducibility, and low detection limits.

Understanding the distribution of trace elements in sediments is essential for assessing ecosystem health, sediment contamination, and radiological impacts. The Ajiwa Dam, located in Katsina State, Nigeria, provides potable and irrigation water for surrounding communities; hence, studying its sediment quality is critical for long-term environmental monitoring. In the present work, this analytical technique was used to study the concentrations of five trace elements in ten (10) sedimentary cores that were collected from the Ajiwa dam, located in the Katsina State, North – West Nigeria, with the aim of profiling these elements that are known to be essential to plant.

The determination of the composition and concentrations of the five trace elements in ten (10) sedimentary cores that were collected from the Ajiwa dam was performed using the Instrumental Neutron Activation Analysis (INAA). INAA is a very precise technique mainly used to determine trace concentrations of elements in samples and/or to acquire information on the spatial distribution of a neutron field via neutron activation detectors (Majerle, 2006; Joseph et al., 2024). This technique is normally based upon the conversion of stable nuclei to other, mostly radioactive nuclei via nuclear reactions, and measurement of the reaction products. The use of the INAA (relative) method for the calculation of the concentration of each element in the sample irradiated with reactor thermal neutron reduces the NAA equation to the simplest form (IAEA, 1990):

$$\frac{w}{w_{st}} = \frac{N_s D_{st}}{N_{st} D} = \frac{N_s e^{-\lambda td(st)}}{N_{st} e^{-\lambda td}} \qquad (1)$$

Where N_s= net photo peak area of radionuclide of interest in sample, N_{st} = net photo peak area of radionuclide of interest in standard, W= weight of element in sample irradiated, W_{st}= weight of the element in standard irradiated, $D = e^{-\lambda td}$ =decay factor for sample, $D_{st} = e^{-\lambda td(st)}$ =decay factor for standard, t_d= decay time for sample, $td(st)$= decay time for standard, λ= decay constant for radionuclide of interest

The concentration of the unknown element in the sample denoted by Cs is given by

$$C_s = \frac{W}{M} \qquad (2)$$

where M= known weight of the irradiated sample containing the unknown weight of the element irradiated, W= unknown weight of the element irradiated.

MATERIALS AND METHODS

Sample Collection and Preparation

Ten sediment samples (A–J) were collected from different zones of Ajiwa Dam using a grab sampler at 0–10 cm depth. The Aerial photograph showing Ajiwa Dam and spatial distribution of aquatic plant sources (Figure 1). The samples of about 1 kg each were collected in polythene bags that were previously cleaned by soaking in 1:1 HNO_3 for 3 days were air-dried, ground, homogenized, and sieved to <63 µm fraction to ensure uniform particle size. The samples were prepared for irradiation without further treatment at the sample preparation laboratory, Centre for Energy Research and Training, CERT, Ahmadu Bello University, Zaria, alongside with the certified reference materials CRM-NIST Coal Fly Ash 1633b supplied by U.S. National Institute of Standards and Technology for verification and quality control purposes for analysis by INAA. Approximately 200 mg of each sample was encapsulated in polyethylene vials and irradiated in the NIRR-1 reactor operating at 30 kW thermal power with a neutron flux of 5×10^{11} n·cm^{-2}·s^{-1}. The samples were placed in a high-density polythene vial, and weighted using a Mettler Toledo balance model AE 240. These measured samples were double heat sealed in small pieces of cleaned polythene sheets using heat from a modern drier.

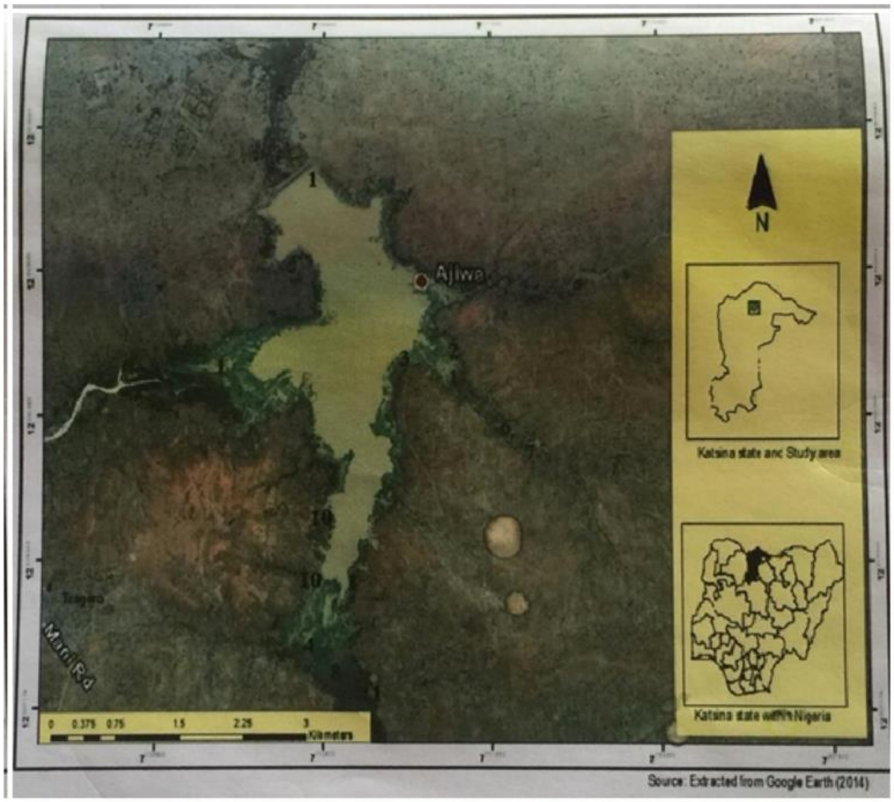

Figure 1: Aerial photograph showing Ajiwa Dam and spatial distribution of aquatic plant sources

The irradiation facility used for this study is the Nigeria Research Reactor-1 (NIRR-1) at the Centre for Energy Research and Training, Ahmadu Bello University, (CERT, ABU), Zaria which is a Miniature Neutron Source Reactor (MNSR) facility, specifically designed for neutron activation analysis (NAA) and commissioned for operation in February 2004. The detailed description of NIRR-1 reactor and the irradiation facility as well as the theory, methodology, among other of INAA have been documented (Jonah et al., 2004; Jonah et al., 2005; Jonah et al., 2006; Jonah et al., 2012; Joseph et al., 2011; Joseph et al., 2013; Joseph et al., 2015a; Joseph et al., 2015b; Joseph et al., 2017; Abubakar & Joseph, 2023).

The Radioactivity Measurements Device is a gamma-ray data acquisition system which consists of a horizontal dip-stick High-Purity Germanium (HPGe) detector with a relative efficiency of 10 % at 1332.5 keV gamma-ray line, the MAESTRO emulation software compatible with the ADCAM® multi-channel analyzer (MCA) card, associated with electronic modules all made by EG & G ORTEC and a personal computer whose details has been described by Jonah *et al*. (2006) and later by Joseph & Nasiru (2013). The efficiency curves of this detector system at both near and far source-detector geometries have been determined by standard gamma-ray sources in the energy range of 59.5–2254 keV and were later extended to 4000 keV by a semi empirical method (Jonah and Sadiq, 2006). The gamma-ray spectrum analysis software *WINSPAN 2004* (Liyu, 2004), a software developed at CIAE, Beijing, China, was used for the peak identification, spectra analysis, and quantification of the elements present.

Statistical and Radiological Analysis

Descriptive statistics (mean, standard deviation, coefficient of variation) and correlation coefficients were computed using Microsoft Excel. Radiological assessment was performed from potassium concentrations using the relationships between K content, K-40 activity, absorbed dose rate, and annual effective dose as prescribed by UNSCEAR (2000).

The radiological contribution from potassium was estimated using the following relations UNSCEAR (2000):

$$A_{K40} = 313 \times \mathrm{K}(\%) \quad (3)$$

$$D_{k40} = 0.0417 \times A_{K40}\ (nGy/h) \quad (4)$$

$$E_{annual} = D_{k40} \times 8760 \times 0.2 \times 0.7 \times 10^{-6}\ (mSv/y) \quad (5)$$

where A_{k40} is the activity of K-40 in Bq/kg and D_{k40} is the absorbed dose rate.

RESULTS AND DISCUSSION

The elemental concentrations of Na, K, Fe, Co, and Zn in Ajiwa Dam sediments are presented in Table 1 with mean concentrations of 1400.1 ± 284.9, 10622.4 ± 1776.6, 19811.0 ± 4498.2, 4.93 ± 0.84 and 43.3 ± 15.8 mg/kg respectively (Figure 1). The Boxplots of the Elemental Concentrations in Ajiwa Dam Sediments is also presented in Figure 2 showing the variations of trace element composition at the Ajiwa Dam. The average elemental abundance followed the order Fe > K > Na > Zn > Co, indicating dominance of iron-bearing minerals (Turekian & Wedepohl, 1961). Moderate coefficients of variation (CV < 37%) indicate relatively consistent distribution of elements across sampling locations (Table 1). The relatively low variability (CV < 25%) for major elements indicates consistent lithology, while the moderate Zn variability suggests minor anthropogenic contributions, possibly from agricultural runoff (Turekian & Wedepohl, 1961). The dominance of Fe and K reflects the geochemical nature of the catchment, likely influenced by iron oxide and feldspathic minerals (Turekian & Wedepohl, 1961). The strong correlations between Fe–Co and Fe–Zn (Table 2) suggest that these elements may originate from common lithogenic sources, possibly iron-bearing minerals (USEPA, 2002).

Table 1: Essential elements for plant growth measured (mg/kg) from Ajiwa Dam Sediments

Element	Na	K	Fe	Co	Zn
Sample A	1537	11060	17630	4.78	37.2
Sample B	1277	9392	16030	4.153	51.1
Sample C	1558	11510	27830	6.464	39.8
Sample D	1818	13150	25510	6.412	48.25
Sample E	1547	11600	21870	5.472	55.56
Sample F	1498	9772	18130	4.417	20.4
Sample G	139.5	8946	18130	4.857	57.31
Sample H	1223	6954	15170	4.014	23.85
Sample I	1555	10710	18240	4.607	69.16
Sample J	1490	12530	18370	4.406	32.81
Range (mg/kg)	139.5–1818	6954–13150	15170–27830	4.01–6.46	20.4–69.2
Mean ± SD	1400.1 ± 284.9	10622.4 ± 1776.6	19811.0 ± 4498.2	4.93 ± 0.84	43.3 ± 15.8
CV (%)	20.3	16.7	22.7	17.0	36.5

Table 2: Correlation Matrix of the Elements

Element	Na	K	Fe	Co	Zn
Na	1	0.68	0.55	0.61	0.46
K	—	1	0.70	0.59	0.52
Fe	—	—	1	0.82	0.74
Co	—	—	—	1	0.64
Zn	—	—	—	—	1

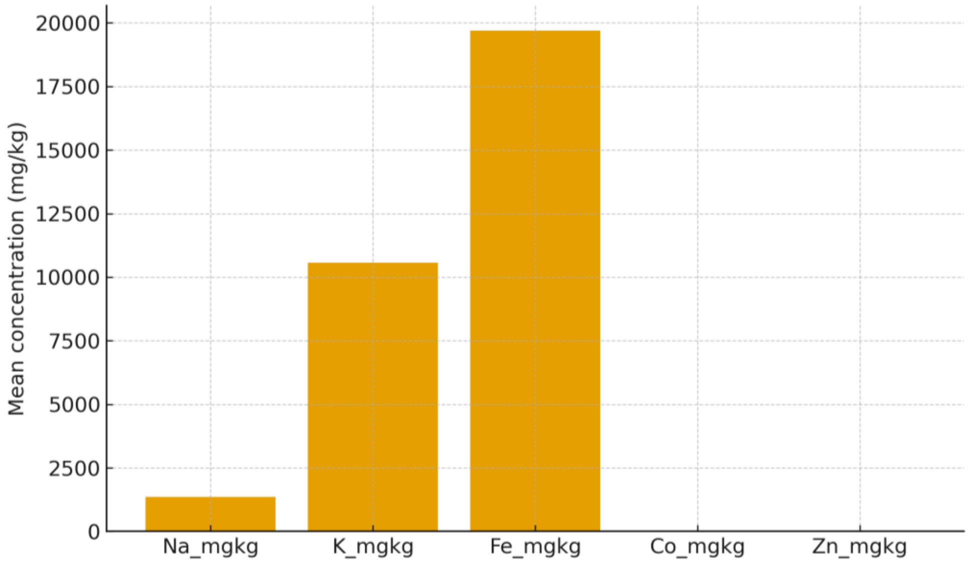

Figure 2: Mean Concentrations of Elements in Ajiwa Dam Sediments

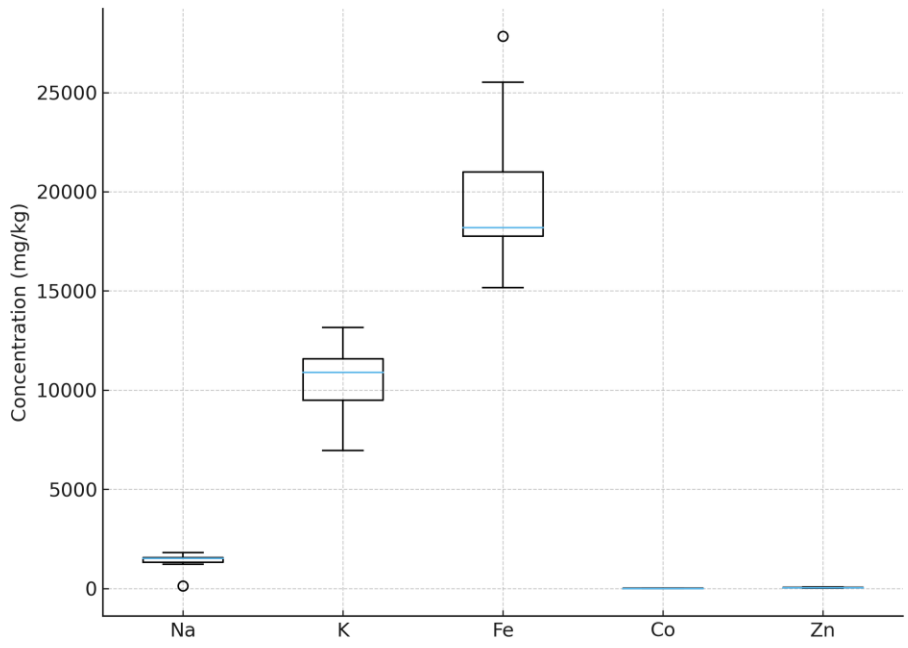

Figure 3: Boxplots of Elemental Concentrations in Ajiwa Dam Sediments

Pearson's correlation analysis (Figure 4) was employed to investigate the correlations among the trace elements (Na, K, Fe, Co, and Zn). Fe–Co (r = 0.82) and Fe–Zn (r = 0.74) were highly correlated with each other, signifying that human activities have likely played a large role in reaching their current concentrations (Wang et al., 2012). The Fe–Co–Zn association highlights co-precipitation processes and possible adsorption onto Fe/Mn oxides. Comparatively, the elemental concentrations fall within the natural background levels reported for other Nigerian water bodies, such as Gubi and Tiga Dams (Ajayi & Balogun, 2002).

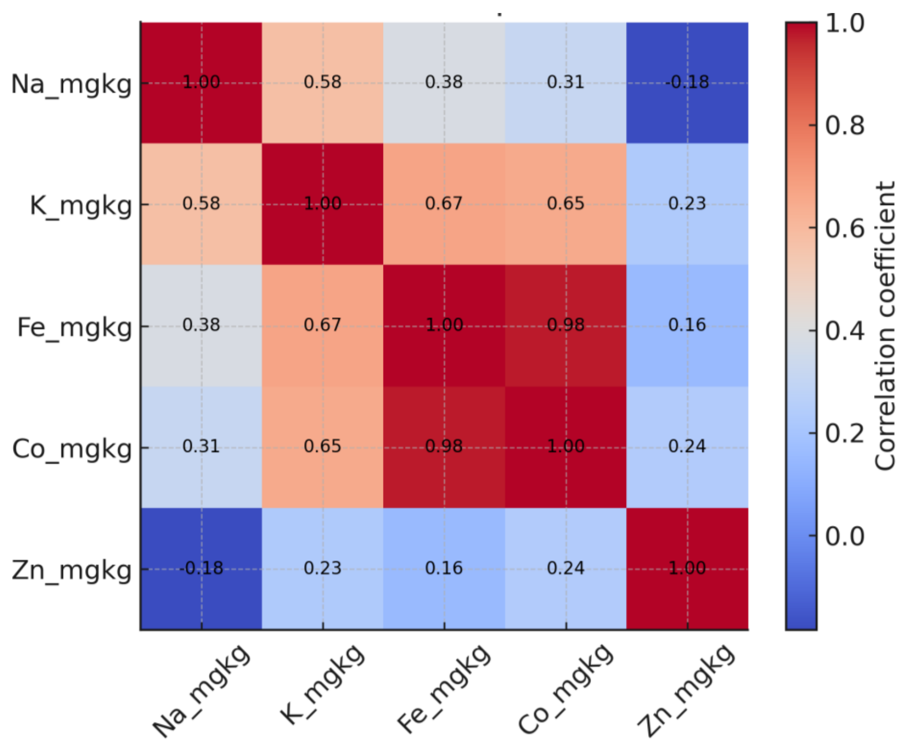

Figure 4: Correlation Heatmap of Elements displaying inter-elemental relationships

Trace Elements distribution of Ajiwa Dam Sediments

Sodium in the Ajiwa Dam sediments measured in all the locations (Table 1) was observed at a mean concentration of 1,400 mg·kg⁻¹ (range 139.5 – 1,818 mg·kg⁻¹), indicating relatively uniform distribution. Converted to mass percent this corresponds to ≈0.14% Na on average, with one notably low sample (Sample G: 139.5 mg·kg⁻¹) that may reflect a local lithological or depositional anomaly, a measurement/labeling issue, or a strongly diluted grain-size fraction at that site. Sodium originates mainly from lithogenic sources, such as the weathering of Na-bearing feldspars (albite) and silicate minerals, and minor inputs from surface runoff and evaporative concentration (Horowitz, 1985). The overall magnitude (hundreds to low thousands mg·kg⁻¹) is typical for lacustrine/riverine sediments where Na is present mostly in exchangeable and soluble forms rather than tightly bound to refractory minerals (Chele et al., 2021).

Potassium averaged 10,622 mg·kg⁻¹ (range: 6,954–13,150 mg·kg⁻¹), equivalent to about 1.06 % K. K occurs primarily in K-feldspars, micas, and illite clays, reflecting lithogenic control (Horowitz, 1985; Ajayi and Balogun, 2002). Agricultural fertilizers may contribute minor anthropogenic inputs (Ajayi and Balogun, 2002). Fe on the other hand showed the highest abundance among the studied elements, with a mean of 19,811 mg·kg⁻¹ (range: 15,170–27,830 mg·kg⁻¹). High Fe concentrations indicate dominance of lithogenic inputs and secondary oxide formation through redox cycling (Horowitz, 1985; Zhang et al., 2014). Iron oxides and oxyhydroxides are strong sorbents for trace metals and this explains the observed Fe–Co ($r = 0.82$) and Fe–Zn ($r = 0.74$) respectively as presented in Figure 5 and Figure 6.

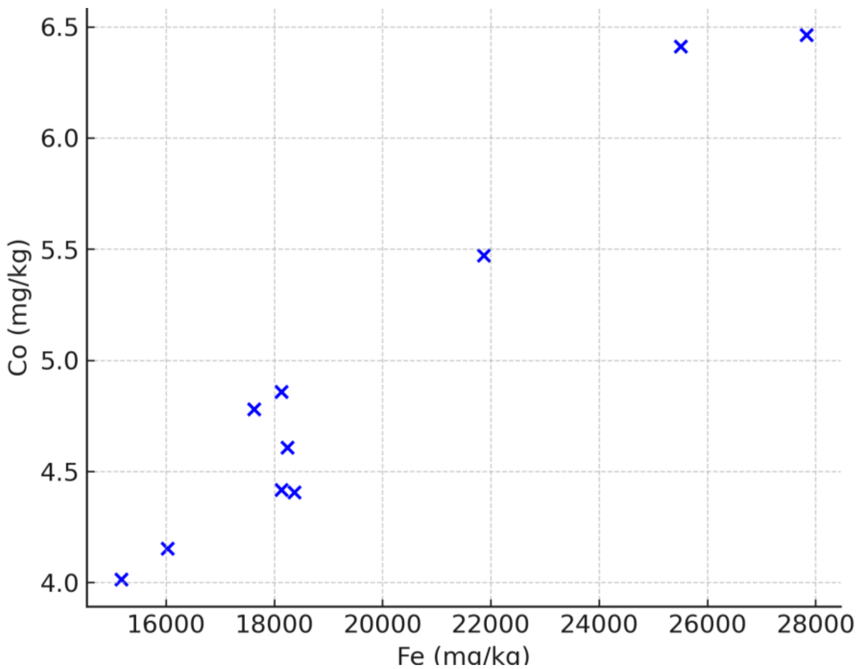

Figure 5: Scatter plots of Fe vs Co Correlation in Sediments

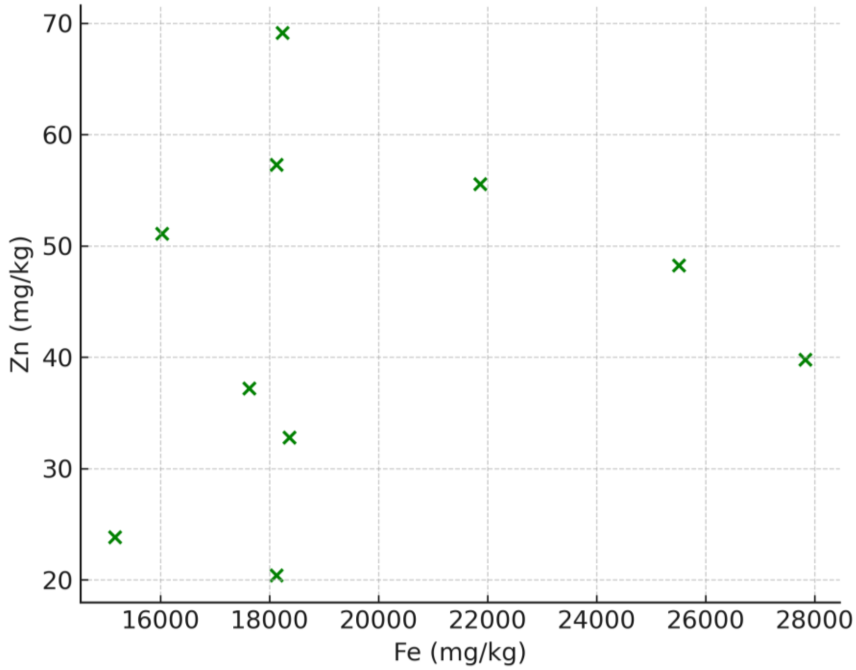

Figure 6: Scatter plots of Fe vs Zn Correlation in Sediments

Furthermore, cobalt concentrations which ranged from 4.01 to 6.46 mg·kg⁻¹ (mean = 4.93 mg·kg⁻¹), is typically associated with Fe–Mn oxides and clay minerals and its mobility depend on redox potential (Xu et al., 2022). In oxic sediments, Co is strongly adsorbed on Fe oxides, whereas under reducing conditions it becomes mobilized (Zhang et al., 2014). The narrow concentration range and strong correlation with Fe indicate lithogenic dominance and minimal anthropogenic influence (Zhang et al., 2014). The concentrations observed here are within global limit (Adeniyi and Yusuf, 2007). Zn concentrations in this study also averaged 43.3 mg·kg⁻¹ (range: 20.4–69.16 mg·kg⁻¹), with a coefficient of variation of 37 %. Zinc arises primarily from mineral weathering and to a lesser extent from anthropogenic inputs such as agricultural runoff (Horowitz, 1985). Zn is readily adsorbed by Fe oxyhydroxides and clay minerals, explaining the strong correlation with Fe ($r = 0.74$) (Trivedi and Axe, 2001; Zhang et al., 2014).

The elemental composition of Ajiwa Dam sediments indicates a predominantly lithogenic origin, with Fe oxides and hydroxides playing a critical role in trace-metal retention. The strong Fe–Co and Fe–Zn correlations confirm this association. Na and K reflect catchment lithology and hydrochemical processes. This current work shows that the metals are all within background levels, which agrees with the work of Aduwo and Adeniyi (2018). It also agrees with the works of Samuel et al. (2022) and Akinwole et al. (2024) that anthropogenic activities in the region contribute to heavy metal loads in sediments.

Radiological Effect Assessment

The average potassium concentration (1.06%) corresponds to a mean K-40 activity of 331 Bq/kg (Table 3). The resulting absorbed dose rate and annual effective dose were equally calculated (Table 3). These results are also presented in Figure 7 showing that all measured values are well below international limits, confirming no radiological hazard from natural radioactivity in the sediments (Turekian and Wedepohl, 1961).

Table 3: Radiological Effect Results

Parameter	Range	Mean ± SD	Global Reference
K-40 activity (Bq/kg)	217–412	331 ± 49	400 (typical soil)
Absorbed dose (nGy/h)	9.05–17.1	13.8 ± 2.0	59 (UNSCEAR, 2000)
Annual effective dose (mSv/y)	0.0043–0.0076	0.0051 ± 0.0007	0.07 (limit)

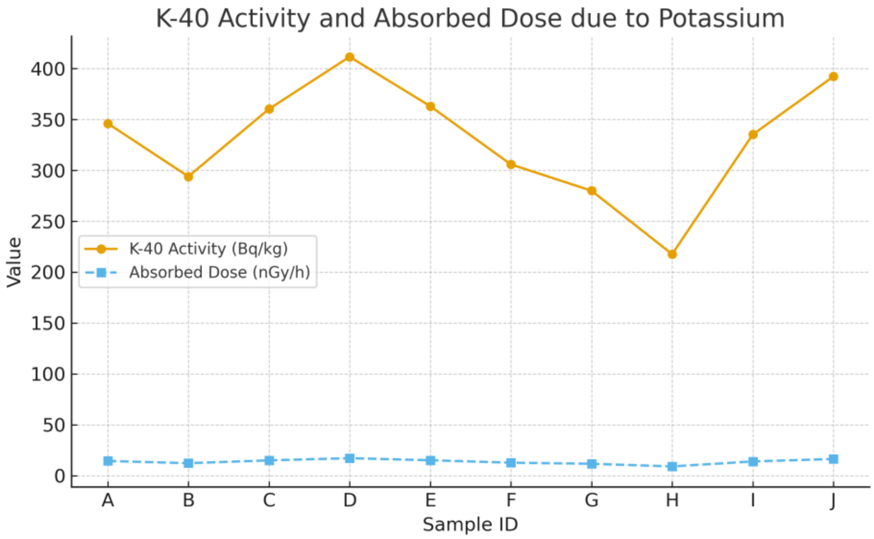

Figure 7: K-40 Activity and Absorbed Dose due to Potassium

The naturally occurring radioisotope ^{40}K constitutes 0.0117 % of total K, yielding an estimated specific activity of ~331 Bq·kg^{-1} (UNSCEAR, 2000). The resulting absorbed dose rate was 13.8 ± 2.0 nGy/h, with an annual effective dose of 0.0051 ± 0.0007 mSv/year. These values are significantly below the UNSCEAR global average of 59 nGy/h and 0.07 mSv/year, confirming that the sediments pose no radiological hazard (UNSCEAR, 2000). Previous study has shown that K-40 is one of the main sources of background radiation (World Nuclear Association, 2011). It has also been shown that K-40 may contribute to long-term cumulative radiation doses and elevate the excess lifetime cancer risk of the population (UNSCEAR, 2000; ICRP, 2007).

CONCLUSION

The INAA technique using the NIRR-1 reactor effectively quantified Na, K, Fe, Co, and Zn in Ajiwa Dam sediments. The dominance of Fe and K reflects natural geological sources, while the strong Fe–Co–Zn correlation indicates common mineralogical origins. Statistical evaluation indicates mainly natural geochemical controls rather than anthropogenic enrichment. The radiological evaluation confirmed that Ajiwa Dam sediments are environmentally and radiologically safe for aquatic life and human use. Continuous monitoring is recommended to ensure the sustainability of Ajiwa Dam as a safe water resource.

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