



<https://doi.org/10.33003/fjorae.2025.0201.90>

INAA-Based Assessment of Elemental Contamination and Ecological Risks in Sediments Associated with Local Open-Site Fertilizer Blending Activities in Kankara, Northwestern Nigeria

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ABSTRACT

Open-site fertilizer blending activities can release potentially toxic and environmentally relevant elements into surrounding sediments, posing risks to local agro-ecosystems through runoff, dust dispersion and subsequent accumulation. This study employed instrumental neutron activation analysis (INAA) to determine the concentrations of nine elements (As, Cr, Zn, Co, V, Mn, U, Th and Ba) and to evaluate associated ecological risks in sediments linked to such activities in Kankara Local Government Area, Katsina State, northwestern Nigeria. Six composite sediment samples (SS1–SS6) were collected, processed and analysed. Analytical accuracy was confirmed using the IAEA-158 marine sediment reference material, which yielded recoveries of 96.6–111.2 % and Z-scores within the acceptable range ($|Z| \leq 0.96$). Marked spatial variation was observed, with elevated concentrations of V (up to 241 mg kg⁻¹), Mn (up to 2615 mg kg⁻¹), Cr (up to 106 mg kg⁻¹) and Ba (up to 890 mg kg⁻¹) at sites nearest the blending operations. Contamination factors and geoaccumulation indices indicated low to moderate enrichment for several elements at these locations, while the partial potential ecological risk index based on As, Cr and Zn ranged from 3.68 to 14.35, indicating low potential ecological risk under the adopted screening framework. The localised enrichment of Mn and V, however, warrants continued monitoring. The results provide a baseline for future assessments of fertilizer-related sediment contamination in the region and underscore the need for improved containment, dust suppression and routine environmental surveillance to minimise mobilisation of potentially toxic and environmentally relevant elements into adjacent sediments and the wider agro-ecosystem.

Keywords: INAA, Potentially toxic elements, Sediment contamination, Ecological risk, Fertilizer blending, Kankara, Nigeria

1.0 INTRODUCTION

Potentially toxic elements (PTEs) originating from both anthropogenic and geogenic sources pose persistent threats to ecological integrity and human health because of their non-biodegradability, bioaccumulation potential and toxicity even at low concentrations (McLaughlin et al., 1999; MacDonald et al., 2000; Salmanighabeshi et al., 2015). These elements enter terrestrial and aquatic systems through multiple pathways, including atmospheric deposition, surface runoff, industrial discharges and agricultural inputs, and may subsequently transfer into food chains via crops and livestock (Sun et al., 2013). Chronic exposure has been linked to neurological disorders, gastrointestinal illness and developmental effects, particularly in vulnerable populations (USEPA, 1986; Singh et al., 2010; Ljung et al., 2006).

Priority PTEs identified by environmental agencies include arsenic (As), cadmium (Cd), chromium (Cr), nickel (Ni), lead (Pb) and zinc (Zn) (Tóth et al., 2016). In aquatic and semi-aquatic environments, the majority of these metals are sequestered in the particulate phase of sediments under stable physicochemical conditions; however, changes such as flooding, dredging or bioturbation can remobilise them into the water column (Martínez-Santos et al., 2015; Zhang et al., 2014).

Nigeria's extensive agricultural lands make soil and sediment quality critical to food security. Fertilizer blending and distribution activities, especially when conducted in open sites with limited containment, constitute a recognised pathway for PTE introduction into surrounding soils and sediments. Although several studies have documented heavy-metal contamination in Nigerian soils and sediments (Ahaneku & Sadiq, 2014; Opaluwa et al., 2012; Abdullateef et al., 2014; Orisakwe et al., 2012), data specifically addressing sediments associated with local open-site fertilizer blending operations in northwestern Nigeria remain scarce.

The present work therefore aims to determine the concentrations of selected potentially toxic and environmentally relevant elements (As, Cr, Zn, Co, V, Mn, U, Th and Ba) in sediments collected near a local open-site fertilizer blending area in Kankara, Katsina State, using instrumental neutron activation analysis (INAA); validate the analytical performance of the INAA protocol; and evaluate the degree of elemental contamination and potential ecological risk using standard indices. The results are intended to support environmental management and regulatory oversight of open-site fertilizer handling in the region.

2.0 MATERIALS AND METHODS

2.1 Study Area

Kankara Local Government Area covers approximately 1 462 km² in the southern part of Katsina State, northwestern Nigeria (latitudes 11°42'–11°51' N; longitudes 7°30'–7°39' E). It lies about 143 km south of the state capital and shares boundaries with Zamfara State (west), Faskari LGA (south), Danmusa LGA (north), Musawa LGA (east) and Malumfashi LGA (southeast) (Figures 1 and 2). The population is predominantly Hausa and Fulani, with farming and livestock rearing as the main livelihoods. Mean annual rainfall ranges from 800 to 1 000 mm. The study focused on sediments associated with local open-site fertilizer blending activities within the LGA.

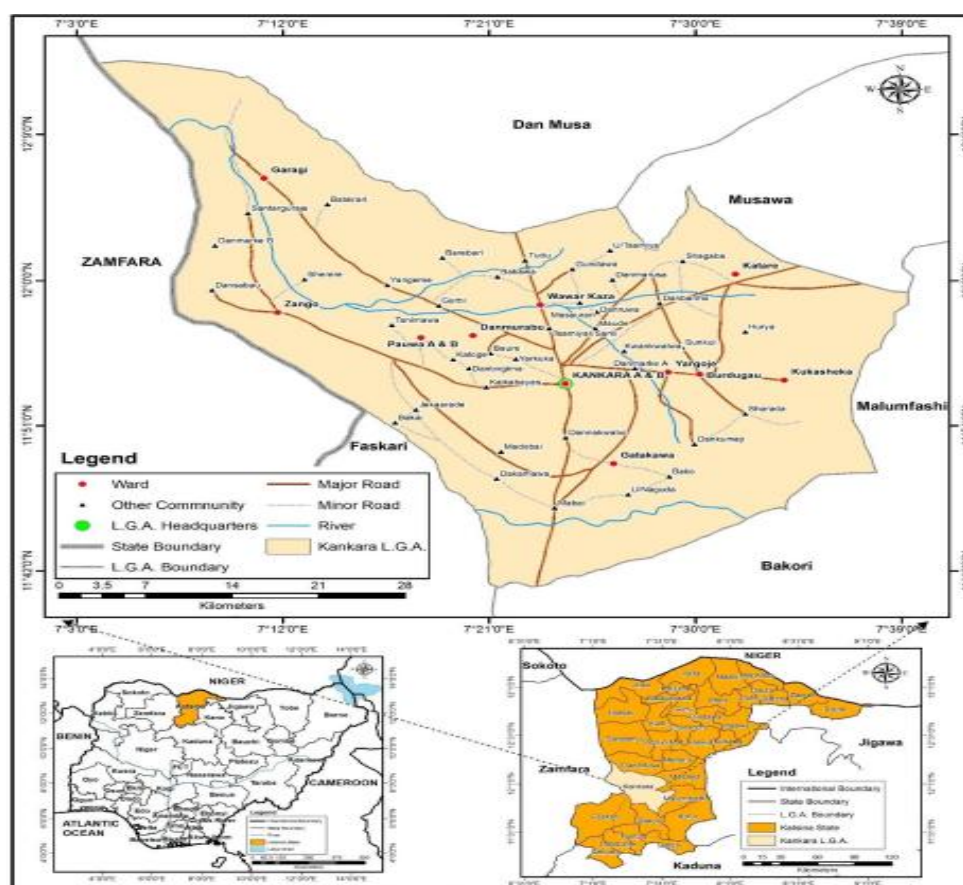


Figure 1: Map of Kankara Local Government (Source: Adapted from the administrative map of Katsina State (2014))



Figure 2: Google Map showing the Location of the local open-site fertilizer blending area in Kankara, Katsina State, Nigeria

2.2 Sample Collection and Preparation

Six composite sediment samples (SS1–SS6) were collected from distinct locations around the fertilizer blending site using a clean plastic shovel and transferred into labelled, pre-cleaned polyethylene containers. Samples were air-dried at room temperature in a clean laboratory, then oven-dried at 105 °C for 6 h to constant weight (Inuwa et al., 2007). The dried material was crushed and sieved through a 20 µm plastic mesh to obtain a homogeneous fine fraction (Inuwa et al., 2007; IITA, 1979).

For INAA, approximately 150–200 mg of each sample was accurately weighed on a METTLER TOLEDO AE 240 balance and double-heat-sealed in cleaned high-density polyethylene vials at the NAA Sample Preparation Laboratory, Centre for Energy Research and Training (CERT), Ahmadu Bello University, Zaria. Certified reference material IAEA-158 Marine Sediment was prepared identically for quality control.

2.3 Instrumental Neutron Activation Analysis

Irradiations were performed at the Nigeria Research Reactor-1 (NIRR-1). The theory, irradiation and counting schemes, and quantitative methodology of instrumental neutron activation analysis (INAA) are well established and have been comprehensively described elsewhere (Jonah et al., 2006; Greenberg et al., 2011; Bode, 2017; Joseph et al., 2024). Short irradiations (1 min) were followed by two counting periods: 600 s after 2–15 min cooling (geometry H2, 5 cm) and a second 600 s count after 3–4 h cooling (geometry H1, 2 cm). Long irradiations (6 h) were counted for 1 800 s after 4–5 days decay and again for 3 600 s after 10–15 days decay, both at geometry H1. Dead time was maintained below 5 % by appropriate choice of decay time and geometry (Jonah et al., 2006).

Gamma-ray spectra were acquired with a high-purity germanium (HPGe) detector (relative efficiency 10 % at 1 332.5 keV) coupled to a multichannel analyser. Elemental concentrations were quantified by the relative method using the certified reference materials.

2.4 Analytical Quality Assurance and Validation

Analytical quality control was performed using the IAEA-158 marine sediment reference material, which was prepared and analysed under conditions identical to those of the sediment samples. The reference material served to assess the agreement between measured concentrations and the certified (assigned) values.

Analytical performance was evaluated using percentage recovery, relative error and the Z-score. Percentage recovery was calculated as:

$$\text{Recovery (\%)} = \frac{C_m}{C_r} \times 100 \quad (1)$$

Relative error was obtained from:

$$\text{RE (\%)} = \frac{C_m - C_r}{C_r} \times 100 \quad (2)$$

and the Z-score was evaluated according to:

$$Z = \frac{C_m - C_r}{SD_r} \quad (3)$$

where C_m is the measured concentration, C_r is the certified reference concentration and SD_r is the standard deviation associated with the reference value. A Z-score within the range $|Z| \leq 2$ was regarded as satisfactory for the assessment of analytical performance.

2.5 Ecological Risk Assessment

Contamination and ecological risk were assessed using the following indices.

The contamination factor (CF) was calculated as:

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

where C_{sample} is the measured concentration of the element in the sediment and $C_{\text{background}}$ is the corresponding background value.

The geoaccumulation index (I_{geo}) was computed according to:

$$I_{\text{geo}} = \log_2 \left(\frac{C_{\text{sample}}}{1.5 \times C_{\text{background}}} \right) \quad (5)$$

The potential ecological risk factor for each element (E_r^i) was obtained from:

$$E_r^i = T_r^i \times \text{CF} \quad (6)$$

where T_r^i is the toxic-response factor of the element (As = 10, Cr = 2, Zn = 1, Co = 5, V = 2, Mn = 1, and similarly for the remaining elements).

The integrated potential ecological risk index (RI) was then calculated as the sum of the individual risk factors:

$$\text{RI} = \sum E_r^i \quad (7)$$

Classification followed established criteria: $\text{CF} < 1$ (low contamination), $1 \leq \text{CF} < 3$ (moderate contamination), $\text{CF} \geq 3$ (considerable contamination); $I_{\text{geo}} \leq 0$ (uncontaminated), $0 < I_{\text{geo}} \leq 1$ (uncontaminated to moderately contaminated), and higher classes accordingly; $\text{RI} < 150$ (low ecological risk), $150 \leq \text{RI} < 300$ (moderate ecological risk), and $\text{RI} \geq 300$ (considerable to high ecological risk).

3.0 RESULTS AND DISCUSSION

3.1 Analytical Validation

Table 1 summarises the INAA validation results obtained for selected elements using the IAEA-158 marine sediment reference material. Recoveries ranged from 96.56 % (V) to 111.16 % (U), with relative errors generally within ± 5 %, except for U (+11.16 %). All calculated Z-scores fell between -0.68 and +0.96 and were therefore well within the acceptable limit of $|Z| \leq 2$ (ISO, 2015; Thompson et al., 2006). These results confirm that the measured concentrations are statistically consistent with the certified reference values and demonstrate that the analytical protocol is fit for purpose.

Table 1: INAA validation results for selected elements using IAEA-158 Marine Sediment reference material

Element	INAA Value (mg kg ⁻¹)	IAEA Ref. (mg kg ⁻¹)	IAEA SD	Recovery (%)	Rel. Error (%)	Z-score
Ba	1029 ± 42.22	1028	46	100.10	+0.10	+0.02
Co	9.20 ± 0.28	9.20	1.10	100.00	0.00	0.00
Cr	74.4 ± 2.83	74.4	5.8	100.00	0.00	0.00
U	2.69 ± 0.09	2.42	0.28	111.16	+11.16	+0.96
Th	8.89 ± 0.21	8.89	0.58	100.00	0.00	0.00
V	70.49 ± 3.66	73.0	3.7	96.56	-3.44	-0.68
Zn	140 ± 11.48	140.6	9.5	99.57	-0.43	-0.06

3.2 Concentrations of Potentially Toxic Elements

Concentrations of the nine potentially toxic elements (PTEs) determined in the six sediment samples are presented in Table 2. Marked spatial variation is evident across the sampling locations. Arsenic ranged from 1.43 ± 0.23 mg kg⁻¹ (SS5) to 5.03 ± 0.40 mg kg⁻¹ (SS2). Chromium was highest at SS1 (106 ± 4 mg kg⁻¹) and lowest at SS5 (15.6 ± 1.6 mg kg⁻¹). Zinc followed a similar pattern, with maxima at SS2 (130 ± 13 mg kg⁻¹) and minima at SS5 (24.5 ± 5.6 mg kg⁻¹). Cobalt peaked at SS3 (30.9 ± 0.5 mg kg⁻¹). Vanadium showed a particularly elevated value at SS1 (241 ± 7 mg kg⁻¹), while manganese reached an extreme of 2615 ± 16 mg kg⁻¹ at SS3. Uranium and thorium were generally low (< 3 mg kg⁻¹ and < 16 mg kg⁻¹, respectively), and barium ranged from 183 ± 24 mg kg⁻¹ (SS4) to 890 ± 42 mg kg⁻¹ (SS2).

The higher concentrations of V, Mn, Cr, Zn and Ba recorded at SS1–SS3 relative to SS4–SS6 are consistent with proximity to the open-site fertilizer blending operations. Phosphate rocks and the P-based fertilizers derived from them are well-documented sources of a suite of PTEs, including elevated levels of Cr, V, Zn, Mn, U and associated elements (Chen et al., 2015; Nziguheba & Smolders, 2008; Jiao et al., 2012). Handling of phosphate and multi-nutrient fertilizers, together with dust generation and surface runoff from the open blending yard, provides a plausible pathway for the mobilisation of these elements into adjacent sediments (Alloway, 2013; Kratz et al., 2016).

Comparison with consensus-based sediment quality guidelines indicates that most measured concentrations of As and Zn remain below the threshold effect levels (TEL) commonly applied for freshwater sediments (MacDonald et al., 2000). Chromium at SS1 (106 mg kg⁻¹), however, approaches or exceeds certain threshold effect concentrations reported for Cr, while the extreme Mn value at SS3 substantially exceeds typical background levels for sedimentary environments. Vanadium concentrations at SS1 are also notably elevated and many reported sediment backgrounds (Turekian & Wedepohl, 1961; Alloway, 2013). Uranium and thorium remain within the lower range of values often associated with phosphate-related inputs, consistent with the selective enrichment of U in P-fertilizers relative to the parent phosphate rock (Jiao et al., 2012).

In all, we can infer that the spatial pattern of enrichment likely supports an anthropogenic contribution linked to local open-site fertilizer blending activities, superimposed on the regional geochemical background. Continued monitoring is warranted, particularly for Mn, V and Cr at the more impacted sites, because changes in redox conditions or pH could increase the mobility and bioavailability of these elements (Zhang et al., 2014).

Table 2: Concentrations of potentially toxic and environmentally relevant elements in sediments (mg kg⁻¹)

Element	SS 1	SS 2	SS 3	SS 4	SS 5	SS 6
As	4.93 ± 0.33	5.03 ± 0.40	4.04 ± 0.43	1.87 ± 0.25	1.43 ± 0.23	4.91 ± 0.34
Cr	106 ± 4	82.7 ± 3.2	86.1 ± 3.3	17.4 ± 1.9	15.6 ± 1.6	29.3 ± 2.3
Zn	119 ± 14	130 ± 13	102 ± 11	25.8 ± 7.0	24.5 ± 5.6	72.3 ± 7.4
Co	23.1 ± 0.4	19.6 ± 0.4	30.9 ± 0.5	5.10 ± 0.21	2.30 ± 0.16	7.78 ± 0.26
V	241 ± 7	118 ± 5	123 ± 5	41.8 ± 3.85	153 ± 5	23.0 ± 1.8
Mn	712 ± 3	575 ± 3	2615 ± 16	221 ± 2	366 ± 3	240 ± 2
U	1.90 ± 0.18	2.53 ± 0.28	2.97 ± 0.23	0.78 ± 0.12	0.99 ± 0.12	1.25 ± 0.16
Th	9.46 ± 0.25	12.2 ± 0.2	16.0 ± 0.3	2.77 ± 0.13	2.53 ± 0.12	4.52 ± 0.10
Ba	497 ± 41	890 ± 42	828 ± 54	183 ± 24	269 ± 26	300 ± 26

3.3 Ecological Risk Evaluation

Using literature background values based on composition or uncontaminated regional sediments (Turekian & Wedepohl, 1961; Håkanson, 1980; Rudnick & Gao, 2014), contamination factors (CF) for the majority of elements at SS4–SS6 remained in the low range ($CF < 1$). In contrast, at SS1–SS3, CF values for Cr, Zn, Co, V and Mn frequently fell within the moderate contamination category ($1 \leq CF < 3$). Manganese at SS3 and vanadium at SS1 approached or exceeded the threshold for considerable contamination ($CF \geq 3$), indicating clear local enrichment relative to background levels (Håkanson, 1980; Kowalska et al., 2018). Geoaccumulation indices (I_{geo}) similarly classified most sampling sites as uncontaminated to moderately contaminated ($0 < I_{geo} \leq 1$), consistent with the classification scheme of Müller (1969). Isolated higher I_{geo} values were recorded for Mn and V at the sites closest to the blending operations, reinforcing the spatial pattern already observed in the absolute concentrations.

Potential ecological risk factors (E_r^i) were generally low for Zn, Cr and Mn because of their relatively low toxic-response factors ($T_r^i = 1-2$). Arsenic and cobalt, which carry higher toxic-response factors ($T_r^i = 10$ and 5, respectively), contributed more substantially to individual risk at the more enriched sites (Håkanson, 1980). Nevertheless, the integrated potential ecological risk index (RI) remained below 150 at all six locations. According to the conventional classification of Håkanson (1980), this places the sediments in the low ecological risk category overall.

Despite the low integrated risk, the elevated single-element enrichment of Mn and V at selected sites warrants continued monitoring. Both elements can be remobilised under changing redox or pH conditions, potentially increasing their bioavailability to benthic organisms and the wider aquatic food web (Zhang et al., 2014; Eggleton & Thomas, 2004).

The observed spatial pattern of moderate enrichment at SS1–SS3 is consistent with the open handling of fertilizer materials. Phosphate rock and the P-based fertilizers derived from it are recognised sources of a suite of trace elements, including V, U, Th, Cr and associated metals (Nziguheba & Smolders, 2008; Jiao et al., 2012; Ullah et al., 2024). Micronutrient additives and process dust generated during open-site blending further contribute to the local PTE load. Surface runoff and wind-blown particulates from the blending yard constitute the most plausible vectors transferring these elements into the surrounding sediments (Alloway, 2013; Kratz et al., 2016).

Taken together, the CF, I_{geo} and RI results indicate that, while the current overall ecological risk remains low, localised enrichment linked to fertilizer blending activities is already detectable. Implementation of improved dust control, containment and routine environmental monitoring would help prevent further accumulation and potential future elevation of ecological risk.

4.0 CONCLUSION

This study applied instrumental neutron activation analysis (INAA) to determine the concentrations and spatial distribution of As, Cr, Zn, Co, V, Mn, U, Th and Ba in sediments associated with local open-site fertilizer blending activities in Kankara, northwestern Nigeria. Analytical validation using IAEA-158 marine sediment reference material demonstrated satisfactory performance, confirming the reliability of the elemental concentration data. The investigated elements exhibited marked spatial variability across the sampling stations. Contamination factor and geo-accumulation index analyses indicated predominantly low-to-moderate contamination. However, Mn at SS3 showed the most pronounced enrichment, with $CF = 3.38$ and $I_{geo} = 1.17$, while V at SS1 recorded $CF = 2.48$ and $I_{geo} = 0.73$. Pollution load indices further identified SS1–SS3 as the relatively enriched stations, with SS3 recording the highest PLI of 1.40, whereas SS4–SS6 had values below unity. The partial potential ecological risk index based on As, Cr and Zn ranged from 3.68 to 14.35, indicating low potential ecological risk under the adopted screening framework. In all, the observed spatial pattern is consistent with localized elemental enrichment associated with the open fertilizer blending environment, particularly at SS1–SS3. However, fertilizer blending cannot be established as the exclusive source of the measured elements. Natural geochemical variability and lithogenic contributions from underlying parent materials may also influence their occurrence, particularly for geochemically associated elements such as U and Th. The enrichment may therefore reflect combined anthropogenic and geogenic influences. The study identifies SS3 as the principal enrichment hotspot, followed by SS1 and SS2. Improved fertilizer-handling practices, control of material spillage and surface runoff, and periodic environmental monitoring are recommended. Future studies should incorporate uncontaminated local background sediments, direct characterization of fertilizer materials, increased spatial and seasonal sampling, and source-apportionment techniques to distinguish fertilizer-related inputs from natural geochemical contributions more conclusively.

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