Journal of Science Research and Reviews

PRINT ISSN: 1595-9074
E-ISSN: 1595-8329
DOI: https://doi.org/10.70882/josrar.2025.v2i1.33
Homepage: https://josrar.esrgngr.org

Original Research Article

PIXE Analysis of Vegetation Samples from Mining Site, Dange-Shuni LGA, Sokoto State

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KEYWORDS

PIXE,
Mining sites,
Vegetation Samples,
Iullemeden Sokoto basin.

CITATION

Ahijjo, Y. M., & Baba-kutigi, A. N. (2025). PIXE Analysis of Vegetation Samples from Mining Site, Dange-Shuni LGA, Sokoto State. *Journal of Science Research and Reviews*, *2*(1), 92-98. https://doi.org/10.70882/josrar.2025.v2i1.33

ABSTRACT

This study has used Proton induced X-ray Emission (PIXE) to analyze fifteen vegetation samples collected from mining sites in Dange-Shuni LGA, Sokoto State. It is in a bid to ascertain trace elemental concentration in the vegetation samples as a result of their exposure to unearthed soil from deeply excavated mining activities in the mining communities of Dange-Shuni LGA. The elements; Al, Mn, Zn, Pb, Ti, Cr, Ni, Co, Cu, Fe, and As, were identified via the PIXE analysis of the vegetation samples from fifteen mining sites in a stratified random sampling by proportions. The results of PIXE through intrinsic efficiency of the detector indicates fair elevations of the elemental concentrations from the samples. The results are a justification of potential toxicity relevance in the samples as a response to the unabated mining activities in the localities. Caution must be applied on open grazing of animals and usage of vegetation of these localities for medicinal and other domestic applications to avert toxicity in human especially children of tender ages.

INTRODUCTION

Trace elements have remained vital components of all biological systems if lives must be sustained. In their useful amounts, they are sometimes referred to as micronutrients with vital roles to play during anabolism and catabolism (Zhou *et al.,* 2018; Onumejor *et al.,* 2021 and Kvasniak *et al.,* 2023). Some studies are of the views that about fifteen of the trace elements out of several tens of them are really sort after based on necessity for virtuous life cycles of plants and animals.

A ground breaking study that was conducted by Ahijjo, 2019 in the famous Iullumeden basin shows that elevated concentration of trace elements could cause health concerns and therefore should be nib in the bud. Other studies have also reported some disturbing facts about their presence in environmental sources such as air, water, soil and vegetative samples. Globally, scientists are coming together with concerted efforts in order to address issues related to contaminations of particles with potential danger to inhalation, ingestion and skin contacts from the environment. Some of these realities have compelled the careful choice of this study in a view to conducting Particle Induced X-ray Emission (PIXE) analysis on vegetation samples in and around mining sites in the localities of Dange-Shuni LGA, Sokoto state (figure 1).

Dange-Shuni is geologically categorized into a sub-set of the famous Iullumeden basin also referred to Sokoto basin. It is one of the homes to mining activities in the basin due to relative abundance of precious elements such as Kaolin, Gypsum, Gold, Silver, Diamond (Okosun and Alkali, 2013; Hussain *et al.,* 2019 and Mustapha *et al.,* 2022) etc.

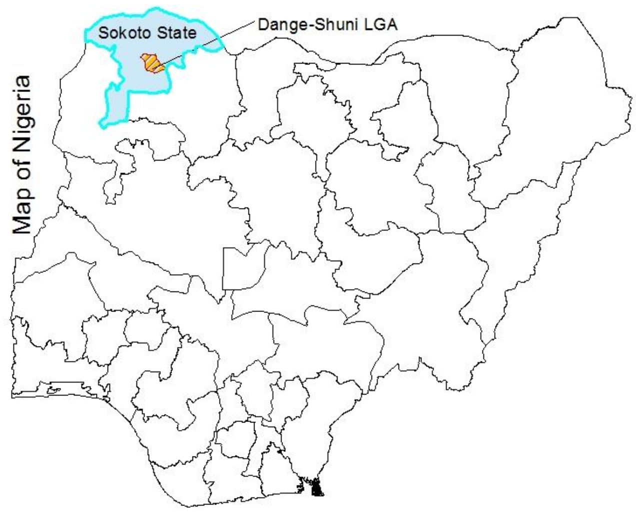


Figure 1: Map of Nigeria featuring Sokoto State and Dange-Shuni LGA

As such, mining becomes the order of the day to the inhabitants in most parts of the remote dwelling of this geographical location (The Sun Nigeria, 2019; Federal Government of Nigeria, 23; ENACT Africa, 2020 and Kebbi State Government, 2023). Figure 2 is an illustration of some majorly mined minerals. It lies on the geographical coordinates of 12.7956^{0}N and 5.4359^0 respectively. The features of semi-arid climate are very prominent in most cases according to Koppen climate classification.

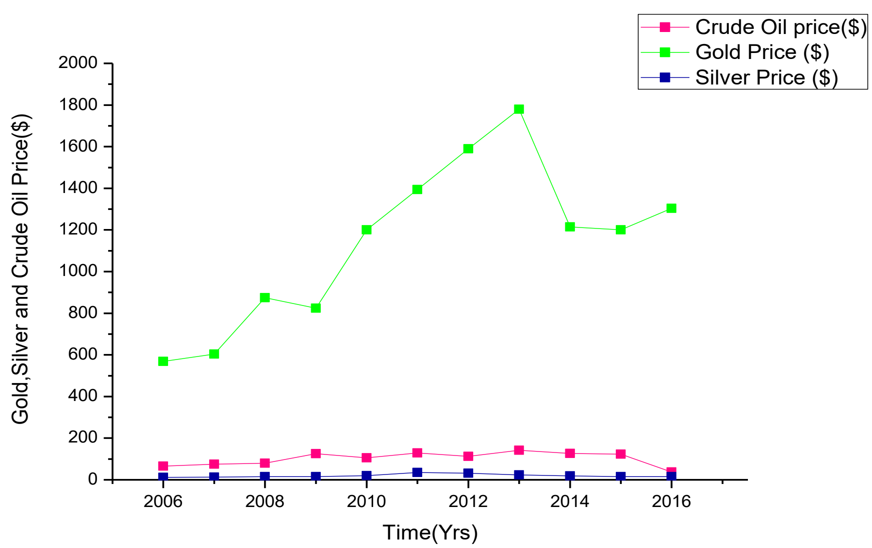


Figure 2: Price Comparison and fluctuation chart of majorly mined minerals (Ahijjo, 2019)

Nuclear methods that involve atomic analysis has gained acceptance in physical and life science particularly when environmental samples demand adequate sensitivity in handling. In PIXE, analytical procedure has been employed in this précis of the study in its entirety since this article is just a little fraction of the larger study conducted in order to ascertain radiological impacts of mining activities in the basin. So, PIXE was used to analyze the trace elements concentration in vegetation samples that were collected from mining sites of Dange-Shuni LGA, Sokoto state. PIXE is a well – known technique for trace elemental analysis that employs several tens to hundreds of MeV ion beam to achieve high accuracy. Usually, very thin target is required for irradiation of beam particles in the form of protons (Khan and Khan, 2022).

This article is an extract of a larger research conducted over a vast range of location, where samples such as soil, water and vegetation were collected so as to ascertain the level of environmental toxicity embedded in the samples as a result of rampant mining activities in the localities. The main aim was therefore achieved via high precision and sensitivity method of nuclear and nuclear analytical related methods.

MATERIALS AND METHODS

Sampling and Sample Preparations

Vegetation samples were chosen for the PIXE analysis. They were collected from fifteen different mining sites within Dange-Shuni LGA based on stratified random sampling by proportions. The sampling approach was faced with enormous challenges inform clumsy and difficult to unravel and thereby it was tackled by being broken down based on mutually exclusive and collectively exhaustive expression of equation 1;

$$\Sigma_{\sigma} = \frac{S_{\rho}}{\sigma}(N_k) \quad (1)$$

Where, $\Sigma_{\sigma}$is the sum of the real-world contributions, $S_{\rho}$is the number of sample points, $\sigma$is the sum of the sample points and N_k, is the sum of all the real-world contributions from the inhabitants. The validity of the equation was ensured for its break through and innovative approach to sampling.

This was done in order to overcome the earlier avoided clumsiness in the randomly selected strata and to improve collection of sample representation thereby reducing sampling error and biasness. For each randomly selected mine, the most virtual parts of the vegetation were considered for collection due to their rural as well as urban importance. They often serve as animal feed during grazing, additive in medicinal ingredients and herbs preparation, dispersed into water bodies and soak it contents or get decay and ingested by either domestic animals or humans (Zeb *et al.,* 2017; Wongke and Tippawan 2021 and Khan and Khan2022).

This is worthy to note that about 0.6$kg$of the vegetation samples was collected from each sample point. They were carefully washed with deionized water and packed in a clean labeled polyethylene bags and then conveyed to Center for Advanced Studies in Physics (CASP), GC, University, Lahore, Pakistan for PIXE analysis. Subsequently, they were oven dried under a temperature of about 80^0 for 21,600 sec.It is worth noting that available parts of plants were collected to form the vegetation samples for this study (Khan and Khan 2022). They were crushed, homogenized with agate mortar and a pestle in order to achieve uniformity. This was to as well enhance optimum exposure under proton beam according to Hussain *et al,* 2019. The crushed vegetation samples were sieved through 150μm mesh and known weight of uniform size sample of 0.3$g$was mixed via 1.5cm diameter funnel with 0.075$g$of a high purity graphite powder in the ratio 4:1 of weight so as to avoid charade build up in PIXE measuring vacuum. Sample insulation, charge collection of samples and good surface area to charge ratio were achieved. The samples were enriched in a 1.5$g$of baric acid powder to bind the components tightly together. The targets were press together in their muddled state by a 100KN press in circular die of 25mm in diameter to obtain a 3mm thick pallet for PIXE (Rahman *et al.,* 2005; Dasgupta-Schubert *et al.,* 2007; Acharya *et al.,* 2023).

Experimental Set-up

When A 4 MeV proton beam of the pellet accelerator at CASP, GCU, Lahore, Pakistan was used for PIXE on the vegetation samples. The beam flounce was set at 1μC and a model spectrum was fitted to optimize area and conversion to elemental concentrations. The peaks were found at the optimum duration earlier outlined and saved as characteristic constant of detector. The standardization of the X-ray energy dependent value was based on characteristic of the detector and the absorber. The sample pellets were then measured by insertion on the ladder made of 2.5mmthick Aluminum plates that can take maximum of twelve pellets of 3mmat a time. They were confined to an anti-scatter collimator. The X-ray energy of the target was detected by Canberra Si(Li) detector under a low beam current of about 2-5ηA at 135° to the plane of the ladder.

The spectra information of eleven elements was acquired sufficiently within 180sec maximum exposure time earlier set. A kepton filter of thickness 10$\mu m$was used to reduce the high X-ray intensity in the spectra due to K and Ca $K\alpha$X-ray and $K\beta$X-ray lines. The matrix effect, proton stopping power, X-ray mass attenuation coefficient and the secondary fluorescence were all combined used to obtain the spectrum fitting. The relationship in equation 2 below was used for the X-ray intensity yield principal line ($Y(Z,M)$) for element Z (atomic number) in a matrix M (Lee *et al.,* 2021; Nedjma and Lakhdar, 2021; Abo El-Maali and Amr, 2022; Sung and Kim, 2022 and Pini et al., 2023)

$$Y(Z,M) = Y1t(Z,M) \times Cz \times Q \times f_q \times \Omega \times \delta \times T \quad (2)$$

where; $Y1t(Z,M)$ is the theoretical intensity or yield$\mu C$of charge per unit concentration per steradian, $Cz$is the actual concentration of the element$Z$in the matrix M, Ωis the measured beam charge or fraction between $\mu C$and 1.0 electron suppression ensured at the target, $\delta$is the intrinsic efficiency of the Si(Li) detector, and $T$is the transmission through any filter or absorber between the sample and detector.

Data acquisition was enabled by Canberra Multi-Channel Analyzer (MCA) interfaced with PC for the X-ray under the preamplifier pulse height mode. GUPIX program was employed to harness the information from the X-ray spectra so as to suppress the linear background and modify the Gaussian Peak Shape (GPS). The acquired data in turn was compared with theoretical spectrum and tested via Chi-square criterion to obtain the best fit based on channel weighing. The X-ray intensities were converted to elemental concentrations via earlier standardization and further expressed in equations 3 and 4.

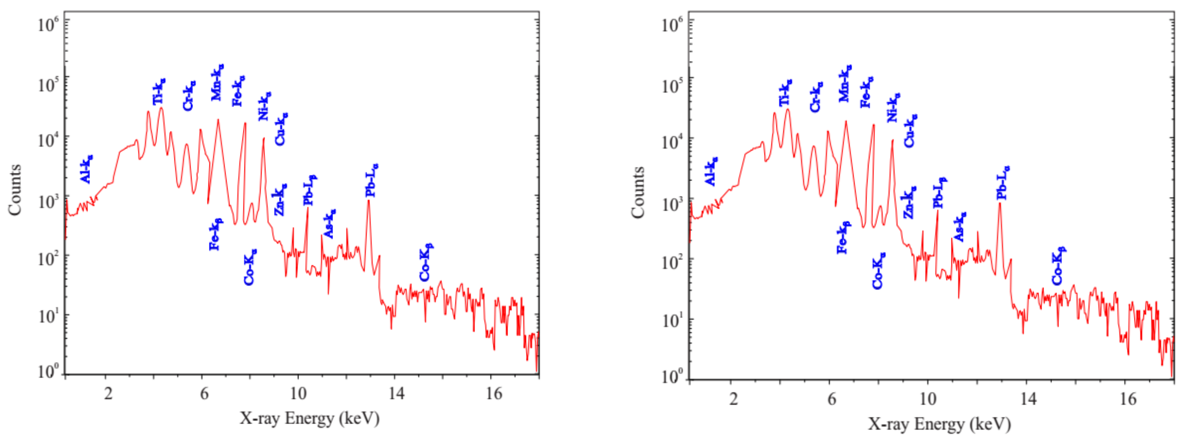
$$Y(Z,M) = Y1t(Z,M) \times Cz \times Q \times H \times \delta \times T \quad (3)$$

$$Cz = Y(Z,M)/Y1t \times Q \times H \times \delta \times T \quad (4)$$

Where, H is the X-ray energy dependent value. H value may also depend on the X-ray energies due to imperfect secondary electrons suppression, error from the thickness of the absorber and efficiency of the detector, the standard H value were utilized in the data analysis of the samples.

RESULTS AND DISCUSSION

A typical PIXE spectrum obtained in this study can be seen below in figure 3 a. and b. respectively. They are the typical illustrations of identified particles from the vegetation samples. They featured the outputs of Al, Mn, Zn, Pb, Ti, Cr, Ni, Co, Cu, Fe, and As, (i.e. eleven elements of interest) in ppm.



Figures 3 a and b: PIXE Spectrum of vegetation sample from ROI

PIXE spectra results shown in figures 3 a and b above were found corresponding to different vegetation samples in accords with elemental concentrations in ppm. They were achieved via standard techniques which involves fundamental parameters, predetermined instrument constants and input parameters such as solid angle, charge collection.

The corresponding limits of detection (LD) to the background within the characteristics peak of spectra which are of the order of $mg.kg^{-1}$ for the element detected. PIXE spectra is embedded with different elements identified in the general characteristics. This also show that the background is dominated at energies beginning from 0 to a few MeV are of the bremsstrahlung for the secondary electrons, and by x- and gamma – ray they feature at higher energies. The mean concentration of the eleven elements in all the samples have been compared and presented on Table 1.

The results of PIXE analysis of eleven elements in from fifteen vegetation samples presented on Table 1 shows that Al exhibits its highest concentration at sample point coded as Dan9 which is 1954 ppm and lowest concentration at Dan15 with just 69 ppm. On the other hand, Mn was found with its highest concentration at Dan4 which is exactly 182 ppm while its lowest concentration fell below detection limit at Dan1 and Dan11 respectively. Zn has its highest concentration at Dan10 with a value of 93 ppm comparing with its lowest concentration below detectable limits at Dan4 and Dan14 respectively. Never the less, Pb was noticed with its highest concentration at sample code Dan1 with a value of 3552 ppm and its lowest concentration at Dan7 which falls at 298 ppm. It is also worthy of noting that Ti was found with highest concentration at sample point Dan1 with 450 ppm while its lowest concentration was at Dan14 with just 39 ppm.

Table 1: PIXE elemental Concentration Results in Fifteen vegetation Samples from Dange-Shuni LGA

Code	Identified Elements										
	Al	Mn	Zn	Pb	Ti	Cr	Ni	Co	Cu	Fe	As
Dan1	819	-	85	3552	450	-	14	-	13	11492	100
Dan2	2081	132	81	1812	41	19	10	12	38	10092	82
Dan3	2321	84	83	345	432	11	23	27	19	11432	25
Dan4	2262	182	-	2108	356	-	18	15	34	9674	30
Dan5	1403	97	77	1940	390	23	21	19	41	12491	74
Dan6	96	160	90	3102	52	14	19	21	-	10341	103
Dan7	2100	64	79	298	314	21	15	13	19	1979	97
Dan8	831	129	84	3601	49	17	12	24	35	11029	102
Dan9	1954	87	59	2003	409	12	20	16	21	710	79

Dan10	1214	129	93	1408	295	16	13	26	18	14219	85
Dan11	904	-	71	534	48	22	17	14	18	541	94
Dan12	2631	181	38	1028	365	31	21	17	43	1040	101
Dan13	89	62	82	3133	215	18	11	29	-	2102	311
Dan14	971	79	-	354	39	10	-	16	32	10413	-
Dan15	69	123	48	300	54	22	16	25	39	2119	102

Cr was found with highest concentration at point Dan12 with 31 ppm and lowest at Dan1 and Dan4 below detectable limit. On the other hand, Ni was noticed with the highest concentration of 23 ppm at Dan3 and its lowest occurrence was found below detectable limit at Dan14. Co was as well found with its highest concentration at Dan13 with a value of 29 ppm and a corresponding lowest concentration below detectable limit at Dan1. Also, Cu, Fe and As were found with their highest concentrations of 43, 1979 and 311 ppm at sample points Dan12, Dan7 and Dan13 with their corresponding lowest values below detectable limit at Dan6 and Dan13, 541 ppm at Dan11 and below detectable limit at Dan14 respectively.

However, the results presented on Table 1 above and discussed could be observed with eleven elements of biological, environmental and toxicological concerns. The occurrence of Al could be noticed with significant prominence which followed by concentrations of Pb, Fe and Ti respectively. Mn on the other hand appeared fairly high in concentration even though its concentrations is not a serious toxicological concern for both for biotoxicity and environmental presence due to its function as micronutrients status (Zhou *et al.,* 2018; Liu *et al.,* 2022; Joo *et al.,* 2023 and Khosravi and Torkzadeh, 2023). But its excess concentration could be of a concern in plants and animals as well.

Nevertheless, Zn being another vital micronutrient could alter metabolism in plants if the concentration exceeds the required amounts. Symptoms like chlorosis in leaves could be noticed in plants when Zn concentration is deemed high. The high concentration reported in this study could be due to mining activities rampant in Dange-Shuni LGA.

Pb concentrations can easily find its way into the food chains of humans through plant and other animals' consumptions. It is a known pollutant of both biological and environmental concerns. In plants, it affects the morphology, growth and process of photosynthesis (Wang *et al.,* 202). The locality makes up of the geology and unabated exploration of soil in form of mining could also triggers its concentrations in the environment.

Ni concentration have also been accented on due to its prevalence in mining activities. Its elevated concentration is often linked to chlorosis in plants while respiratory disorder in human can be frequently established on the rise in the content of Ni in seldom times (Kumar and Sinha, 2022 and Mishra and Sinha, 2023).

Co on the other hand was found fairly low compared to other elemental concentration in the PIXE analysis for vegetation sample from mining communities in Dange-Shuni LGA, Sokoto State. Elevated concentration of Co has been linked to death of plants aside from causing chlorosis. The potential deep excavation of soil during mining activities can expose the public to its toxic's concentration through ingestion of vegetative parts of the plants.

Cu being a micronutrient in its naturally moderate amount but could at certain concentrations lead to liver and kidney damage if elevated concentration is ingested. According to Saha and Saha, (2023) it can slow down the development of young plants and lead to leaf blight (Sadeghi *et al.,* 2022 and Zhou *et al.,* 2023).

CONCLUSION

It is worthy of noting that this study of PIXE technique has been achieved the ascertaining the concentration of eleven trace elements from vegetation samples that were collected from fifteen mining sites in Dange-Shuni LGA, Sokoto state. The elements identified by the high resolution PIXE analysis on the vegetation samples are Al, Mn, Zn, Pb, Ti, Cr, Ni, Co, Cu, Fe and As respectively. The results were obtained by using GUPIX via peak area calculation in the X-ray spectra by programing the background bremsstrahlung. This was enabled by spectral corrections interferences based on multi-elemental standards. In so doing, biological samples such as plants particles or vegetation samples whose matrix is made up of low-Z element with sort of undetectable via common X-ray spectrometer can be easily identified by PIXE. This was enhanced by relatively high K and Ca components. However, since soil is the dependent factor for plant and vegetation in general, the activities of mining for metal of economic importance in these localities will further expose the inner layer of the soil that harbors most primordial core elements to the surface for easy absorption to the vegetations and by elevating their concentrations to plants, man and other animals. This study therefore presents a baseline data for future research in this direction.

ACKNOWLEDGEMENTS

The authors are thankful to the supports of Federal Government of Nigeria, TETfund, Usmanu Danfodiyo University, Sokoto, Government College University, Lahore, Pakistan, the Laboratory personnel at Center for Advanced Studies in Physics, GC University, Lahore and Sadiq Shehu, Maniru Road, Sokoto. They were all very instrumental to the success of the PhD benchwork 2015/2016 that produces this research.

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