



DETERMINATION OF LEAD FROM GOLD ORE USING PROTON INDUCED X-RAY EMISSION TECHNIQUE

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ABSTRACT

The impetus for this research work arose from alleged signs of Lead (Pb) poisoning from Medicines Sans Frontiers (Doctors without Borders). These poisonings were narrowed down to areas of solid minerals mining and extraction in Northern Nigeria. The aim of this research work is to identify mining Sites with ores having high Pb concentration. Fifteen samples were collected from areas located at approximately latitudes 007008.690° E and longitudes 090 34' 224'' N and interrogated using Proton induced X-ray emission (PIXE) technique for their elemental content. PIXE was chosen because of its sample nondestructive and it does not contaminate the environment. The result obtained varied between 24.3 – 632303.3 ppm. The world Health Organization recommends that sites with Pb concentration above 400 ppm are inimical to human health and ordered that children be evacuated from such areas. Exposure to Pb poisoning may cause anemia, weakness, and Kidney and brain damage; particularly in children.

Keywords: Pb, Medecins Sans Frontiers, 400 ppm.

INTRODUCTION

To mitigate the dwindled revenue from petroleum, the government and people of Nigeria were prompted into mining of solid minerals as source of foreign exchange earner (Olasupo, 2018). The mining of solid minerals however comes with unintended hazards of Lead (Pb) poisoning (Utembe *et al.*, 2015). These hazards are believed to manifest in the form of strange illnesses associated with old and recent mining sites. The impetus for this work was to investigate what is the cause of these strange illnesses which have been superstitiously referred as a mysterious disorder caused by the gods.

This work collected samples from mining sites in Kebbi State in North Western Nigeria and assay them using proton induced-X ray emission (PIXE) technique. PIXE technique of assay is a powerful yet non-destructive, simultaneous trace multi-element analytical technique (Bello *et al.*, 2015). This technique bombards the collected sample with ions. Bombardment with ions of sufficient energy (usually MeV protons) produced by an ion accelerator, will cause inner shell ionization of atoms in a specimen. Outer shell electrons drop down to replace inner shell vacancies, however only certain transitions are allowed. X-rays of a characteristic energy of the element are emitted.

In the PIXE technique the energy of the characteristic X-rays identifies the element, and the number of emitted X-rays with characteristic energy peak is a measure of elemental concentration for specific element. In this technique, accelerated ions beam like alpha particles, protons and other heavy ions have many applications for the analysis in different research fields like archaeological, environmental, biological, geological etc. In PIXE technique, the accelerated proton beam is used due to its low bremsstrahlung radiations, high fluorescence yield and X-rays production cross-section (Johansson *et al.*, 1995). The

standardization of PIXE is a process in which materials of already known composition and concentration (Standard Reference Materials (SRM's)) are analysed for finding the elemental concentration and then compared with the SRM data. For this process it is necessary to set up a reliable calibration of the analytical system. The trace elements are those whose concentration are in the parts per billion (ppb) or tenth of parts per million (ppm) in a sample. For standardization of trace elements, accurate knowledge of beam parameters (energy, current and collected charge) and calibration parameters (fluorescence yield, sensitivity, ionization cross section, background radiation and absorption attenuation etc.) are required.

The assumption of this research work is that there may be Lead (Pb) poisoning in the areas where minerals are been extracted (from crushed boulders) via washing.

MATERIAL AND METHOD

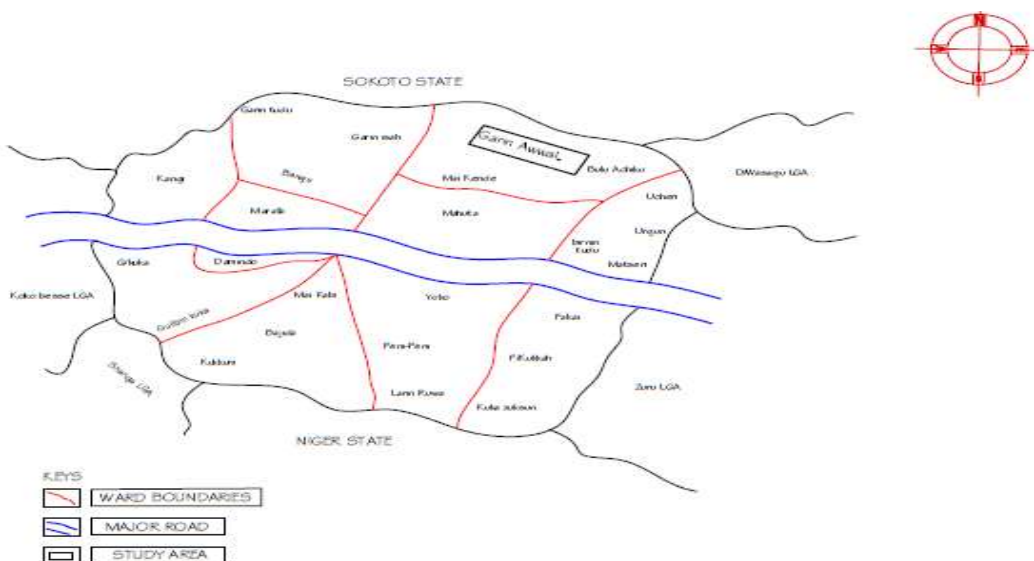
MATERIALS

The materials used include: Four rock samples, Global Positioning System (GPS), Hillquist thin section machine, Chemflex TM and GUPIX Software

METHOD

Fifteen samples were collected from GarinAwwal which is Located at approximately latitudes 007008.690° E and longitudes 090 34' 224'' N of North Eastern Nigeria. Of the fifteen samples collected two were biological (samples L and M). The samples collected were taken to Centre for Energy Research and Development (CERD), ObafemiAwolowo University, Ile-Ife. Osun State, Nigeria. Each bulk sample was

crushed and grinded into powdered form and then labelled separately to avoid contamination.



After thorough mixing of the powdered materials with some binding agent such as chemflex TM, pellets are prepared with a hydraulic press. Fifteen pellets are made and thereafter fastened to the specimen holder (special ladder akin to a slide projector, which enables the analysis of many (100) in sequence). The aluminium foil paper was placed behind the pellets before it was fastened to the special ladder to avoid the masking tape sticking to the pellets. It was then meticulously lowered in to the specimen chamber. Once the specimen was securely placed in the specimen chamber, the chamber is made vacuum by a special vacuum pump affixed to the chamber.

PIXE Calculations

The formula for calculating concentration [Y (Z)] in PIXE is given as:

$$Y(Z) = \frac{N_{av}\omega_Z b_Z t_Z \epsilon}{A_Z} N_p C_Z \int_{E_2}^{E_1} \sigma_Z \frac{E_y T^2(E)}{S_M(E)} dE \quad 2.1$$

Where N_p is the number of protons, N_{av} Avogadro's number, and $\sigma_Z(E)$ the K-shell ionization cross section for the proton energy E corresponding to depth x . The number of K X-rays in a particular spectral line is then obtained via the fluorescence yield $\omega_{k,z}$ and line intensity fraction $b_{k,z}$.

$$\begin{aligned} \frac{CZ(SP)}{CZ(ST)} &= \frac{Y_Z(SP)}{Y_Z(ST)} \\ &= \frac{I_{Z,ST}}{I_Z(ST)} \end{aligned} \quad 2.2$$

Standards are usually single elements or very simple compounds containing the elements of interest or their near neighbours in the chart of nuclides. The merit of this ratio process is its

cancellation of instrumental factors such as solid angle, efficiency, and calibration factors for charge integration (Aung *et al.*, 2002). This is important given the practical difficulties in obtaining accurate knowledge of the detector's line shape and intrinsic efficiency at the low X-ray energies characteristic of the light elements that are so often the major elements in environmental specimens (Sven *et al.*, 1995).

RESULTS AND DISCUSSION

Quality Control

Table 3.1 shows the results of irradiation carried out on a standard (NIST) 278 (Obsidian Rock). The table contains the analyte, standard and certain values. The observation from the table reveals that the certain values of the analyte silicon (Si), potassium (K), iron (Fe) and rubidium (Rb) respectively are of the standard. Analyte chlorine (Cl), vanadium (V) and zirconium (Zr) respectively are below the standard. Observation shows that PIXE is efficient for the analysis.

Table 1 (NIST) 278 (Obsidian Rock)

ANALYTE	STANDARD	CERT. VALUES (ppm)
Si	341397.3 ± 6281.71	341436
Cl	584.5 ± 88.03	-
K	34511.7 ± 106.99	34530
Ca	7020.8 ± 90.57	7026
Ti	1439.7 ± 22.75	1469
V	30.7 ± 15.76	-
Mn	401.2 ± 10.99	403
Fe	14275.1 ± 51.39	14268
Cu	6.8 ± 3.02	5.9
Zn	55.8 ± 5.96	55
Rb	127.5 ± 18.47	127.5
Sr	64.2 ± 13.67	.5
Zr	375.8 ± 39.46	-
Ba	1222.2 ± 165.49	1140
Ce	67.1 ± 42.94	62.2

The following are tables of the analyses of the fifteen samples collected from the mining sites in the field.

Table 2 tables of the analyses of the fifteen samples collected from the mining sites in the field

ANALYTE	SAMPLES (ppm)									
	A	B	C	D	E	F	G	H	I	J
S	5512.5	1134.5	4332.7	6319.2	451.1	1355.0	621.8	5102.8	-	7413.6
K	20605.3	7808.1	21195.4	10365.5	9133.4	20231.7	20144.2	3120.5	41904.2	3842.5
Ca	248.6	522.1	-	1439.1	267.5	1574.5	147.9	123.5	1663.7	155.9
Cr	751.5	550.1	736.0	614.1	604.8	747.5	1141.6	1281.5	832.9	1339.0
Fe	18568.8	19161.3	33595.0	18804.3	15148.7	50798.9	24904.1	22837.6	12407.2	33870.6
Cu	2451.8	97.5	272.4	402.3	34.5	408.1	61.7	46.3	7.4	465.2
Zn	32.7	25.4	30.4	35.9	-	291.5	34.7	28.4	52.1	-
Rb	52.5	50.1	82.9	33.2	-	122.1	87.8	48.4	88.3	-
Sr	57.2	63.0	127.9	80.8	-	502.5	104.2	33.4	207.1	39.6
Zr	138.8	76.0	175.7	240.3	148.9	206.0	167.3	-	480.8	
Ba	607.8	382.2	625.2	-	409.4	-	549.8	329.0	1331.4	538.8
Pb	942.9	444.5	3279.1	2119.7	529.4	3826.3	791.3	864.3	-	1030.2
P	-	879.8	-	-	-	1178.0	-	2120.7	663.9	1090.8
Ti	703.2	332.6	2148.3	1517.7	644.3	13570.1	723.4	97.3	1954.5	153.1
V	-	36.3	-	47.6	-	346.6	-	-	-	
Mn	-	96.1	146.1	-	66.7	399.3	-	-	102.5	
Cl	-	116.9	92.0	-	-	-	202.7	-	106.8	330.0
Ce	-	-	-	99.6	-	-	-	-	-	
Ni	-	-	-	-	7.6	14.2	-	-	12.6	

Table 2 continue;

ANALYTES	SAMPLES (ppm)				
	K	L	M	N	O
S	-	791.1	522.8	1404.4	957.9
K	-	5987.0	6168.6	16938.3	17451.5
Ca	226.3	1642.3	2092.1	5440.0	6537.6
Cr	112.6	186.2	190.3	400.2	399.6
Fe	389.5	1065.6	1198.6	2852.5	3136.3
Cu	57.6	20.1	16.7	30.2	24.0
Zn	-	26.6	30.7	40.3	47.5
Rb	-	864e-3	-	-	-
Sr	-	-	61.0	-	18.7
Zr	-	-	-	-	-
Pb	632303.3	892.7	105.8	214.0	24.3
Cl	5087.2	1910.3	1856.9	3923.6	3892.3
Ce	63.5	-	-	-	-
Ti	-	56.3	68.8	167.7	177.5
Mn	-	28.5	21.1	57.7	39.8

Pb which is the primary element of interest is found to be present in all the samples except sample I. Sample K has the highest concentration of Pb with the value of 632303.3 ppm. This value is so high that no human or a living thing should leave within such a surrounding because anything above 400 ppm (Utembe, 2015) is frowned upon as inimical to human health particularly children who in their formative years. Only samples of tables M, O and N have values below 400 ppm. Outside the three tables mentioned above, children are expected to be removed in all other areas the tables stipulated the Pb concentration is above 400 ppm. The site where sample K is collected is of great concern because Pb concentration is dangerously high.

CONCLUSION

This research work concluded that because Pb presences were in excess of what was recommended in sites where this work was undertaken, the authorities in the concern areas should be notified: they in turn will then create consciousness of the imminent health hazard, the authority should make provisions to cleanse areas already affected and prevent the creation of new ones and Constituted authorities should enforced mining ethics and ensured that they are been observed strictly.

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