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THE ELEMENTAL INVESTIGATION OF GOLD MATRIX BELIEVED TO BE LEAD-RICH USING PIXE TECHNIQUE

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ABSTRACT

To mitigate the dwindling of revenue from petroleum, the government and people of Nigeria added the mining of solid minerals as source of foreign exchange earner. The mining of solid minerals however comes with unintended hazards of Lead (Pb) poisoning. This work assay samples of gold matrix believed to be Pb rich from mining sites and determine the elemental content. Proton induced X-ray emission technique was used to interrogate the samples because of its low detection limit as well as its non-destructive and it does not contaminate the environment. The results showed that Pb content in all the samples but one, are way high above the USA limit of minimum permissible level of 400 ppm for areas with children. The results value variation is between 333 – 2575 (ppm).

Keywords: *Pb , PIXE, 400 ppm.*

1. INTRODUCTION

The price of petroleum – been the mainstay of Nigerian economy – is not stable, even more worrisome is often the low price occasioned by the gloat in the international market. This consequently prompted the country to search for other sources of revenue: hence the move to explore and exploit solid mineral deposits to mitigate the fall in revenue. This new search for new sources of revenue comes with its concomitant hazards.

This search for mineral was in time past primarily undertaken by artisans but lately international players are engaged by the government. As rewarding as these mineral ore are, they do come along with elements that are hazardous to the environment and to the people around the exploiting sites.

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. This technique bombard 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 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 motivation that informed this research was the widespread mining artisans undertake without recourse to the ethics of mining: not to harm the environment and the living things therein. More often animals and human beings in search of water fall into the dug deep pits leading to loss of lives. The poisoning of the water source that both animals and human beings drink which would have cumulative adverse effect on all that demography.

The assumption of this research work is that there may be Lead (Pb) poisoning in the areas where gold ore are been extracted (from crushed boulders) via washing; sometimes even the use of mercury to remove desire elements from crushed ore matrix tend to contaminate and poison source of drinking water.

2. MATERIAL S AND METHOD

A. MATERIALS

a) Four rock samples

b) Global Positioning System (GPS)

c) Hill quist thin section machine

d) Chemflex TM

e) GUPIX Software

B) METHOD

Four rock samples were collected from the site. The site is located at the range latitudes 007008.690'E and longitudes 090 34'224''N in Fakai local Government Area, Kebbi State, Nigeria. It is North to Sokoto State, East to DankoWasagu L.G.A, South to Niger State and West to Koko Besse L.G.A.

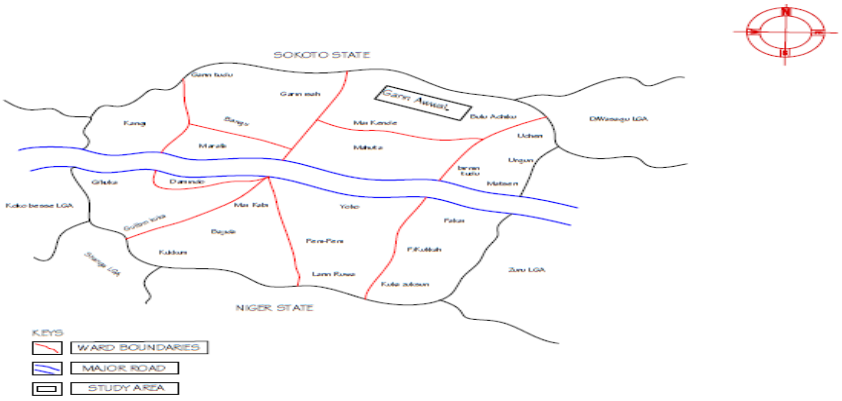

Figure2.1: Map of Fakai Local Government Area in Kebbi State, Nigeria.

C) SAMPLE PREPARATION AND IRRADIATION

The four geological samples collected were taken to Centre for Energy Research and Development (CERD), Obafemi Awolowo University, Ile-Ife,Osun State, Nigeria.

Each boulder of 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. Four 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 aluminum 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 vacuous by a special vacuum pump affixed to the chamber. It was now ready for irradiation.

Irradiation has to do with the bombardment of samples with energetic protons ion beam, Johansson et al, (1995). . With PIXE, Irradiation and spectrum generated were sequential. Each sample was irradiated and counted for ten minute (10 min).The projectile (proton) accelerated with 2.5 MeV Tandem accelerators at the (CERD). The proton hit the target atoms in the sample which led to the atomic interactions that give off electromagnetic radiation of wavelengths in the X-ray part of the electromagnetic spectrum specific to the element contained in the sample. The number of emitted X-rays with characteristic energy peak is a measure of elemental concentration for specific element. The X-ray spectrums emitted was passed through a window detected by silicon detector. Subsequently, the spectrum obtained was stored for qualitative and quantitative calculation at a later date. Irradiation was done together with quality control for relative quantitative calculation. But first, Irradiation for quality control was undertaken to assured us of the efficacy of the method in use.

D) PIXE Calculations

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

$$Y(Z) = \frac{N_{av}\omega_z b_z t_z \epsilon}{A_z} N_p C_z \int_{E_2}^{E_1} \sigma z \frac{EyT2\ (E)}{S_M(E)} dE \qquad 2.1$$

Where Np is the number of protons, Nav Avogadro's number, and σz (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 ωk,z and line intensity fraction bk,z.

If we generalized angle α and ΘTO for proton impact and X-ray take off en route to the detector, the X-ray intensity from the element of the path indicated suffers a transmission factor When specimens are thick enough to stop the beam, then we have Ef = 0 in Equation 1. The matrix (M) effect (i.e., those due to proton slowing and X-ray attenuation) are contained integral, which we denoted by IZ (M) where M denotes the specimen (SP) or the standard (ST)

$$\frac{CZ\ (SP)}{CZ\ (ST)} = \frac{Y_z\ (SP)}{Y_z\ (SP)} = \frac{I_{z_{ST}}}{I_z\ (ST)} \qquad 2.2$$

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.*, (2013). 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).

3. RESULTS AND DISCUSSION

QUALITY CONTROL

Table 3.1 shows the results of irradiation carried out on standard (NIST) 278 (Obsidian Rock). The table contains the analyte, standard and certify values. The observation from the table reveals that the certify 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 3.1 (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

Table 3.2 Average Concentration (ppm) of Elements in Sample A

Element	Oxide	Element Concentration (ppm)	Oxide Concentration (ppm)
Pb	PbO	756	814
Na	Na_2O	3503	4723
Mg	MgO	2660	4411
Al	Al_2O_3	46037	86985
Si	SiO_2	401090	858058
P	P_2O_5	714	1637
S	SO_3	78481	195964
K	K_2O	25060	30186
Ti	TiO_2	533	889
V	V_2O_3	61	90
Cr	Cr_2O_3	538	787
Mn	MnO	137	177
Fe	FeO	90510	116440
Ni	NiO	14	18
Cu	Cu_2O	35	39
Zn	ZnO	351	436
Se	SeO_2	64	91
Br	Br	153	153
Rb	Rb_2O	123	135
Y	Y_2O_3	65	82
Zr	ZrO_2	117	158
Ba	BaO	807	901
Dy	Dy_2O_3	2309	2650
Au	Au_2O_3	457	513

Table 3.3 Average Concentration (ppm) of Elements in Sample B

Element	Oxide	Element Concentration (ppm)	Oxide Concentration (ppm)
Pb	PbO	2390	2575
Na	Na_2O	57	77
Mg	MgO	545	904
Al	Al_2O_3	7254	13706
Si	SiO_2	73577	157404
S	SO_3	253510	633001
K	K_2O	2492	3002
Ca	CaO	200	280
Ti	TiO_2	394	657
Cr	Cr_2O_3	231	338
Mn	MnO	74	95
Fe	FeO	267407	344015
Ni	NiO	81	103
Cu	Cu_2O	24	27
Zn	ZnO	2880	3585
Se	SeO_2	396	556
Rb	Rb_2O	240	263
Sr	SrO	26	31
Dy	Dy_2O_3	12356	14181
Au	Au_2O_3	1579	1771

Table 3.4 Average Concentration (ppm) of Elements in Sample C

Element	Oxide	Element Concentration (ppm)	Oxide Concentration (ppm)
Pb	PbO	309	333

Na	Na_2O	537	724
Mg	MgO	49749	82488
Al	Al_2O_3	87232	164823
Si	SiO_2	242029	517776
P	P_2O_5	1517	3476
Cl	Cl	334	334
K	K_2O	53960	64999
Ca	CaO	35294	49382
Ti	TiO_2	18844	31432
V	V_2O_3	493	725
Cr	Cr_2O_3	335	490
Mn	MnO	1772	2288
Fe	FeO	141279	181753
Cu	Cu_2O	41	46
Zn	ZnO	580	722
Br	Br	62	62
Rb	Rb_2O	278	304
Sr	SrO	195	231
Zr	ZrO_2	53	71
Nb	Nb_2O_5	219	314
Mo	MoO_3	2682	4024
Dy	Dy_2O_3	354	406
Au	Au_2O_3	254	285

Table 3.5 Average Concentration (ppm) of Elements in Sample D

Element	**Oxide**	**Element Concentration (ppm)**	**Oxide Concentration (ppm)**
Pb	PbO	588	633
Na	Na_2O	281	378
Mg	MgO	795	1319
Al	Al_2O_3	9468	17890
Si	SiO_2	434689	929936
P	P_2O_5	601	1378

S	SO_3	5471	13662
K	K_2O	4127	4971
Ca	CaO	97	136
Ti	TiO_2	243	405
V	V_2O_3	17	25
Cr	Cr_2O_3	1371	2004
Mn	MnO	66	86
Fe	FeO	18284	23522
Ni	NiO	3	4
Cu	Cu_2O	6	7
Zn	ZnO	85	106
Se	SeO_2	56	79
Br	Br	68	68
Rb	Rb_2O	22	24
Sr	SrO	32	37
Y	Y_2O_3	65	82
Sn	SnO_2	2220	2819
Au	Au_2O_3	42	47
Th	ThO_2	467	532
U	UO_3	53	63

Beside Pb element which is the motivation of this research, other sough-after minerals are observed to be present in commercial quantity at the sites. These available minerals are likely to bring more revenue than the Au sought after. These mineral include chromium, iron, Thorium, zirconium, Uranium and gold.

Of the four samples analyzed, only one sample (sample C) has concentration (309 ppm) below the world health recommendation of 400 ppm. Any region or place observed to have values above world health recommendation, children are advised to be immediately evacuated from them because of the cumulative adverse health effect it may have on them later in life. These health challenges may include stunted growth, imbecility, and others.

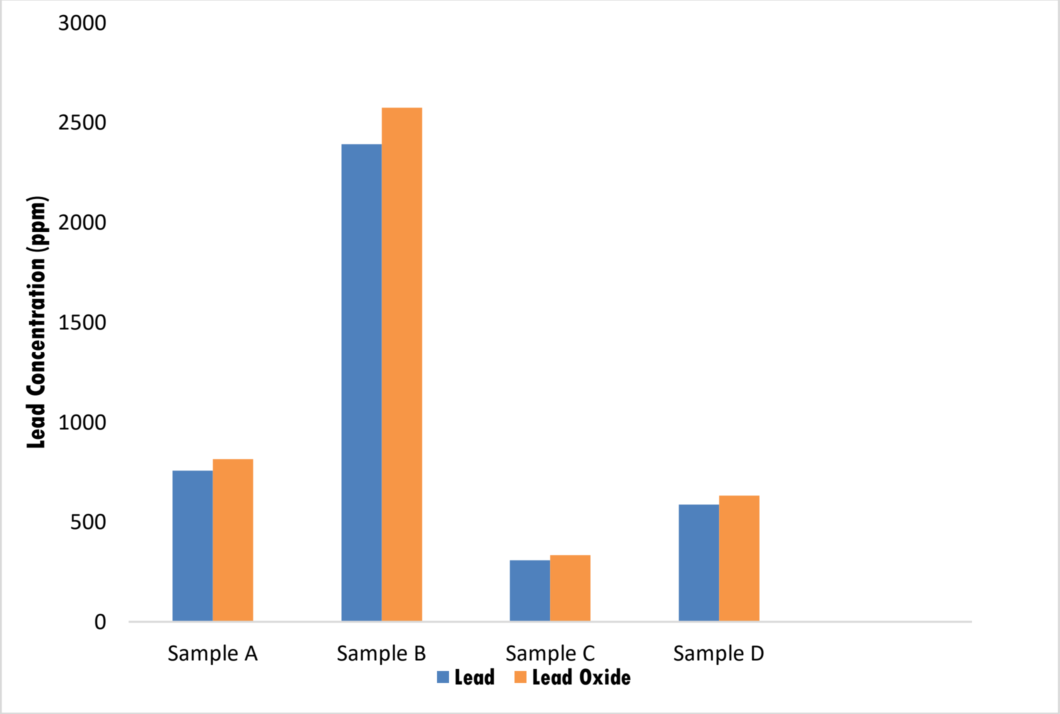

Figure 3.1: Concentration of lead in samples

Gold were found in all the four samples as speculated, even in concentrations that this researcher will advise specialists be recommended to undertake reconnaissance study in preparation for commercial exploitation.

4. CONCLUSION

The elemental assay of gold matrix believed to be lead-rich using pixe technique. Table 3.2 has lead concentration to be 814 ppm: This is way too high above the World Health Organization (WHO) recommendation. Equally Figure 3.1 illustrated that apart from sample C, all other samples in this research work lead concentration above recommended figure. Therefore this research work concluded that because Pb presence were in excess of what was recommended in sites where this work was undertaken, the authorities in the concern areas should be notified:

1) They in turn will then create consciousness of the imminent health hazard.

2) The authority should make provisions to cleanse areas already affected and prevent the creation of new ones.

3) Constituted authorities should enforced mining ethics and ensured that they are been observed strictly.

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