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**TRACE METALS ANALYSIS OF SOIL AND WATER SAMPLES FROM A LIMESTONE-MINING SITE IN SOKOTO STATE NIGERIA.**

<sup>a</sup>Itumoh, Emeka J. \*; <sup>b</sup>Izuagie, Thompson.; <sup>a</sup>Omaka, Ndukaku O.; <sup>c</sup>Uba, Ahmad.; <sup>b</sup>Shuaibu, Malami.; <sup>b</sup>Dogonyaro, Ismaila. A.; <sup>b</sup>Isah, Abdullahi.; <sup>b</sup>Dange, Attahiru. U. and <sup>b</sup>Isah, Saidu I.

<sup>a</sup>Department of Industrial Chemistry, Ebonyi State University, Abakaliki

<sup>b</sup>Department of Natural Sciences, Sokoto State Polytechnic, Sokoto

<sup>c</sup>Department of Pure and Applied Chemistry, Usmanu Danfodiyo University, Sokoto

\*E-mail: [ray.chukwuemekajude@yahoo.com](mailto:ray.chukwuemekajude@yahoo.com)

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**ABSTRACT**

Atomic absorption spectrophotometric determination of metal contents of soil and water samples of a limestone-mining site is presented in this report. The work is based on the concern that the concentration of metals in soils, ground and surface water increases as a result of mining and other industrial activities. Soil and water samples collected from the site in Kalambaina were analyzed for metals (Fe, Mn, Zn, Ni, Pb, and Cu) by AAS model 210 VGP after pre-sample treatments. The results yielded  $1.57 \pm 0.4 \text{ mgL}^{-1}$  and  $0.29 \pm 0.02 \text{ mgL}^{-1}$  Zn for soil and water samples respectively. Pb ( $0.03 \pm 0.007$ )  $\text{mgL}^{-1}$ , Mn ( $0.16 \pm 0.02$ )  $\text{mgL}^{-1}$  were detected in the soil sample only, while Ni, Cu and Fe were not detected in both the soil and water samples. Temperature, pH, electrical conductivities, turbidity, total alkalinity and hardness of the soil and water samples were analyzed to help in the study of the work. The result suggests that the metal concentrations of the site might have resulted from atmospheric depositions, traffic activities, bedrock geochemistry and mining activities. The metal concentrations of the studied site were all below the maximum permissible limits and thus poses no contamination threat to water flowing from it.

**Keywords:** Limestones, mining, trace metals, Kalambaina, Sokoto

**INTRODUCTION**

On-going researches in chemistry and other environmental subject areas are concerned with the study of possible pollution effects arising from man's activities on the environment such as mining, excessive fertilizer and pesticide use, irrigation and pollution by waste materials. Researches are also probing into the health effects of the pollution on man as a result of using ground and surface waters, plants and livestock exposed to these pollutions. Trace metal pollution is widely reported in literature <sup>[1-7]</sup> as one of the profound alteration of our environment. However, anthropogenic activities like mining and other industrial activities are recognized as some of the sources of metals in the environment <sup>[5,8-9]</sup> and metals released to the atmosphere from such activities travel for long distances and are deposited onto soil, vegetation and water. These metal ions, which are not degraded, persist and are retained in the ecosystem indefinitely <sup>[10-11]</sup>. Investigation has revealed that trace metal pollution in soils (lands) is a threat to surrounding aquatic ecosystems especially during storm water events <sup>[3,9]</sup>. Six (6) metals were chosen for the study for various reasons. Ni and Pb can pose a health risk to human via direct injection of soil (especially Pb) <sup>[12]</sup>. Ni is also phytotoxic <sup>[13]</sup>.

Fe, Zn and Mn are essential plant micronutrients but can be toxic at high concentrations [12]. Na, K, Mg and Ca were also analyzed because they are essential nutrients in soil, human body and in plant [14] and indicate the state of the potable water for human consumption. This paper is aimed at carrying out analytical studies on soils and water samples of a limestone-mining site in Sokoto, Nigeria in order to ascertain the extent of trace metal pollution on the soils as well as the water flowing from the site.

### **Area Description**

The study area, Kalambaina (13° 21' 16" N and 5° 5' 37" E) in Wamakko local government of Sokoto State is one of the notable areas because of the large deposits of limestone and other minerals. A cement plant (the Cement Company of Northern Nigeria) is located in the area, which is about 6 km from the capital – Sokoto. The State is in the illumedden basin, which is surrounded to the east and south by the Precambrian basement complex, covered by a series of sedimentary rocks, deposited over the basement complex. The sedimentary rock in the study area is classified as Sokoto group, which included the Kalambaina formation made up of limestone [15]. The climatic setting of the area is semiarid zone with savannah-type vegetation as part of the sub-Saharan Sudan belt of West Africa. The area is characterized by a prolonged dry season (October to May) and short wet season (mid-May to September). The annual mean precipitation varies from 350 mm (at Kalmalo in the extreme north) to 670 mm (at Sokoto airport) [16]. With an annual average temperature of about 39 – 41°C, Sokoto is one of the hottest cities in the world. The highest temperature occurs between April and July, the lowest in August (during the rainy season).

Mineral deposit surveys in Nigeria reveal that the Dange-Shuni belt down to Wamakko and Kalambaina, about 30 km in length, is rich in limestone, phosphate and gypsum [17]. The area's lifeline for growing crops is the food plains of the Sokoto-Rima River system, which are covered with alluvial soil. Crops like millet, maize, rice and other cereals, beans; vegetables, like tomatoes are grown in the area usually with irrigation. The limestone mining operations in Kalambaina area is dated back to 1960s. The site is under daily mining activities and operations for limestone used for cement manufacturing, with heavy-duty machines. Atmospheric dusts generated from the activities are transported to the nearby villages.

### **MATERIALS AND METHODS**

**Sample collection:** Soil and water samples were collected in April 2011 between 6:00 am to 8:00 am local time. The soil samples were collected with an improvised soil augur (a home-made hydrochloric acid sterilized PVC pipe) to a depth of 30cm [12] from areas approximately 1 m<sup>2</sup> [18] chosen to be representative of the land cross-section at the site. 50 samples were randomly taken from different spots and homogenized. The soil sample were placed in dust-free bags and zipped. Water samples were collected from the main source of the running water at the site with sterilized sampling containers. The water samples were placed in a cooler (with ice). All the samples were transported to the Sokoto State Water Board chemical laboratory, and chemistry laboratory, Usmanu Danfodiyo University, Sokoto

for various analyses. The grid references of the sampled spots were mapped using a portable Global Positioning System (GPS) (Table 1).

### **SAMPLE PREPARATION AND PHYSICOCHEMICAL ANALYSIS**

**Soil samples:** The soil sample was dried at ambient temperature (25°C) <sup>[19]</sup> in a GallenKlamp oven, and was passed through a 1 mm plastic sieve to remove large material and gravel-size materials <sup>[18]</sup>. The dried sample were then homogenized with a polypropylene mortar and sieved through a mesh sieve (diameter 60 µm). Small fraction of the soil was used for the determination of selected soil physicochemical properties. Soil pH was measured using a pH meter probe according to <sup>[20,21]</sup>. The soil temperature and conductivity were measured using probes <sup>[22]</sup>.

**Water samples:** In the laboratory the water sample was filtered. Turbidity, temperature, pH, electrical conductivity, total alkalinity and hardness of the water were determined according to standard procedures <sup>[22]</sup>

**Elemental and nutrient determinations:** The nitrate, phosphate, ammonium, chloride and sulphate contents of the soil and water samples were determined using standard methods <sup>[22]</sup>. Sodium, potassium, calcium and magnesium were determined using FES <sup>[22]</sup>.

**Trace metal determination:** The soil sample was dried in a GallenKlamp oven at 105°C for 6 hours. 1 g of the sample was placed in a beaker and 10 ml 1:3 mixture of hot HNO<sub>3</sub> and HCl (*aqua regia*) was added. The solution was filtered and the filtrate diluted to 40 ml with distilled water. The water sample was spiked with concentrated nitric acid according to <sup>[12]</sup> and filtered using Whatman no. 542 filter paper. The concentrations of the metals were analyzed using AAS Model 210 VGP.

**Quality Controls, Accuracy and Uncertainties:** All the analytical procedures preceding the instrumental measurements (cleaning of filters and all plastic items, sample treatments, preparation of standard solution) were carried out on clean benches in the chemistry laboratories. Sterilized and disposable rubber gloves were also used. Plastics (polypropylene and HDPE) containers were used for storing samples prior to analysis. All HDPE bottles for nutrient analysis were washed with nutrient-free detergent and were sterilized by soaking with 10% HCl AnalaR<sup>®</sup> (BDH, Poole, England) overnight and then rinsed several times with distilled water (except the containers for chloride determination). The bottles were air-dried prior to use. The HCl was used in order to prevent the sorption of suspended sediment and phosphorus compound onto interior surfaces of the bottles <sup>[23]</sup>. The uncertainties in the analyses were checked by analyzing replicates of the samples used in the experiment and averages were obtained. All reagents used were of analytical grade, AnalaR<sup>®</sup> (BDH, Poole, England).

## **RESULTS AND DISCUSSION**

**The physicochemical parameters:** The results obtained from the physicochemical analysis of the soil and water samples are presented in table 3. The soil pH was ( $6.7 \pm 0.3$ ) and that of the water was ( $7.51 \pm 0.04$ ). The soil temperature was above  $25^{\circ}\text{C}$ , which was effective for faster plant germination <sup>[24]</sup>. The water sample is colourless, odourless and tasteless. The results also show that the turbidity and the total hardness were within the maximum permitted limit of the European Union (EU), World Health Organization (WHO) and Nigerian Industrial Standards (NIS) for drinking water <sup>[25,26]</sup>. The normal hardness of the water may be because the excavated pit from which the water flows out has reached the water table and the water was pumped to the villagers without contact with the limestone soil.

**The nutrients and other major cations and anions:** The nutrient analysis was necessary because soil-bound nutrients create a reserve pool which, under specific conditions, are actively exchanged between the soil and the water (by storm water events), which leads to enrichment effects (eutrophication) <sup>[20,27-28]</sup>. Soil biogeochemistry and high atmospheric depositions represent the sources of N, P, and Cl in the samples analyzed. This study showed that the soil sample contained high concentration of chlorine ( $43.2 \pm 2.3$  mg/l) (see table 3), although the concentration in the water sample was below the maximum permitted level in drinking water (250 mg/l) <sup>[26]</sup>. Nevertheless, it is worrisome because, most of the chlorine in the soil evaporates into the air or combines with other chemicals to form stable compounds. Chlorine-containing chemicals that seep through the soil down into groundwater can remain unchanged for many years <sup>[29]</sup>. However, P forms apatite-complexes with metals [e.g  $\text{Ca}_5(\text{PO}_4)_3(\text{OH})$ ], and this could have resulted in low concentrations of P in the soil <sup>[20,28]</sup>.

**The trace metals:** Table 2 shows the results of the trace metals analysis. The transportation mechanism of trace metals is of interest to both environmental and soil scientists because of the possibility of groundwater contamination through metal leaching <sup>[30]</sup>. The concentration of Pb in the sample may be due to mineralization and mining <sup>[8]</sup>. Also, the traffic activities, and atmospheric depositions on the site could be an important sources of Pb in the samples analyzed. Tire wear might have contributed significantly to Zn concentrations <sup>[3]</sup>. The concentration of Zn may be related to atmospheric depositions. Zn concentrations may also be derived from the mechanical abrasion of vehicles and from Zn compounds used extensively as antioxidants (e.g., zinc carboxylate complexes and zinc sulphonates) as detergent/dispersant improvers for lubricating oils <sup>[18]</sup>. Zn existed in all the samples because it is a more abundant anthropogenic pollutant <sup>[31]</sup>. The concentration of Mn from the analysed samples could be attributed to background soil concentration because Mn is widespread in the environment, occurring in all rocks and soils <sup>[32]</sup>.

**The implications of bioaccumulation of analyzed elements in human systems:** It is obvious from the results that the concentrations of all the elements are within the maximum allowable levels and maximum permissible limits for agricultural soils and drinking water respectively. However, bioaccumulation of various elements with time is a major source of

concern. It is important to mention that chronic low-level intakes of these metals have adverse effects on human beings and other animals because there is no effective mechanism for their elimination from the body [33]. Excess concentrations of nutrients (especially N and P) cause fouling of natural waters as a result of algal blooms, which lead to toxic cyanobacteria [28]. Elevated nitrate concentrations in drinking water sources present a potential risk to public and animal health and concentrations greater than 10 mg l<sup>-1</sup> nitrate as nitrogen (NO<sub>3</sub><sup>-</sup>-N) can be fatal to infants under 6 months of age [34]. Nitrate can react with secondary amines present in the body resulting in the formation of carcinogenic nitrosoamines [35]. Mn enrichment in water may be toxic to fish and human, impair drinking water quality and can cause unpleasant metallic taste [36].

Cadmium is a cumulative poison, (i.e. the danger lies primarily in the regular consumption of foodstuffs with low contamination) [33,37]. Cd intake has been reported to cause renal, prostate and ovarian cancers [33]. Pb toxicity leads to anaemia both by impairment of haem biosynthesis and acceleration of red blood cell destruction and depression of sperm count [6]. Nervous system is susceptible to concentrations of lead and copper and the effects vary from brain damage (encephalopathy) to damage to the peripheral nerves, causing uncoordinated muscular control and poor eyesight [10]. Zn is potentially hazardous and excessive concentrations in soil lead to phytotoxicity (zinc sulphate is a weed killer) [6].

## **CONCLUSION**

The results presented in this work imply no serious environmental or health challenges since all the metals analyzed are within the maximum allowable levels and maximum permissible limits for agricultural soils and drinking water respectively. The analyses have also shown that the soil and water contains relative amount of some macro elements (cations and anions) like potassium, magnesium, calcium, phosphorus, nitrogen and chlorine. Thus, the paper concludes that trace metal concentrations of the studied site poses no contamination threat to water flowing from and through it and that, the soil could be applied for agricultural purposes. Future work: Different techniques and methods will be employed and data generated compared, to examine the effects on recovery of both digested and other samples.

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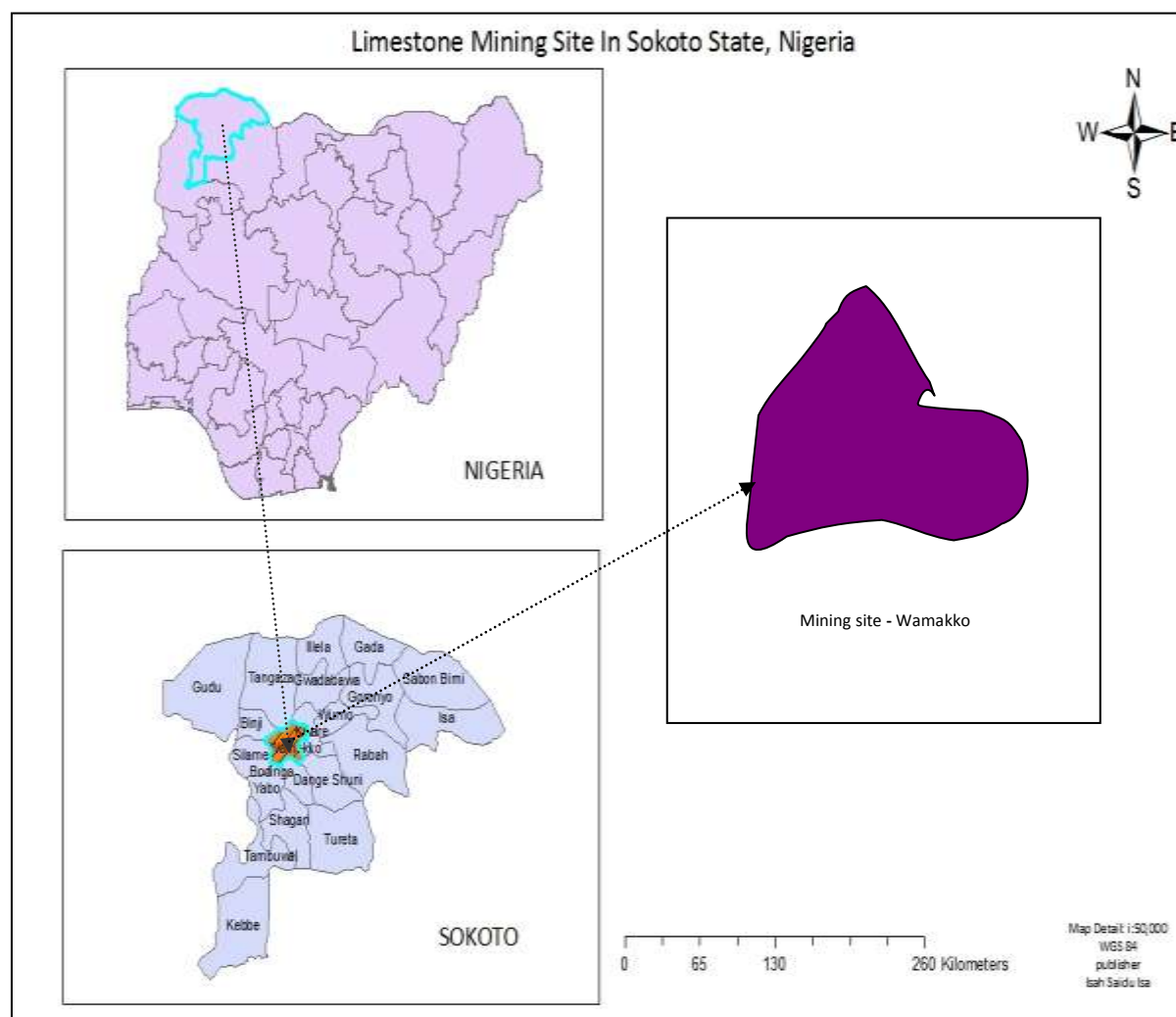


Fig. 1: The map of the study area - Sokoto, showing the mining site in Kalambaina, Wamakko local government area

**Table 1: Site Description with GPS**

Sample	Location N and E
Soil	13° 24' 38" N, 4° 34' 6" E
Water	13° 24' 38" N, 4° 34' 6" E

**Table 2: The Results of the Trace Metals (and the Maximum Allowable Levels (MAL) for agricultural soils and the Maximum Permissible Limit (MPL) for Drinking Water) [ $\pm$  SD,  $n = 3$ ]. N.D = not detected**

Metal	Soil	Water	MAL (soil) [12]	MPL (water) [25-26]
Zn	1.57 $\pm$ 0.4	0.29 $\pm$ 0.02	150-300	3
Pb	0.013 $\pm$ 0.007	0.001 $\pm$ 0.02	50-300	0.01
Mn	22.11 $\pm$ 0.02	ND	-	0.2
Ni	ND	ND	30-75	0.02
Cu	ND	ND	50-140	1
Fe	ND	ND	-	0.3

**Table 3: The Results of the Physicochemical Parameters, Nutrients, Major Cations and Anions (and the Maximum Permissible Limit (MPL) for Drinking Water). [ $\pm$  SD,  $n = 3$ ]**

Parameter	Soil	Water	MPL (Water) [25-26]
Temperature (°C)	27.2 $\pm$ 1.1	31.3 $\pm$ 0.6	Ambient
pH	6.7 $\pm$ 0.3	7.51 $\pm$ 0.04	6.5-8.5
Electrical Conductivity ( $\mu$ S/cm)	162.5 $\pm$ 6.3	24.9 $\pm$ 0.4	1000
Phosphate (mg/L)	0.07 $\pm$ 0.2	0.05 $\pm$ 0.002	-
Nitrate (mg/L)	1.0 $\pm$ 0.2	4.04 $\pm$ 1.12	50
Potassium (mg/L)	1.3 $\pm$ 0.2	0.53 $\pm$ 0.06	200
Sodium (mg/l)	1.5 $\pm$ 0.1	1.07 $\pm$ 0.06	-
Ammonium (mg/L)	0.4 $\pm$ 0.02	-	-
Chloride (mg/L)	43.2 $\pm$ 2.3	0.73 $\pm$ 0.03	250
Calcium (mg/L)	1.75 $\pm$ 0.3	-	-
Magnesium (mg/L)	1.0 $\pm$ 0.2	0.2 $\pm$ 0.01	0.20
Colour (TCU)	-	Colourless	15
Odour	-	Odourless	Unobjectionable
Taste	-	Tasteless	Unobjectionable
Turbidity (NTU)	-	1.3 $\pm$ 0.2	5
Total alkalinity (mg/L)	-	2.28 $\pm$ 0.03	500
Total hardness (mg/L)	-	10.7 $\pm$ 0.1	150