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Sequential Extraction Analysis of Cu, Pb, Ni and Zn in Cereals and Soils

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***Abstract*:** The estimation of Cu, Pb, Ni and Zn in guinea corn, maize and soil sample from Kaduna metropolis, Nigeria was carried out. The objective of the study was to determine the bioavailable Cu, Pb, Ni and Zn in guinea corn, maize and soil samples by flame Atomic absorption spectrometry (FAAS) using sequent extraction techniques. The concentrations of the metals in the cereals and soils vary from one sampling site to another. The t-test p = 0.000<0.05 for Ni, Cu, Zn and Pb respectively indicated a significant difference between the concentrations of metals in maize and guinea corn for all the four types of metals. It showed that maize contains more Ni than guinea corn contained more Zn than in guinea corn with a mean of 166.99mg/kg against 5.42mg/kg. Maize contained more than guinea corn with a mean of 37.62mg/kg against 3.17mg/kg. On the other hand, it showed that guinea corn contained more Cu than maize with a mean of 46.2mg/kg aganist21.88mg/kg. Similarly, it showed that maize soil contained about ten times more Ni than guinea corn soil with a mean of 38.23mg/kg against 3.91mg/kg. It also showed that maize soil contained more Zn than guinea corn soil contained more Pb than guinea corn soil with a mean of 28.57mg/kg against 3.32mg/kg. The test also indicated that the guinea corn contained more Ni than Cu with a mean of 28.45mg/kg against 3.68mg/kg.

Keywords: Sequential extraction, bioavailability, AAS, guinea corn, maize.

I. INTRODUCTION

Heavy metals occur in soils of the world and are essential to plants and animals. We ingest them through food, water and air. Their concentrations are usually low and hazardous with little or no environmental disturbance[1]. Most heavy metals remain in the soil nearly indefinitely as soils have long memories and do not degrade them. Ruinoffs as well as dust falls can result in accumulation in the roadside dusts/soils at levels that are toxic to organisms in the surrounding environments. These heavy metals remain bound to organic matter unless they are remobilized as windblown dust [2]. Living organisms require trace amounts of some metals like, cobalts, copper and zinc but their excess levels can be detrimental[3]. Other heavy metals such as mercury, lead and cadmium have no known or beneficial effects on organisms and their accumulation overtime in the bodies of mammals can cause illness[3].

The tetra alkyl lead accounts for 20% of lead used by mankind and accounts for the main environmental lead pollution problem. Street dusts and roadside soils on busy streets may contain Pb in the range of 1000 to 4000mgKg^{-1} [4]. Other sources of Pb would be attributed to mining and smelting of the ores, effluent from storage battery industries, emissions from automobiles exhausts [1] lead, affects every organ system in the body. It is absorbed into the body and distributed to the blood, soft tissue and bones [5].

Municipal refuse, automobiles and agricultural use of pesticide and fungicides containing $ZnSO_4$ are sources of Zn environmental pollution [6]. Zinc influences growth rate and bone development, the integrity of the skin, the development and function of the reproductory organs. It also helps in wound healing capacity [1].

The sources of nickel pollution are the air borne effluent from industrial units, emission of nickel into the atmosphere by the burning of fossil fuels and other anthropogenic practices [7]. Initial symptoms of toxicity due to nickel are nausea, dizziness, headache, chest pain, etc [7].

Iron and steel industries, fertilizer industry, burning of wood, disposal of municipal and industrial wastes are some of the sources of copper pollution [8]. Defects in pigmentation, bone formation, reproduction, myelin of the spinal cord, cardiac function and connective tissue formation are manifestations of copper deficiency [8].

In this research, Cu, Pb, Ni and Zn concentrations in guinea corn and maize samples were analyzed. The concentrations of these metals in the corresponding soil samples in the cereals were extracted using the chemical reagents such as the mixture of HNO_3/H_2O_2; 1.0mol L^{-1} oxalic acid, 0.05mol L^{-1} EDTA and 1.0 mol L^{-1} acetic acid. The relation between the concentrations of these metals in the cereals and soil samples was also investigated.

II. MATERIALS AND METHODS

A flame atomic absorption spectrophotometer model 8010 Young Lin was used for Cu, Pb, Ni and Zn determinations. In the extraction procedures, 1.0mol L^{-1} oxalic acid, 0.05mol L^{-1} EDTA and 1.0mol L^{-1} acetic acid were used.

Preparation of Samples:
The research covered seven agricultural sites in Kaduna, Nigeria. The sites are: Nasarawa (NS), Sabon Tasha (ST), Unguwar Mu azu(UM), Tudun Wada (TW), Kakuri (KK), Mando (MD), Kabala West (KB) and Kachia (KC). The samples were collected during the harvest season (Oct- Nov., 2008, 2009 and 2010). The soil samples were collected from the different areas enumerated at a depth of about 10cm below the surface [9]. The guinea corn and maize samples were collected at each of the locations. Guinea corn and maize samples were chosen for the purpose of this research work as they are the staple food being produced and consumed in these areas, Kachia. A town situated about 130km away from Kaduna was taken as a control, (Fig. 1). The cereals were thoroughly washed with water and allowed to drain on a filter paper. Both the cereal and soil samples were dried at 85°C. All the analysis were carried out in the Analytical Laboratory of Department of Applied Science, College of Science and Technology, Kaduna Polytechnic, Kaduna-Nigeria.

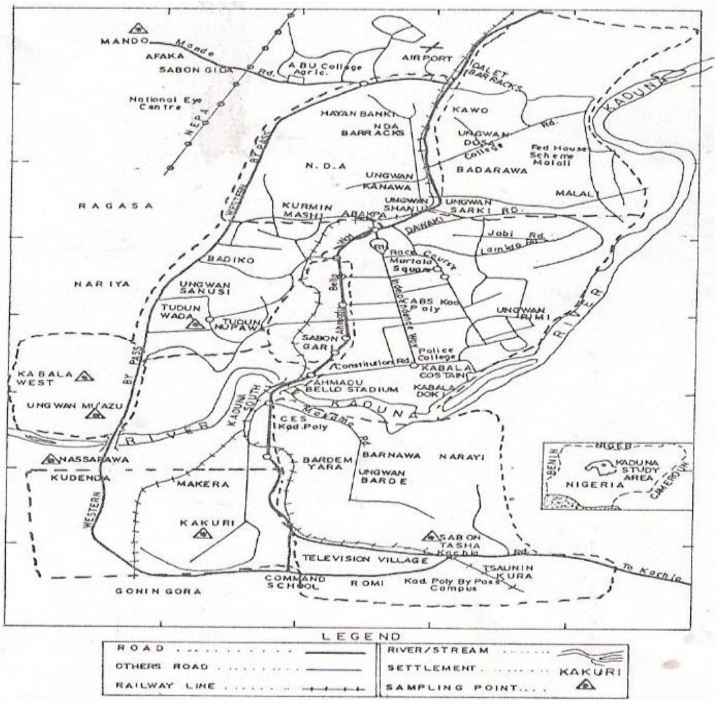

Fig. 1: Map of Kaduna Metropolis showing the sampling locations

Wet ashing of Cereals:

Five(5)g each of oven dried guinea corn and maize samples were accurately weighed into an evaporating dish and a steel at 480°C in an ashing furnace for 4h. Ten cm^3 of a mixture of nitric acid- hydrogen peroxide (2+1) was added to the ashed sample and dried with occasional shaking on a hot plate and cooled, 4cm^3 of 1.5mol L^{-1} nitric acid was then added and centrifuged sixty cm^3 water was added to the clear digest and was filtered. This was analysed for Cu, Pb, Ni and Zn, using FAAS model 8010 Young Lin. A blank digest was carried out in the same way.

Digestion and extraction:
A modified sequential extraction method [10] developed by Yaman was used [9]. Ten cm³ of a mixture of nitric acid-hydrogen peroxide (2+1) was added to 5g of soil sample and dried with occasional shaking on a hot plate and cooled. Four cm³ of 1.5mol L^{-1} nitric acid was added to the reminder, centrifuged and diluted to 60cm³ with water and filtered. The clear digest was analysed for Cu, Pb, Ni and Zn. Using FAAS model 8010 Young Lin. A blank digest was carried out in the same way. Soil extracts were obtained by shaking separately, 5g of soil samples with 10cm³ of 0.05mol L^{-1} EDTA (for carbonate and organically bound phase), 1.0mol L^{-1} oxalic acid (for oxide phases) and 1.0mol L^{-1} acetic acid (for carbonate phases). The mixture was evaporated with occasional shaking on a hot plate. Four cm³ of 1.5mol L^{-1} nitric acid was added to the reminder and centrifuged. The digest was diluted to 60cm³ with water and filtered and then analyzed for Cu, Pb, Ni and Zn using FAAS model 8010 Young Lin. A blank digest was carried out in the same way.

III. RESULTS AND DISCUSSION

Metal content in cereals and soils
The metal content in cereals and soils vary from one sampling site to another. Some sampling sites had higher concentration of the metal in cereals than the corresponding soils, while in others, the concentration of the metals was higher in the soils than the corresponding cereals. The copper and nickel contents of the soils from some of the sampling locations were above the tolerable limit. The higher concentrations of these metals in the areas could pose a problem to plant nutrition and food chain. Thus, constituting a direct health hazard [11].
The t-test $p = 0.000 < 0.05$, for Nickel, Copper, Zinc and Lead respectively indicated a significant difference between the concentration of metals in maize and guinea corn for all the four types of metals. It showed that maize contains more Nickel than guinea corn with a mean of 43.24 mg/kg against 2.87 mg/kg. Similarly, maize contained more Zinc than in guinea corn with a mean of 166.99 mg/kg against 5.42 mg/kg. Maize contained more Lead than guinea corn with a mean of 37.62 mg/kg against 3.17 mg/kg. On the other hand, it showed that guinea corn contained more Copper than maize with a mean of 46.21 mg/kg against 21.88 mg/kg (Figs. 2 and 3).

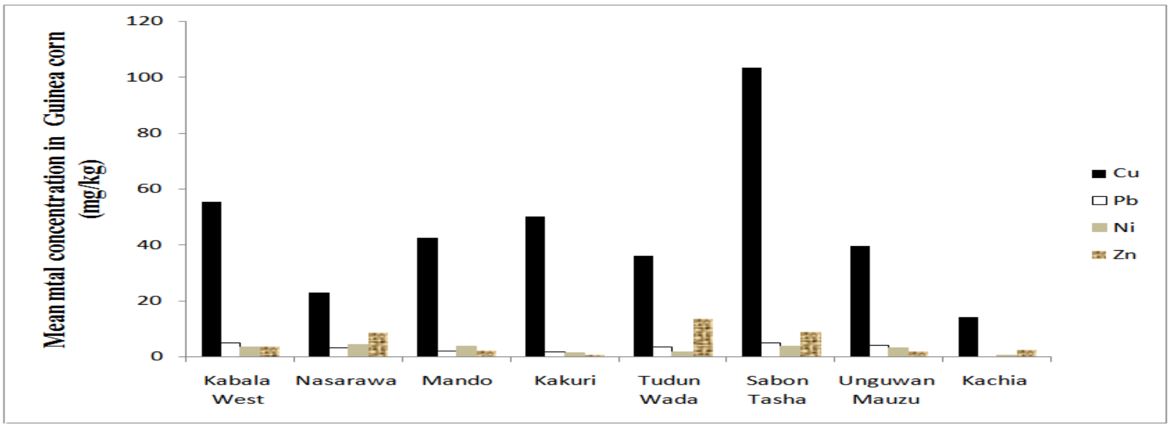

Fig. 2: Plot for mean Cu, Pb, Ni, Zn in guinea corn.

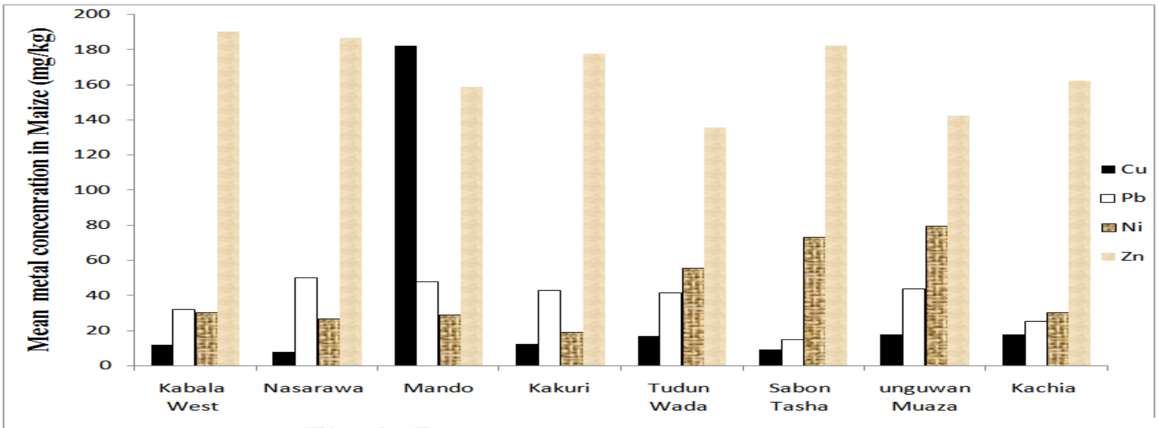

Fig. 3: Plot for mean Cu, Pb, Ni, Zn in maize.

Metal Speciation

The distribution of copper, lead, nickel and zinc in the soils vary from one sampling location to another. The metals generally exist in the forms; residual, oxide, carbonate/organic and Fe-Mn oxide.

The values of these elements recorded from the various sites revealed that they are either residual, oxide, carbonate/organic or Fe-Mn oxide species. The metal species associated with Fe-Mn oxide fraction is tightly held and bound and thus not readily bioavailable. Its release into the soil solution depends on strong depletion of minerals content of the soil solution [12].

The t-test $p = 0.000 < 0.05$ for Nickel, Copper, Zinc and Lead respectively indicated a significant difference between the concentrations of the metals in maize soil and guinea corn soil for all the four types of metals. It showed that maize soil contained about ten times more Nickel than guinea corn soil with a mean of 38.23 mg/kg against 3.91 mg/Kg. Similarly, it showed that maize soil contained more Zinc than guinea corn soil with a mean of 28.45 mg/Kg against 3.88 mg/Kg. Again, maize soil contained more Lead than guinea corn soil with a mean of 28.57 mg/Kg against 3.32 mg/Kg. On the other hand, it showed that guinea corn contains more Nickel than Copper with a mean of 28.45 mg/Kg against 3.68 mg/Kg (Figs. 4 and 5).

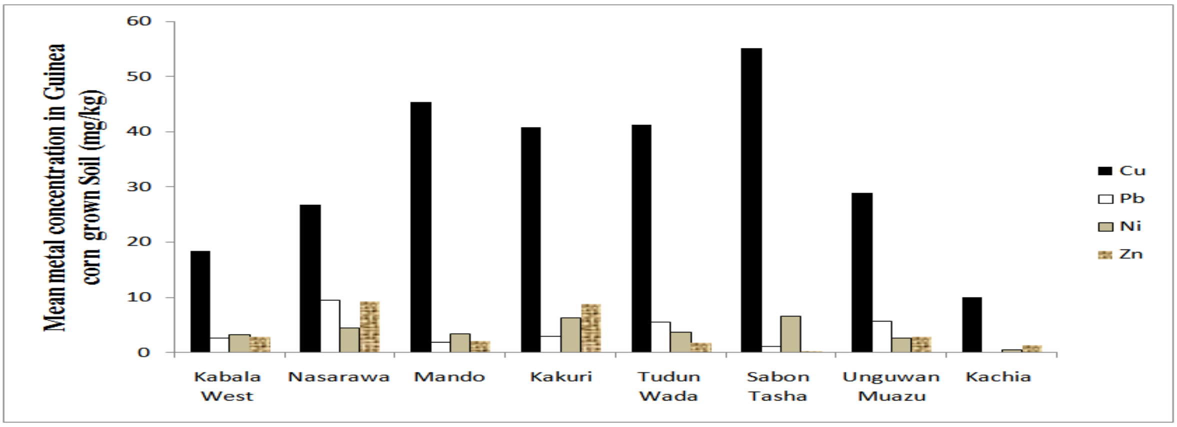

Fig.4: Plot for mean Cu, Pb, Ni, Zn in Guinea corn grown soils.

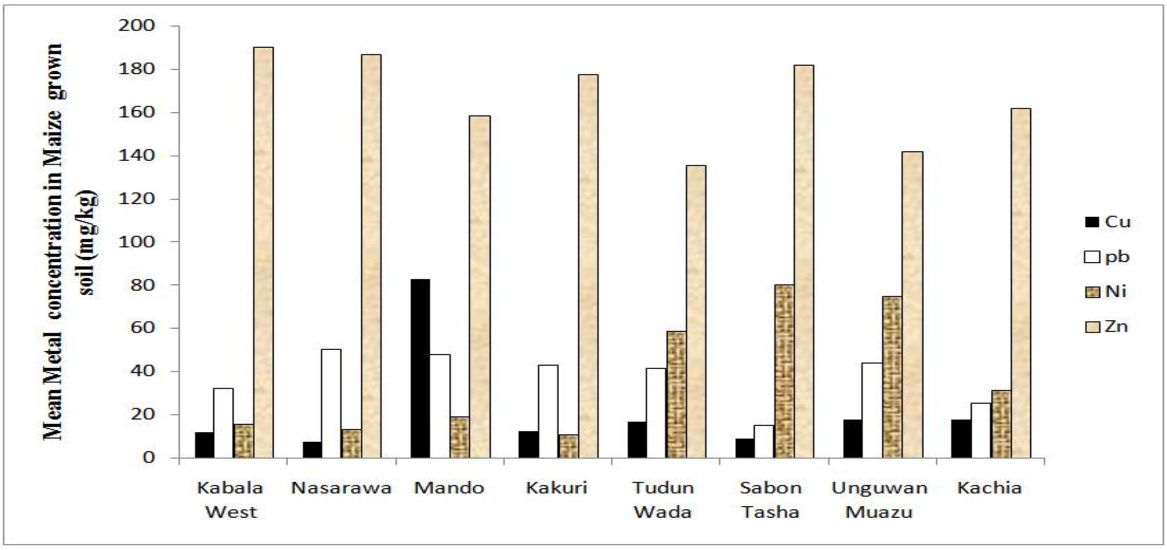

Fig.5: Plot for mean Cu, Pb, Ni, Zn in maize grown soils.

IV. CONCLUSION

Measurement of the metals concentration in soils is useful for determining the extent of contamination and for measuring any net change in soil metal concentration overtime. The method doesn't however give an indication as to the chemical form of the metal in the soil system [13]. A more commonly used procedure is the hot nitric acid-hydrogen peroxide procedure[9] . Which is a partial digestion of the soil solid phase.

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