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Identification of Chemical Forms of Lead in Soil and Fruits

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ABSTRACT: *The estimation of Pb in fruit and soil samples from Yelwa in Keffi Local Government Area of Nasarawa State, Nigeria was carried out. The objective of the study was to determine bioavailable Pbin these fruits and soil samples by flame atomic absorption spectrometry (FAAS) using sequential extraction technique. The concentrations of the metal in the fruit and soils vary from one sampling site to another. The soil samples related to the fruits where digested and extracted using different digestion and extraction reagents. The result revealed that the soil samples obtained from various locations contain varying amount of Pb and was distributed between residual, oxide, carbonate and exchangeable fractions. The result also showed that the concentration of Pb in the soil samples recorded was within the allowable limits of 200mg/kg and the ANOVA (p=0.000<0.05), showed that there is significant difference in the concentration of Pb in mango, orange, cashew and pawpaw Soils. Similarly, from the Duncan post hoc test, in the second homogenous subset, cashew soil has the highest concentration of Pb in their soils. On the other hand, in the second homogenous subset,mango, pawpaw and orange have less concentration of Pb in their soils.*

Keywords: *Sequential extraction, bioavailability, FAAS, fruit, soil, Pb*

I. INTRODUCTION

Apart from natural weathering processes, the sources of lead pollution are mining and smelting of the ores, emissions from automobile exhaust and effluent from storage battery industries use of glazed earthen wax-containers, bad pipes and lead pigments [1].Organic lead, as tetraethyl and tetra methyl lead is more acutely poisonous than inorganic lead. Lead affects every organ system in the body. Inhalation and ingestion are the two routes of exposure, and the effects from both are the same. Pb accumulates in the body organs (i.e., brain), which may lead to poisoning (plumbism) or even death. The gastrointestinal tract, kidneys, and central nervous system are also affected by the presence of lead. Children exposed to lead are at risk for impaired development, lower IQ, shortened attention span, hyperactivity, and mental deterioration, with children under the age of six being at a more substantial risk. Adults usually experience decreased reaction time, loss of memory, nausea, insomnia, anorexia, and weakness of the joints when exposed to lead. Lead can cause serious injury to the brain, nervous system, red blood cells, and kidneys[2]. Exposure to lead can result in a wide range of biological effects depending on the level and duration of exposure. Lead performs no known essential function in the human body and is particularly dangerous chemical element, as it can accumulate in individual organisms, but also in entire food chains. The most serious source of exposure to soil lead is through direct ingestion (eating) of contaminated soil or dust. The risk of lead poisoning through the food chain increases as the soil lead level rises above this concentration. Even at soil levels above 300 ppm, most of the risk is from lead contaminated soil or dust deposits on the plants rather than from up take of lead by the plant [3].This research is aimed at investigating the levels of Pb and relation to its concentrations in fruits.

II. MATERIALS AND METHODS

Study Area

The study area is located at Yelwa in Keffi town which is in the northern part of Nigeria. Yelwa is about 50km from Abuja, the Federal Capital Territory (FCT) and about 118km from Lafia, the Nasarawa State Capital, Nigeria. Yelwa is situated on latitude 10° 5’, north of the equator and longitude 10° 35’ west. This location is close to a major road, a mechanic workshop and some meters away surrounded by houses.

Instrumentation, Apparatus and Reagents

A flame atomic absorption spectrometer (model 8010: Young Lin) was used for Pb, Cd, Cr and Ni determinations.

An electro thermal oven (model: DHG) was used for drying the fruits samples, a 90 mics (0.09 mm) Standard Test Sieve was used for sieving the soil samples, funnel, filter paper, measuring cylinder, analytical balance, conical flask (pyrex), mortar and pestle, evaporating dish and centrifuge.

All of the reagents used to digest samples and for sequential extraction were of analytical reagent grade. 10 cm^3 of a mixture of nitric acid and hydrogen peroxide (2+1) was used for the digestion of fruit and soil samples. In the extraction procedures, 1.5 mol L^{-1} niric acid, 1.0 mol L^{-1} oxalic acid, 0.05 mol L^{-1} EDTA, and 1.0 mol L^{-1} magnesium chloride were used.

Sample Collection

Random sampling was used in the collection of both the fruits and soil samples. The samples were collected in October, 2015. The soil samples were obtained at 10cm depth and 100cm away from the trees where the fruits were obtained [5] and were put into separate polythene bags and labeled accordingly.

Sample Preparation

The fruit samples collected were washed thoroughly, rinsed with tap water, and allowed to drain. The fruits were peeled and then sliced into smaller pieces and the seeds removed. The peels were dried at 85˚C; using an electro thermal oven, model DHG. The oven dried fruits samples were stored in sample containers respectively and ready for ashing.

The soil samples were also oven dried at 85˚C; size reduced by the use of mortar and pestle; sieved using a Standard Test Sieve of 90 mics (0.09 mm) and then stored in samples containers respectively and ready for digestion and extraction.

Most samples require digestion before analysis so as to reduce organic interference by destroying all or most of the organic matter present in the sample into such a form that they can be analyzed with minimal interference.

Samples Digestion

Wet Ashing

Five grams (5g) each of oven dried fruit samples were accurately weighed using analytical balance into an evaporating dish and heat at 480˚C in an ashing furnace for 4 hours (4hrs). 10 cm^3 of a mixture of nitric acid-hydrogen peroxide (2+1) was added to each of the ashed samples and dried with occasional shaking on a hot plate and cooled, 4 cm^3 of 1.5 mol L^{-1} nitric acid was then added, and centrifuged and 6 cm^3 distilled water was added to the clear digest ashed samples and were filtered [4]. These samples were analyzed for Pb, using FAAS model 8010 young Lin. Blank digests were also carried out in the same ways.

Digestion and Extraction of Soil samples

A modified sequential extraction method [5] developed by Yaman was used (Tessier*et al.*, 1979). 10 cm^3 of a mixture of nitric acid-hydrogen peroxide (2+1) was added to 5 g of the soil samples and dried with occasional shaking on a hot plate and cooled. 4 cm^3 of 1.5mol L^{-1} nitric acid was added to the reminders, centrifuged and diluted to 60 cm^3 with water and were filtered. The clear digests were analyzed forPb using FAAS model 8010 Young Lin. Blank digests were carried out in the same way. Soil extracts were obtained by shaking separately, 5g of soil samples with 10 cm^3 of 0.05 mol L^{-1} EDTA (for carbonate and organically bound phases), 1.0 mol L^{-1} oxalic acid (for oxide phases) and 1.0 mol L^{-1} $MgCl_2$ (for exchangeable phases). The mixtures were evaporated with occasional shaking on a hot plate. 4 cm^3 of 1.5 mol L^{-1} nitric acid was added to the reminders, centrifuged and diluted to 60 cm^3 with water and were filtered. The clear digests were analyzed forPb using FAAS model 8010 Young Lin. Blank digests were carried out in the same ways.

III. RESULTS AND DISCUSSION

Content of Lead (Pb) in Soils and Fruits

The results of Pb concentration in fruit and soil samples are shown in Table **1.** The results showed that mango, orange, pawpaw and cashew have lower concentrations of Pb as compared to their corresponding soils. This could be attributed to the nature of soil, agricultural practices, nearness of the sampling location to major road and other anthropogenic activities within the locations. Most human activities such as driving, burning and dumping of electronic/metal waste like solder, storage batteries and empty cans into these locations contribute to the elevation in Pb levels[6]. The concentration of Pb in the location ranged from 2.673 to 8.400 mg/kg in the fruits. This may be interpreted that if the level of these metals in soils is significantly increased, the plants have the potential of showing increased uptake of the metals. This is also supported by studies that plants grown on soils possessing enhanced metal concentrations have increased heavy metal ion content [7,8]. This may be interpreted that if the level of this metal in soils is significantly increased, the plants have the potential of showing increased uptake of the metal. The level of Pb in the soils was found to be within the accepted limit of 200 mg/kg [9].

Lead (Pb) Speciation

The distribution of Pb in the soil samples showed that Pb exists in the forms; residual, carbonate, oxide and exchangeable phases. The concentration of Pb bound to the residual fraction is higher in cashew soil as compared to its concentration in the other extraction media. [10] Similar results for Pbwere found in regions influenced by anthropogenic activities, with trace metals that were mostly found in the labile fraction. This possibly cause a release of Pb into the soil solution and available for plant uptake. Thus, Pb is bio-available and mobile in the soil, similar results were reported by other researchers [4,11]. The concentration of Pb bound to the exchangeable fraction is higher in mango and pawpaw soils as compared to its concentration in the other extraction media; $\mathbf{Pb^{2+} + 2Cl^{2-} \rightarrow PbCl_2}$

Therefore, Pb is said to be an exchangeable species [12]. The concentration of Pb bound to the oxide fraction is higher in orange soil as compared to its concentration in the other extraction media and hence, Pb is said to be an oxide species and available for plant uptake [4].

$\mathbf{Pb^{2+} + O^{2-} \rightarrow PbO}$

The levels of Pb found in this study are lower than those from other studies in similar or related field [13].

The HNO_3/H_2O_2, EDTA, $H_2C_2O_4$, and $MgCl_2$extractablesare considered available in the locations.

Comparing the Concentration of Lead in Different Soils

Here we need to compare the concentration of Lead in mango, orange, cashew and pawpaw Soils. The one-way analysis of variance (ANOVA) is hereby applied for the test.

Table 1: Shows Results of Pb Concentrations in Fruit and Soil Samples

Results of mean value (mg/kg) ± STD DEV (n=3)

Sample	Pb Conc. in fruit sample (mg/kg) HNO_3/H_2O_2(2+1)	Pb Conc. in soil sample (mg/kg) HNO_3/H_2O_2(2+1)	Oxalic acid	EDTA	$MgCl_2$
Mango	2.673±0.142	8.493±1.200	19.680±0.300	25.340±1.500	26.400±0.600
Orange	4.160±0.730	9.333±0.800	15.400±0.500	13.890±0.200	6.213±0.200
Cashew	8.400±0.600	18.810±0.300	2.010±0.900	14.250±0.900	10.880±0.500
Pawpaw	3.520±1.600	8.500±1.200	19.700±0.320	25.34±1.542	26.360±0.600

Table 2: Lead Concentration in Soils (mg/kg)

Soils	N	Mean	SD
Mango	3	8.49	1.19
Orange	3	9.33	0.76
Pawpaw	3	8.49	1.19
Cashew	3	18.81	0.29

Table 3: ANOVA of Lead Concentration in Soils

Source of Variation	Sum of Squares	Df	Mean Square	F	Sig.
Between Groups	227.914	3	75.971	87.441	0.000
Within Groups	6.951	8	0.869		
Total	234.864	11			

From Table 3 above, since $p=0.000<0.05$, we conclude there is significant difference in the concentration of Lead in mango, orange, cashew and pawpaw Soils. The real difference is further investigated by the following Duncan multiple range post hoc test as shown in Table 4 below.

Table 4: Duncan test for Lead Concentration in Soils

Type of fruit	N	Subset	
		1	2
Mango	3	8.49	
Pawpaw	3	8.49	
Orange	3	9.33	
Cashew	3		18.81
Means for groups in homogeneous subsets are displayed.			

From Table 4 above, Duncan post hoc test, in the second homogenous subset, showed that cashew soil has the highest concentration of Lead in their soils. On the other hand, in the second homogenous subset,mango, pawpaw and orange have less concentration of Lead in their soils. This is shown in Fig. 1 below.

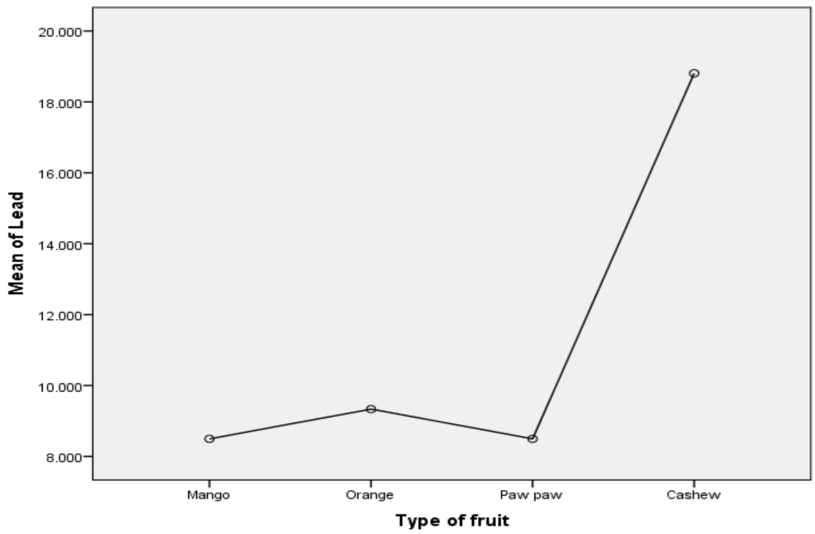

Fig. 1: Themean concentration of nickel against the type of fruit

IV. CONCLUSION

Total trace metal composition of soil is of little importance in determining its uptake by plants and consequently, in contaminating the food chain since the different forms have different mobilities, bioavailabilities and potential environment contamination potential. The results on heavy metal speciation in the study indicated that the soil samples collected from various areas contain varying amounts of the metal. The metal was distributed between residual, oxide, exchangeable and carbonate fractions. An increase of the metal concentration in some areas could be attributed to various agricultural practices in those areas.

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