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Zinc Speciation in Maize and Soils

Ayodele J.T. and Mohammed S.S.

Department of Pure and Applied Chemistry Bayero University, Kano - NIGERIA
Department of Applied Science College of Science and Technology Kaduna Polytechnic, Kaduna – NIGERIA

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Abstract

The levels of trace elements in food and agricultural samples have been shown to influence human and plant metabolism. The study of heavy metal speciation is of interest for the evaluation of their mobility, bioavailability and ecotoxicity. In this study, the zinc concentrations of the cereal and soil samples were determined using flame atomic absorption spectrometry (FAAS). The soil samples related to the cereal were digested and extracted using different digestion and extraction reagents. The results indicate that the soil samples collected from various locations, contain varying amounts of the metal, and it was distributed between Residual, Oxide, and Carbonate fractions. The results of the study also showed that the lowest value of total Zn concentration in soil was in sampling points KC1 (1.49 mg/kg) and the highest value was in the sampling points KK5 (207.2 mg/kg) and MD5 (207.2 mg/kg). It was found out that in all the sampling locations, the Zn concentration is within the tolerable range (10 – 300 mg/kg). The relationship between the cereal Zn and soil-extractable Zn concentrations was significant ($P < 0.05$).

Keyword: *Speciation, zinc, soil, cereal, flame atomic absorption spectrometry.*

Introduction

Zinc is essential for the function of more than 300 enzymes, phosphates, alcohol dehydrogenase, Superoxide dismutase, carboxypeptidase, δ – aminolevulinic acid dehydratase, carbonic anhydrase, and deoxyribonucleic acid polymerases[1]. Severe zinc deficiency in animals has been associated with reduced fertility, fetal nervous system malformations and growth retardation in late pregnancy[2].

Zinc contamination in soils and vegetation is derived from several anthropogenic activities[3]. Soils are the receptacles for metals released from industrial activities, municipal waste sludge, urban composts, road traffics, atmospheric deposits and agro-chemicals[4].

Heavy metals are persistent in the environment and are non-biodegradable thus readily accumulate to toxic levels[5]. The heavy metal content alone does not provide predictive insights on the bioavailability, mobility and fate of heavy metal contaminants [6]. It is the chemical form or species of the heavy metal that is a factor in assessing their impacts on the environment because it is their chemical forms that control its bioavailability or mobility[7].

The approach to soil speciation is to separate the soil into different chemical reagents or solvent fractions and analyse in each fraction the amount of element combined or associated with each fraction or phase[8]. Research on nutrient intake have shown that low dietary Zn poses a potential nutritional problem[9]. Therefore, the accurate Zn determination in food and soil is very important.

This study reports Zn concentrations in maize and soil samples by flame atomic absorption spectrometry (FAAS).

Material and Methods

The soil samples were dissolved by using the chemical reagents such as mixture of HNO_3/H_2O_2, $(COOH)_2$, Na_2EDTA and CH_3COOH. The relation between the maize contents and the Zn contents of soil extracts was also investigated.

In the digestion and extraction procedures, concentrated HNO_3, H_2O_2, 1.0M $(COOH)_2$, 0.05M Na_2EDTA and 1.0M CH_3COOH were used. Stock solution of Zn (1000mg/L) was prepared by dissolving Zn $(NO_3)_2$ in 1.0mol/L nitric acid.

Preparation of Samples: The study covered seven agricultural sites in Kaduna, Nigeria (fig. 1). The sites are: Nasarawa (NS), Sabon Tasha (ST), Unguwar Muazu (UM), Tudun Wada (TW), Kakuri (KK), Mando (MD) and Kabala West (KB). The samples were collected during the harvest season (Oct – Nov., 2008, 2009 and 2010). The soil samples were collected from different areas enumerated at a depth of 10cm below the surface[10]. The maize sample (maize) were collected at each of the locations. Kachia, a town situated about 130km away from Kaduna was taken as control. The sample was washed with water and was allowed to dry on filter papers. Both samples were dried at 85ºC. All the analyses were carried out in the analytical laboratory of the department of Applied Science, College of Science and Technology, Kaduna Polytechnic, Kaduna-Nigeria.

Wet Ashing of Maize: 5g of oven dried crushed and sieved maize sample was weighed into an evaporating dish and ashed at 480ºC for 4 hr. $10cm^3$ 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 1.5 mol/dm^3 nitric acid was then added and centrifuged. The digest was diluted to $60cm^3$ of water and filtered. This was analyzed for Zn using FAAS model 8010 Young Lin. A blank digest was carried out in the same way.

Digestion of Soil: Soil pH was measured (1:5, w/v) using microprocessor pH meter model, pH 210. $10cm^3$ of 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, $4cm^3$ of 1.5 mol/dm^3 nitric acid was added and centrifuged and diluted to $60cm^3$ with water and was filtered. The clear digest was analyzed for Zn using FAAS model 8010 Young Lin. A blank digest was carried out in the same way.

Extraction of Soil: Soil extracts were obtained by shaking separately, 5g of soil samples with $10cm^3$ of $0.05Mol/dm^3$ Na_2EDTA (for carbonate and organically bound phases), $0.1Mol/dm^3$ oxalic acid (for oxide phases), and $1.0Mol/dm^3$ acetic acid (for carbonate phases). The mixture was evaporated with occasional shaking on a hot plate. $4cm^3$ of $1.5mol/dm^3$ nitric acid was added to the remainder and centrifuged. This is referred to as hot extraction. The digest was diluted to $60cm^3$ and analysed for Zn using FAAS model 8010 Young Lin. A blank digest was carried out in the same way.

Results and Discussion

Zn content in Samples: The Zn content for the samples collected from the eight different locations at Kaduna, Nigeria is shown in Tables 1 to 8. The results of the Zn concentrations from each location indicate higher concentrations of the metal in maize samples than the soil samples and in agreement with results from other investigators[11-12].

The lower Zn concentration for some soil sites compared to others could be due to the lesser impact of anthropogenic sources[11]. Similar findings were observed by other investigators[13]. The higher Zn values obtained for the maize samples could be attributed to the various agricultural practices[14].

The pH of the soil samples from the various locations is acidic. Similar results were also reported by many authors[14-16].

Metal Speciation: The distribution of Zn in soil samples from the various locations showed that the metal exist in three operationally defined geochemical fractions; the residual, oxide and carbonate (Table 1 – 8). The concentration of the metal bound to oxide is generally higher than the carbonate or residual, except in Nasarawa (NS), where the carbonate is higher compared to the other two fractions. The predominance of the oxide fraction in these areas is in agreement with the result as reported by other investigators[11,17]. The dominance of the metal concentration in the carbonate fraction from NS is in agreement with the results of other investigators[10].

In case of total Zn concentration in the soil, the lowest value was in point KC1 and the highest was in sampling points KK_5 and MD_5. In all the sampling locations, the Zn concentration is within the range (10–300mg/kg) as reported[18]. The $(COOH)_2$ extractable, CH_3COOH extractable, EDTA extractable and HNO_3/H_2O extractable Zn have been considered as available Zn[18]. These Zn species should measure available concentration of Zn in the soils from the various locations[19].

The results of correlation calculations (Table 9) show that the HNO_3/H_2O_2, EDTA, $(COOH)_2$ and CH_3COOH_3 extractable and total Zn content in maize positively and significantly correlate with each other at 0.05 and 0.01 levels. Hence it could be deduced that the various reagents used were efficient for the extraction of this metal from the soil at these levels.

Conclusions

Different selective chemical reagents and the modified sequential extraction procedures used in this work were found useful in determining the mobility and chemical forms of Zn in soil. The results obtained show that the metal is distributed between Residual, Oxide and Carbonate fractions.

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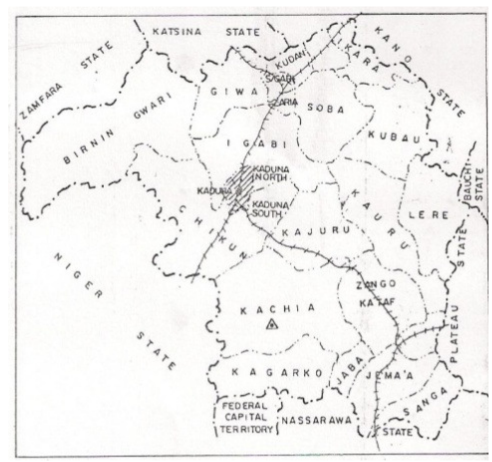

Figure: (A) Map of Kaduna State showing

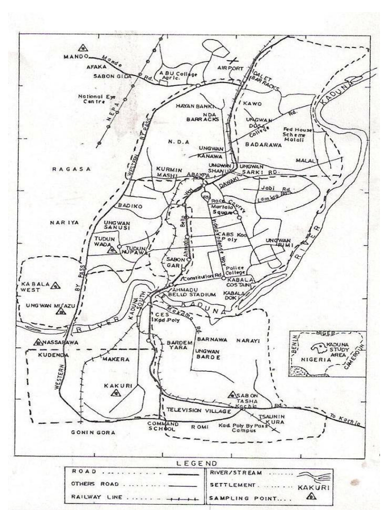

Figure: (B) Map of Kaduna Metropolis

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Table-1: Results of Zn Concentrations of Maize and Soil Samples in Kabala West
The results are mean values (mg/kg) ± standard deviation n = 3

	Metal conc in maize sample	Metal conc in soil sample	Hot Extraction			
Sample site Metals	Maize	HNO_3/H_2O_2	EDTA	Oxalic Acid	Acetic Acid	pH
	Samples	(2+1)	0.05M	1.0M	1.0M	
KB_1	38.76±0.7	10.44±0.7	11.18±0.7	3.73±0.7	9.69±0.7	5.16
KB_2	239.25±0.7	2.24±0.7	11.18±0.7	18.63±1.5	8.20±0.7	5.46
KB_3	245.96±7.5	2.98±0.7	11.93±1.5	14.91±7.5	8.94±0.7	5.36
KB_4	39.50±0.7	11.18±1.5	11.93±0.7	4.47±0.7	10.43±0.7	5.26
KB_5	240.00±0.7	2.98±0.7	11.93±0.7	19.38±1.5	8.94±0.7	5.66
KB_6	240.75±1.5	3.73±0.7	12.67±0.7	19.63±0.9	9.69±0.7	6.12
KB_7	240.00±0.7	3.73±0.7	11.93±0.7	20.12±1.5	8.94±0.7	6.12
KB_8	240.00±0.7	3.73±0.7	12.67±0.7	19.38±0.7	8.94±0.7	6.12

Table-2: Results of Zn Concentrations of Maize and Soil Samples in Nasarawa
The results are mean values (mg/kg) ± standard deviation n = 3

	Metal conc in maize sample	Metal conc in soil sample	Hot Extraction			
Sample site Metals	Maize	HNO_3/H_2O_2	EDTA	Oxalic Acid	Acetic Acid	pH
	Samples	(2+1)	0.05M	1.0M	1.0M	
NS_1	11.93±0.7	8.20±1.5	8.20±1.5	8.94±0.7	10.44±0.7	5.49
NS_2	241.49±1.5	2.24±1.5	55.90±1.5	6.71±0.7	13.42±0.7	6.12
NS_3	242.24±0.7	2.98±0.7	57.39±0.7	7.45±0.7	13.42±0.7	5.33
NS_4	13.42±0.7	8.94±0.7	8.94±1.5	9.69±0.7	11.18±1.5	5.92
NS_5	242.24±0.7	2.98±0.7	56.65±0.7	7.45±0.7	14.16±0.7	6.12
NS_6	242.98±0.7	3.73±0.7	57.39±1.5	8.20±0.7	14.91±1.5	6.45
NS_7	242.98±1.5	2.98±0.7	12.67±1.5	7.45±0.7	14.91±0.7	6.45
NS_8	242.98±0.7	4.47±0.7	58.88±0.7	7.45±0.7	14.16±1.5	6.45

Table-3: Results of Zn Concentrations of Maize and Soil Samples in Mando
The results are mean values (mg/kg) ± standard deviation n = 3

	Metal conc in maize sample	Metal conc in soil sample	Hot Extraction			
Sample site Metals	Maize	HNO_3/H_2O_2	EDTA	Oxalic Acid	Acetic Acid	pH
	Samples	(2+1)	0.05M	1.0M	1.0M	
MD1	11.93±0.7	11.93±0.7	13.42±0.7	11.93±0.7	12.67±0.7	4.15
MD2	206.46±1.5	20.87±0.7	33.54±1.5	51.43±0.7	35.03±1.5	4.45
MD3	205.71±0.7	21.61±0.7	34.29±1.5	52.17±7.5	35.03±1.5	4.56
MD4	13.42±1.5	12.67±1.5	14.91±7.5	13.42±1.5	14.16±1.5	4.35
MD5	207.20±0.7	207.20±0.7	34.29±0.7	51.43±1.5	35.78±0.7	4.26
MD6	207.95±1.5	22.36±0.7	35.03±1.5	52.92±0.7	36.52±0.7	4.75
MD7	207.20±0.7	22.36±1.5	34.53±0.9	52.17±0.7	36.52±1.5	4.75
MD8	210.43±5.0	22.36±0.7	34.29±0.7	51.43±1.5	35.78±0.7	4.75

Table-4: Results of Zn Concentrations of Maize and Soil Samples in Kakuri
The results are mean values (mg/kg) ± standard deviation n = 3

	Metal conc in maize sample	Metal conc in soil sample	Hot Extraction			
Sample site Metals	Maize	HNO_3/H_2O_2	EDTA	Oxalic Acid	Acetic Acid	pH
	Samples	(2+1)	0.05M	1.0M	1.0M	
KK1	10.44±0.7	11.18±0.7	11.18±0.7	11.93±0.7	13.42±0.7	5.08
KK2	257.14±1.5	27.58±1.5	30.56±0.7	41.74±1.5	23.85±1.5	4.98
KK3	258.63±1.5	28.32±0.7	31.30±0.7	42.24±1.6	25.09±1.6	5.14
KK4	13.42±1.5	12.67±1.5	14.9±17.5	13.42±1.5	14.16±1.5	5.14
KK5	207.20±0.7	207.20±0.7	34.29±0.7	51.43±1.5	35.78±0.7	5.18
KK6	207.95±1.5	22.36±0.7	35.03±1.5	52.92±0.7	36.52±0.7	5.58
KK7	207.20±0.7	22.36±1.5	34.53±0.9	52.17±0.7	36.52±1.5	5.08
KK8	258.63±0.7	28.32±0.7	32.05±0.7	42.48±1.5	25.34±0.7	4.98

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Table-5: Results of Zn Concentrations of Maize and Soil Samples in Tudun Wada
The results are mean values (mg/kg) ± standard deviation n = 3

	Metal conc in maize sample	Metal conc in soil sample	Hot Extraction			
Sample site Metals	Maize	HNO_3/H_2O_2	EDTA	Oxalic Acid	Acetic Acid	pH
	Samples	(2+1)	0.05M	1.0M	1.0M	
TW1	10.44±0.7	11.18±0.7	11.18±0.7	11.93±0.7	13.42±0.7	5.07
TW2	174.41±1.5	48.45±1.5	41.74±1.5	43.23±1.5	34.29±1.5	5.12
TW3	181.86±0.7	49.19±0.7	41.74±0.7	43.98±1.5	35.03±0.7	5.25
TW 4	15.65±0.7	13.42±0.7	15.65±0.7	14.91±7.5	12.67±1.5	5.27
TW 5	175.16±0.7	175.16±0.7	42.48±1.5	43.98±1.5	35.03±1.5	5.17
TW 6	175.90±0.7	49.94±0.7	43.23±0.7	44.72±1.5	35.78±0.7	5.49
TW 7	175.90±0.7	49.19±0.7	43.23±1.5	43.98±0.7	35.78±0.7	5.49
TW 8	174.41±0.7	47.70±0.7	40.99±0.7	43.73±1.6	34.29±0.7	5.49

Table-6: Results of Zn Concentrations of Maize and Soil Samples in Sabon Tasha
The results are mean values (mg/kg) ± standard deviation n = 3

	Metal conc in maize sample	Metal conc in soil sample	Hot Extraction			
Sample site Metals	Maize	HNO_3/H_2O_2	EDTA	Oxalic Acid	Acetic Acid	pH
	Samples	(2+1)	0.05M	1.0M	1.0M	
ST1	20.87±0.7	16.40±0.7	14.16±0.7	16.40±0.7	16.40±0.7	5.14
ST2	234.78±1.5	24.60±0.7	35.03±1.5	32.05±0.7	29.07±1.5	5.2
ST3	235.53±1.5	25.34±0.7	35.78±0.7	32.80±1.5	29.81±0.7	5.24
ST4	22.36±7.5	20.62±1.6	25.34±0.7	20.12±1.5	20.87±1.5	5.82
ST5	235.53±0.7	25.34±0.7	35.78±0.7	32.80±1.5	29.81±1.5	5.25
ST6	236.27±0.7	26.09±0.7	36.52±1.5	33.54±0.7	30.56±0.7	6.1
ST7	236.27±1.5	25.34±1.5	36.52±0.7	32.80±0.7	29.81±1.5	6.1
ST8	235.53±0.7	25.34±0.7	35.78±0.7	32.80±1.5	29.81±0.7	6.1

Table 7: Results of Zn Concentrations of Maize and Soil Samples in Unguwan Muazu

The results are mean values (mg/kg) ± standard deviation n = 3

	Metal conc in maize sample	Metal conc in soil sample	Hot Extraction			
Sample site Metals	Maize	HNO_3/H_2O_2	EDTA	Oxalic Acid	Acetic Acid	pH
	Samples	(2+1)	0.05M	1.0M	1.0M	
UM1	176.65±1.5	163.98±7.5	11.18±1.5	18.63±1.5	17.14±1.5	4.10
UM2	177.39±0.7	17.14±0.7	11.18±1.5	19.38±0.7	17.89±1.5	4.22
UM3	23.11±0.7	20.87±1.5	26.09±0.7	20.87±0.7	21.59±1.5	4.74
UM4	177.39±0.7	17.14±0.7	11.93±0.7	19.38±0.7	17.89±0.7	4.11
UM5	178.14±1.5	17.89±0.7	12.67±0.7	20.12±1.5	18.63±0.7	4.54
UM6	178.14±0.7	17.14±0.7	12.67±1.2	19.38±0.7	18.63±2.1	4.54
UM7	177.39±0.7	17.89±0.7	12.67±0.7	19.38±1.5	17.89±0.7	4.54
UM8	172.65±1.5	161.98±7.5	11.18±1.5	18.63±1.5	17.14±1.5	4.54

Table 8: Results of Zn Concentrations of Maize and Soil Samples in Kachia

The results are mean values (mg/kg) ± standard deviation n = 3

	Metal conc in maize sample	Metal conc in soil sample	Hot Extraction			
Sample site Metals	Maize	HNO_3/H_2O_2	EDTA	Oxalic Acid	Acetic Acid	pH
	Samples	(2+1)	0.05M	1.0M	1.0M	
KC 1	164.72±0.75	1.49±0.75	7.45±0.75	14.91±1.49	36.52±1.49	6.16
KC 2	166.96±0.7	2.24±1.5	4.22±1.1	4.47±0.7	6.71±0.7	6.24
KC 3	161.74±0.7	10.43±0.7	17.14±0.7	24.60±0.7	17.14±1.5	6.15
KC 4	161.74±0.7	10.43±0.7	17.14±0.7	24.60±0.7	17.14±1.5	6.08
KC 5	158.01±0.7	36.52±0.7	24.60±0.7	25.34±0.7	17.14±0.7	6.07
KC 6	163.23±0.7	20.87±0.7	27.58±0.7	24.60±0.7	21.61±0.7	6.14
KC 7	158.01±0.7	13.42±0.7	7.45±0.7	14.16±0.7	14.16±1.5	6.01

Table-9: Correlation Matrix for maize and different Zn extractable species in soil

(A)

Components	**Maize Sample**	**HNO_3/H_2O_2**	**EDTA**	**Oxalic Acid**	**Acetic Acid**	**pH**
Maize Sample	1	-.963**	.777*	-.886**	.935**	.478
HNO_3/H_2O_2		1	-.716*	.936**	-.854**	-.350
EDTA			1	-.683	.614	.202
Oxalic Acid				1	-.706	-.284
Acetic Acid					1	.686
PH						1

*Correlation is significant at the 0.05 level (2-tailed).
**Correlation is significant at the 0.01 level (2-tailed).

(B)

Components	**Maize Sample**	**HNO_3/H_2O_2**	**EDTA**	**Oxalic Acid**	**Acetic Acid**	**pH**
Maize Sample	1	.273	.998**	.999**	.999**	.672
HNO_3/H_2O_2		1	.280	.271	.281	-.357
EDTA			1	.999**	.999**	.680
Oxalic Acid				1	.999**	.671
Acetic Acid					1	.683
PH						1

**Correlation is significant at the 0.01 level (2-tailed).

(C)

Components	**Maize Sample**	**HNO_3/H_2O_2**	**EDTA**	**Oxalic Acid**	**Acetic Acid**	**pH**
Maize Sample	1	.213	.920**	.873**	.675**	.071
HNO_3/H_2O_2		1	.354	.389	.456	.768*
EDTA			1	.989**	.903**	.344
Oxalic Acid				1	.949**	.413
Acetic Acid					1	.576
PH						1

**Correlation is significant at the 0.01 level (2-tailed).

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(D)

Components	**Maize Sample**	**HNO_3/H_2O_2**	**EDTA**	**Oxalic Acid**	**Acetic Acid**	**pH**
Maize Sample	1	.517	.995**	.999**	.998**	.439
HNO_3/H_2O_2		1	.527	.523	.522	-.100
EDTA			1	.998**	.994**	.476
Oxalic Acid				1	.996**	.467
Acetic Acid					1	.453
PH						1

**Correlation is significant at the 0.01 level (2-tailed).

(E)

Components	**Maize Sample**	**HNO_3/H_2O_2**	**EDTA**	**Oxalic Acid**	**Acetic Acid**	**pH**
Maize Sample	1	.937**	.929**	.988**	.973**	.196
HNO_3/H_2O_2		1	.996**	.979**	.992**	.357
EDTA			1	.972**	.987**	.365
Oxalic Acid				1	.997**	.278
Acetic Acid					1	.315
PH						1

**Correlation is significant at the 0.01 level (2-tailed).

(F)

Components	**Maize Sample**	**HNO_3/H_2O_2**	**EDTA**	**Oxalic Acid**	**Acetic Acid**	**pH**
Maize Sample	1	.176	-.987**	-.559	-.909**	-.552
HNO_3/H_2O_2		1	-.290	-.697	-.509	-.242
EDTA			1	.642	.954**	.610
Oxalic Acid				1	.785*	.452
Acetic Acid					1	.641
PH						1

*Correlation is significant at the 0.05 level (2-tailed).
**Correlation is significant at the 0.01 level (2-tailed).

(G)

Components	**Maize Sample**	**HNO_3/H_2O_2**	**EDTA**	**Oxalic Acid**	**Acetic Acid**	**pH**
Maize Sample	1	.372	.991**	.981**	.260	-.627
HNO_3/H_2O_2		1	.403	.342	.959**	-.195
EDTA			1	.986**	.303	-.646
Oxalic Acid				1	.251	-.571
Acetic Acid					1	-.260
PH						1

**Correlation is significant at the 0.01 level (2-tailed).

(H)

Components	**Maize Sample**	**HNO_3/H_2O_2**	**EDTA**	**Oxalic Acid**	**Acetic Acid**	**pH**
Maize Sample	1	-.708	-.379	-.536	.098	.923**
HNO_3/H_2O_2		1	.784*	.630	-.127	-.464
EDTA			1	.873*	.089	-.154
Oxalic Acid				1	.256	-.397
Acetic Acid					1	.004
PH						1

*Correlation is significant at the 0.05 level (2-tailed).
**Correlation is significant at the 0.01 level (2-tailed).