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Zinc Fractionation of Soils of Different Parent Materials and their Relationships with Some Soil Properties

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ABSTRACT

Understanding the distribution of various fractions of micronutrients in soils is important for effective and efficient management of soils for optimum crop production and environmental conservation. Distribution of various Zn fractions in soils of different parent materials (coastal plain sands, alluvium, false bedded sandstones and Imo clay shale) in Imo State, South-eastern Nigeria was evaluated using sequential extraction procedure, comprising six fractions: water soluble, exchangeable, specifically sorbed/CO_3 bound, Fe-Mn bound, organic matter (OM) bound and residual bound. Soil samples were sequentially extracted and the various Zn fractions determined using atomic absorption spectrophotometer. Also, various Zn fractions were correlated to selected soil properties. On average value basis, zinc fractions in mg/kg decreased in the order of residual bound (0.695) > Fe-Mn oxide bound (0.598) > specifically sorbed/CO_3 bound (0.454) > exchangeable (0.241) > water soluble (0.209) > OM bound (0.143) in soils of Coastal Plain Sands; specifically sorbed/CO_3 (0.408)> water soluble (0.378) > exchangeable (0.375) > residual bound (0.250) > OM bound (0.217) > Fe-Mn oxide bound (0.121) in soils of alluvium; specifically sorbed/CO_3 bound (0.581)> residual bound (0.560) > Fe-Mn oxide bound (0.464) > OM bound (0.402) > exchangeable (0.283) > water soluble (0.182) in soils of false bedded sandstones; and residual (1.163) > specifically sorbed/CO_3 bound (1.086)> exchangeable (0.587) > Fe-Mn oxide bound (0.389) > OM bound (0.364) > water soluble (0.154) in soils of Imo clay shale. Available zinc concentrations were low and varied among the soils of different parent materials in a decreasing order of alluvium (0.753 mg kg^{-1}) > Imo clay shale (0.741 mg kg^{-1}) > false bedded sandstones (0.464 mg kg^{-1}) > coastal plain sands (0.449 mg kg^{-1}) while total zinc concentrations were in decreasing order of Imo clay shale (3.742 mg kg^{-1}) > falsebedded sandstones (2.471 mg kg^{-1}) > coastal plain sands (2.340 mg kg^{-1}) > alluvium (1.749 mg/kg). Zn fractions correlated among each other and with pH, OM, ECEC, available P, Ca and clay.

Keywords: Zinc fractions, parent materials, soil properties, South-eastern Nigeria

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INTRODUCTION

Zinc is one of the eight trace elements that are essential for normal healthy growth and reproduction of plants. It is required as a structural component of a large number of proteins such as transcription factors and metallo-enzymes (Figueiredo *et al.*, 2012). Sadeghzadeh (2013) noted that zinc is required for the functioning of all the six enzyme classes (oxidoreductase, transferases, hydrolases, lyases, isomerases and ligases) in plants.

Zinc deficiency is prevalent worldwide in temperate and tropical climates (Slaton *et al.*, 2005; Prasad, 2006; Fageria *et al.*, 2011). Many cases of its deficiency occur in developing (Third world) countries, where there is an urgent need to increase food production in order to feed their population without relying on food imports. Efforts are being made to reduce Zn deficiency in soils as it is not only a barrier to achieving crop yield goals but also results in low Zn content in grains and straw leading to poor Zn nutrition for humans and animals, a subject which recently received considerable attention (Schardt, 2006).

Zinc bioavailability is reported to be associated with its transformation in soils and plant continuum through various mechanisms, such as adsorption by clay surfaces, hydrous oxide minerals, organic matter and so forth, which affect Zn uptake by crops (Soltani *et al*., 2015). For a better understanding, total soil Zn can be broadly classified into five mechanistic fractions using sequential or batch fractionation schemes (Saffari *et al.*, 2009). These fractions include a water soluble pool, present in the soil solution, exchangeable pool with ions bound to soil particles by electrical charges, organically bound pool consisting of ions adsorbed, chelated or complexed with organic ligands, pool of zinc sorbed non-exchangeably onto clay minerals and insoluble metallic oxides and pool of weathering primary minerals (Alloway, 2008). These fractions provide broad information on the biological, geological and chemical processes occurring in a soil and are useful for predicting the availability of Zn for plant uptake. It has been reported that the residual Zn and oxide bound Zn are the more stable fractions while the exchangeable Zn and water soluble Zn fractions are rather more soluble and available to plants (Rahmani, *et al*. 2012).

Distributions of micronutrient forms vary with parent materials and profile depths (Verma *et al.,* 2005). It has been reported that soils derived from shale are rich in carbonate bound trace metal fractions (Hiller, 2006) while soils derived from false bedded sandstones are usually high in Fe-Mn oxides bound trace metal fractions (Gideon *et al.*, 2014).

The extent to which each fraction is present and the transformation in equilibrium between fractions is influenced by soil properties such as pH, cation exchange capacity, texture and soil organic matter (Ramzan *et al.*, 2014), Thus, the chemistry and effect of the aforementioned properties appear to be of major importance in determining the concentration of Zn fractions (Naik and Das, 2007). For instance, it has been reported that increasing soil pH increases concentration of carbonate bound zinc fraction (Meki *et al.*, 2012), whereas a decrease in pH increases concentration of water soluble zinc fraction (Kabata-Pendias and Pendias, 1999).

Despite several studies on zinc fractionation in soils, there have been few studies on zinc fractionation of soils from different parent materials. Therefore, the major objective of this study was to determine zinc fractionation of soils from different parent materials and their relationships with some soil properties.

MATERIALS AND METHODS

Study Area

The study was conducted in four different locations in Imo State, Southeastern Nigeria, where soils are derived from four different parent materials of coastal plain sands, alluvium, Imo clay shale and false bedded sandstones (Orajaka, 1975). The four study locations of different parent materials include Ihiagwa (coastal plain sands) located between latitude 5^0 21^1 and 5^0 27^1 N and longitude 7^0 02^1 and 7^0 15^1 E, Egwe (alluvium) located between latitude 5^0 42^1 and 5^0 46^1 N and longitude 6^0 47^1 and 6^0 49^1 E, Amauro (Imo clay shale) located between latitude 5^0 48^1 and 5^0 53^1 N and longitude 7^0 20^1 and 7^0 25^1 E and Mbato (false bedded sandstones) located between latitude 5^0 55^1 and 5^0 58^1 N and longitude 7^0 02^1 and 7^0 08^1. Imo State, South-eastern Nigeria lies between latitude 4^0 $40^1$and 8^0 15^1 N and longitude 6^0 40^1 and 8^0 15^1 E (Federal Department of Agricultural Land Resources, 1985) and is within the humid tropics. Temperatures are high and change slightly during the year (mean daily temperature of about 27^0 C). The average annual rainfall is about 2400 mm and there is a distinct dry season of about 3 months. Imo State has rainforest vegetation characterised by multiple tree species (Onweremadu *et al*., 2007). Agriculture and cottage industries are major socio-economic activities in the study area. Agricultural crops mostly cultivated in the study area include yam (*Dioscorea spp*), cassava (*Manihot spp*), oil palm (*Elaies guineensis*) and maize (*Zea mays*).

Soil Sampling and Routine Laboratory Analyses

A profile pit was dug in each parent material group, namely, coastal plain sands (Owerri), alluvium (Egwe), Imo clay shale (Amauro) and falsebedded sandstones (Mbato). Siting of profile pits was guided by the geological map of the study area. Soil samples were collected from soil horizons identified, starting from the bottom to the top to avoid contamination. The soil samples were air-dried, sieved using a 2-mm sieve and subjected to laboratory analyses. Routine analyses were conducted for particle size (Gee and Or, 2002) and pH in 1: 2.5 solute/suspension ratio using glass electrode of a pH meter (Thomas, 1996). Exchangeable cations (Ca^{2+}, Mg^{2+}, K^+, Na^+) were extracted with NH_4OAc buffered at pH 7.0 (Thomas, 1982). Exchangeable K^+ and Na^+ contents of extracts were read on flame photometer while exchangeable Ca^{2+} and Mg^{2+} were determined using atomic absorption spectrophotometer. Exchangeable acidity (Al^{3+} and H^+) was extracted with 1 N KCl (Thomas, 1982) and determined by titrating with 0.5 N NaOH using phenolphthalein indicator. Effective cation exchange capacity was obtained by summation of basic and acidic cations, organic matter was determined by wet oxidation method (Nelson and Sommers, 1982) while available P was determined by Bray II method (Olson and Sommers, 1982).

Zinc Sequential Fractionation Procedures

Water soluble, exchangeable, specifically sorbed/carbonate bound, Fe-Mn oxide, organic matter bound and residual bound Zn fractions were determined using the sequential extraction procedure as described by Salbu *et al.* (1998). The procedure is outlined as follows:

Water Soluble Fraction (F1): This was extracted as follows: Two grams of soil sample were weighed into a 50-mL polycarbonate centrifuge tube and extracted using 20 mL of de-ionised water for 1 h at 20^0 C on a rolling table.

Exchangeable Fraction (F2): In this procedure, residue obtained from water soluble extraction was washed with 10 mL de-ionised water and the washes discarded. The washed residue was transferred into a 50-mL polycarbonate centrifuge tube and extracted with 20 mL of 1M NH_4OAc solution buffered at pH 7 for 2 h.

Specifically Sorbed and Carbonate Bound Fraction (F3): In this procedure, residue obtained from extraction of exchangeable form was washed with 10 mL de-ionised water and the washes discarded. The washed residue was transferred into a 50-mL polycarbonate tube and extracted with 20 mL of 1M NH_4OAc solution buffered at pH 5 for 2h.

Fe-Mn Oxide Bound Fraction (F4): In this procedure, the residue obtained from the extraction of specifically sorbed and carbonate bound form was washed with 10 mL de-ionised water and the washes discarded. The washed residue was transferred into a 50-mL polycarbonate tube and extracted with 20 mL of 0.04 M $NH_2OH.HCl$ in 25% HOAc for 6 h in a water bath at 60^0 C.

Organic Matter Bound Fraction (F5): In this procedure, residue obtained from the extraction of Fe-Mn oxide bound form was washed with 10 mL deionised water and transferred into a 50-mL polycarbonate tube and extracted with 15 mL of 30% H_2O_2 at pH 2 (Adjusted with HNO_3) for 5.5 h in a water bath at 80^0 C. The content was allowed to cool and 5 mL of 3.2 M NH_4OAc in 20% HNO_3 was added and diluted to 20 mL with deionised water.

Residual Fraction (F6): In this method, 1g of the residue obtained from the extraction of organic matter bound form was dried after which it was digested in a conical flask with 10 mL of 7 M HNO_3 on a hot plate for 6 h. After evaporation, 1 mL of 2 M HNO_3 was added and the residue dissolved. Thereafter, it was diluted using 10 mL de-ionised water.

After each successive extraction, the mixture was centrifuged at 1000 rpm for 30 min and the supernatant decanted into polyethylene bottles, acidified to pH <2 and stored for analysis. The various chemical fractions of Zn were determined using ICE 3300 atomic absorption spectrophotometer at 324.8 nm.

Available and Total Zinc Determination

Available zinc was calculated as sum of water soluble and exchangeable fractions while total zinc was calculated as sum of all the fractions determined (Ramzan *et al.*, 2014).

Statistical Analysis

Data generated were subjected to coefficient of variation to determine variability in distribution of Zn fractions in the soil profiles studied and ranked according to the method of Wilding et al. (1994). The relationship between selected soil properties and Zn fractions was estimated using simple linear correlation analysis.

RESULTS AND DISCUSSION

Physico-chemical Properties of the Soils of Different Parent Materials

The results of physico-chemical properties of the soils are presented in Table 1. Particle size distribution analysis indicated predominance of sand particles (94.7%) over clay (4%) and silt (1.3%) particles in the soils of alluvium. A similar distribution trend was observed for soils of coastal plain sands and false bedded sandstones with sand, silt and clay particles having mean values of 85.6%, 3.6% and 10.8%, respectively, in soils of coastal plain sand soils and 54.2%, 17.4% and 28.4%, respectively, in soils of false bedded sandstones. But in soils of Imo clay shale, clay (42.8%) particle size was the highest followed by sand (34%) and silt (23.2%) The high clay content of the soils of Imo clay shale could be attributed to the clayey nature of the shale parent material from which the soils are derived from. Generally, clay particle size increases with depth and could be due to the illuviation pedogenic process that may have taken place in the location. Soils of coastal plain sands and alluvium were dominated by sandy loam and sand texture, respectively (Table 1). That of soils derived from false bedded sandstones and Imo clay shale were dominated by sandy clay loam and clay texture, respectively. Soil pH ranged from 4.28 - 5.63 in coastal plain sand soils, 5.32 - 5.62 in alluvial soils, 5.12 - 5.41 in false bedded sandstone soils but in the Imo clay shale soils, it was in the range of 5.55 - 5.98. These values varied from extremely (<4.5) acidic to moderately (5.6 - 6.5) acidic (FAO, 2004). The low pH of the soils could be due to high amounts of rainfall in the study area, resulting in leaching of basic cations; this has led to the exchange complex of the soils to be dominated by acidic cations. Similar pH results have been reported by Eshett *et al.* (1990) in soils of South-eastern Nigeria.

TABLE 1
Physico-chemical properties of the soils studied

Horizon	Soil Depth (cm)	Sand →	Silt (%)	← Clay	OM (g/kg)	Ca (cmol$_c$ kg^{-1})	pH (H_2O)	Avail. P (mg/kg)	ECEC (cmol$_c$ kg^{-1})	TC
					Coastal plain sands					
Ap	0-17	90	4	6	13.99	1.22	4.28	0.86	4.14	S
AB	17-36	88	4	8	1.28	1.61	4.89	0.66	4.7	S
Bt1	36-53	86	2	12	1.28	1.22	5.63	0.73	3.75	LS
Bt2	53-91	84	4	12	6.41	2.03	4.91	0.47	5.11	LS
Bt3	91-150	80	4	16	1.56	1.81	5.26	0.56	5.09	SL
	Mean	**85.6**	**3.6**	**10.8**	**4.99**	**3.79**	**0.66**	**0.66**	**4.56**	
					Alluvium					
Ap	0-4	94	2	4	2.27	1.22	5.36	2.19	4.03	S
BC	4-84	96	0	4	1.91	1.61	5.62	1.44	3.81	S
C	84-100	94	2	4	1.91	2.01	5.32	2.56	4.67	S
	Mean	**94.7**	**1.3**	**4**	**2.03**	**1.61**	**5.43**	**2.06**	**4.17**	
					False bedded sandstones					
Ap	0-9	68	14	18	38.51	3.41	5.41	0.92	6.47	SL
AB	9—28	50	20	30	5.72	1.4	5.41	0.76	4.14	SCL
Bt1	28-49	52	18	30	13.63	1.83	5.21	0.36	4.69	SCL
Bt2	49-73	52	16	32	2.24	1.61	5.12	0.27	4.88	SCL
Bt3	73-170	49	19	32	1.91	1.61	5.31	0.23	5.14	SCL
	Mean	**54.2**	**17.4**	**28.4**	**12.4**	**1.97**	**5.29**	0.51	**5.06**	
					Imo clay shale					
Ap	0-11	44	28	28	48.87	2.04	5.56	3.36	5.37	CL
AB	19—11	30	26	44	47.95	1.02	5.56	0.39	2.89	C
Bt1	19-36	26	24	50	47.78	3.23	5.55	0.23	6.59	C
Bt2	36-55	28	12	60	43.28	2	5.98	0.96	4.79	C
Bt3	55-83	42	26	32	46.03	2.61	5.85	1.56	6.78	CL
	Mean	**34**	**23.2**	**42.8**	**46.78**	**2.18**	**5.7**	1.3	**5.28**	

OM- organic matter, Avail. P- available phosphorus, ECEC-effective cation exchange capacity, TC-textural class,S-sand,LS-loamy sand, SL-sandy loam, SCL- sandy clay loam,CL-clay loam, C- clay,

Effective cation exchange capacity (ECEC) of the soils was higher in Imo clay shale soils (5.28 cmol$_c$ kg^{-1}), attributable to higher clay content of the soils (Table 1) followed by false bedded sandstones soils (5.08 cmol$_c$ kg^{-1}), coastal plain sands soils (4.56 cmol$_c$ kg^{-1}) and alluvial soils (4.09 cmol$_c$ kg^{-1}). These values are low compared with the critical limit (6 cmol$_c$ kg^{-1}) recommended by Esu (1991) for arable crop production. This property is found to be an important predictor of metal retention, movement (Udom *et al.*, 2004) and extractability (Rieuwerts *et al*, 2005) in soils. Hence, the low ECEC of the soils indicates that the soils have low metal retention capacity. Onweremadu *et al*. (2011) also reported low ECEC in soils derived from coastal plain sands, alluvium and false bedded sandstones in South-eastern Nigeria which is attributed to parent material, climate and land use interactions. Higher ECEC values were recorded mostly in the surface horizons (Ap) and could be due to higher organic matter content of the horizons. Organic matter (OM) content of all the soils studied was low (FAO, 2004) with mean values of the soils ranging from 2.03 g kg-1 to 46.78 g kg^{-1}. The low organic matter concentrations of the soils could be attributed to high temperature and rainfall in the study area which encourages rapid mineralisation, erosion and leaching of organic matter. Generally, OM decreased with soil depth which is in line with the report of Uzoho *et al.* (2014). With the exception of soils derived from Imo clay shale where exchangeable calcium attained a moderate level (2.16 $cmol_c$ kg^{-1}), other soils had low exchangeable calcium (<2 $cmol_c$ kg-^{1}). Onweremadu *et al.,* (2011) also reported low exchangeable calcium in soils of South-eastern Nigeria derived from coastal plain sands and alluvium and attributed the low results to the sandiness of the soils which encourages leaching of calcium. Available P concentrations of the soils were low (0.23-3.36 mg kg^{-1}) and below the critical limit (10 mg kg^{-1}) recommended by Esu (1999) for arable crop production. The low values of available P in all the soils could be due to low pH of the soils which could have resulted in fixation of P by sesquioxides in the

Fractions and Distribution of Zinc in the Soils

The results of zinc fractions distribution of soils of different parent materials studied are presented in Table 2. Water soluble zinc fraction of the soils differed and on a mean value basis, it was highest in alluvial soils (0.378 mg kg^{-1}), followed by coastal plain sands soils (0.209 mg kg^{-1}), false bedded sandstones soils (0.182 mg kg^{-1}) and Imo clay shale soils (0.154 mg kg^{-1}). Compared with other fractions, water soluble Zn was the least fraction in the soils of coastal plain sands (8.93% of total Zn), false bedded sandstones (7.36% of total Zn) and Imo clay shale (4.11% of total Zn) but was intermediate fraction in soils of alluvium, attaining 21.61% of the total Zn. Ramzan *et al.* (2014) also obtained least Zn concentration in the water soluble fraction. The low concentration of water soluble zinc when compared with other fractions could be partially due to losses from leaching and plant uptake, since this represents the fraction that is most bioavailable and mobile in soil (Filgueiras *et al.*, 2002). It could also be due to the poor extractive strength of water (Mbila *et al.*, 2001). Kabata-Pendias and Pendias (1999) reported that the concentration of water soluble zinc in soils ranges from 0.004 - 0.27 mg kg^{-1}which is very low compared with the average total concentrations of about 50-80 mg/kg. However, in very acid soils, soluble concentration of about 7 mg kg^{-1} has been obtained, indicating that solubility is strongly, but inversely linked to soil pH. Therefore values of water soluble zinc fraction in the soils that were above the soil range reported could be due to the acidic nature of the soils. The least concentration of water soluble zinc fraction recorded in Imo clay shale soils could be due to higher pH of the soils (Table 2). In most of the soils, higher values were recorded in the upper horizons and could be due to highly decomposable organic matter content of the horizons which favoured chelation of Zn^{2+}. Alloway (2008) noted that when soils are rich in rapidly decomposable organic matter, zinc may become more available due to the formation of soluble organic zinc complexes which are mobile and also probably capable of absorption into plant roots. Its distribution in the Pedons of different parent materials varied from low to high as evident in high coefficient of variations recorded (Table 2).

Exchangeable zinc fraction was higher than water soluble Zn and varied from 0.147-0.455 mg/kg in coastal plain sands soils, 0.120-0.837 mg kg^{-1} in

TABLE 2

Fractions And Distribution of Zinc (mg kg^{-1}) In The Soils Studied

Horizon	**Depth** (cm)	Water soluble	**Exchangeable**	**Available**	**specifically sorbed/CO$_3$ bound**	**Fe-Mn bound**	**OM Bound**	**Residual Bound**	**Total**
					Coastal plain sands				
Ap	0—17	0.458	0.455	0.913	ND	0.570	0.104	1.347	2.934
AB	17-36	0.173	0.227	0.400	0.431	0.317	0.216	0.981	2.345
Bt1	36-53	0.125	0.190	0.315	ND	0.359	0.140	0.724	1.538
Bt2	53-91	0.136	0.147	0.283	1.342	1.428	0.108	ND	3.161
Bt3	91-150	0.151	0.185	0.336	0.495	0.317	0.148	0.425	1.721
	Mean	**0.209**	**0.241**	**0.449**	**0.454**	**0.598**	**0.143**	**0.695**	**2.340**
	%Total	**8.93%**	**10.29%**	**19.19%**	**19.40%**	**25.55%**	**6.11%**	**29.70%**	
	%CV	**67.4**	**51.1**	**58.4**	120.9	**79.50**	**31.4**	**26.4**	51.9
					Alluvium				
Ap	0-4	0.461	0.168	0.629	0.136	ND	0.210	0.224	1.199
BC	4—84	0.482	0.837	1.319	0.154	0.187	0.175	0.330	2.165
C	84-100	0.190	0.120	0.310	0.933	0.176	0.265	0.196	1.880
	Mean	**0.378**	**0.375**	**0.753**	**0.408**	**0.121**	**0.217**	**0.250**	**1.749**
	%Total	**21.61%**	**21.44%**	**43.05%**	**23.33%**	**6.92%**	**12.41%**	**14.29%**	
	%CV	**43.1**	**106.9**	**68.5**	**111.6**	**86.7**	**20.9**	**28.3**	9.2
					False bedded sandstones				
Ap	0-9	0.226	0.065	0.291	0.722	0.375	0.356	ND	1.743
AB	9—28	0.139	0.755	0.894	0.622	0.618	0.194	1.074	3.402
Bt1	28-49	0.234	0.147	0.381	0.556	ND	0.857	0.341	2.135
Bt2	49-73	0.166	0.229	0.395	0.973	0.579	0.602	0.380	2.929
Bt3	73-170	0.143	0.218	0.361	0.033	0.748	ND	1.006	2.148
	Mean	**0.182**	**0.283**	**0.464**	**0.581**	**0.464**	**0.402**	**0.560**	**2.471**
	%Total	**7.36%**	**11.45%**	**18.78%**	**23.51%**	**18.78%**	**16.27%**	**22.66%**	
	%CV	**25.0**	**96.2**	**52.4**	**59.4**	**62.9**	**83.8**	**24.1**	63.5
					Imo clay shale				
Ap	0-11	0.146	0.096	0.242	1.184	0.340	0.954	2.431	5.151
AB	11—19	0.151	0.065	0.216	1.008	0.504	0.550	0.138	2.416
Bt2	19-36	0.166	0.803	0.969	1.007	0.417	0.142	1.310	3.845
Bt2	36-55	0.144	0.945	1.089	1.106	0.359	0.174	0.310	3.038
Bt3	55-83	0.164	1.024	1.188	1.124	0.324	ND	1.626	4.262
	Mean	**0.154**	**0.587**	**0.741**	**1.086**	**0.389**	**0.364**	**1.163**	**3.742**
	%Total	**4.11%**	**15.69%**	**19.80%**	**29.02%**	**10.39%**	**9.73%**	**31.08%**	
	%CV	**6.6**	**79.9**	**63.9**	**7.1**	**18.9**	**106.5**	**81.8**	28.4

ND- no detection

alluvial soils, 0.065-0.755 mg kg^{-1} in false bedded sandstones soils and 0.065-1.024 mg kg^{-1} in Imo clay shale soils (Table 2). Distribution among the sites on a mean value basis was in a decreasing order of Imo clay shale (0.587 mg kg^{-1}) > alluvium (0.375 mg kg^{-1}) > false bedded sandstones (0.283 mg/kg) > coastal plain sands (0.241 mg kg^{-1}). When compared with other zinc fractions, it was low in the soils of coastal plain sands and false bedded sandstones, attaining about 10.29% and 11.45% of total Zn, respectively whereas in soils of alluvium and Imo clay shale, it was an intermediate fraction, attaining 21.44% and 15.69% of total Zn, respectively. Schulte (2004) noted that soils contain exchangeable zinc fraction between the range of 2-25 mg kg^{-1}, with a larger proportion held in iron and manganese oxides. The values of exchangeable Zn recorded in the soils were below the range and could be due to the sandiness of some of the soils, low pH of the soils as decreasing pH decreases cation exchange capacity of soils (Das, 2011) as well as low organic matter content of the soils, hence exchange sites for attraction of Zn^{2+} were in small quantity. The highest concentration recorded in soils of Imo clay shale could be attributed to higher pH (5.7) and clay (42.8%) contents of the soils as increasing levels of both the soil properties increases cation exchange capacity of soils (Brady and Weil, 2010). Except in soils of alluvium and false bedded sandstones where its distribution in the soil profiles did not follow a definite pattern, in soils of coastal plain sands, its concentration decreased with soil depth and could be due to decreasing organic matter content down the profile whereas in soils of Imo clay shale, it increased down the profile and could be due to increasing clay content with depth (Table 2). Das (2011) noted that organic matter and clay are known to constitute exchange sites in soils. The results further indicate high variation in its distribution in the soils of different parent materials as evident in the high coefficient of variations recorded (Table 2).

Specifically sorbed/CO_3 bound zinc fraction differed in the soils and was two times more than exchangeable Zn fraction. Mean values of 0.454 mg kg^{-1}equivalent to 19.40% of total Zn, 0.408 mg kg^{-1} equivalent to 23.33% of total zinc, 0.581 mg kg^{-1} equivalent to 23.51% of total zinc and 1.086 mg kg^{-1} equivalent to 29.02% of total zinc were recorded in soils of coastal plain sands, alluvium, false bedded sandstones and Imo clay shale, respectively (Table 2). Distribution among the sites decreased as Imo clay shale > false bedded sandstones > coastal plain sands > alluvium. It was a dominant fraction in soils of alluvium and Imo clay shale, intermediate fraction in soils of coastal plain sands and the highest fraction in soils of false bedded sandstones. It was higher in soils of Imo clay shale and could be due to high carbonate content of the shale parent material from which the soils were derived from (Hiller, 2006) as well as higher pH of the soils as increasing pH increases concentration of carbonate bound zinc (Ramzan *et al.*, 2014). Rajakumar (1994) noted that carbonate bound zinc is usually seen in soils with high pH and lime content. Except in soils of alluvium where its concentration increased with soil depth, its distribution in other soils followed an irregular pattern (Table 2). The results further indicated high variation in its distribution in soils of coastal plain sands (CV= 120.9%), alluvium (CV= 111.6%)

and false bedded sandstones (CV= 59.4%), an indication of uneven distribution whereas low variation was recorded in soils of Imo clay shale (CV= 7.1%).

Fe-Mn oxide bound zinc fraction, being among the non-residual fraction, was found to be the major Zn fraction with values ranging from 0.317-1.428 mg kg^{-1} in coastal plain sands soils, 0-0.187 mg kg^{-1} in alluvial soils, 0-0.748 mg kg^{-1} in false bedded sandstones soils and 0.324-0.504 in Imo clay shale soils. When compared with other zinc fractions of the soils, it was dominant in soils of plain sands, low in soils of alluvium but intermediate fraction in soils of false bedded sandstones and Imo clay shale. However, these values are low when compared with values ranging from 2.18 to 7.65 mg kg^{-1} (mean= 4.53 mg/kg) reported in Gangavati taluk soils of Dharwad, India which is derived from mixed parent materials (Wijebandara *et al.,* 2011) and the findings of Shober (2007) in soils of Pennsylvania, U.S.A. derived from limestone (Ciolkosz *et al.*, 1995) and could be attributed to low total zinc content of the soils under study. It was lower in soils of alluvium, attributable to sandiness of the soils as oxides of Fe and Mn are usually present in the clay fraction of soils (Kabata-Pendias, 2011). Its distribution pattern was irregular in soils of coastal plain sands and Imo clay shale but increased and decreased with soil depth in soils of false bedded sandstones and alluvium, respectively (Table 2). The results further indicate moderate variation in its distribution in soil profile of Imo clay shale (CV= 18.9%) whereas high variations were recorded in soil profiles of coastal plain sands (CV= 79.5%), false bedded sandstones (CV= 62.5%), and alluvium (CV= 86.7%). Generally, Fe-Mn oxide bound zinc fraction of the soils of different parent materials decreased in the order of coastal plain sands > false bedded sandstones > Imo clay shale > alluvium.

Organic matter bound zinc fraction followed a similar distribution trend with water soluble Zn as it was low in most of the soils. Mean organic matter bound Zn concentration decreased in the order of false bedded sandstones (0.402 mg kg^{-1}) > Imo clay shale (0.346 mg kg^{-1}) > alluvium (0.217 mg kg^{-1})> coastal plain sands (0.143 mg kg^{-1}). It was the least Zn fraction in soils of coastal plain sands (6.11% of total zinc), low in soils of alluvium (12.41% of total zinc) and Imo clay shale (9.73% of total zinc) but intermediate fraction in soils of false bedded sandstones (16.27% of total Zn) (Table 2). The values were low when compared with the report of Ideriah *et al.,* (2013) in alluvial soils of Niger Delta, Nigeria and could be due to low total zinc contents of the soils (<10 mg kg^{-1}) as well as low organic matter (<50 g kg^{-1}) contents of the soils under study. It was higher in the surface horizon and could be due to higher organic matter content of the horizon. Additionally, the results indicated moderate variation in its distribution in soils of coastal plain sands (CV= 31.4%) and alluvium (CV= 20.9%) whereas its distribution followed high variation in soils of false bedded sandstones (CV= 83.8%) sandstones and Imo clay shale (CV= 106.5%).

Residual bound zinc dominated most of the soils. Mean value of 0.695 mg/kg equivalent to 29.70% of total zinc was recorded in coastal plain sands soils, 0.250 mg/kg equivalent to 14.29% of total zinc in soils of alluvium, 0.560 mg kg^{-1} equivalent to 22.66% of total zinc in false bedded sandstone soils and 1.163

mg kg^{-1} equivalent to 31.08% of total zinc in soils of Imo clay shale. Distribution among the sites decreased in the order of Imo clay shale > coastal plain sands > falsebedded sandstones > alluvium. It was the highest Zn fraction in soils of coastal plain sands and Imo clay shale but dominant in soils of false bedded sandstones and alluvium. The results are in agreement with the report of Ramzan *et al.,* (2014) and Aydinalp (2009) who obtained highest concentration of zinc in the residual fraction. The greater percentage of Zn in the residual fraction in most of the soils indicated its greater tendency to become unavailable to plants (Ramzan *et al.*, 2014). This is because the residual bound fraction represents metals incorporated into the crystalline lattices of clays and appears inactive (Kabala and Singh, 2001). It was more in soils of Imo clay shale and could be due to higher clay content of the soils but lower in soils of alluvium, attributable to low clay content of the soils as residual bound fraction of metals is known to be embedded in silicate clays (Ma and Rao, 1997). Furthermore, in the soil profiles of different parent materials, its distribution pattern was irregular and moderate variations were recorded in soils of coastal plain sands (CV= 26.4%), alluvium (CV= 28.3%), false bedded sandstones (CV= 24.1%) while high variation was noted in soils of Imo clay shale (CV= 81.8%).

Generally, fractional distribution of zinc varied in each of the soil profiles of different parent materials. Zinc fractions decreased in the order of residual bound > Fe-Mn oxide bound > specifically sorbed/CO_3 bound > exchangeable > water soluble > OM bound in coastal plain sands soils, specifically sorbed/CO_3 bound > water soluble > exchangeable > residual bound > OM bound > Fe-Mn Oxide bound in soils of alluvium, specifically sorbed/CO_3 bound > residual bound > Fe-Mn Oxide bound > OM bound > exchangeable > water soluble in soils of falsebedded sandstones and residual bound > specifically sorbed/CO_3 bound> exchangeable > Fe-Mn Oxide bound > OM bound > water soluble in soils of Imo clay shale.

Distribution of Available and Total Zinc in the Soils

For available zinc fraction, mean values of 0.241 mg kg^{-1}, 0.753 mg kg^{-1}, 0.464 mg kg^{-1} and 0.741 mg kg^{-1} were recorded for coastal plain sands, alluvium, false bedded sandstones and Imo clay shale, respectively. For arable crop production, Esu (1991) classified available zinc as low, medium and high when concentrations varied in the order <0.8, 0.8-2.0 and >2.0 mg kg^{-1}, respectively. Using the mean values, all the soils were low in available zinc. The low values of available zinc recorded in the soils could be due to low total zinc concentrations (Table 2) of the soils as total concentration of an essential element is an indication of its availability, sandiness of some of the soils, high amount of rainfall in the area as well as the acidic nature of the soils which encourages leaching losses of zinc. According to Alloway (2008), sandy soils and acid highly leached soils with low total zinc concentrations are highly prone to zinc deficiency. Eteng *et al.,* (2014) also reported low available zinc in coastal plain sand soils of South-eastern, Nigeria. It was more in soils of alluvium followed by soils of Imo clay shale, false bedded sandstones and coastal plain sands. The highest concentration recorded in soils of alluvium could be due to very low organic matter content of the soils as increasing organic matter content in soils decreases zinc availability due to increased sorption of zinc by organic ligands and components (Alloway, 2004). It could also be due to sandiness of the soils which reduced adsorption sites for Zn^{2+}. Additionally, its distribution in all the soils of different parent materials did not follow a regular pattern. The results further indicate high variation in its distribution in the soil profiles of different parent materials under study (Table 2). Concentration of total zinc in the soils ranged from 1.538-3.161 mg/kg (mean= 2.340 mg kg^{-1}), 1.880 -2.165 mg kg^{-1} (mean= 1.749 mg kg^{-1}), 1.743-3.402 mg kg^{-1} (mean= 2.471 mg kg^{-1}) and 2.416 - 5.151 mg kg^{-1} (mean= 3.742 mg kg^{-1}) in soils of coastal plain sands, alluvium, false bedded sandstones and Imo clay shale, respectively (Table 2). Kabata-Pendias (2011) reported that zinc content of agricultural soils varies between 10 - 300 mg kg^{-1}. Based on the report, the soils were considered low in total zinc since total zinc concentrations of the soils were below the range. This could be due high intensity of rainfall in the area which may have triggered weathering and leaching losses of weathered materials. These findings are in line with the report of Onweremadu *et al*. (2008) who obtained low total zinc in coastal plain sands (2 - 5 mg kg^{-1}) and Imo clay shale soils of Imo State, South-eastern, Nigeria. It was higher in Imo clay shale soils followed by false bedded sandstones soils, coastal plain sands soils and alluvial soils. Higher values recorded in soils of Imo clay shale could be due to high zinc content of the shale parent material as reported by Havlin *et al.* (2012). However, total zinc concentration of the soils did not attain toxic level since it was below 300 mg/kg maximum permissible agricultural soil concentration of total zinc recommended by Kabata-Pendias and Pendias (2001). The results further indicated that its distribution followed low variation in soils of alluvium (CV= 9.2%), moderate variation in soils of Imo clay shale (CV= 28.4%) and high variation in soils of coastal plain sands (CV= 51.9%) and false bedded sandstones (CV= 63.5%).

Table 3 shows the relationships existing among zinc fractions and between zinc fractions and selected soil properties (OM, ECEC, Clay, Ca and pH) using simple linear correlation analysis. The results of the analysis indicated significant ($p<0.05$) negative correlation between exchangeable zinc and organic matter bound zinc ($r = 0.46$). This implies that a decrease in organic matter bound zinc will significantly result in an increase in exchangeable zinc. However, water soluble zinc was found to have a significant and negative ($p<0.05$) relationship with organic matter bound zinc ($r = -0.46$) whereas specifically sorbed/CO_3 bound zinc correlated significantly ($p<0.05$) and negatively with water soluble zinc ($r = -0.55$) while it had a significant ($p<0.05$) and positive relationship with Ca ($r = 0.48$) and clay ($r = 0.51$) (Table 3). In addition, organic matter had a significant ($p<0.05$) and positive correlation with exchangeable zinc ($r = 0.56$) and specifically sorbed/CO_3 bound zinc ($r = 0.47$) (Table 3). The implication of these results is that an increase in organic matter content of the soils will result in an increase in exchangeable and specifically/CO_3 bound zinc fractions. However,

TABLE 3
Simple Linear Correlation Among Zinc Fractions and Between Zinc Fractions and Selected Soil Properties (n= 18)

	Exch. Zn	W-S Zn	Specifically sorbed/CO_3 bound Zn	Fe/Mn bound Zn	OM bound Zn	Residual Zn
Exch. Zn	-	-	-	-	-	-
W-S Zn	0.11ns	-	-	-	-	-
Specifically sorbed/CO_3 bound Zn	0.11 ns	-0.55*	-	-	-	-
Fe/Mn bound Zn	-0.07 ns	-0.36 ns	0.30 ns	-	-	-
OM bound Zn	-0.45*	-0.11 ns	0.31 ns	0-.28 ns	-	-
Residual bound Zn	0.28 ns	-0.13 ns	0.05 ns	-0.04 ns	0.11 ns	-
Avail. P	-0.06 ns	0.24 ns	0.14 ns	-0.39 ns	0.26 ns	0.35 ns
Ca	0.23 ns	-0.24 ns	0.48*	-0.01 ns	-0.04 ns	0.10 ns
Clay	0.39 ns	-0.53*	0.51*	0.08 ns	0.18 ns	0.16 ns
ECEC	0.25 ns	-0.30 ns	0.42 ns	0.07 ns	-0.12 ns	0.33 ns
OM	0.56*	-0.16 ns	0.47*	-0.09 ns	-0.07 ns	0.29 ns
pH(H_2O)	0.41ns	-0.32 ns	0.311 ns	-0.33 ns	0.03 ns	0.02 ns

W-S- water soluble, Avail. P – available phosphorus, ECEC- effective cation exchange capacity, OM significant - organic matter, * significant at 5% probability level, ** - significant at 1% probability level, ns- not significant, soil pH had no serious association with any of the zinc fractions as no significant correlation between soil pH and zinc fractions was recorded.

CONCLUSIONS

The results of this study indicated that the concentration of Zn fractions varied among the soils of different parent materials and decreased in the order of residual bound > Fe-Mn Oxide bound > specifically sorbed/CO_3 bound > exchangeable > water soluble > OM bound in coastal plain sands soils, specifically sorbed/CO_3 > water soluble > exchangeable > residual bound > OM bound > Fe-Mn Oxide bound in alluvial soils, specifically sorbed/CO_3 > residual bound > Fe-Mn Oxide bound > OM bound > exchangeable > water soluble in false bedded sandstones soils and residual bound > specifically sorbed/CO_3 > exchangeable > Fe-Mn Oxide bound > OM bound > water soluble in Imo clay shale soils. Available zinc concentrations were low and varied among the soils of different parent materials in a decreasing order of alluvium > Imo clay shale > false bedded sandstones > coastal plain sands, while total zinc concentrations were in decreasing order of Imo clay shale > falsebedded sandstones > coastal plain sands > alluvium, respectively. Zinc forms correlated among each other and with pH, Ca, clay, ECEC, available P and OM.

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