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IMPACT OF TEXTURE ON SESQUIOXIDE DISTRIBUTION IN SOUTHEASTERN NIGERIAN SOILS

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Abstract

Sesquioxides which play significant roles in soil classification and dominant soil properties vary in soils with particle size fractions. Using randomized complete block design, this study was conducted in the month of June 2018 to evaluate the impact of texture on sesquioxide distribution in humid rainforest soils of Southeastern Nigeria. Four mini-pedons were dug at four landscape positions (upslope, midslope, downslope and flat surface) and triplicate soil samples taken from 0-20, 20-40 and 40-60 cm depths, summing to total of 36 samples. Crystalline and amorphous Al, Fe, Mn and Si oxides were then determined at a depth of 0-20 cm using Dithionate Citrate Bicarbonate (DCB) and ammonium oxalate reagents. Dithionate and oxalate fractions in bulk soil ranged between 9300-11,400 (Fe_d), 390-1580 (Fe_{ox}), 4600-6700 (Al_d), 660-890 (Al_{ox}), 3300-5600 (Mn_d), 350-580 (Mn_{ox}) and 9600-13,500 (Si_d) and 1690-1790 mg kg^{-1} (Si_{ox}), with dithionate superior indicating high crystallinity and low mobility of the sesquioxides. Ratios of bulk soil oxalate/dithionate fractions were low and ranged between 0.04-0.17 (Fe_{ox}/Fe_d), 0.11-0.15 (Al_{ox}/Al_d), 0.10-0.16(Mn_{ox}/Mn_d) and 0.13-0.18 (Si_{ox}/Si_d) and confirming their crystallinity. Low Fe_{ox}/Fe_d ratios signified that soils were well drained (< 0.35), old (< 0.65) and highly weathered (< 0.50) with the order being a decreasing sequence of upslope > flat surface > down slope > midslope. Sesquioxide contents and reactivity in soil particle size fractions (sand, silt and clay) varied with landscapes. Regression models indicated that particle size fractions accounted for 50% of 2/3rd of the bulk soil sesquioxide concentrations and that averaged over landscapes, sand fraction was more enriched with various sesquioxides than the other soil particle size fractions. Correlations between most bulk soil sesquioxide fractions and sesquioxide fractions with selected soil properties (sand, silt, clay, moisture content, total porosity, organic matter, pH, Ca and ECEC) were significant ($P < 0.05$).

Key words: Sesquioxides, Grain size fraction, Landscape, Humid tropics, Southeastern Nigeria

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Introduction

Sesquioxides are oxides, hydroxides and oxy-hydroxides of Al, Fe, Mn, Si and Ti that are present as coatings or discrete particles on the soil surfaces (Uzoho, 2014; Maniyunda *et al*., 2015). They are crystalline or amorphous in nature, even though a small fraction exists as organic complex and together they influence soil physiochemical properties (Osodeke *et al*., 2005). Sesquioxides play important roles in soil cation exchange reactions, anion adsorption, surface charge, nutrient transformation, swelling and aggregate formation, pollutant retention and precipitation of phosphorus (P) into insoluble compounds (Fe-P and Al-P) thereby restricting their mobility and availability in soils (Hassan *et al*., 2005; Jelic *et al*., 2011; Uzoho, 2014*)*. Also, they are useful in the estimation of the degree of weathering, pedogenesis, conferment of soil tints and the cementation of tropical soils thereby influencing their plasticity and compressibility (Essoka and Esu, 2000)

Reactivity of Sesquioxides varies with their level of crystalline fraction of sesquioxide, with the less crystalline or amorphous fractions more reactive than the highly crystallized fractions. It has been reported that P and metal sorption are greater in the amorphous Fe and Al oxides than the crystalline oxides (Owusu-Bennoah and Acquaye, 1989). Reactivity of sesquioxides is also associated with the ratio or relative amounts of the poorly ordered and the crystalline Fe and Al oxide fractions in soils (Hassan *et al*., 2005). Research has indicated that poorly drained soils have high ratios and thus more reactive than the well-drained soils (Ibia, 2001*)*.

Chemical reagents especially ammonium oxalate and dithionite-citrate-bicarbonate could be used for the estimation of amorphous and crystalline oxide fractions respectively in soils and sediments. It has been indicated that dithionite extractable Fe and Al constitutes a reasonable estimate of total pedogenic free Fe and Al oxides while oxalate extractable is for the amorphous Fe and Al oxides (Hassan *et al*., 2005). Crystalline (free) or dithionate extractable iron oxides represent total pedogenic iron that are present as geothites, lepidocricite and haematite while aluminium occurs as gibbsite in soils (Osodeke *et al*., 2005; Maniyunda *et al*., 2015). It has also been noted that extractable dithionite-citrate manganese and silicon constitutes easily reducible manganese and silicon forms in soils respectively (*NSSC, 1995*).

Distribution of sesquioxides varies with landscape positions. For instance, variation with landscape positions have been reported in soils of inland valleys (Essoka and Esu, 2000), contrasting land units (Uzoho, 2014*)*, toposequences (Osodeke *et al*., 2005; Enya *et al*., 2011), granitic and basement complex rocks (Hassan *et al*., 2005; Maniyunda *et al*., 2015; Olatunji *et al*., 2015) and soil profile depths (Essoka and Esu, 2000). According to Wei et al. (2010), landscape influence could be associated with the topography, drainage condition, parent materials and land use types and affects soil chemical properties and water movement conditions. Within each landscape, sesquioxide concentrations may vary with soil particle size fractions. In related studies, variation amongst soil particle size fractions have been reported for amino sugars (Zhang *et al*., 1999), carbohydrates (Spaccini *et al*., 2001; Uzoho and Igbojionu, 2014), nitrogen (Uzoho *et al*., 2016) and potassium (Igwe *et al*., 2008; Ajiboye and Ogunwale, 2012*;* Uzoho *et al*., 2016*)*. Spaccini *et al*. (2001) ascribed the differences to variations in soil surface area and organic matter contents.

Several physiochemical properties especially sand, silt, clay, silt/clay ratio, TN, OM, P, Ca, K, Mg, Na, H, ECEC and % BS have been reported to affect sesquioxide contents of soils (Uzoho, 2014*).* Maniyunda *et al.* (2015) obtained strong relationships between sesquioxides with clay and soil organic matter contents while Osodeke *et al.* (2005) noted positive relationships between DCB-Al with clay and sand and oxalate Fe with clay and organic matter contents of soils along a toposequence in Umudike area of southeastern, Nigeria,.

Though sesquioxide concentrations of most Nigerian, soils have been studied (Osodeke *et al.*, 2005; Hassan *et al.*, 2005; Enya *et al.*, 2010; Uzoho,2014; Maniyunda *et al.*, 2015), there appears to be a dearth of information on their distribution in soil particle size fractions. The objectives of this study were therefore to evaluate the impact of texture on sesquioxide contents of soils of contrasting landscapes in a humid tropical rainforest, relationship between bulk soil and particle size sesquioxide concentrations and correlation between sesquioxide contents and selected physicochemical properties of the bulk soil.

Materials and Methods

Study Location

The study location was Ihiagwa on latitudes $5^{o}\,24^{1}\,0^{11}$ N and longitudes $7^{o}\,10^{1}\,0^{11}$ E and an altitude of 117m above sea level. The annual rainfall average range between 2000-2250 mm, daily temperature average range between 26-27^{o}C and relative humidity average range was 83-85%. Its rainfall pattern is bimodal with peaks in July and September and a short dry spell in August known as the August break. The soil type is Arenic Kandidiult (Uzoho *et al.*, 2014) derived from Coastal Plain Sands (Orajiaka, 1975). Economic activities of the area consisted of farming, trading and sand mining.

Sample Collection, Preparation and Laboratory Analysis

Guided by Randomized Complete Block Design (RCBD), triplicate soil samples were collected from specific depths (0-20, 20-40 and 40-60 cm) of the mini-pedons dug at the four landscape positions (upslope, mid-slope, down-slope and flat surface), summing to a total of 36 samples used for the study. The three samples from the same depths of each landscape position were then bulked to obtain a composite. The samples were air dried, sieved using a 2 mm diameter mesh and the fine earth fractions subjected to routine laboratory analyses in the Teaching and Research Soil Laboratory of the Federal University of Technology, Owerri Nigeria. Particle size fraction was determined after dispersion with sodium hexametaphosphate (Gee and Or, 2002), organic matter (Nelson and Sommers, 1996), available P (Olsen and Sommers 1982), effective cation exchange capacity (Thomas, 1996), total nitrogen (Bremner, 1996), bulk density (Blake and Hartage, 1986), pH in1:2.5 soil/water ratio (Thomas, 1996) and total porosity from bulk density value as TP = 100(1-Db/Dp) where Db = bulk density and Dp = particle density equivalent to 2.65g cm^{-3}.

Soil Particle Size Fractionation

Subsamples of fine earth soil fractions of the 0-20cm depths (topsoil) for each landscape position were fractionated into sand (<0.002 mm), silt (0.002-0.02 mm) and clay (0.02-2.0 mm) fractions according to the method described by Sequaris and Lewandowski (2003). The procedure consisted of a three stage process. In the first stage, 100g fine earth soil sample was weighed into a 1000 ml glass bottle containing 200 ml distilled water and shaken on a horizontal shaker for 6 hrs at a revolution of 15 x 1000 g. Then 600 ml of distilled water was added, agitated by hand for 1 minute, left undisturbed for 6 minutes to sediment after which about 700 ml of the supernatant was transferred into another glass bottle. The sediments left in the first glass bottle were then dried at 40 ^{o}C to obtain the 0.02-2.0 mm fraction.

In the second stage, the 700 ml supernatants from stage 1 was shaken for 2 minutes and then left undisturbed for 12 hrs after which 600 ml was removed. The sediments left were dried at 40 ^{o} C to obtain the 0.002-0.02 mm fraction while in the third stage the 600 ml supernatants from stage 2 was transferred into 250 ml centrifuge tubes and centrifuged for 90 minutes at 12000 x g and 20 ^{o}C. The sediment remaining after the supernatant was discarded, was then dried at 40 ^{o}C to obtain the < 0.002mm fraction.

Determination of Sesquioxide Content of the Soils

Sesquioxides contents in the bulk soil and particle size fractions of the 0-20 cm soil depths were determined as follows: Crystalline (free) Al, Fe, Mn and Ti oxides were determined using the Dithionate Citrate Bicarbonate (DCB) reagents (Mehre and Jackson, 1960) while amorphous Al, Fe, Mn and Si oxides were determined using ammonium oxalate reagents (McKeague and Day, 1966). All extracts were analyzed using an Alpha 4 Atomic Absorption Spectrophotometer (AAS).

Calculations

$$\text{Sesquioxide reactivity or ratios} = \frac{\text{Oxalate fractions } (Y_{ox})}{\text{Dithionate citrate bicarbonate fraction } (Y_{d})} \text{ (Galez et al., 1975) --- (1)}$$

$$\text{Enrichment Factor} = \frac{\text{Sesquioxide content of particle size fractions}}{\text{Sesquioxide content of Bulk Soil}} \text{ (Zhang et al., 1999) ------------------------ (2)}$$

Statistical Analysis

All data generated were subjected to Analysis of Variance (ANOVA) and treatment means separated using Least Significant Difference (LSD) at 1% probability level. Also correlation analysis was used to determine the relationship between sesquioxide fractions and between sesquioxide fractions with selected soil properties. Equally, regression analysis was used to determine the relationship between bulk soil and particle size sesquioxide contents. All statistical analyses were conducted using Genstat statistical package version 11 (Buysse *et al*., 2004).

Results and Discussion

Soil Characterization

Sand, silt and clay contents ranged from 859.60-949.60 (mean = 904.60), 12.80-32.80 (mean = 22.80) and 37.60-137.60 (mean = 87.60 g kg^{-1}) respectively and decreased (sand), increased (clay) or irregular (silt) with soil depth (Table 1).

Table 1. *Physical and Chemical Properties of the Selected Soil*

Landscape	Soil Depth	TP	Bd	MC	Sand	Silt	Clay	pH	Avail P	TN	OM	Ca	ECEC
Position	Cm	%	g cm^{-3}		g kg^{-1}			(H$_2$O)	mg kg^{-1}	g kg^{-1}		Cmol kg^{-1}	
Upslope	0-20	10.26	1.12	350.00	899.60	32.80	77.60	5.12	9.66	1.38	34.10	1.44	8.79
	20-40	13.36	1.15	360.00	889.60	12.80	87.60	4.65	3.99	1.36	18.80	0.80	7.13
	40-60	25.17	1.32	430.00	859.60	22.80	137.60	4.37	0.77	1.04	10.3	0.64	6.67
	LSD (0.01)	1.31	0.03	13.54	21.52	7.85	46.83	0.06	1.06	0.89	8.50	0.45	1.64
Mid slope	0-20	2.80	1.07	300.00	919.60	32.80	87.60	5.02	1.52	1.60	16.60	0.40	8.27
	20-40	8.11	1.22	370.00	869.60	12.80	97.60	5.01	1.12	1.29	8.90	0.24	5.93
	40-60	19.43	1.30	410.00	869.60	12.80	117.60	4.98	0.84	1.21	5.20	0.16	4.79
	LSD (0.01)	1.31	0.53	21.26	19.47	5.56	19.63	0.05	0.97	0.68	0.80	0.07	0.44
Down lope	0-20	3.85	1.15	280.00	942.60	12.80	44.60	5.35	8.81	2.6	15.50	1.20	7.33
	20-40	13.66	1.22	280.00	929.60	22.80	87.60	4.94	8.46	2.41	12.00	0.32	5.21
	40-60	3.85	1.27	320.00	889.60	12.80	97.60	4.85	7.57	2.25	10.30	0.32	5.72
	LSD (0.01)	1.31	0.02	23.34	18.54	7.45	6.40	0.06	0.15	0.85	6.00	0.24	0.59
Flat Surface	0-20	9.66	1.17	270.00	949.60	12.80	37.60	5.05	2.52	1.74	17.10	1.04	10.06
	20-40	10.19	1.22	280.00	899.60	22.80	77.60	5.02	3.43	1.63	15.90	0.16	8.32
	40-60	11.92	1.27	300.00	889.60	20.00	90.40	4.58	1.96	1.43	14.40	1.04	5.97
	LSD (0.01)	4.41	0.92	26.45	21.63	2.61	8.50	0.06	0.06	0.66	5.10	0.03	0.31

Increased clay content with soil depth has been attributed to clay illuviation and translocation by erosion at the surface horizon (Obi *et al*., 2009; Maniyunda et al., 2015). Sandiness of the soils was due to the nature of their parent materials which is Coastal Plain Sands (Enwezor *et al*., 1990; Uzoho *et al*., 2007). Mean bulk density (1.20 g cm^{-3}) was low and increased with depth attributable to soil overburdening weight and decreased organic matter content with depth (Brady and Weil, 2002; Uzoho *et al*., 2007; Hargreaves and Warman, 2009). Total porosity (3.85-25.17%) increased with depth probably due to the high clay accumulation and increased microporosity. Soils were acidic (pH = 4.37-5.35) with acidity higher down the soil depth (Table 1) ascribable to low organic matter content and lose of basic cations through runoff and erosion (Enwezor *et al*., 1990; Uzoho *et al*., 2007). Low mean pH (4.86) value of less than 5.5 for the soils indicated that they could suffer from aluminium toxicity (Ernani *et al*., 2002; White *et al*., 2006; Uzoho *et al*., 2007). Other soil properties

(Available P, total N, exchangeable Ca and ECEC) were low and decreased with soil depth as the soil OM indicating that fertility of the soils could be related to their organic matter contents (Enwezor *et al.*, 1990; Noma *et al.*, 2004*)*.

Sesquioxide Concentrations in the Soils

Dithionate and oxalate iron (Fe_d and Fe_{ox}), aluminium (Al_d and Al_{ox}), Manganese (Mn_d and Mn_{ox}) and Silicon (Si_d and Si_{ox}) in the bulk soil and soil particle size fractions deferred with landscape positions. Concentrations of Fe_d, Fe_{ox} and iron activity (Fe_{ox}/Fe_d ratio or active Fe) in the bulk soil ranged from 9300.00-11, 400.00, 390-1580 mg kg^{-1} and 0.04-0.17 respectively (Table 2) and low compared to ranges reported by other workers (Osodeke *et al.*, 2005; Uzoho, 2014; Olatunji *et al.*, 2015; Maniyunda *et al.*, 2015). However, compared to ranges of Fe_d (Ojo-Atere and Ajowon, 1985; Essoka and Esu 2000) and Fe_{ox} (Hassan *et al.*, 2005) those of the soils were high. Amongst landscapes, best bulk soil Fe_d was at the downslope while Fe_{ox} and Fe_{ox}/Fe_d were at the upslope positions. Generally, Fe_d was higher than Fe_{ox}, giving low Fe_{ox}/Fe_d ratio (0.04-0.17) and indicating high crystallinity and good drainage (Udo, 1980; Ibia, 2001). It has been noted that Fe_{ox}/Fe_d ratio or iron activity is useful in evaluating the age, drainage conditions, degree of weathering and amounts of crystalline or free amorphous oxides in soils (Blume and Schwertmann, 1969; Alamdari *et al.*, 2010*)*. Also, research has shown that ratios greater than 0.35 signify poor drainage while lower values represent well drained condition (Udo, 1980; Essoka and Esu, 2000; Uzoho, 2014). Equally, ratios less than 0.50 have been associated with high degree of weathering (Alexander, 1974) while those of 0.65 are younger than soils with 0.45 or 0.55 (Dolui and Mustafi, 1997). Thus, soils studied could be said to be well drained, old and highly weathered, with the degree decreasing as Upslope > flat surface > down slope > midslope. Similar observation has been reported for soils of the studied area (Enwezor *et al.*, 1990; Omenihu *et al.*, 1994). Variation amongst landscape positions could be related to differences in topography, water movement and drainage conditions (Wei *et al*., 2010). Amongst particle size fractions, values of Fe_d in the sand fraction were uniform at all landscape positions exception being upslope while that in the silt and clay fractions were higher at the down slope position.

Table 2. *Dithionate Citrate Bicarbonate, Oxalate Iron (mg kg^{-1}), Active Iron and Enrichment Factors (EF) in Bulk and Particle Size Fractions of the Soils*

Landscape Position	Bulk soil	Sand	Silt	Clay	EFsand	EFsilt	EFclay
			Dithionate Citrate Bicarbonate Fe (Fe_d)				
Upslope	9300	7600	890	590	0.82	0.10	0.06
Mid slope	11000	9000	8500	7200	0.82	0.77	0.65
Down slope	11400	9000	11200	9700	0.79	0.98	0.85
Flat Surface	11200	9000	8700	7800	0.80	0.78	0.70
LSD (0.01)	1078	989	652	230	0.09	0.10	0.10
			Oxalate Fe (Fe_{ox})				
Upslope	1580	440	340	390	0.28	0.22	0.25
Mid slope	390	150	150	370	0.38	0.38	0.95
Down slope	590	180	120	340	0.31	0.20	0.58
Flat Surface	620	510	260	370	0.82	0.42	0.60
LSD (0.01)	126.60	98.91	98.91	8.99	0.09	0.06	0.05
			Active Fe				
Upslope	0.17	0.06	0.38	0.66	0.34	2.25	3.89
Mid slope	0.04	0.02	0.02	0.05	0.47	0.50	1.45
Down slope	0.05	0.02	0.01	0.04	0.39	0.21	0.68
Flat Surface	0.06	0.06	0.03	0.05	1.02	0.54	0.86
LSD (0.01)	0.06	0.03	0.01	0.03	0.03	0.15	0.13

EF = Enrichment factor

Whereas enrichment of the sand fraction was better at the upslope, that of the silt and clay fractions was at the down slope position, with the sand more enriched than other particle size fractions averaged over landscape positions. Also, Fe_{ox} content of the sand fraction was better at the flat surface while that in the silt and clay fractions were at the upslope position. Its enrichment in the sand and silt fractions was better at the Flat surface while that in the clay fraction was at the mid slope and with enrichment of the clay superior to the silt and sand fractions, averaged over landscape positions. Equally, Fe_{ox}/Fe_d ratio or iron activity in the sand fraction was higher at the upslope or flat surface while that in the silt and clay fractions was at the upslope position and with enrichment of the sand fraction better at the flat surface and the silt and clay fractions in the upslope position. Enrichment of the clay was superior to other size fractions averaged over landscape positions.

Table 3. *Dithionate Citrate Bicarbonate, Oxalate Aluminium (mg kg^{-1}), Active and Enrichment Factors (EF) in Bulk and Particle Size Fraction of Soils*

Landscape Position	Bulk soil	Sand	Silt	Clay	EFsand	EFsilt	EFclay
			Dithionate Citrate Bicarbonate Al (Al_d)				
Upslope	6700	4800	3900	5600	0.72	0.58	0.84
Mid slope	4600	3000	2800	3500	0.65	0.61	0.76
Down slope	6200	480	4400	5100	0.08	0.71	0.82
Flat Surface	5500	4800	2800	4300	0.87	0.51	0.78
LSD 0.01	1000	994	1001	100	0.01	0.10	0.02
			Oxalate Al (Al_{ox})				
Upslope	750	660	550	590	0.88	0.73	0.79
Mid slope	660	430	350	480	0.65	0.53	0.72
Down slope	780	630	470	560	0.81	0.60	0.72
Flat Surface	830	690	430	520	0.83	0.52	0.63
LSD 0.01	103	273	425	99.9	0.12	0.12	0.12
			Active Al				
Upslope	0.11	0.14	0.14	0.11	1.23	1.26	0.94
Mid slope	0.14	0.14	0.13	0.14	1.00	0.87	0.96
Down slope	0.13	1.31	0.11	0.11	10.43	0.85	0.87
Flat Surface	0.15	0.14	0.15	0.12	0.95	1.02	0.80
LSD 0.01	0.07	0.08	0.09	0.08	0.80	0.71	0.34

EF = Enrichment factor

The Al_d, Al_{ox} and Al_{ox}/Al_d ratio of the bulk soil ranged between 4600-6700 mg kg^{-1}, 750-830 mg kg^{-1} and 0.11-0.15 respectively (Table 3), with Al_d range high relative to values reported by other workers (Hassan *et al*, 2005; Osayande *et al.*, 2013; Uzoho, 2014) but low compared to ranges of other studies (Osodeke *et al.*, 2005;Olatunji *et al.*, 2015). Also, Al_{ox} range was high compared to others (Hassan *et al.*, 2005; Osodeke *et al.*, 2005; Osayande *et al.*, 2013; Uzoho, 2014) but low relative to that of Olatunji *et al.* (2015). In general, bulk soil Al_{ox} was less than Al_d ascribable to the neoformation process of silicate clays or the increased alteration of weathering products with time, thus serving as an index or degree of weathering (Dolui and Mustafi, 1997; Hassan *et al.*, 2005). Equally, Al_{ox}/Al_d ratio was low relative to those of other workers (Osodeke *et al.*, 2005; Uzoho, 2014; Olatunji *et al.*, 2015) indicating that soils were well drained and contained highly crystalline Al. Compared to Fe_{ox}/Fe_d ratio, the Al_{ox}/Al_d was higher and signifying that chemistry of the soils were dominated by aluminium. Amongst landscapes, bulk soil Al_d, Al_{ox} and Al_{ox}/Al_d ratio were significantly (LSD 0.05) higher at the upslope, mid slope and flat surface positions respectively. Also, amongst particle size fractions, Al_d in sand fraction was significantly higher at the upslope or flat surface landscapes while that in the silt and clay fractions were at the down slope and upslope positions respectively, but with enrichment of the sand, silt and clay particles better at the flat surface, down slope and upslope positions respectively. Averaged over landscapes, clay fraction was more enriched in Al_d than other fractions. Equally, sand fraction Al_{ox} was better at the flat surface whereas silt and clay contents were at the upslope positions but with enrichment of all particle size fractions higher at the upslope than other landscape positions. Sand fraction was more enriched than particle fractions, averaged over landscape positions. Furthermore, sand and silt fraction Al_{ox}/Al_d ratio were better at the flat surface while clay was at the down slope position but with enrichment of the fractions better at the midslope (sand), down slope (silt) and upslope (clay) respectively. Averaged over landscapes, enrichment of the Al_{ox}/Al_d ratio decreased as sand > silt > clay.

Bulk soil Mn_d, Mn_{ox} and Mn_{ox}/Mn_d ratio ranged from 3300-5600mg kg^{-1}, 530-690 mg kg^{-1} and 0.10-0.16 respectively (Table 4), with the Mn_d and Mn_{ox} low compared to values of other study (Uzoho, 2014*)* but high relative to those reported by Maniyunda *et al.* (2015). Also, the range for Mn_{ox}/Mn_d ratio was high relative to values reported by the above authors. The Mn_d, Mn_{ox} and Mn_{ox}/Mn_d in the bulk soil were significantly (LSD 0.05) higher at the upslope, flat surface and mid slope positions respectively. Amongst particle size fractions, sand fraction Mn_d was significantly (LSD 0.05) higher at the upslope whereas the silt and clay fractions were at the down slope positions but with enrichment of the sand fraction higher at the flat surface while that of the silt and clay fractions were at the down slope position. Equally, whereas sand and silt fraction Mn_{ox} were significantly (LSD 0.05) higher at the flat surface, the clay was at the upslope landscape but with the sand, silt and clay fractions more enriched at the down slope, flat surface and upslope positions respectively. Finally, the sand, silt and clay fraction Mn_{ox}/Mn_d ratios were significantly (LSD 0.05) higher at the down slope, flat surface and upslope positions respectively, but with enrichment of the sand fraction better at the down slope whereas the silt and clay fractions were at the upslope position. Averaged over landscape positions, the silt size fraction was better enriched than the other size fractions. Ranges of bulk soil Si_d, Si_{ox} and Si_{ox}/Si_d ratio were 9600-13,500, 1690-1790 mg kg^{-1} and 0.13-0.18 respectively, and significantly (LSD 0.05) higher at the upslope, flat surface and mid slope positions respectively (Table 5). Amongst particle size fractions, sand, silt and clay fraction Si_d were significantly (LSD 0.05) higher at the upslope position, but with the enrichment of the size fractions better at the flat surface (sand), down slope (silt) and upslope or mid slope (clay) positions. Also, sand and silt fractions Si_{ox} were significantly (LSD 0.05) higher at the flat surface while that of the clay was at the upslope position, but with enrichment of the sand and clay fractions better at the upslope while that of the silt fraction was at the upslope or flat surface positions. Equally, sand, silt and clay fractions Si_{ox}/Si_d ratios were significantly (LSD 0.05) higher at the mid slope or down slope, flat surface and down slope positions respectively but with enrichment of the sand and clay fractions better at the down slope while the silt fraction was at the flat surface landscape position. Averaged over landscapes, the silt fraction was more enriched than others. In general, variations in concentrations and enrichment with particle size fractions could be due to differences in affinity for surfaces.

Table 4. *Dithionate Citrate Bicarbonate, Oxalate Manganese (mg kg^{-1}), Active Manganese and Enrichment Factors (EF) in Bulk and Particle Size Fraction of Soils*

Landscape Position	Bulk soil	Sand	Silt	Clay	EFsand	EFsilt	EFclay
			Dithionate Citrate Bicarbonate Mn (Mn_d)				
Upslope	5600	4500	2100	3700	0.80	0.38	0.66
Mid slope	3300	2800	2200	2400	0.85	0.67	0.73
Down slope	4700	2800	3500	4200	0.60	0.74	0.89
Flat Surface	4700	4200	2600	3900	0.89	0.55	0.83
LSD 0.01	554	430	287	353.12	0.05	0.05	0.06
			Oxalate Mn (Mn_{ox})				
Upslope	580	500	430	480	0.86	0.74	0.83
Mid slope	530	350	260	290	0.66	0.49	0.55
Down slope	570	510	390	450	0.89	0.68	0.79
Flat Surface	690	580	640	470	0.84	0.93	0.68
LSD 0.01	34.13	26.14	33.87	31.89	0.03	0.03	0.02
			Active Mn				
Upslope	0.10	0.11	0.20	0.13	1.07	1.98	1.25
Mid slope	0.16	0.13	0.12	0.12	0.78	0.74	0.75
Down slope	0.12	0.18	0.11	0.11	1.50	0.92	0.88
Flat Surface	0.15	0.14	0.25	0.12	0.94	1.68	0.82
LSD 0.01	0.02	0.02	0.01	0.01	0.07	0.08	0.05

EF = Enrichment factor

Table 5. *Oxalate and DCB Silicon (mg kg^{-1}), Active Si and Enrichment Factors (EF) in Bulk and Particle Size Fraction of Soils*

Landscape Position	Bulk soil	Sand	Silt	Clay	EFsand	EFsilt	EFclay
			Dithionate Citrate Bicarbonate Si (Si_d)				
Upslope	13500	11800	8900	10400	0.87	0.66	0.77
Mid slope	9600	6200	5400	7400	0.65	0.56	0.77
Down slope	10900	7000	8600	6700	0.64	0.79	0.61
Flat Surface	11200	11000	5900	7600	0.98	0.53	0.68
LSD 0.01	1054.24	1033.18	879.23	1005.21	0.12	0.12	0.11
			Oxalate Si (Si_{ox})				
Upslope	1690	1580	1490	1550	0.93	0.88	0.92
Mid slope	1770	1350	1290	1410	0.76	0.73	0.80
Down slope	1730	1560	1380	1470	0.90	0.80	0.85
Flat Surface	1790	1640	1570	1430	0.92	0.88	0.80
LSD 0.01	53.16	121.63	121.14	54.34	0.01	0.01	0.01
			Active Si				
Upslope	0.13	0.13	0.17	0.15	1.07	1.34	1.19
Mid slope	0.18	0.22	0.24	0.19	1.18	1.30	1.03
Down slope	0.16	0.22	0.16	0.22	1.40	1.01	1.38
Flat Surface	0.16	0.15	0.27	0.19	0.93	1.66	1.18
LSD 0.01	0.02	0.02	0.02	0.02	0.04	0.03	0.03

EF = Enrichment factor

Relationships amongst bulk soil sesquioxide forms, between sesquioxide forms and soil properties and between bulk soil and particle size sesquioxides

Relationships amongst sesquioxide forms, between sesquioxide forms and soil properties and sesquioxide forms in bulk soil and particle size fractions are presented in Tables 6, 7 and 8. In Table 6, Al_d correlated significantly ($P < 0.05$) with Al_{ox}, Mn_d and Mn_{ox}, Al_{ox} with all other sesquioxide forms exception being Mn_d while Al_{ac} was also with other sesquioxide forms except Si_d and Mn_d. Also, Fe_d and Fe_{ox} correlated significantly with other sesquioxide forms besides Mn_d while Fe_{ac} correlated significantly ($P < 0.05$) with all other sesquioxide forms exception being Si_d and Mn_d.

Table 6. *Correlation Matrix between various Sesquioxides in Bulk Soil*

Sesquioxide Fractions	Al_d	Al_{ox}	Al_{ac}	Fe_d	Fe_{ox}	Fe_{ac}	Si_d	Si_{ox}	Si_{ac}	Mn_d	Mn_{ox}	Mn_{ac}
Al_d	1											
Al_{ox}	0.50	1										
Al_{ac}	-0.04	0.84	1									
Fe_d	0.14	0.80	0.84	1								
Fe_{ox}	0.16	-0.56	-0.76	-0.94	1							
Fe_{ac}	-0.44	-0.90	-0.77	-0.95	0.79	1						
Si_d	0.09	-0.82	-0.10	0.82	0.75	0.74	1					
Si_{ox}	0.38	0.88	0.78	0.97	-0.84	-0.10	-0.75	1				
Si_{ac}	0.31	0.89	0.77	0.98	-0.86	-0.99	-0.81	0.99	1			
Mn_d	0.87	0.14	-0.37	0.03	0.16	-0.27	0.13	0.23	0.13	1		
Mn_{ox}	0.63	0.95	0.71	0.84	-0.61	-0.97	0.93	0.94	0.93	0.39	1	
Mn_{ac}	0.23	0.96	0.96	0.88	-0.73	-0.90	-0.94	0.89	0.93	-0.10	0.88	1

Table 7. *Correlation Matrix between Sesquioxides and Soil Properties*

Soil Properties	Sesquioxides											
	Al_d	Al_{ox}	Al_{ac}	Fe_d	Fe_{ox}	Fe_{ac}	Si_d	Si_{ox}	Si_{ac}	Mn_d	Mn_{ox}	Mn_{ac}
Sand	0.71	0.11	-0.33	-0.47	0.74	0.18	0.35	-0.26	-0.29	0.48	0.08	-0.15
Silt	-0.29	0.25	0.49	0.78	-0.93	-0.59	-0.48	0.65	0.66	-0.1	0.37	0.44
Clay	-0.85	-0.06	0.46	0.40	-0.66	-0.09	-0.49	0.16	0.23	-0.76	-0.15	0.23
MC	-0.73	0.06	0.54	0.56	-0.79	-0.27	-0.56	0.34	0.39	-0.63	0.02	0.34
TP	-0.09	0.42	0.56	0.88	-0.96	-0.74	-0.54	0.79	0.78	0.05	0.55	0.56
Bd	0.85	0.05	-0.05	-0.40	0.65	0.09	0.51	-0.16	-0.23	0.78	0.15	-0.24
OM	0.42	0.78	-0.97	0.94	-0.83	-0.97	-0.61	0.98	0.96	0.37	0.91	0.78
pH	-0.20	-0.55	-0.53	-0.90	0.89	0.86	0.50	-0.88	-0.86	-0.30	0.72	-0.62
Ca	0.66	0.87	0.61	0.84	-0.63	-0.96	-0.56	0.94	0.91	0.51	0.98	0.79
ECEC	0.59	0.22	-0.09	0.44	-0.37	-0.54	0.14	0.54	0.46	0.83	0.52	0.12

Table 8. *Relationship between Sesquioxide Contents of Bulk Soil and Particle Size Fractions*

Parameter	Regression Equation	R^2
bAl_{ac}	$Y(bAl_{ac}) = 14.16 + 1.32$ Al clay + 0.18 Al silt - 2.62 Al sand	0.52
bMn_{ac}	$Y(bMn_{ac}) = 1.84$ Mn sand + 0.74 Mn silt + 1.02 Mn clay - 17.94	0.61
bFe_{ac}	$Y(bFe_{ac}) = 2.92$ Fe clay + 0.77 Fe sand - 0.76 Fe silt - 31.75	0.74
bSi_{ac}	$Y(bSi_{ac}) = 0.14 + 0.50$ Si sand + 0.51 Si silt + 0.14 Si clay	0.72
bAl_d	$Y(bAl_d) = 172.46 + 0.82$ Al sand + 1.07 Al clay - 1.08 Al silt	0.47
bAl_{ox}	$Y(bAl_{ox}) = 1581.21 + 1.34$Al clay - 0.10 Al sand - 0.50 Al silt	0.64
bFe_d	$Y(bFe_d) = 8188.2 + 14.95$ Fe silt - 3.93 Fe sand - 25.54 Fe clay	0.69
bFe_{ox}	$Y(bFe_{ox}) = 5183.16 + 0.41$Fe clay + 0.54 Fe sand - 0.23 Fe silt	0.26
bMn_d	$Y(bMn_d) = 438.95 + 0.12$ Mn sand + 0.47 Mn silt -0.25 Mn clay	0.54
bMn_{ox}	$Y(bMn_{ox}) = 9781.97 + 4.25$ Mn clay - 2.18 Mn sand - 4.81 Mn silt	0.48
bSi_d	$Y(bSi_d) = 2653.07 + 0.18$ Si silt - 0.06 Si sand - 0.74 Si clay	0.67
bSi_{ox}	$Y(bSi_{ox}) = 3406.21 + 0.28$ Si sand +0.40 Si silt + 0.31 Si clay	0.49

b = bulk soil, ac = active,

Besides Mn_d, Si_d, and Si_{ox}, Si_{ac} correlated significantly ($P < 0.05$) with other sesquioxide forms. Other workers have reported correlations between Fe_d and Fe_o (Jaworska *et al.*, 2016), Fe_{ac} with Fe_o, Fe_d, and Al_d and Fe_o with Al_d (Olatunji *et al.*, 2015). Relationship between soil properties and sesquioxide forms (Table 7) showed that sand was significantly ($P < 0.05$) correlated with Al_d and Fe_{ox}, silt with Fe_d, Fe_{ox}, Fe_{ac}, Si_{ox} and Si_{ac}, clay with Al_d, Fe_{ox} and Mn_d while MC was with Al_d, Al_{ac}, Fe_d, Fe_{ox}, Si_d and Mn_d. Also there was significant ($P < 0.05$) correlation between TP with Al_{ac}, Fe_d, Fe_{ox}, Fe_{ac}, Si_d, Si_{ox}, Si_{ac}, Mn_{ox} and Mn_{ac}, Bd with Al_d, Fe_{ox}, Si_d and Mn_d and while OM and pH were with all sesquioxide forms exception being Al_d and Mn_d. Equally, whereas exchangeable Ca correlated significantly ($P < 0.05$) with all sesquioxide forms (Al_d, Al_{ox}, Al_{ac}, Fe_d, Fe_{ox}, Fe_{ac}, Si_d, Si_{ox}, Si_{ac}, Mn_d, Mn_{ox} and Mn_{ac}), ECEC correlated significantly ($P < 0.05$) with only Al_d, Fe_{ac}, Si_{ox}, Mn_d and Mn_{ox}. Significant correlation have been reported between clay and OC with Fe_d and Fe_o (Jaworska *et al.*, 2016), sand and clay with Al_d, OM and clay with Fe_o (Osodeke *et al.*, 2005), OM and clay with all sesquioxide forms (Maniyunda *et al.*, 2015) and various soil properties (sand, silt, clay, silt/clay ratio, TN, OM, P, Ca, K, Mg, Na, H, ECEC and % BS) with sesquioxide forms (Uzoho, 2014). Regression equation

(Table 8) indicated that sesquioxide concentrations in soil particle size fractions accounted for more than 55% of 2/3 of that in the bulk soil. Contributions of particle size fractions to various bulk soil sesquioxides showed that sand fraction dominated bulk soil Al_{ac}, Mn_{ac} and Fe_{ox} with coefficients of 2.62, 1.84 and 0.54 respectively, silt fraction in bulk soil Si_{ac} (0.51), Al_d (1.08), Mn_d (0.47), Mn_{ox} (4.81) and Si_{ox} (0.41) and clay fraction in bulk soil Fe_{ac} (2.92), Al_{ox} (1.34), Fe_d (25.54) and Si_d (0.74). Thus, silt fraction dominated particle size inputs to bulk soil sesquioxides.

Conclusions

Bulk soil oxalate sesquioxide was lower than dithionate forms indicating low mobility and high crystallinity. Also, bulk soil oxalate/dithionate ratios were generally low. From the low ratios, it can be inferred that the soils were well drained, old and highly weathered, with the degree being a decreasing sequence of Upslope > flat surface > down slope > mid slope. Amongst landscape positions, sesquioxide contents and re-activities in bulk soil and particle size fractions differed. Equally, enrichment of various particle size fractions with sesquioxide forms differed with landscape position, with sand more enriched averaged over landscape positions. A regression model explained that particle size fractions accounted for about 50% of 2/3 of bulk soil sesquioxides, with silt dominant over other size fractions. Correlation between most sesquioxide forms and sesquioxide forms with selected soil properties (sand, silt, clay, MC, TP, OM, pH, Ca and ECEC) were significant.

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