



FORMS AND DISTRIBUTION PATTERN OF IRON OXIDE IN SOILS FORMED UNDER FOUR DIFFERENT PARENT MATERIALS IN SOUTHERN WESTERN NIGERIA.

I. P. Egharevba and a. O. Bakare

Department of Soil Science and Land Management,
University of Benin, Benin City, Nigeria.
anthonia.bakare@uniben.edu

ABSTRACT

This study investigated the forms and distribution of Iron (Fe) oxide in soils developed from four different parent materials in Nigeria, and their relationship with some soil physical and chemical properties. Composite soil samples were collected at soil depths of 0–30cm, 30–60cm, 60–90cm and 90–120cm from Idanre (Basement complex) in Ondo State, Koko (Alluvium) in Delta State, Nigeria Institute for Oil Palm Research (NIFOR) (Coastal Plain Sands) and Uhonmora (Shale) both in Edo State, Nigeria. The soils were analyzed for the physical and chemical properties, using standard laboratory techniques. The concentrations and forms of Fe were extracted using the total Ammonium Oxalate Extract (Amorphous forms), Citrate – Bicarbonate Dithionite Extract (Crystalline forms) and the Pyrophosphate solution (Amorphous organic forms). Data obtained were subjected to statistical analysis and the mean separation was carried out using the Duncan Multiple Range Test (DMRT). Results showed that the concentrations of iron oxides in the soils were higher than those of aluminum and manganese in all the locations. The crystalline free oxides dominated the soils in Shale, Hydromorphic, Coastal plain sand and Basement Complex. They had mean concentrations ranging from 1.46–5.71mgkg⁻¹ (mean of 3.40mgkg⁻¹). The Amorphous forms of Fe oxides were generally lower than other oxides in the four locations. The lowest mean concentration for all the oxide forms were found in Coastal plain sand compared to shale which had the highest concentration. Correlation studies showed that there was a positive and significant ($p>0.5$) correlation between clay and other parameters, suggesting that the oxide concentration increased with the prevalence of clay in all the location studied.

Keywords: Iron (Fe) oxide, Composite soil, Basement complex, Coastal Plain sands, Shale and Alluvium



INTRODUCTION

Iron oxide is generally referred to as sequioxide (Essoka and Esu, 2000). They are commonly found in tropical rain forest and formed as a result of leaching caused by heavy rainfall (Greenwood and Earnshaw 1997). During rainfall, there is slight to moderate leaching that results in the removal of the mobile cations leaving behind compound such as aluminum oxides, iron oxide, quartz (SiO_2) and some silicate mineral. Forms of this oxides fraction may be present as organic complexes and together they influence several soil physical and chemical properties and the parent materials (Hassan *et al.*, 2005). Relevance of Iron oxides in soil physical and chemical processes vary depending on their crystallinity, with the less crystalline or amorphous fractions more reactive than the highly crystallized forms. For instance, it has been indicated that P and metal sorption is higher with amorphous Fe than the crystalline forms (Uzoho and Oti 2005). According to Hassan *et al.*, (2005), the dithionite extractable Fe have been considered to give a reasonable estimate of the total pedogenic free Fe while oxalate extractable forms represent the amorphous Fe in soils. Reactivity of oxides depends

on the ratio of the oxalate to the dithionite extractable Fe oxides and describes the relative amount of the poorly ordered and crystalline Fe oxides in the soil (Omenihu *et al.*, 1994). Poorly drained soils are more reactive with higher ratios than well-drained soils. It has been established that the content of different sesquioxides in tropical soils increases as the soil ages, largely due to the influence of weathering (Osodeke *et al.*, 2005), thus their distribution has been used as a yardstick to indicate and determine the degree and stage of soil development. The distribution of Fe oxides in some Nigerian soils has been variously studied. However, there is a dearth of information with respect to these oxides in soils selected based on the parent material from which they were formed. This study was therefore undertaken to provide information on the distribution of dithionite and acid ammonium oxalate extractable, pyrophosphate as well as the total forms of Fe in selected soils of Ondo, Delta and Edo states formed on basement complex, shale, coastal plain sands and hydromorphic.

Materials and Methods

3.1. Description of the Study Area

- i. The Soil samples for the experiment were collected from four different parent materials in



Nigeria; the first location was in NIFOR, a rainforest agro-ecological zone of Nigeria and lies between latitude 06°33'N and longitude 05°37'E (Fig 1). Humid tropical climate prevails with average annual rainfall of 1500 - 2500 mm. In the raining season, the rainfall pattern is bimodal and falls between April and October, while the dry season is between November and March. Mean minimum and maximum daily temperatures are 22°C and 31°C. The soils developed from Coastal Plain Sands are acidic and classified as ultisols. The soils of the study area have been reported to belong to the Orlu series and classified as Rhodic Paleudult (Fagbami, 1985). The second location was at Uhonmora (Shale) lies between Latitude 6° 30'45"N and Longitude 5°50'26"E, Rainfall is between 1575 mm to 1725 mm across the year have previously been described as Rhodic tropudualf (Fagbami, 1985). The third location was at Idanre (Basement Complex), it lies between latitude 6° 43'0"N and longitude 5°6'0"E. Topographically, Idanre is composed of undulating lands and hills. It is within the rainforest ecological zone, with two distinct seasons – the rainy (April to

October) and dry (November to March) seasons. The mean annual rainfall is 1500 mm and the mean annual temperature is 26°C and the fourth location was at Koko (Freshwater swamp) lies within latitude 06°0'04"N and longitude 05° 28'03"E, with a mean annual rainfall of 1500 mm. The rainfall distribution is bimodal with peak in July and September and a low amount of precipitation in August. The mean annual temperature is about 24 °C with about 37 °C as maximum. The relative humidity is about 77.3 % while the mean monthly soil temperature at 100 cm depth is 28°C. Sunshine generally stands at 4.8 bars (Meteorological Station Anwai Asaba Campus). Soils are hydromorphic and humic with high water table.

Soil Sampling, Preparation and Analysis Procedure

Composite samples were collected from three (3) replicate in each of four locations (shown in Fig 1) at a soil depth of 0-30 cm, 30-60 cm, 60-90 cm and 90-120 cm, to give 12 samples per location, a total of 48 samples were collected across the locations.

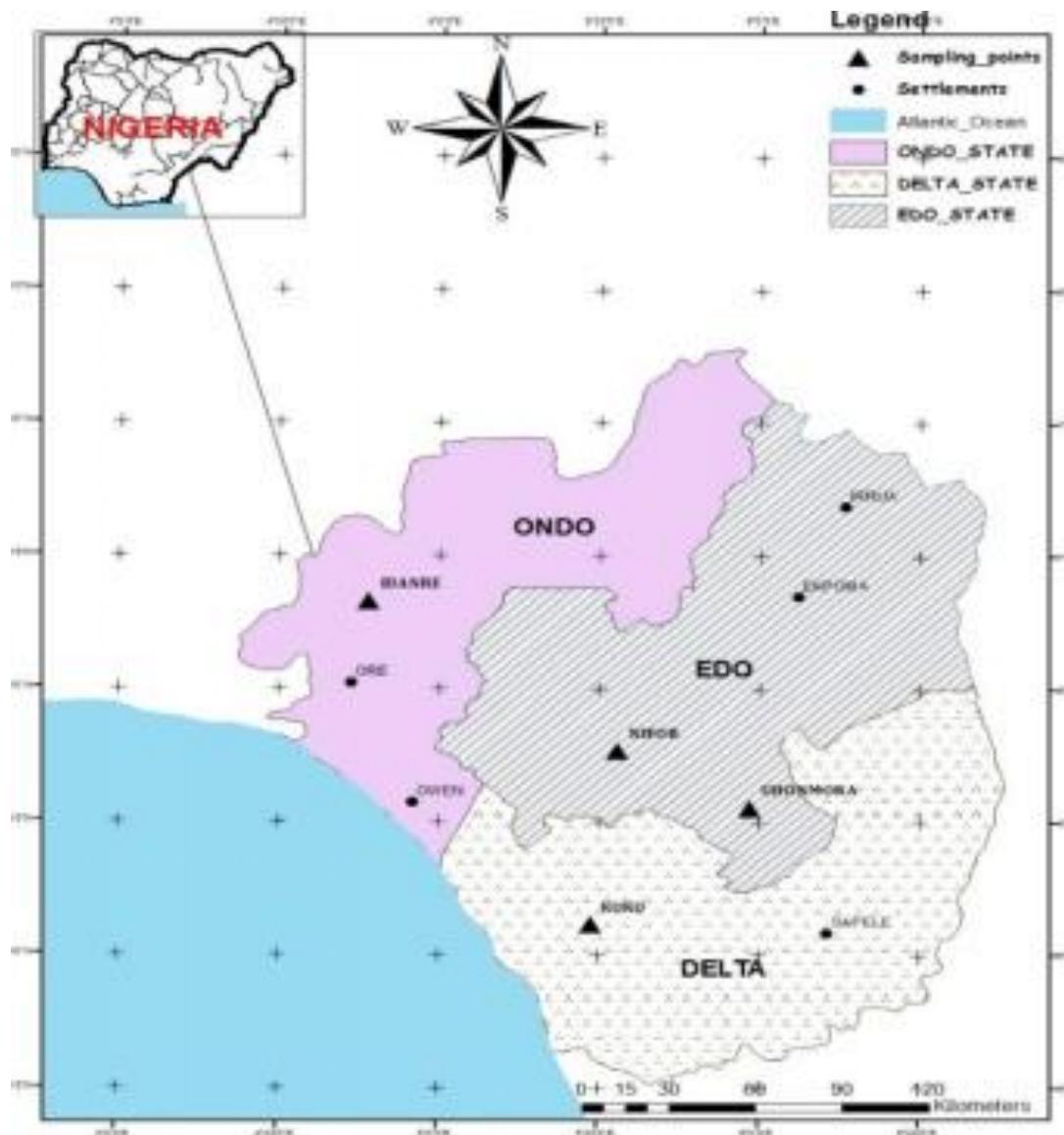


Figure 1: Map of Edo, Ondo and Delta States showing the sample locations

The soil samples were collected using a soil auger, air dried, sieved through 2 mm sieve and also a 0.5mm mesh sieve. They were

stored in well labeled polythene bags for subsequent analysis.

Particle size distribution was determined by the hydrometer



method according to Okalebo *et al.*, (2002), Soil pH was measured in 1:2 soil water suspension (Macleans, 1982), Organic Carbon was determined by wet dichromate acid oxidation method (Nelson and sommers, 1982), Total N was determined using the micro-kjeldahl method (Anderson and Ingram, 1993), Exchangeable acidity (EA) was extracted using 1M KCl (IITA 1979). Exchangeable cations (Ca, Mg, K and Na) were extracted with 1N NH₄OAC at pH 7.0. Potassium and Na were determined with a flame emission photometer while Ca and Mg were determined with atomic absorption spectrophotometer (Anderson and Ingram, 1993). Effective cation exchange capacity (ECEC) was calculated by the summation of exchangeable bases and exchange acidity (Anderson and Ingram, 1993).

Total iron (Fe) was extracted with double acid (mixture of 3 parts of HCl to 1 part of HNO₃) as described by IITA (1979). Ammonium oxalate extractable Fe was determined as describe Jackson *et al.*, 1986, Ammonium oxalate Fe extract was determined by Inductive coupled plasma technique. Dithionite-Citrate Fe was determined using the methodology by Soil Survey Staff (2006) determination of Fe was done by ICP-AES (inductive

coupled plasma). The amorphous organic forms of Fe, Al and Mn were extracted using the McKeague and Day (1966) method.

Data obtained were analyzed using Genstat statistical package (10th edition) package. Mean separation was carried out by Duncan's Multiple Range Test (DMRT).

Results and Discussion

The sand and silt contents at the four soil depths in Uhunmora decreased with increase in soil depth from 633.30 g kg⁻¹ to 533.30 g kg⁻¹ for sand and 120.00 g kg⁻¹ to 70.00 g kg⁻¹ while the clay content increased from 216.70 g kg⁻¹ to 350.00 g kg⁻¹ (Table 1) Koko and Idanre locations however showed no significant difference among the different particle sizes irrespective of soil depth. In the Coastal plain sand of NIFOR the sand content of the top soil decreased significantly ($P < 0.05$) from 786.70 g kg⁻¹ at 0 – 30 cm depth to 630.00 g kg⁻¹ at 90 – 120 cm depth. Whereas the silt and clay contents at the respective soil depth examined were not significantly different.

Sand dominated the particle size fraction of the fine earth (< 2 mm) portion in all the soils formed from the different parent materials. This agrees with studies on soils formed on basement complex



rocks as well as other parent materials in different regions of Nigeria (Malgwi *et al.*, 2000; Odunze, 2006; Fasina *et al.*, 2007; Obi and Akinbola, 2009; Ande, 2010). Generally, there was a decrease in sand with depth with clay accumulating in the sub soils. The soil chemical properties indicated that the soils were slightly acidic to extremely acidic (Table 2) and fall within range reported on soils in Southern Nigeria (Fasina, *et al*, (2007)). The acidic properties may be due to lack of oxygen in the soils and decomposition of organic matter to release organic acids (humic and

fluvic acids). The soils were generally low in organic carbon (range 5.73- 8.59 g/kg) and total Nitrogen (0.27-0.61 g/kg). Available phosphorus varied between 6.58-11.3 mgkg⁻¹ in content and rated low to high. The low content of organic carbon, total N and available phosphorus in these soils were attributed to continuous cultivation, bush burning, high rate of mineralization and crop removal without incorporating back to soils (Odunze, 2006). Also, low Organic carbon might be attributed to continuous

**Table 1: Particle size distribution of the soils in Uhonmora and Koko**

Location	Depth (cm)	Parent materials	Sand	Silt	Clay	Textural Class
				g kg ⁻¹		
Uhonmora	0-30	Shale	663.30 _a	120.00 _a	216.70 _b	SCL
	30-60		633.30 _{ab}	96.67 _{ab}	270.00 _{ab}	SCL
	60-90		580.00 _{ab}	70.00 _b	350.00 _a	SC
	90-120		533.30 _b	76.67 _b	390.00 _a	SC
KOKO	0-30	Hydromorphic	843.30 _a	66.67 _a	110.00 _a	LS
	30-60		843.30 _a	56.67 _a	113.30 _a	LS
	60-90		830.00 _a	46.67 _a	116.70 _a	LS
	90-120		816.70 _a	36.67 _a	120.00 _a	LS
NIFOR	0-30	Coastal Plain Sand	786.70 _a	43.33 _a	170.00 _b	SL
	30-60		693.30 _{ab}	30.00 _a	276.70 _a	SCL
	60-90		646.70 _b	30.00 _a	323.30 _a	SCL
	90-120		630.00 _b	30.00 _a	340.00 _a	SCL



Idanre	0-30	Basement complex	633.30 _a	160.00 _a	206.70 _a	SCL
	30-60		623.30 _a	156.70 _a	220.00 _a	SCL
	60-90		583.30 _a	180.00 _a	236.70 _a	
	90-120		583.30 _a	146.70 _a	270.00 _a	SCL

Mean value (s) with the same coloum letter(s) in the column are not significantly different from one another at 5% level of probability.

Table 2: Chemical properties of the soil in Uhonmora and Koko Locations

Locations	Depths (cm)	Parent materials	pH	Org C	N	P	Ca	Mg	K	Na	EA	E C E C	BS(%)
				(g kg ⁻¹)	(g kg ⁻¹)	(mg kg ⁻¹)	←	cmol kg ⁻¹					
Uhonmora	0-30	Shale	5.87a	10.43a	0.33a	11.28a	3.58a	0.67a	1.51a	0.33a	0.90a	6.18a	98.60a
	30-60		5.83a	4.92b	0.26b	13.15a	2.5b	0.69a	1.05b	0.26b	0.10a	4.66b	97.77a
	60-90		5.53a	4.06b	0.24b	13.27a	2.52b	0.62a	0.90b	0.24b	0.12a	4.40b	97.45a
	90-120		5.27a	3.52b	0.23b	7.72a	2.88ab	0.52a	0.81b	0.23b	0.24a	4.68b	94.91a
KOKO	0-30	Hydromorphic	4.80a	10.56a	0.75a	8.91a	0.58a	0.10a	0.47a	0.10a	1.25a	2.50a	51.73a
	30-60		4.57a	6.42b	0.46b	5.96b	0.47a	0.06a	0.23b	0.06b	0.88ab	1.70ab	49.04a
	60-90		4.57a	3.68b	0.26b	5.94b	0.43a	0.04a	0.14c	0.08b	0.62ab	1.32b	52.88a



90-120	4.43a	3.52b	0.25b	5.49b	0.39a	0.06a	0.17ab	0.07b	0.52b	1.22b	56.52a
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Mean value (s) with the same letter(s) in the same column are not significantly different from one another at 5% level of probability. LSD = Least significant difference

Table 2 (cont.): Chemical properties of the soils studied in NIFOR and Idanre Location

Location s	Depth s (cm)	Parent material s	pH	Org C g kg ⁻¹	N g kg ⁻¹	P mg kg ⁻¹	Ca	Mg	K	Na	EA	ECE C	BS	
							(cmol kg ⁻¹)					%		
NIFOR	0-30	Coastal Plain Sand	4.70 a	11.05a 0.79a		9.89a		1.23a	0.27a	1.11a	0.20a	0.56 a	3.36a	83.97a
	30-60		4.43 a	5.51b b	0.39	8.05a		0.78b	0.45a	0.52b	0.12b	0.99 a	2.87a	67.52a b
	60-90		4.30 a	5.28b b	0.38	7.53a		0.69b	0.15a	0.38b	0.09b a	1.62 a	2.92a	46.02b
	90- 120		4.30 a	4.27b b	0.31	6.93a		0.74b	0.08a	0.32b	0.08b a	1.68 a	2.91a	43.16b
Idanre	0-30	Baseme nt Comple x	6.23 a	15.13 a	1.08 a	14.48a		3.70a	1.73a	1.40a	0.36a	0.05 a	7.23a	99.14a
	30-60		5.93 a	7.60b a	0.54 a	11.85a		2.43a b	0.48ab	0.99a	0.24a b	0.06 a	4.19a b	98.03a
	60-90		5.87 a	6.13b a	0.44 a	8.64a		1.71b	0.13b	0.76a	0.19a b	0.07 a	2.85b	97.24a
	90- 120		5.77 a	5.51b a	0.39	8.51a		1.48b	0.15b	0.62a	0.16b a	0.24 a	2.66b	92.10a

Mean value (s) with the same letter(s) in the same column are not significantly different from one another at 5% level of probability.

LSD = Least significant difference



cultivation, bush burning, high rate of mineralization. The low contents of organic C, total N, and available P obtained in the sites studied could also be partly related to their inherent low status with low weatherable mineral reserve necessary for nutrient recharge (Bakare *et al.*, 2022), coupled with the presence of tree crops with little litter droppings, high temperatures across all the locations facilitating the rapid decomposition and mineralization of organic materials. However, the values of organic carbon, total nitrogen and available phosphorus content at 0-15cm depth were significantly higher than the other depths. This may be attributed to accumulation and decomposition of organic matter on surface horizons. Similar content and trend were reported by Esu *et al.*, (1987). The parent material seemed to have influence on the ECEC indicating high leaching of exchangeable bases in the soils. The organic carbon content of the soils reduced with depth, and ranged between 5.73 – 8.59 g/kg.

Analysis of the free, amorphous and organic matter bound forms of the oxides (Table 3) showed that the free/crystalline, that is the Dithionite Citrate bi-carbonate (DCB- Fe_2O_3) forms were highest form of Fe oxides and in Hydromorphic soils (4.49-6.12 mg/kg) than the amorphous that is the Ammonium

oxalate (OX- Fe_2O_3), organic matter bound forms Pyrophosphate (P- Fe_2O_3) and Total Iron Oxide (T- Fe_2O_3). The values obtained for pyrophosphate extractable iron were higher than those values reported by Raji *et al.* (2000), but lower than the values reported in wetland soils of Nigeria (Olaleye *et al.*, 2000). The variation in P- Fe_2O_3 values may be due to difference in the amount of organic matter generated by vegetations in the different ecological zones of the various studies.

**Table 3: Extraction methods and Iron oxides distribution along the soil depth in Uhonmora, Koko, Nifor and Idanre locations**

Locations	Depths (cm)	Parent materials	DCB-Fe ₂ O ₃	Ox-Fe ₂ O ₃	P-Fe ₂ O ₃	T-Fe ₂ O ₃	Locations	Depths (cm)	Parent materials	DCB-Fe ₂ O ₃	Ox-Fe ₂ O ₃	P-Fe ₂ O ₃	T-Fe ₂ O ₃
mg/kg													
Uhonmora	0-30	Shale	3.93a	2.76a	2.86a	9.59a	Nifor	0-30	Coastal Plain Sand	1.50b	0.81a	1.04a	3.40a
	30-60		3.94a	2.74a	2.87a	9.58a		30-60		1.65a	0.60b	0.70b	2.98b
	60-90		3.89a	2.94a	2.83a	9.68a		60-90		1.31d	0.61b	0.70b	2.66c
	90-120		3.85a	3.10a	2.80a	9.81a		90-120		1.39c	0.50c	0.65c	2.57c
Koko	0-30	Hydromorphic	6.12a	1.66c	2.10c	10.67ab	Idanre	0-30	Basement complex	2.90a	1.70b	2.65b	7.45b
	30-60		6.43a	2.17b	2.74b	12.27b		30-60		2.48a	2.43ab	3.83a	8.80a
	60-90		5.71b	2.89a	4.18a	13.00a		60-90		2.58a	2.55a	3.99a	9.20a
	90-120		4.59c	2.89a	3.96a	12.00b		90-120		2.06a	2.97a	4.08a	8.50ab

Mean value (s) with the same letter(s) in the same column are not significantly different from one another at 5% level of probability. LSD = Least significant difference

DCB – Dithionite Citrate bi-carbonate

OX – Ammonium oxalate

P – Pyrophosphate

T – Total



The values obtained (Table 3) were within range reported in established forest in Southern Guinea Savanna of Nigeria (Samndi *et al.*, 2006). The soils generally had irregular distribution of P- Fe_2O_3 in their profiles and were similar to those reported by Abdourahamane and Yaro (2007). In Koko, the relative accumulation of DCB- Mn_2O_3 in the subsurface horizons agrees with the finding of Maniyunda *et al.*, (2015) and is attributed to eluviation-illuviation processes, and probably related to release of Mn oxides from silicates (Blume and Schwertmann, 1969; Nahon *et al.*, 1989). The illuviation and accumulation of total free manganese oxide in subsurface horizons may be significantly influenced by clay content.

Significant positive and negative relationships were observed in the Correlation between the Fe oxides forms and the soil properties studied (Table 4). Most of these relationships were positive with clay, suggesting that the oxide concentrations increased with the prevalence of clay. The DCB- Fe_2O_3 however showed significant and negative correlation in NIFOR (Coastal plain sands) and Koko (hydromorphic). Crystalline and total Al correlated significantly in the coastal plain sands of NIFOR while all forms of Fe in the basement complex soils of Idanre had significant relationships with

available P. Oyeyiola and Omueti (2016) got similar results when DCB Fe_2O_3 correlated with phosphorus of some Alfisols in Southwestern Nigeria and reported that Fe_2O_3 , Al_2O_3 and clay are dominant factors influencing P fixing capacity of those soils. Free iron oxide was found to be responsible for up to 80% while free Al_2O_3 and clay accounted for 77 and 79% of P fixed respectively.

**Table 4: Correlation coefficient between some soil physical and chemical properties and the extractable forms of Iron oxides Uhommora (Shale) and Koko location**

Uhommora					Koko				
Soil properties	DCB Fe ₂ O ₃	Ox-Fe ₂ O ₃	P-Fe ₂ O ₃	T-Fe ₂ O ₃	Soil properties	DCB Fe ₂ O ₃	Ox-Fe ₂ O ₃	P-Fe ₂ O ₃	T-Fe ₂ O ₃
Sand	0.132	-0.023	0.339	0.008	Sand	-0.033	-0.21	-0.278	-0.213
Silt	0.102	-0.052	0.182	-0.035	Silt	-0.098	0.169	0.252	0.141
Clay	-0.132	0.033	-0.315	0.004	Clay	0.447	0.163	0.119	0.268
pH	-0.489	0.557*	0.078	0.572*	Ph	0.306	-0.427	-0.257	-0.178
O.C	0.204	-0.131	0.273	-0.114	O.C	0.495	0.807**	0.844**	0.910**
N	0.204	-0.131	0.273	-0.114	N	0.495	0.807**	0.844**	0.910**
A.P	-0.273	0.393	-0.124	0.401	A.P	0.096	0.02	-0.105	-0.004
K	-0.136	0.245	0.223	0.269	K	0.392	0.829**	0.936**	0.930**
Ca	0.006	0.163	0.086	0.192	Ca	0.26	0.575*	0.616*	0.645*
Mg	0.566*	-0.561*	0.092	-0.561*	Mg	0.21	0.334	0.431	0.443
Na	0.275	-0.14	0.304	-0.119	Na	-0.199	0.755**	0.770**	0.595*
ECEC	0.272	-0.155	0.202	-0.087	ECEC	0.371	0.654*	0.737**	0.771**

B – Dithionite Citrate bi-carbonate

OX – Ammonium oxalate

P – Pyrophosphate

T – Total

* – Correlation () is significant at 0.05 level**
 – Correlation is significant at 0.01 level

**Table 4 (cont): Correlation coefficient between some soil physical and chemical properties and the extractable forms of iron oxide in Nifor and Idanre location**

Nifor					Idanre				
Soil properties	DCB Fe ₂ O ₃	Ox-Fe ₂ O ₃	P-Fe ₂ O ₃	T-Fe ₂ O ₃	Soil properties	DCB Fe ₂ O ₃	Ox-Fe ₂ O ₃	P-Fe ₂ O ₃	T-Fe ₂ O ₃
Sand	0.582*	-0.794**	-0.762**	-0.379	Sand	-0.304	-0.865**	-0.736**	-0.854**
Silt	0.205	-0.31	-0.337	-0.047	Silt	0.376	0.526	0.497	0.634*
Clay	-0.590*	0.810**	0.784**	0.372	Clay	0.262	0.873**	0.732**	0.835**
pH	0.447	-0.662*	-0.561*	-0.244	pH	-0.441	-0.920**	-0.758**	-0.901**
O.C	0.404	-0.842**	-0.795**	-0.687*	O.C	0.702*	-0.019	-0.312	0.159
N	0.404	0.842**	-0.795**	-0.687*	N	0.702*	-0.019	-0.312	0.159
A.P	0.308	-0.442	-0.395	0.157	A.P	-0.631*	-0.795**	-0.713**	-0.909**
K	0.482	-0.825**	-0.773**	-0.554	K	0.457	-0.009	-0.109	0.194
Ca	0.304	-0.832**	-0.763**	-0.664*	Ca	-0.231	-0.719**	-0.807**	-0.734**
Mg	0.466	-0.425	-0.39	0.147	Mg	-0.229	-0.620*	-0.761**	-0.682*
Na	0.526	-0.845**	-0.786**	-0.534	Na	0.094	-0.414	-0.497	-0.309
ECEC	0.660*	-0.832**	-0.765**	-0.325	ECEC	-0.099	-0.584*	-0.723**	-0.569*

B – Dithionite Citrate bi-carbonate

OX – Ammonium oxalate

P – Pyrophosphate

T – Total

* – Correlation is significant at 0.05 level

** – Correlation is significant at 0.01 level



Conclusion and Recommendations

The results indicated that the crystalline iron oxides clearly influenced the availability of nutrient components such as potassium and phosphorus of these soils. Their high amounts also suggest that there might be deterioration of physical and chemical properties. These high values indicate that overtime there will be formation of nodules, concretions and plinthites in the soils. The consequence of this is that the soils may eventually be rendered unsuitable for cultivation of crops in all the parent materials. Based on these findings, it is recommended that soil management practices that improve the organic matter level of soils such as green manuring, alley cropping, improved fallowing, etc. should be adopted. Also, deficient nutrients in the soils studied should be replenished through the use of organic and inorganic fertilizers as the soils studied had low nutrient status.

From the correlation tables, in Uhonmora, Koko, NIFOR and Idanre the total pyrophosphate iron forms correlate highly with the organic carbon showing the importance of amorphous organic materials as major banks for nutrient exchange.

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