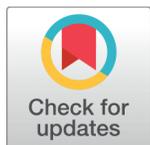


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Mineralogy and Geochemistry of some Phosphate Deposits for Possible Rare Earth Elements Mineralization Potentials within Sokoto Basin, Northwestern Nigeria

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Abstract

Objective: Phosphate rocks found in some parts of Sokoto Sedimentary basins in Nigeria were studied to understand their mineralogy and geochemistry specifically to determine their abundance in Rare Earth Elements hosting minerals notably Apatite, Monazite and Xenotime as there is an increasing demand for these elements globally in the production of green technology.

Methods: Field observations were carried out in places with reported phosphate occurrences and seven (7) representative phosphate samples were collected, crushed, pulverized, and analyzed using X-Ray Diffraction (XRD) analysis for their mineralogical composition while samples with significant fluorapatite concentrations were further analyzed with Lithium Borate fusion Inductively Coupled Plasma Mass Spectrometry (ICPMS) for their elemental composition. **Findings:** The phosphates typically occur as nodules intercalated with Shale occurring at depth of about 0.25 – 7m with a mineralogical composition including Fluoroapatite, Calcite, Smectite, Quartz, Kaolinite, Goethite, and Palygorskite. A high concentration of Fluoroapatite was observed at Miyalyako (79.28%) and Dilingo (36.60%) while interpretation of the relationship between P₂O₅ with other major oxides and trace elements revealed that the phosphates were primary type and typically formed in an oxidizing environment with fluctuating pH in the presence of calcite or gypsum. **Novelty:** The study pinpoints that, of the seven locations, only two showed high concentrations of fluoroapatites enough to suggest that they are areas of interest for further investigation in the search for REEs mineralization in Nigeria.

Keywords: Rare Earth Elements; Phosphate; Mineralisation; Fluoroapatite; Sokoto

1 Introduction

The drastic global shifts from the production of technologies with huge carbon emissions to eco-friendly and green technologies have become inevitable because of the numerous adverse effects of carbon emissions on the environment. Notably among the raw materials used in green technologies are the Rare Earth Elements (REEs) including Lanthanum (La), Cerium (Ce), Praseodymium (Pr), Neodymium (Nd), Promethium (Pm), Samarium (Sm), Europium (Eu), Gadolinium (Gd), Terbium (Tb), Dysprosium (Dy), Holmium (Ho), Erbium (Er), Thulium (Tm), Ytterbium (Yb), Lutetium (Lu), Scandium (Sc) and Yttrium (Y). These elements are widely used currently as metal alloys in the production of rechargeable batteries, cell phones, catalysts, ceramics, glass polishing, metallurgy, magnets, and fluorescent lighting among several others^(1,2).

Geologically, the REEs occur in Carbonatites, Alkaline Igneous rocks, ion absorption clay deposits, monazite-xenotime-phosphate deposits, Fluoroapatites, some pegmatites, tailings of red muds produced during bauxite mining, and as placer deposits in some laterites due to weathering of REEs rich rocks. All these sources are being explored in the production of REEs globally, with China being the world's largest producer accounting for about 62%, followed by the United States (12.2%), Myanmar (10.3%), Australia (9.9%), India (1.4%) and other countries (4.2%). The increasing demand for REEs has led researchers into more exploration works on alternative sources to Carbonatites, with phosphates being one of such⁽³⁾. The Nigerian Phosphate deposits are dominantly found within the Sokoto and Dahomey basins in the Northwestern and Southwestern parts of the country (Figure 1). The Sokoto Basin is characterized by a gently undulating plain with an average elevation of between 250 to 400m above sea level and origin believed to be the subsidence of the cratons because of change in mantle thermal regime and flexural of the lithosphere in response to sediments and water loading⁽⁴⁻⁶⁾.

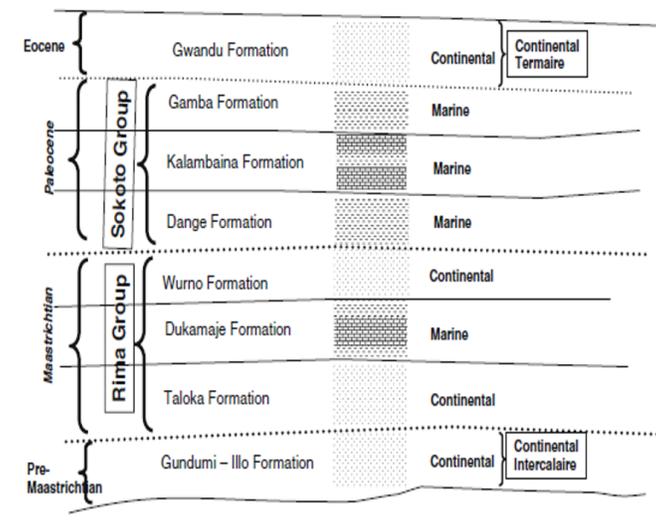


Fig 1. Stratigraphic succession of Nigeria session of Iullummenden Basin showing Dunkamaje and Dange Formations hosting the phosphate deposits⁽⁷⁾.

These Sokoto phosphates have been studied for their agricultural usage, but no information about the potential for REEs mineralizations has ever been mentioned⁽⁸⁻¹⁰⁾. This study, therefore, aims at understanding the mineralogy and geochemistry of phosphate deposits found in some parts of Sokoto Sedimentary Basins in the Northwestern part of Nigeria to determine their enrichment levels in REEs hosting minerals.

2 Study Area Description

The sample locations lie within the Sokoto Basin and are defined by Latitudes 13°34'00" – 13°36'00"N and Longitudes 5°34'00" – 5°37'00"E. Identified villages within the area include Chimmola, Miyalyako, Gidan Fako, Gaigawo, Dillingo and Bang'awge (Figure 2). The climate of the area is Sudan climate characterized by Rainfall between 500-1000mm per annum, a high temperature throughout the year in the range of 21°C-33°C, and a long period of little or no rainfall, while the vegetation is also Sudan savannah with short, scanty grasses and many droughts resistant plants. Accessibility is through major roads and footpaths.

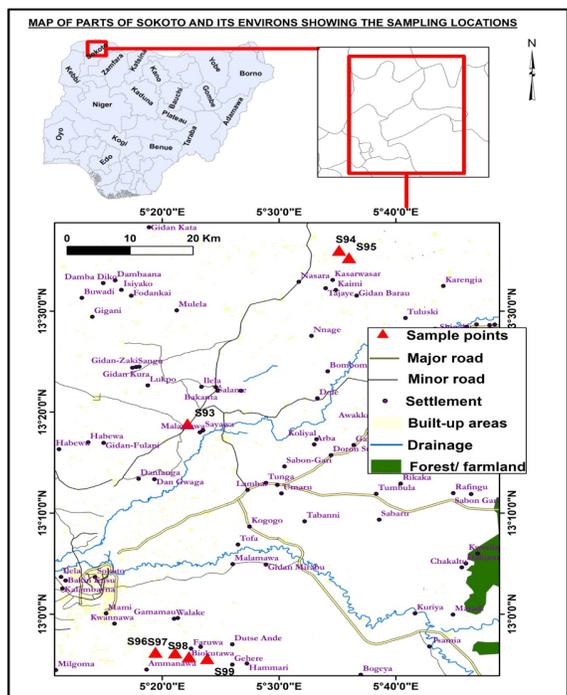


Fig 2. Map of parts of Sokoto and its environs showing the sampling locations

3 Methodology

Ground trotting of the areas was done to physically identify notable places with phosphate occurrences and the locations' geographic coordinates were determined using the Global Position System (G.P.S). A detailed description of each location was done by recording in-situ field characters such as depth of road cut exposures and pits, observable change in color, and lithology down the pits excavated to phosphatic layers. Representative phosphate samples were separated and handpicked from the road cuts/excavated pits at depths ranging from surface level to about 7m and bagged in well-labeled sample bags before being transported to the Laboratory (Figure 3). The phosphate samples were air-dried at room temperature, pulverized using an agate mortar and pestle to disaggregate into the finer size, and sieved to extract the < 65-micron fraction. The mortar, pestle, and sieve were cleaned with methylated spirit for each sample while sieved fractions were packaged in re-sealable sachets and sent to the Laboratory for X-Ray Diffraction analysis to determine mineralogical constituents at the University of Poitier Laboratory, France. The method of sample preparation is non-destructive and the sample fractions can be recovered for further testing or refinement. Approximately 1g was kept as a reference, 5g are taken for bulk sample preparations, and the remainder was used in the preparation of decalcified, fractionated 2-20 μ , and <2 μ samples.

Separate sets of pulverized portions of the phosphate samples were also analyzed for total elements contents using Inductively Coupled Plasma Mass Spectrometry (ICPMS) at Activation Laboratory Limited, Canada. Pulverized samples were mixed with excess lithium borate and heated until the mixture formed a homogenous mass then dissolved in 5% HNO₃ before analysis. Blank fusion solutions were provided so that calibration standards and blanks could be matric matched while quality assurance and quality control procedures were carried out using standard reference materials: OREAS101b, NCSDC86318, BCR-2, USZ42-2006, REE-1, and W-2b. Detection limits for MnO and TiO₂ were 0.001%; SiO₂, Al₂O₃, Fe₂O₃(T), MgO, CaO, Na₂O, K₂O, and P₂O₅ were 0.01%; Bi 0.4 μ g/g; Ag, Sb, and Cs was 0.5 μ g/g; Sc, Be, Y, Co, Ga, Ge, Nb, Sn, and W was 1 μ g/g; Ba, Sr, Zr, Rb, and Mo was 2 μ g/g; V, As and Pb was 5 μ g/g; Cu 10 μ g/g; Cr and Ni were 20 μ g/g while Zn was 30 μ g/g. Duplicate samples and blank samples were also performed throughout the experiment.



Fig 3. Field occurrences of phosphates in the study area: a) Road cut exposure at Chimmola village showing the shales intercalated with phosphates. b) Hand dug pits where artisanal mining of phosphates is ongoing at Dillingo village. c) Artisanal miner hand picking phosphate nodules at Bang'nawge village. d) Handpicked phosphate nodules ready for sale

4 Results and Discussion

4.1 Phosphate occurrence in Sokoto Basin

Phosphates occur in all sample locations as nodules in shale with or without gypsum that can be easily handpicked occurring either as disseminated nodules at the surface most likely due to reworking of the environments or at some depth in the excavated pits. Phosphate occurrence at Chimmola village was at a roadcut session along the major road with a profile of about 1m overburden of reddish-brown ferrogitized Ironstone overlying about 4m of light-brown Shale intercalated with phosphate nodules which are easily handpicked with a base of about 2m of dark-brown Shale which was almost turning to Marl due to erosion.

Phosphates at Miyal'yako village occur as surface exposure of about 500m by 300m together with Gypsum indicating that the area has been reworked. The area also had a significant amount of mud cracks showing a high level of evaporation while some of the phosphates had been calcified. The logged pit showed about 1.4m reddish-brown Ironstone overburden and about 7m Shale intercalated with clay and Gypsum. Two pits were logged at Gidan Fako with reddish-brown Ironstone overburden of depth ranging between 0.5-0.8m and Shale having intercalation of minute phosphates with a depth of 2-4m while the pit at Gaigawo village was characterized by 0.2m overburden of reddish-brown Ironstone and about 1m of light-brown Shale occurring together with minute phosphate and Gypsum.

Pit logged at Dillingo village had 0.25m overburden of reddish-brown Ironstone and about 2m of shale intercalated with phosphate and Gypsum was absent while Bang'nawge pit had Reddish-brown Ironstone overburden as low as 0.5m followed by Shale intercalated with phosphate. The phosphates are very close to the surface and artisanal mining and sale of phosphate are ongoing in this location (Figure 4).

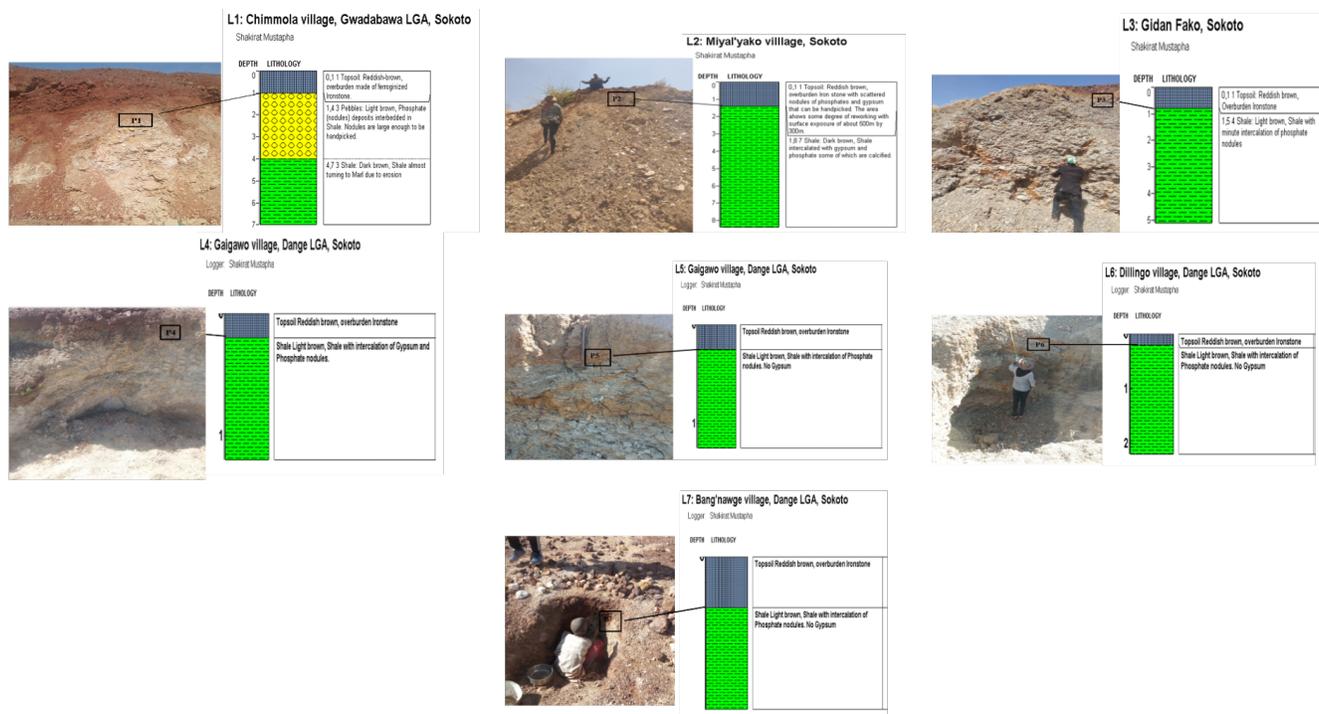


Fig 4. Field description and pit logs of phosphate sample locations within the Sokoto Basin

4.2 Mineralogy of phosphate samples

The mineralogical composition (Table 1 and Figure 5) includes Smectite ranging from 1.78-13.13 %, Quartz 0.44-10.46%, Calcite 42.22-97.89%, Fluorapatite 1.33-79.28%, Goethite 0.92-2.59%, and Kaolinite 1.7-2.39% with only one sample containing Palygoskite of 2.3%. Fluorapatite is the main mineral in the phosphates, while Calcite constitutes the main gangue in samples with high fluorapatite content. This may be an indication that the phosphates were formed through diagenesis as a partial replacement of carbonate muds by apatite⁽¹¹⁾. The presence of clay minerals notably Kaolinite and Palygoskite indicates some level of weathering while the absence of pyrite and low Goethite content indicates that the environment of formation was highly oxidizing⁽¹²⁾.

Table 1. Mineralogical composition of phosphate samples

Location	Lab Label	Smectite	Palygoskite	Quartz	Calcite	Kaolinite	Fluorapatite	Goethite
Chimmola	P1	-	-	0.67	96.95	2.39	-	-
Miyal'yako	P2	13.13	-	5	-	-	79.28	2.59
Gidan Fako	P3	-	-	0.44	97.89	-	1.67	-
Gaigawo	P4	1.78	-	0.47	97.74	-	-	-
Gaigawo	P5	7.89	2.3	8.68	79.8	-	1.33	-
Dillingo	P6	9.03	-	10.46	42.22	1.7	36.6	-
Bang'nawge	P7	2.39	-	4.7	83.59	-	8.41	0.92
	Min	1.78	2.3	0.44	42.22	1.7	1.33	0.92
	Max	13.13	2.3	10.46	97.89	2.39	79.28	2.59
	Mean	6.84	2.3	4.35	83.03	2.05	25.46	1.76
	SD	4.26	0	3.79	19.61	0.35	29.87	0.84

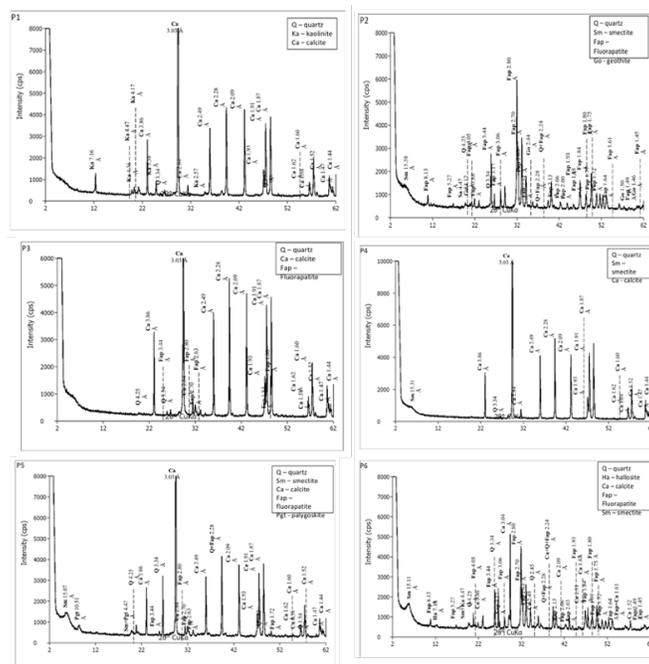


Fig 5. Mineralogical composition of phosphate using X-Ray Diffraction

4.3 Geochemistry of phosphate samples

The results for the major, trace, and rare earth elements analysis are presented in Table 2. The oxides had average concentrations in the descending order of CaO (46.73wt%), P₂O₅ (11.17wt%), SiO₂ (8wt%), Al₂O₃ (2.23wt%), Fe₂O₃T(1.84wt%), MgO (0.57wt%), MnO (0.21wt%), TiO₂ (0.17wt%), Na₂O (0.15wt%) and K₂O (0.12wt%). Trace elements had the highest average concentrations of V (38 μg/g) followed by Zn (30 μg/g), U (27.52 μg/g), Cr and Ni (24 μg/g), Cu (12 μg/g), Pb (8.2 μg/g), and Co (4.7 μg/g).

Concentrations of P₂O₅ were correlated with values of other oxides and trace elements to determine their relationship (Figures 6 and 7). Apatite content of samples is evident in the high P₂O₅ and CaO with a positive correlation which may indicate diagenetic phosphatization where PO₄ displaced CO₂ during precipitation of phosphorites in an oxidizing environment with fluctuating pH leading to the formation of carbonate fluorapatite although higher content of CaO may also indicate the presence of calcite or gypsum. The P₂O₅/CaO trend was similar to those reported by⁽¹³⁾ who worked on the geochemistry of Ogun phosphate deposits indicating a similar environment of deposition. The positive relationship between P₂O₅ and Na₂O may also either be a result of CO₃ being replaced by PO₄ during phosphatization or mild weathering of the phosphorites while a negative correlation with Al₂O₃ might be due to ionic substitution in the apatite lattice under high alkaline conditions⁽¹⁴⁾. K₂O had a negative relationship suggesting the samples may be primary phosphorites and the presence of minute K₂O content outside the apatite crystal lattice⁽¹⁵⁾. The strong relationship of Fe₂O₃T/P₂O₅ may be due to the deposition of Fe₂O₃ and FeO during the process of phosphatization which is also peculiar with primary phosphorites⁽¹⁶⁾ while the negative correlation with SiO₂ reflects a mutual substitution before the final precipitation within the depositional environment. A positive correlation with MnO indicates some level of affinity with P₂O₅ and occurrence within the apatite structure.

A very high positive correlation between P₂O₅ and U may be due to the co-precipitation of phosphorus and Uranium during phosphatization and also further confirm the oxidizing state of the environment⁽¹⁷⁾. A strong positive correlation between V and Cr in phosphorites associated with Ironstones may indicate affinity of Cr with Fe in the Ironstones and they may be absorbed in the ferruginous Iron-oxide and clayey minerals during phosphatization. Cr's positive relationship with the group of elements like V-Ni and Zn indicates natural affinity typical of organic matter while the positive correlation between Ni and Cu supports marine environment sediments^(18,19). Low Uranium content, similar to reports from Ogun phosphate samples, can be interpreted as deposition in a shallow marine environment in an arid region and the negative correlation of U and V indicate no proper substitution of these elements within the apatite lattice.

Table 2. Geochemical composition of the phosphates in the study area (Major oxides in wt% while trace and rare earth elements in $\mu\text{g/g}$)

Location	Miyal'yako	Gidan Fako	Gaigawo	Dillingo	Bang'nawge
Lab Label	P2	P3	P5	P6	P7
SiO ₂	11.95	3.35	15.71	4.25	4.56
Al ₂ O ₃	2.51	1.26	4.36	1.11	1.91
Fe ₂ O ₃ T	1.4	1.67	1.63	1.01	3.5
MnO	0.22	0.27	0.04	0.15	0.37
MgO	0.42	0.33	1.54	0.3	0.24
CaO	44.84	50.89	39.03	50.98	47.9
Na ₂ O	0.19	0.15	0.04	0.11	0.24
K ₂ O	0.17	0.08	0.18	0.06	0.09
TiO ₂	0.3	0.08	0.23	0.1	0.12
P ₂ O ₅	14.42	11.04	0.29	7.8	22.29
Ni	20	30	30	10	30
V	35	29	54	27	45
U	24.6	35.8	2	21.2	54
Cr	20	10	70	10	10
Cu	10	10	10	10	20
Pb	10	3	5	6	14
Co	1	3	2	0.5	17
Zn	15	30	50	15	40

	P ₂ O ₅	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃ T	MnO	MgO	CaO	Na ₂ O	K ₂ O	TiO ₂
P ₂ O ₅	1									
SiO ₂	-0.538	1								
Al ₂ O ₃	-0.513	0.94	1							
Fe ₂ O ₃ T	0.707*	-0.245	-0.001	1						
MnO	0.955*	-0.682	-0.614	0.703	1					
MgO	-0.773	0.838	0.924	-0.199	-0.797	1				
CaO	0.467*	-0.96	-0.995	0.008	0.593	-0.888	1			
Na ₂ O	0.992*	-0.506	-0.516	0.64	0.946	-0.786	0.464	1		
K ₂ O	-0.345	0.964	0.889	-0.131	-0.486	0.715	-0.922	-0.296	1	
TiO ₂	-0.175	0.87	0.694	-0.217	-0.384	0.46	-0.76	-0.11	0.926	1

Fig 6. Correlation matrix of P₂O₅ against other oxides in the phosphate samples
Correlation is significant at **0.01 level, *0.05 level

	P ₂ O ₅	Ni	V	U	Cr	Cu	Pb	Co	Zn
P ₂ O ₅	1								
Ni	0.12*	1							
V	-0.184	0.615*	1						
U	0.936*	0.227	-0.252	1					
Cr	-0.732	0.343	0.785*	-0.78	1				
Cu	0.765*	0.375*	0.345	0.772	-0.3	1			
Pb	0.922*	0.193	0.164	0.781	-0.443	0.86	1		
Co	0.747*	0.487*	0.366*	0.785	-0.275	0.99	0.825	1	
Zn	-0.209	0.816	0.857	-0.091	0.653*	0.363	0	0.438	1

Fig 7. Correlation matrix of P₂O₅ against trace elements in the phosphate sample
Correlation is significant at **0.01 level, *0.05 level

5 Conclusion

The phosphate deposits in the study area occur within the Sokoto Basin, Northwestern Nigeria as nodular intercalations with Shale in the presence/absence of Gypsum at a depth between 0.25 – 7m, are mostly close to the surface and can be easily handpicked and separated from the Shale. Mineralogical and geochemical data were used to better understand the constituents of these samples and ultimately their REEs mineralization potentials.

Their mineralogical composition includes Fluoroapatite, Calcite, Smectite, Quartz, Kaolinite, Goethite, and Palygoskite with the highest concentration of Fluoroapatite at Miyal'yako (79.28%) followed by Dillingo (36.60%) and the lowest at Gaigawo (1.33%). Interpretation of the relationship between P_2O_5 with other major oxides and trace elements revealed that phosphates are the primary type and are typically formed in an oxidizing environment with fluctuating pH in the presence of calcite or gypsum. Low Uranium contents and other similarities with reports from the Ogun phosphates may point to the fact that both deposits were formed in similar depositional.

The high concentrations of fluoroapatites in these two locations out of the seven locations studied are significant as fluoroapatites are known to be rich in REEs and thus a pointer to the possibility of these locations being enriched in REEs. Further studies should be done in these two locations particularly mineral chemistry to ascertain the exact concentrations of their REEs to provide more information for REE exploitation in Nigeria.

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