



Chemical, Geotechnical and Mineralogical Characteristics of a Clay Mineral Deposit in Lokoja, Kogi State, Nigeria

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ABSTRACT

The solid mineral sector provides a viable alternative to expand the economic base of Nigeria which has an abundant clay deposit across the country. The underutilization of clay deposits is in part either due to lack of characterization data for most sites or the absence of it. In this work, the chemical, geotechnical and mineralogical characteristics of the clay mineral deposit at Lokoja, Kogi State were evaluated. The results revealed that the deposit consists of: SiO₂ (21.75 - 38.15%), Al₂O₃ (18.49 - 21.65%), Na₂O (5.50 - 7.70%), Fe₂O₃ (4.43- 7.89%), K₂O (2.3- 5.78%), CaO (0.56-1.01%), MgO (0.50- 1.00%), loss on ignition (7.40-10.00 %), and moisture content (3.40- 4.60%). The mineralogical studies revealed the presence of orthoclase, paracelsian and albite low as the feldspar minerals. The clay minerals detected were Kaolinite, Halloysite 7A, lizardite aluminian, clinochlore IIb, vertumnite, lovdarite and corrensite. Other silicate and siliceous carbonate minerals found were; wollastonite, paraspurrite and alumahydrocalcite. The three layers exhibited medium plasticity, while the middle and bottom layers had refractoriness above 1200°C, and moderate thermal shock resistance, the top layer exhibited refractoriness below 1200°C and low thermal shock resistance.

Keywords: Clay and carbonate minerals, Feldspars, Plasticity, Refractoriness, Thermal shock resistance

INTRODUCTION

Nigeria is the most populous country in Africa with an estimated population of over two hundred million (Yahaya *et al.*, 2020). The high dependence of Nigeria's economy on revenue from oil whose price is highly volatile in the world market coupled with her inability to attain its assigned production quota due to vandalisation of oil infrastructure and other mitigating factors which has negatively affected its economic growth leading to macroeconomic instability and spiralling inflation, there have been calls for the nation to diversify its economy (Eko *et al.*, 2013; Evbuomwan (2016) and Olamide *et al.*, (2019). The solid mineral sector provides a viable alternative to expand the economic base of the nation as Nigeria with proven reserve of several billions of tonnes of clay mineral deposits (RMRDC, 1990). From the socio-economic survey conducted by the National Bureau of Statistics (NBS) on the contribution of solid minerals to employment and poverty alleviation in Nigeria, it was reported that solid mineral if harnessed to its full potential could lead to job creation which will in turn reduce the poverty (Ayodele *et al.*, 2013). Central to their findings was the fact that the exploration of solid mineral could generate employment from; the mining, construction, cement and building industries as well as medium and small-scale business that provide services for these industries.

Clay is a very important solid mineral with wide applications. However, different clay deposit possesses different physical, chemical and mineralogical compositions which ultimately determines its use. An understanding of the chemistry of clay minerals can lead to a wide range of applications which cuts across oil drilling, cement production, drug delivery, bone tissue engineering (BTE), remediation studies, cosmetic industry and so on (Kieling *et al.*, 2022, Logeshwaran *et al.*, 2023, Maksoud *et al.*, 2023). Unfortunately, many of the clay mineral deposits in Nigeria remain unidentified and uncharacterized (Lori *et al.*, 2007). On account of its wide industrial applications, the nature and composition of each clay mineral deposit needs to be evaluated (Onyeobi *et al.*, 2013). The purpose of this work is to evaluate the properties of the clay mineral deposit in Lokoja, Kogi State, Nigeria.

MATERIALS AND METHODS

Sampling collection/preparation

The samples were collected on profile basis in the study site in Lokoja, Kogi State, Nigeria between latitudes N 7° 52.6381' - N7° 52.6444' and longitude E6° 45.5184' - E6° 45.5245'. The profile was determined by colour variation as described by Onyeobi *et al.*, (1994) and a total of three depth {layer(L1); layer 2 (L2) and layer 3 (L3)} were sampled. Fifteen (15) samples were collected per depth across the mapped site with the

aid of a digger and spade. The samples were labelled and air-dried in the laboratory. The physical properties of the clay minerals such as moisture content, loss on ignition, Atterberg limits and linear shrinkage, refractoriness, thermal shock resistance, bulk density tests were carried out in accordance with BS1377 (1990).

Moisture content determination

The moisture content was determined on dry weight basis. The test clay sample (5.0g) was oven dried at a temperature of 105-110°C for three hours and weighed at one hour interval after cooling in a desiccator until a constant weight was obtained. The loss in weight was computed and expressed in terms of percentage (equation 1).

$$\% \text{ Moisture content} = \frac{\text{Loss in weight (g)}}{\text{weight of sample (g)}} \times 100 \quad (1)$$

Loss of ignition (L.I.O)

The clay sample (5.0g) was heated in a muffle furnace at 400C for 4 hours after which it was cooled in desiccators and weighed. The difference in weight gave the loss of ignition. It is expressed in percentage terms (equation 2);

$$\% \text{ Loss of ignition} = \frac{\text{Loss in weight (g)}}{\text{weight of sample (g)}} \times 100 \quad ..(2)$$

ATTERBERG LIMITS DETERMINATION

Liquid Limit

The air-dried clay samples (120.0g) from thoroughly mixed portion of material passing 425 microns sieve were obtained. Distilled water was added to the clay sample in a mixing disc to form uniform paste. A portion of the paste was placed in the cup of liquid limit device and spread into portions with few strokes of spatula, trimmed to a depth of 1cm at the point of maximum thickness. The clay sample in the cup was divided by the firm strokes of the grooving tool along the diameter through the centre line so that clean sharp groove of proper dimensions was formed. The cup was lifted and dropped by turning crack at the rate of 2revolutions/seconds until the two halves of clay sample came in contact with each other for a length of about 1cm by flow only. The number of blows required to cause the groove close for about 1cm was recorded. A representative portion of clay was taken from the cup for moisture content determination. The test was repeated with different moisture contents at least three more times for blows between10-40. A flow curve was obtained by plotting a graph of moisture content (on Y-axis) and number of blows (on X-axis). The moisture content corresponding to 25 drops (blows) was read, which represented the liquid limit.

Plastic Limit (PL) and Plasticity Index (PI)

The sieved clay sample (15.0g) was wet with water until it became plastic. The mixture was rolled on a glass plate with the hand into thread of

about 1.3mm in diameter until the thread showed sign of crumbling. The rolled soil was weighed and placed in an oven for 24hours after which the moisture content was determined. This process was repeated three times and the average %moisture content was determined. The values obtained were recorded as plastic limit to nearest whole number. The plasticity index was then calculated by the difference between and %liquid and the %plastic limits.

Linear shrinkage

The sieved clay sample was wetted by addition of water and then placed in a shrinkage mould of dimension 140mm by 12.5mm and the top smoothed with the aid of a spatula. The mould was placed in an oven set at a temperature range of 105-110°C for 24hours. It was allowed to cool to room temperature after which the length of the oven-dried sample was measured and recorded. The difference in length was recorded (equation 3).

$$\text{Linear shrinkage \%} = \frac{\text{change in length } h}{\text{original length } h} \times 100 \dots(3)$$

Bulk density

The test pieces were prepared and air-dried for 24hours, the clay samples were later oven dried at 110°C for 24 hours, heated to 1100°C, cooled in desiccator and weighed to nearest 0.001g (D), after which the samples were transferred to a beaker filled with water and heated for 30minutes to assist in releasing trapped air. The samples were cooled and soaked weight (W) taken. The samples were then suspended in water using a beaker placed on a balance and suspended weight (S) for each sample was recorded, the bulk density was calculated from equation 4;

$$\text{Bulk Density} = \frac{D}{W-S} \times \rho(\text{g/cm}^3) \dots\dots\dots(4)$$

Where, D = Dried weight,
 W = Soaked weight,
 S = Suspended weight,
 R_w= Density of water

Permeability to air

Test samples were prepared to specification of 5.08cm diameter and 5.08cm height from a standard rammer. The test pieces were air-dried for 24hours and then dried at 110C for 12 hours in an oven. 2000cm³ of water was allowed to pass through the sample from the jar containing water. The time taken for 2000cm³ of water to displace equal volume of air through the test piece was recorded. The pressure difference between the surfaces was measured by a manometer. Permeability was calculated from equation 5;

$$P_A = \frac{V \times h}{A \times p \times t} \dots\dots\dots(5)$$

Where P_A = Permeability number

V = Volume of air

H = Height of specimen

A = Cross sectional area of specimen

P = Pressure of air in cm of water

t = Time in minutes

Apparent porosity

Disk samples were prepared, and air dried for 24 hours. The samples were then oven dried at 110°C, the pieces were fired at 1100°C, cooled and then transferred into desiccator and weighed to the nearest 0.001g (D). The samples were transferred into a 250ml beaker in an empty vacuum desiccator; water was then introduced into the beaker until the test places were completely immersed. The samples were allowed to soak in boiled water for 30 minutes being agitated from time to time to assist in releasing trapped air bubbles. The sample was transferred into an empty desiccator to cool. The soaked weight (W) was taken. The sample was then weighed suspended in water using a beaker placed on a digital weighing balance and this gave suspended weight (S). The apparent porosity was calculated using equation 6;

$$\text{Apparent porosity} = \frac{W-D}{W-S} \times 100 \dots \dots \dots (6)$$

Where W = Soaked weight,

D = Dried weight,

S = Suspended weight

Chemical assay

The chemical compositions of the mineral deposit were determined on dry samples obtained by taking some quantity of the test sample, heated to 105-110°C for one hour to remove moisture, cooled in a desiccator and thereafter kept in a sealed container. The test sample (1.0g) was weighed into a beaker and digested in Aqua-regia. The determination of the chemical components of the samples were carried out using Atomic Absorption Spectrophotometer (Bulk Scientific 210VGP) and flame Photometer (model Sherwood 410) as described by (Ibor and Okunkpolor, 2020) and the total silica content was determined by the standard method prescribed by American Society for Testing and Materials as described by (Ibor and Okolo, 2010).

Mineralogical analysis

The samples were pulverized to fine particles ($\leq 2\mu\text{m}$) and were subjected to X-ray diffraction using GBC Enhanced Mini Material Analyzer (EMMA) X-ray Diffractometer with Cu, $K\alpha$ radiation source. The generator operating conditions were 40KV and 5SMA. The Diffractograms obtained were compared with the standard X-ray powder diffraction file published by the Joint Committee on the Powder Diffraction

Standards JCPDS (1980) for the identification of the different mineral species and Pei-Yuan (1977).

RESULTS AND DISCUSSION

The results of the chemical composition of Lokoja clay are as presented in Table 1. The silica (SiO_2) content varied from 21.75 -38.15% and was found to have increased from top to bottom. The alumina (Al_2O_3) values ranged between 18.49-21.65% with the highest values being recorded at the bottom layer. The sodium oxide (Na_2O) content ranged from 5.50-7.70%, while potassium oxide (K_2O) and iron (iii) oxide (Fe_2O_3) was between 2.38 - 5.78% and 4.43 -7.89% respectively. It was observed that Na_2O , K_2O and Fe_2O_3 had their highest concentration at L2 (second layer). The results of the other metallic oxides had their ranges as follows: CaO (0.56 - 1.01%), MgO (0.50 - 1.00%), TiO_2 (0.05- 0.18%), NiO (0.07- 0.09%), MnO_2 (0.01%) P_2O_5 (0.32 - 0.38%). The moisture content values were (3.40 - 4.60%), and loss on ignition (L.O.I), (7.40 - 10.00%). It was also observed that the values of the moisture content and loss on ignition were highest at the bottom layer. The observation corresponds with similar values of alumina and silica. This shows the presence of higher amount of clay minerals which contain both bound water molecules and hydroxyl groups on one hand and the carbonate minerals on the other hand. The physical properties of the Lokoja clay mineral deposit are as presented in Table 2. The values of the liquid limit were (36.00-50.00%), plastic limit (15.00- 17.00%), plasticity index (21.00 - 33.00%), linear shrinkage (10-11.00%), bulk density (2.26-2.45g/cm³), apparent porosity (20.00-29.00), permeability in air (74.00-96.00), thermal shock resistance (TSR) (11.00-28.00 cycles). Whereas the top layer had refractoriness below 1200°C, the middle and bottom layers had refractoriness above 1200°C. The mineralogical analysis is as presented in Table 3. In the top layer (L1) the silicate minerals present were; orthoclase (KAlSi_3O_8), albite low ($\text{NaAlSi}_3\text{O}_8$), paracelsian ($\text{BeAl}_2\text{Si}_2\text{O}_8$), quartz (SiO_2) and malladrite (Na_2SiF_6). The clay minerals present were; Kaolinite ($\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$), Lizardite aluminian 6(3)T, [(MgAl)₃(SiAl)₂O₅(OH)₄]halloysite ($\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4 \cdot 2\text{H}_2\text{O}$) and clinocllore Iib [(Mg,Al,Fe)₆(Si,Al)₄O₁₀(OH)₈].

The clay minerals present in the second layer (L2) were Vertumnite ($\text{Ca}_4\text{Al}_4\text{Si}_4\text{O}_6(\text{OH})_{24} \cdot 2\text{H}_2\text{O}$), Lovdarite [(NaK)₄Be₂Si₆O₁₆·4H₂O], Sonolite Zincian [(Mn, Zn)₉Si₄O₁₆(OH)₂], Pentagonite [Ca(VO)Si₄O₁₀·4H₂O]. It had the following silicate minerals, Wollastonite (CaSiO_3), orthoclase (KAlSi_3O_8), Paraspurrite [$\text{Ca}_5(\text{SiO}_4)_2\text{CO}_3$] and Quartz (SiO_2). The non-silicate minerals present were Weberite ($\text{Na}_2\text{MgAlF}_7$), Heterogenite ($\text{CaO} \cdot \text{OH}$) and Orelite ($\text{Ni}_5\text{-AS}_2$). The iron mineral present was Lepidocrocite ($\text{FeO} \cdot \text{OH}$). The third layer (L3) had

the following clay minerals corrensite [(MgFe)₉(SiAl)₈O₂₀OH.xH₂O], clinochloreIIB [(Mg,Al,Fe)₆(SiAl)₄O₁₀(OH)₈], halloysite 7A (Al₂Si₂O₅(OH)₄) and palygorskite [MgAlSi₄O₁₀(OH).4H₂O]. The silicate minerals present were wollastonite (CaSiO₃), albite low

(NaAlSi₃O₈), orthoclase (KA1Si₃O₈) and quartz (SiO₂). The carbonate minerals; alumahydrocalcite [CaAl₂(CO₃)₂(OH)₄.3H₂O] and siderite manganooan [(FeMnZn)CO₃] were present. The iron mineral goethite (FeO(OH)) was present and the Nickel mineral Nickeline (NiAs₅) was also present.

Table 1: Chemical composition of the clay mineral deposit in Lokoja

Depth	L1	L2	L3
Moisture content %	3.40±0.30	4.60±0.20	4.20±0.40
Loss of Ignition (L.O.I)%	7.40±0.40	7.40±0.20	10.00±0.60
SiO ₂ %	21.70±0.65	30.05±0.75	38.15±0.25
Al ₂ O ₃ %	19.54±1.31	18.49±0.49	21.65±0.67
Na ₂ O%	5.50±0.50	7.70±0.20	7.15±0.10
K ₂ O%	2.38±0.05	5.78±0.31	3.97±0.15
Fe ₂ O ₃ %	4.43±0.58	7.89±0.16	5.28±0.29
CaO%	0.56±0.07	0.85±0.01	1.01±0.03
MgO%	0.50±0.02	0.76±0.03	1.00±0.02
P ₂ O ₅ %	0.32±0.01	0.38±0.01	0.38±0.01
CuO%	0.10±0.01	0.15±0.01	0.21±0.01
TiO ₂ %	0.18±0.01	0.06±0.01	0.05±0.01
MnO ₂ %	0.20±0.01	0.30±0.02	0.23±0.02

L1= layer 1, L2 = layer 2, L3 = layer 3

Table 2: Physical properties of the clay mineral deposit in Lokoja

Depth	L1	L2	L3
Liquid limit (%)	36.00	38.00	50.00
Plastic Limit (%)	15.00	16.00	17.00
Plastic Index (%)	21.00	22.00	33.00
Linear Shrinkage (%)	10.00	10.00	11.00
Bulk Density g/cm ³	2.40	2.37	2.26
Apparent Porosity	29.00	20.00	24.00
Permeability in Air	74.00	96.00	94.00
Refractoriness (°C)	<1200 °C	>1200 °C	>1200 °C
Thermal Shock Resistance (TSR)	11	28	23
Colour	cream	Grey	light brown

L1= layer 1, L2=layer 2, L3=layer 3

Table 3: Mineralogical composition of the clay mineral deposit in Lokoja

L1	L2	L3
Orthoclase	Vertumnite	Corrensite
Albite, low	Lovdarite	Clinochlore IIB
Paraclesian	Sonolite zincian	Halloysite 7A
Halloysite	Pentagonite	Palygorskite
Quartz	Wollastonite	Wollastonite
Malladrite	Orthoclase	Albite, low
Kaolinite	Parasporrite	Orthoclase
Lizarditealuminian 6(3) T	Quartz	Alumahydrocalcite
Halloysite	Weberite	Siderite manganooan
Clinochlore IIB	Heterogenite	Goethite
	Lepidocrocite	

L1= layer 1, L2 = layer 2, L3 = layer 3

From the results obtained, the percentage of silica in comparison to alumina showed that the deposit falls within the aluminosilicate mineral group (Moore and Reynolds, 1997). The predominant silicate minerals were found to be orthoclase and albite low, both are feldspar minerals known as precursor to the kaolinite

mineral formation (Hassan, 2014; Omang *et al.*, 2019). The presence of kaolinite, halloysite and quartz in the same top layer (L1) indicates the progressive weathering condition prevalent in the layer. This is supported by other workers (Charles and Diane 2008). Although present in small quantity another rare feldspar mineral paraclesian

was detected (Moro *et al.*, 2001). The iron and magnesium oxide appeared to exist as the clay mineral clinocllore IIb a magnesium rich end member of the chlorite group of clay mineral (Aparico *et al.*, 2006). The presence of malladrite, and hexafluorosilicate mineral of diver's industrial application was detected (Chantilly 2011). Lizardite aluminian which is the most common serpentine specie was also detected. Its properties had earlier been described (Howie, 2005). Inlayer 2 (L2), vertumnite clay mineral a calcium rich phyllosilicate whose properties and importance have been described (Rinaldi *et al.*, 1990, Jackson *et al.*, 2014) was detected. Another clay mineral of importance detected was sonolite zincian (Hawthorne *et al.*, 1993, Sangster, 2003, Schneider *et al.*, 2008, Simandi *et al.*, 2011). The other silicate minerals present which obviously account for the increase in the silicate and calcium content which are associated with carbonate rocks are wollastonite and alumahydrocalcite whose properties and industrial applications have been reported (Deer *et al.*, 1997; Stachowicz *et al.*, 2015) and paraspurite (Rabiee *et al.*, 2019) as well as heterogenite (Sophie *et al.*, 2015). The non-silicate mineral weberite whose characteristics and applications have been described (Pauly and Peterson, 1981, Caramanian *et al.*, 2001) was also detected. In the third layer (L3), Na-feldspar albite low was the major silicate mineral. The clay minerals present in this layer were halloysite 7A possible product of weathering from kaolinite alteration (Omang *et al.*, 2019). Amazing variety of the clay minerals rich in magnesium were detected and these were corrensite, a chlorite mineral which is usually formed in diverse sedimentary environments containing among others carbonate rocks (Shan, 1990), clinocllore IIb, also a chlorite type of clay mineral and palygorskite (Aparico *et al.*, 2006). It should be noted that the calcium minerals were predominantly wollastonite (CaSiO₃). The presence of heterogenite, paraspurite and alumahydrocalcite in L2 and L3 indicates the presence of carbonate environment in which weathering of the main silicate mineral (Wollastonite) may have taken place (Stachonowicz *et al.*, 2015; Wood *et al.*, 2023) and the siderite manganoan presence of these carbonate minerals in L3 was corroborated by the high value of loss of ignition recorded in the bottom layer. The iron mineral lepidocrocite (γ -Fe₂O₃) and goethite (FeO.OH) were present L2 and L3 respectively and these no doubt contributed to the colours of the layers (Klein 1993). Silica was present as α -quartz in all the layers sampled thus indicating the weathering of the various feldspar and clay minerals (Charles and Diane, 2008). The liquid limit of the clay minerals was (36 -50%). This indicates high water retention capacity while the plastic index showed that they belong to the medium plastic range as described by other workers (Seed *et al.*, 2003 and Moreno-Maroto *et al.*, 2021). The bulk density decreased from top to

bottom. The third layer (L3) had the highest values of liquid limit, plastic limit, plastic index, linear shrinkage, permeability in air, the top layer had the least plastic index and permeability in air; it however, had the highest value for apparent porosity. The second (L2) and third (L3) layers had refractoriness above 1200°C and high thermal shock resistance whereas the top layer had refractoriness below 1200°C and low thermal shock resistance.

CONCLUSION

The study revealed the presence of different kinds of plagioclase rock forming feldspar minerals and their weathering products. There were different types of clay and carbonate minerals present in the different layers while the top layer (L1) showed refractoriness below 1200°C and poor thermal shock resistance, the other layers (L2 and L3) had refractoriness above 1200°C and high thermal shock resistance.

Declaration of Interest Statement

The authors declare no conflict of interest.

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