

GEOCHEMICAL INVESTIGATION OF CLAY MINERALS IN MARTE, BORNO STATE, NIGERIA.

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Abstract

Clay deposit collected from various locations in Marte (Northern Borno), were studied to determine their physical and chemical characteristics in order to evaluate their suitability for industrial uses. Major and trace element analyses were carried out on clay samples using Inductively Couple Plasma-Optical Emission Spectrometry (ICP-OES) and X-Ray Fluorescence (XRF). The result of the chemical analysis of the ten (10) samples collected showed significant amounts of SiO₂ and Al₂O₃. Silica content ranges from 51.48 to 62.44 % while alumina varies from 12.49 to 19.00 %. The calcium oxide ranges from 1.17 to 3.39 %, Na₂O ranges from 1.1 to 8.61 %, K₂O from 1.54 to 3.66 %, MgO varies from 0.04 to 0.14 %, Fe₂O₃ varies from 0.3 to 2.7 % and MnO ranges from 0.01 to 1.03 %. The result showed that the clays are mainly smectite with quartz and feldspar as the main non-clay minerals. Generally, the geochemical results of the samples do not meet the standard for industrial utilization when compared to the Industrial specifications. However, for industrial utilization, some of the clay samples may be used after necessary beneficiations.

Keywords: Geochemistry, Clay Minerals, Beneficiation, Chad Formation

Introduction

The term clay is generally used to refer to either fine grained earth material with particle size of less than two micron (<2µm) or group of hydrous aluminum silicate minerals which are characterized by sheet silicate structures of composite layers stacked along the c-axis (Grim, 1968). Abba *et al.*, (2006) classified clays as 1:1 or 2:1 layered based on their structure (tetrahedral or octahedral). They are described as 1:1, if they involve units attaching tetrahedral and octahedral sheets, example is kaolinite. The 2:1 clay layer consists of an octahedral sheet sandwiched between two tetrahedral sheets such as illite, smectite, attapulgite and chlorite. Clays are also described or classified as primary and secondary clays based on mode of formation (Abba *et al.*, 2006). Primary clays are clays that are formed in situ, are pure and have not been contaminated by any oxide or form of foreign body and therefore their kaolinite structures not been disturbed. The colours of primary clays are usually white and not so plastic because of their large particle size structures. These clays are known to have a formula of Al₂O₃.2SiO₂.2H₂O and are often referred to as kaolin or china clay. In Nigeria, local occurrences include; Kuba kaolin and Barikin Ladi kaolin in Plateau State, Kankara kaolin in Kastina State. Thus, it is to be noted that this kaolin has different percentages of various minerals, oxides and are likely to have some slight differences in appearance and characteristics (Abba *et al.*, 2006). The secondary or sedimentary clays are clays that have been transported from their places of formation by natural forces such as wind, glacier and water. They are further deposited in strata at lower areas. In these processes of movement, they come in contact with oxides and foreign matter that gets them contaminated. This results in loss of some of their original primary qualities such as colour which changes from white to blue, black, brown or red. In addition, their particle sizes become extremely fine, laminator in structure and are very plastic. These properties have been observed in the area and hence depict that the Marte clay is secondary in origin. In addition to above, secondary clays consist of disordered kaolinite with

one or other clay minerals or oxides such as montmorillonite, halloysite, iron oxide, titanium oxide (anatase). Examples of secondary clays in existence are: ball clay, high alumina clay, terracotta clay, flint clay, adobe clay, gumbo clay and shale clay.

Clays as minerals are generally common and found in most geological settings most especially in fine grained sedimentary rocks such as shale, mudstone and siltstone and in fine grained metamorphic slate and phyllite. Thus, clays are named based on their locational origins, mode of formation, structure, chemical composition and usage (Cuadros and Altaner, 1998). Cuadros and Altaner, (1998) grouped the principal clay minerals into five on the basis of their structures and chemical composition as kaolinite, illites, bentonite (smectite), vermiculites and mixed-layer clay. Several authors have reported the occurrence of clay mineral deposits in some states within the country, most especially in the northeast and southeastern Nigeria. The occurrences of the clay in Northeastern Nigeria are found to occur within the Gongila Formation, the Fika Shale, the Gombe Formation, the Kerri-Kerri Formation and the Chad Formation (Table 1). Counts *et al.*, (2000) analyzed the applicability of clays in Northern Nigeria as refractory materials and glaze.

Table 1: Stratigraphic succession of the Chad Basin in Nigeria

Age	Formation	Lithology	Thickness (m) From borehole data-Carter <i>et al.</i> , (1963)	Thickness (m) from seismic data Avbovbo <i>et al.</i> , (1986)	Thickness (m) from well log Olugbemi <i>et al.</i> , (1997)	Depositional Environment
Pliocene	Chad Formation	Clay, Sand unconformity	Not investigated	800	400	Continental
Pleistocene	Keri-Keri Formation	Coarse Sandstone, Claystone	Not investigated	-----	130	Continental
Maestrichtian	Gombe Sandstone	Shale, Sandstone Siltstone unconformity	Not investigated	0-1000	315	Deltaic, Estuarine
Turonian- Santonian	Fika-Shale	Blue-black- shale	840-1453	0-900	430	Marine
Turonian	Gongila Formation	Sandstone, Shale	162-420	0-800	420	Marine, Estuarine
Cenomanian	Bima Sandstone	Sandstone	716-850	2000	3050	Continental
Pre-Cambrian Basement Crystals						

Samples were subjected to empirical testing for determination of their refractory tendencies and gloss temperatures and the results indicate that the Alkalari clay demonstrated strong refractory tendencies while the Marte clay cracks easily as it lost significant fire strength during firing and cooling, owing to its colloidal nature. Maigari *et al.*, (2005) carried out the mineralogical and geochemical analyses using X-Ray Diffractometry (XRD) and X-Ray Fluorescence (XRF) method on claystone outcrops samples from Gombe Abba and Dukku (Gombe State), Alkalari and Kirfi (Bauchi State), that are within the Kerri-Kerri Formation with the aim of assessing

their economic viability. The diffractograms obtained show that kaolinite is the dominant clay mineral while vermiculite, chlorite, illite and montmorillonite are present only in trace amounts. Quartz and feldspar are the dominant non-clay minerals with subordinate amounts of mica and goethite. Maigari *et al.*, (2005) further observed that due to the high kaolinite content, these clays can serve as basic raw material for pharmaceutical, paints, paper and ceramic industries after beneficiation. However, the average chemical composition of all the deposits showed significant amounts of SiO₂ and Al₂O₃ (approximately 54.07 and 29.80% respectively) while Fe₂O₃, TiO₂, MnO, MgO, CaO, K₂O, Na₂O and P₂O₅ concentrations are relatively low and ranges from 0.01 % to 3.92 %. Bentonites in Borno State are found to be more of Calcium-montmorillonite type, formed by the deposition of transported fine marine and non-marine materials (Abba *et al.*, 2006). The chemical compositions indicate 8.5 molar % Na, 38.4 molar % Ca with very low viscosities, low swelling capacities (L 10 %) and moisture content between 7-16 % (Abaa *et al.*, 2006). This implies that the bentonite must be beneficiated before any satisfactory industrial application. Lori *et al.*, (2007) investigated the mineralogy of clay from the Kankara area in Katsina State, Nigeria by X-Ray diffraction and observed that kaolinite is the dominant mineral phase with minor amounts of quartz, goethite and muscovite. The diffraction pattern of the glycolated clay samples did not indicate the presence of mixed layer clay or swelling clay (bentonite). Shettima, *et al.*, (2017) evaluated the mudstone of the Bama Ridge Complex that represents the upper part of the Chad Formation in terms of its mineralogy and major oxide concentrations using XRD analysis and indicates the occurrence of quartz, anatase, plagioclase, gypsum, k-feldspars, smectite and kaolinite minerals.

Records show that there are five succeeding stratigraphic sedimentary units overlying the Bima Sandstone in the Chad Basin as reported by earlier workers. These consist of the Gongila Formation, the Fika Shale, the Gombe Formation, the Kerri-Kerri Formation and the Chad Formation. The Chad Formation is the most critical to this study as the Marte clays are associated with it. The Chad Formation is the youngest Formation in the Chad Basin with a variable sequence that include Quaternary sediments of lacustrine origin underlying the surface deposits over a vast area of about 15,000 km in Borno, Yobe, Bauchi and Jigawa States (Matheis, 1986) (Figure 1).

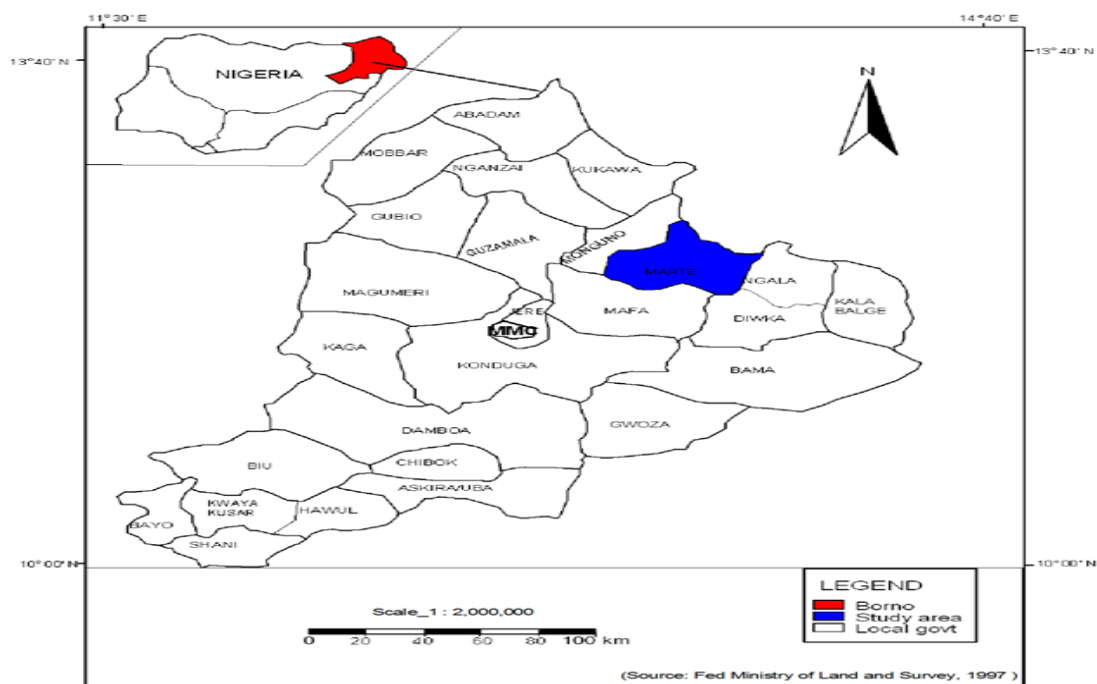


Figure 1: Location map of Marte area

The Chad Formation consists mainly of coarse fluvial sands and thick fluvial lacustrine clays with diatomite (Barber and Jones, 1960). The sand is uncemented with angular and sub angular quartz grains. It is fine to coarse grained and of variable colours from brownish-yellow to whitish-grey. The clay is massive and gritty in texture with variable colours from brownish-grey to dark-grey (Barber and Jones, 1960). The Chad Formation is basically an argillaceous deposit with three well defined arenaceous horizons that provide three main aquifers (upper, middle and lower), the middle and lower being confined, while the upper is semi-confined (Barber and Jones, 1960). The area is underlain by clay, siltstone and sand deposits (Geological Survey of Nigeria, 1982). Field study on geological sampling shows that the area is underlain by sand to the northeastern part (Figure 2) while the Northwestern part and some central portions of the study area are also underlain by clay and sand.

The southern parts are mostly sandy infringing into the central portion of the study area where the samples are dominantly clayey (Figure 3).

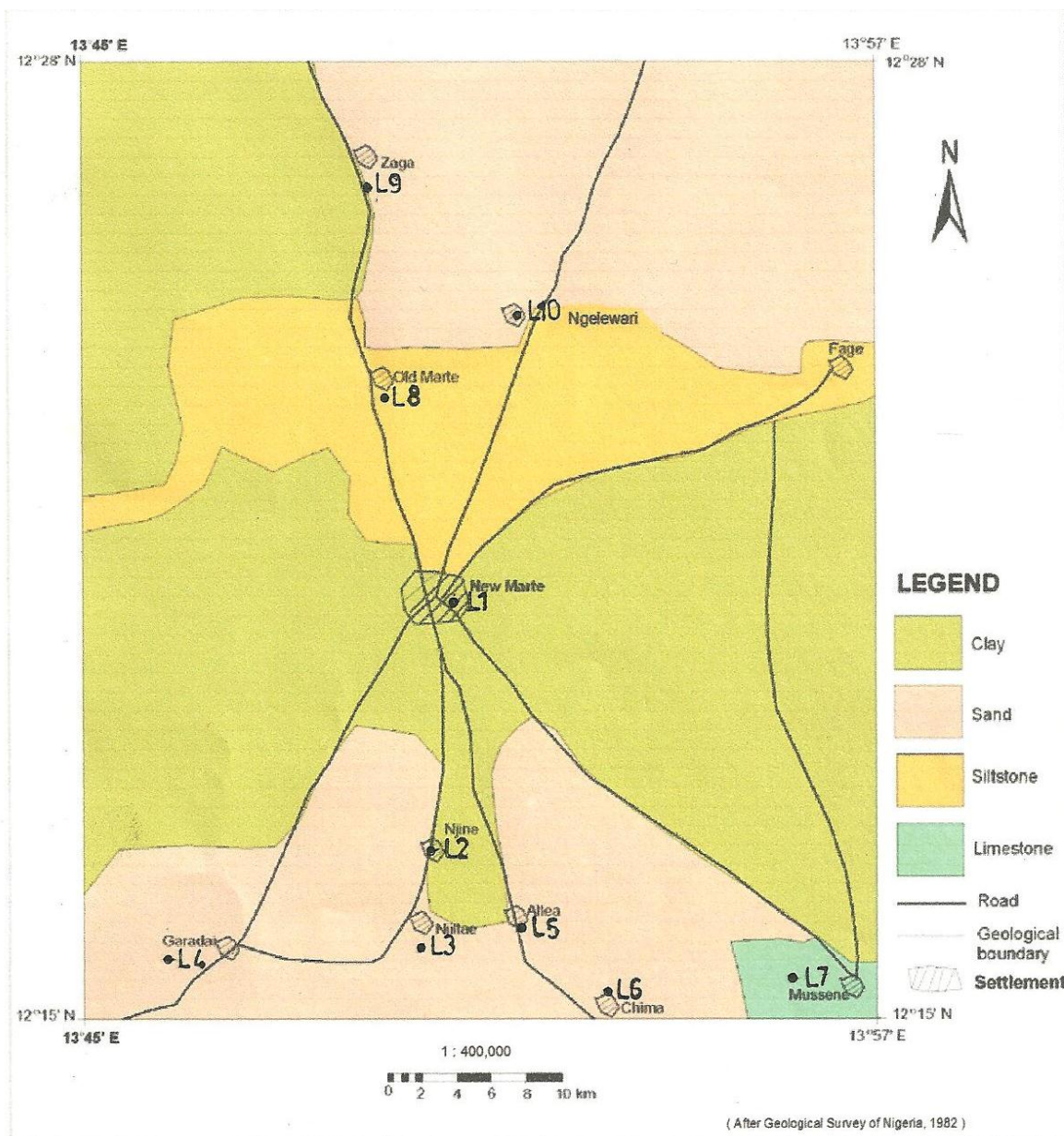


Figure 2: Geological map of Marte area (Adams, 2014)



Figure 3: Clayey flat land at Old Marte

Methodology

Description of the study area

The study area is within Borno State and it is located within sheet 68 Marte NE of the Federal Surveys of Nigeria on a scale of 1:50:000 (Fig.1). The location falls within latitudes $12^{\circ} 15' N$ to $12^{\circ} 28' N$ and longitudes $13^{\circ} 45' E$ to $13^{\circ} 57' E$ (Fig.2).

Materials and Methods

Equipment and materials used for the analyses are inductively couple plasma-optical emission spectrometry (ICP-OES), X-Ray fluorescence (XRF), hot plate, carbolite furnace while the laboratory wares or materials are volumetric flask, funnel, filter paper, porcelain crucible and wash bottles among others. Major and trace element analyses were carried out for clay samples using Inductively Couple Plasma- Optical Emission Spectrometry (ICP-OES) and X-Ray Fluorescence (XRF). With the ICP-OES (Optimal 2000DV), the elements analysed were K, Al, Ca Mg, Fe, Mn, Ti, Cr, Co, Ni and P. The samples were at first ashed and 0.2 g of the sample were digested using concentrated Nitric acid (HNO_3) and concentrated Hydrochloric acid (HCL). The samples were then introduced into the ICP-OES as liquid medium and nebulized as aerosol. The aerosol is dissolved, vaporized and atomized, then excited and ionized in order to obtain characteristic atomic radiation from where the elements and their concentration were recorded. In the XRF analysis, SiO_2 and Al_2O_3 were determined. Five grammes (5g) of the pulverized samples were introduced into the X-Ray chamber of the minimate (Pan Analytical) XRF machine. The machine was calibrated and run, having the results presented in the “result window” of the attached computer in percentage oxide (% oxide). The loss on ignition (LOI) was determined using Carbolite furnace. One gramme (1g) of the sieved sample of clay was transferred to crucibles and placed into a furnace that was set to $1000^{\circ} C$ for about one and a half hours. The samples were left in the furnace to cool and reweighed to determine the loss on ignition.

Results and Discussion

The physical characteristics of the Marte clay at New Marte Table 2 show brown coloration with ferric oxide contamination and traces of organic materials. At Chima, Njine, Mussene and Zaga, the samples appear to be very dark in colour locally associated with carbonaceous materials and few shell fragments. The clays are composed of sandy materials, traces of ferric oxide and carbonaceous matter at Njiltae, Allea and Garadai while at Ngelewari and Old Marte the clays appears with silty texture and commonly grayish in colour.

Table 2: Physical Characteristics of Marte Clay (Adams, 2014)

Location	Description
New Marte (L1)	Brownish containing some organic matter with ferric oxide contamination
Chima (L2)	Dark in colour with traces of organic matter
Njiltae (L3)	Very sandy with slight traces of ferric oxide, brownish to reddish in colour
Njine (L4)	Very dark coloration with slight traces of sand, organic matter and ferric oxide
Allea (L5)	Presence of organic matter and ferric oxide. Brownish in colour very sandy
Mussene (L6)	Dark in colour with slight contamination of carbonaceous material and slight traces amount of shell (limestone).
Ngelewari (L7)	Silty in texture with grayish colour
Garadai (L8)	Very sandy with traces of carbonaceous materials and greyish in colour
Old Marte (L9)	Light brown, very silty with slight traces of mud
Zaga (L10)	Very dark in colour and sandy

The results of geochemical investigations presented in Table 3 indicate that silica (SiO_2) content varies from 51.48 to 62.44 % with an average of 55.82 %. Alumina varies from 12.49 to 19.00 % having an average of 15.2 %. The lime (CaO) content varies between 1.17 and 3.39 % with an average of 2.01 %. Soda (Na_2O) content varies significantly between 1.10 and 8.61 %, having an average of 3.63 %. Potash (K_2O) content ranges from 1.54 to 3.66% with an average of 2.60 %. Ferric oxide varies from 0.30 to 2.70 %, averaging 1.63%. The oxides of Magnesium and Manganese (MgO and MnO) occur in subordinate amounts with the MgO varying between 0.04 to 0.14 % averaging 0.1 % while MnO content on the other hand ranges from 0.002 to 1.03 % with an average of 0.13 %. Titanium oxide (TiO_2) ranges from 0.001 to 0.02 % with an average of 0.01 %. Phosphorous pentoxide (P_2O_5) varies from 0.06 to 2.02 % with an average of 0.79 %. Cr, Ni, and Co, occurs in trace amounts in all the analyzed samples, this is because they are merely adsorbed by the Clay particles. Loss on ignition ranges from 6.32 to 18.95% with an average of 12.61%.

Table 3: Oxide Composition (%) of Marte Clays Samples (Adams,2014)

Location	SiO ₂	Al ₂ O ₃	CaO	Na ₂ O	K ₂ O	MgO	Fe ₂ O ₃	MnO	TiO ₂	P ₂ O ₅	H ₂ O	Cr	Co	Ni	LOI (%)
New Marte	52.62	14.48	2.28	5.21	2.47	0.08	1.74	0.03	0.02	2.02	7.21	0.007	0.0007	0.013	18.95
Chima	53.77	13.43	1.17	4.59	1.54	0.06	0.82	0.01	0.01	0.95	6.60	0.003	0.0003	0.002	16.81
Njiltae	51.48	13.06	1.70	6.52	3.66	0.11	1.45	0.01	0.002	0.52	5.11	0.002	0.0002	0.001	16.43
Njine	54.42	12.49	1.83	2.75	3.10	0.11	1.64	0.03	0.01	1.06	7.50	0.003	0.004	0.002	11.00
Allea	62.44	19.00	1.55	1.58	2.51	0.08	1.73	0.002	0.02	0.70	4.11	0.004	0.0005	0.002	6.32
Mussene	52.42	17.21	2.38	1.50	2.78	0.10	2.70	0.03	0.002	0.77	8.36	0.004	0.0004	0.002	12.41
Ngelewa	61.40	16.80	2.20	2.47	1.69	0.11	2.15	0.04	0.01	0.63	4.58	0.003	0.0001	0.001	7.30
Garadai	58.61	17.42	1.89	1.10	1.85	0.04	0.30	0.01	0.01	0.79	4.01	0.005	0.0007	0.002	14.20
ldMarte	55.20	14.30	1.69	2.00	3.29	0.14	2.64	0.06	0.001	0.06	9.09	0.003	0.0005	0.003	13.30
Zaga	55.80	13.42	3.39	8.61	3.10	0.12	1.13	1.03	0.01	0.40	4.00	0.001	0.0001	0.001	9.40
Total	558.16	151.61	20.08	36.33	25.99	0.95	16.30	1.25	0.095	7.90	60.57	0.035	0.008	0.017	126.12
Average	55.82	15.20	2.01	3.63	2.60	0.10	1.63	0.13	0.01	0.79	6.06	0.004	0.001	0.002	12.61

NOTE: Silica and Alumina were determined by XRF while others by ICP-OES.

Table 4: Chemical Composition of Marte Clays Compared with Specification of some Industrial Clays (Adams, 2014).

Oxides (%)	New Marte	Chima	Njiltae	Njine	Allea	Mussene	Ngelewa	Garadai	Old Marte	Zaga
SiO ₂	52.62	53.77	51.48	54.42	62.44	52.42	61.40	58.61	55.20	55.80
Al ₂ O ₃	14.48	13.48	18.06	12.49	19.00	17.21	16.80	17.42	14.30	13.42
CaO	2.28	1.17	1.70	1.83	1.55	2.38	2.20	1.89	1.69	3.39
Na ₂ O	5.21	4.59	6.52	2.75	1.58	1.50	2.47	1.10	2.00	8.61
K ₂ O	2.47	1.54	3.66	3.10	2.51	2.78	1.69	1.85	3.29	3.10
MgO	0.08	0.06	0.11	0.11	0.08	0.10	0.11	0.04	0.14	0.12
Fe ₂ O ₃	1.74	0.82	1.45	1.64	1.73	2.70	2.15	0.30	2.64	1.13
MnO	0.03	0.01	0.01	0.03	0.002	0.03	0.04	0.01	0.06	1.03
TiO ₂	0.02	0.01	0.002	0.01	0.02	0.002	0.01	0.01	0.001	0.01
Cr	0.007	0.003	0.002	0.003	0.004	0.004	0.003	0.005	0.003	0.001
Co	0.0007	0.0003	0.0002	0.004	0.001	0.004	0.0001	0.0007	0.0005	0.0001
Ni	0.0013	0.002	0.001	0.002	0.002	0.002	0.001	0.002	0.003	0.001
P ₂ O ₅	2.02	0.95	0.52	1.06	0.70	0.77	0.63	0.79	0.06	0.40
H ₂ O	7.21	6.60	5.11	7.50	4.11	8.36	4.58	4.01	9.09	4.00
LOI	18.95	16.81	16.43	11.00	6.32	12.41	7.30	14.20	13.30	9.40

Table 4: Comparison of the Chemical Composition of Marte Clays with Specification of that of some Industries (Adams, 2014) Cont'd

Oxides (wt%)	Refractory bricks Shackelford (2008)	Plastic Fire Clay (Hubers, (1985)	Fuller Earth as Decolorising Agent for Vegetable Oil Ibemesi and Achife, (1990)	Drilling Mud Okogbue and Ene, (2008)
SiO ₂	51-70	57.67	52.26	58-64
Al ₂ O ₃	25-44	24.0	14.33	18-21
CaO	0.1-0.2	0.70	3.03	0.1-1.0
Na ₂ O	0.8-3.5	0.20	0.40	1.5-2.7
K ₂ O	-----	0.50	0.78	0.2-0.4
MgO	0.20-0.70	0.30	-----	2.3-3.2
Fe ₂ O ₃	0.50-2.4	3.23	3.64	2.5-2.8
MnO	-----	-----	-----	0.1-0.2
TiO ₂	1.0-2.8	-----	-----	-----
Cr ₂	-----	-----	-----	-----
Co	-----	-----	-----	-----
Ni	-----	-----	-----	-----
P ₂ O ₅	-----	-----	-----	-----
H ₂ O	-----	10.50	-----	-----
LOI	-----	-----	-----	-----

Generally the geochemical results of the samples do not meet the standard for industrial utilization when compared to the Industrial specifications as indicated in Table 4. However, for industrial utilization some of the clay samples may be used after necessary beneficiations.

For Refractory Bricks: All the samples can be used for refractory bricks after beneficiation being that they meet the Silica content requirement of 51-70 % as specified (Shackelford, 2008). But the alumina content (12.49 -19.00 %) are grossly lower than 25 to 44 % required for refractory bricks (Shackelford, 2008). Thus, the alumina content need to be improved by adding more alumina in form of flux. Also, calcium oxide and potassium oxide content of the samples are higher, ranging between 1.17 to 3.39 % and 1.54 to 3.66 % compared with the standard range of 0.1-0.2 % and 0.0 % respectively. However, the Magnesium oxide and Titanium oxide contents of the clays are lower than the standard values. The MgO content range from 0.04 % - 0.14 % while the standard required is 0.20-0.70 %, TiO₂ value of the samples vary from 0.001 % -0.020 % as compared to the standard range of 1.0-2.8 %. The sodium oxide contents gave mixed values of within range and above, 1.10 % -8.61 % compared with the standard specification of 0.8-3.5 %. Ferric oxide is more or less within the standard range of 0.5 %-2.4 % specified with the exception of that of Mussene and Old Marte that are 2.7% and 2.64% respectively. These oxides can be corrected industrially by mixing with imported fuller earth of calculated proportion.

For Plastic Fire Clay: The SiO₂ do not meet the specification for plastic fire clay except that of Garadai, Allea and Ngelewa (Hubers,1985). The Al₂O₃ and Fe₂O₃ contents are also low compared with that of (Hubers, 1985). The Garadai, Allea and Ngelewa samples can be used as fire clay if the alumina and ferric contents are added in form of flux to upgrade their suitability.

For Fuller Earth: Some clay samples from the study area (New Marte, Njine and Mussene) have SiO₂ content similar to the standard required for fuller earth (Ibemesi and Achife, 1990). The Al₂O₃ content gives mixed values of within and above range (12.49 % -19.00 %) compared with the standard required value of 14.33 %. The New Marte clay with average value of 14.48 % and Old Marte clay with average value of 14.30 % meet the requirement for fuller earth while others are below and above standard (Ibemesi and Achife, 1990).

For Drilling Mud: The SiO₂ contents of Allea, Ngelewari and Garadai samples meet the specification of 58-64 % SiO₂ (Okogbue and Ene, 2008). The Al₂O₃ contents are generally low except that of Allea clay which has an average value of 19 % and therefore falls within the standard range required for drilling mud. Thus, the Allea clay with SiO₂ and Al₂O₃ content of 62.44 % and 19.00 % respectively are geochemically similar to the standard for bentonitic clays used as drilling mud (Okogbue and Ene, 2008). The CaO content of 1.17 % to 3.39 % and K₂O of 1.54 % to 3.66 % are higher as compared with the specification of 0.1 % to 1.0 % and 0.2 % to 0.4 % of CaO and K₂O respectively for drilling mud as indicated by (Okogbue and Ene, 2008).

The SiO₂ and Al₂O₃ in the clays clearly define them as aluminosilicates. The industrial requirement for the oxides vary but the Cr, Ni and Co occur in trace amount in all the locations within the study area, which is due to the fact that they are merely adsorbed by the clay particles and thus have no implication on clay usages. To be used as drilling mud, the samples have to be beneficiated using wet method of beneficiation. The samples are to be crushed and screened with a 2 mm sieve, 100 grammes of resulting samples are to be dispersed in a litre of 10% hydrogen-peroxide (H₂O₂) solution. This is to remove or oxidize any coloured organic and inorganic matter in the samples. The suspension is to be agitated and allowed to stand for 2 hours, after which the clear supernatant liquid decant and clay re-suspended in a liter of 0.5 molar solution of sodium hydroxide (NaOH) for a period of 2 hours with moderate agitation using mechanical stirrer. The resulting suspension is to be diluted with deionized water to siphon off the less than 2µm clays. Thus the clay particles (size <2µm) in the suspension are then extracted by repeated sedimentation and siphoning (Okogbue, *etal.*, 2008). The geochemical results as presented in this study indicates higher concentration of potassium (K), thus implying total absence of illite as one of the clay minerals. This is due to the fact that illite differs from other clay minerals in having more silica and less potassium. The results of Loss on Ignition (LOI) also appear high and therefore point to high structural water content which is normal for clay minerals.

Conclusions

The chemistry of the studied clay samples from Marte area shows that they are mainly smectite type of clays. When compared to the industrial specification, it is shown that they fall below standard and therefore need elaborate treatment to render them suitable for some industrial applications such as pharmaceutical, paint industries etc. However, if properly beneficiated by blending them with imported ones, they can also be utilized for refractory bricks, ceramics, plastic fire clay, bleaching agent, drilling and fuller earth. Presently they are being used locally for pottery and mud building of local houses.

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