CHARACTERIZATION OF CLAY FOR INDUSTRIAL APPLICATION BY PHYSICOCHEMICAL, XRF, AND TGA METHODS

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Abstract: Ndia clay deposits obtained from Takum Local Government Area of Taraba State Nigeria was characterized for industrial application using physicochemical, XRF and TGA techniques. The results of the chemical analyses shows that the clay contains high amount of SiO_2 (61.27±1.232%), Al_2O_3 (18.24±0.024%), Fe_2O_3 (2.44±1.643%) and trace amount of K_2O (2.44±0.23%), CaO (0.17±0.143%), TiO_2 (0.53±0.015%), MnO (0.06±0.014%) and so 3 (0.62±0.016%). The TGA result revealed a weight loss of 9.78% on heating and 98.30% un-decomposed value after degradation which indicates that the clay sample is thermally stable at high temperature. Further, the physical properties shows a moderate plasticity of 3.55 ($Kgf/1cm^2$) on average, shrinkage of about 12.03% on average, pH of 6.80 and a bulk density of 1.44% on average and the colour ranges from grey to red on firing. These properties thus placed the clay deposits as a useful potential in the industrial sector for the production of ceramics, floor tiles, refractory materials, bricks as well as kilns lining and binding agents.

Keywords: Characterization, clay, physicochemical, XRF, TGA

Introduction

Clay is regarded as a kind of natural earth, when mixed with water; it becomes plastic and mouldable and on drying and fired, becomes hard again. Clay is a complex inorganic mixture, whose composition varies widely depending on the geographical location. It is a natural substance occurring in great abundance in nature, being constantly formed on the earth's surface as a result of rock weathering (Hillier, 2003; Kefas *et al*, 2007; Mark, 2010). Clay is composed of silica (SiO_2), Alumina (Al_2O_3) and water (H_2O) plus appreciable concentration of oxides of iron, alkali and alkaline earth metals, and contains groups of crystalline substances known as clay minerals such as quartz, feldspar and mica (Omowumi, 2001; Churchman *et al.*, 2006).

Clay minerals are the most important industrial minerals. Millions of tons are utilized yearly in various applications including uses in geology, process industries, agriculture, environmental remediation and construction. The reason for utilization of certain clay minerals in specific application is that the physical and chemical properties of a particular clay mineral are dependent on its structure and composition. The structure and composition of kaolins, smectites, and palygorskite and sepiolite are very different even though they each have octahedral and tetrahedral sheets as their basic building blocks. However, the arrangement and composition of these octahedral and tetrahedral sheets account for major and minor differences in the physical and chemical properties of kaolin, smectites and palygorskite clay (Bergaya, 2000; Nnuka and Enejor, 2010; Osabor *et al.*, 2009).

Clays have received considerable attention especially as potential adsorbents for environmental research. Also many researchers around the world, have beamed their search lights on the phase developments that occurred by sintering clay in the presence of some oxides (Rahman *et al.*, 2013; Malu *et al.*, 2013).

In Nigeria, deposits of clay raw material are widely distributed in Nigeria. In order to determine the profitability of utilizing clay from a particular deposit for any application, it is of paramount importance to examine the microstructural morphology, determine the mineralogical composition and analyses the various available phases in such clay deposit (Gray *et al.*, 2013; Nweke and Egwu, 2007). Clay is an abundant raw material. However, certain high grade types of clay deposits are limited in geographic occurrence and extent. Clays containing a preponderance of the clay mineral kaolinite are known as kaolinite clays. Several commercial clays are composed predominantly of kaolinite. These are China clays, kaolin's, ball clays, fireclays, and flint clays. The terms China clay and kaolin are used interchangeably in industries (Akinbode, 1996; Murray, 2007; Ekosse, 2001). China clays are high grade white kaolin found in the South-eastern United States, England, and many other countries. In Georgia and South Carolina, the largest producing states, the deposits are composed almost wholly of the mineral kaolinite and occur as lenticular-shaped bodies generally 1.8-7.5 m in thickness (Sabri *et al.*, 2003; Nora, 2009). Many grades of kaolin are used in the manufacture of ceramics (white wares, refractories, and porcelain), paper, rubber, paint, plastics, insecticides, adhesives, catalysts, and ink. They are also used for many other industrial purposes (Churchman *et al*., 2006; Manukaji, 2013).

Kaolin has the desirable properties of being white in color, very fine in particle size, nonabrasive (hardness 2-2.5 on mohs scale), and chemically inert in most uses. The individual kaolin particle is a thin, flat, pseudo-hexagonal platelet, so tiny that approximately 10,000,000 spread on a postage stamp would form a layer thinner than the thickness of a human hair (Nwajugu and Aneke, 2001; Omowumi, 2001; Kefas *et al*., 2007). This thin, flat particle shape is a distinct advantage in many uses. The grades of kaolin commercially available are generally based on particle size and colour, or brightness. In white wares and sanitary wares, kaolin provides a white body, gives easy moulding properties, and adds strength, dimensional stability, and smooth surfaces to the finished product. Refractoriness, dimensional stability and chemical inertness make the kaolins uniquely suitable for special refractories. In addition to the above properties, the excellent dielectric properties of kaolin are very suitable for porcelain electric insulators (Omowumi, 2001; Nnuka and Enejor, 2010). Those clays that are composed mainly of the clay mineral montmorillonite and are formed by the alteration of volcanic ash are known as bentonites. They are used in many industries; the most important uses are as drilling muds and catalysts in the petroleum industry, as bonding clays in foundries, as bonding agents for taconite pellets, and as adsorbents in many industries (Oden *et al*., 2001; Rahman *et al.*, 2013; Ekpunobi *et al*., 2013).

Materials and Methods

Location of investigated clay deposit

The clay used in this research work was collected from Ndia community of Takum Local Government Area of Taraba

State, North-Eastern Nigeria. It is locally used by the people around the area for molding and bricks making.

Methods of sampling

Five pits coded A1, A2, A3, A4, and A5 were dug within an area in the location covering a total of 200 m^2. The holes were dug using a hoe at intervals of 20 m from one another. The clay samples were collected at a depth of 2 m in each of the pits. The collected clay samples were placed in a dry clean polythene bag (Osabor *et al*., 2009; Malu *et al*., 2013)

Sample pre-treatment and preparation

The collected clay samples were taken to the laboratory and sun dried for five days after which they were crushed using ceramic mortar and pestle. The pulverized clay was sieved using 1 mm mesh and packaged in a plastic container, scrubbed and de-limed (thoroughly washed with water to remove impurities such as clay and soluble materials) and finally with distilled water (Ahmed *et al.*, 1986; Emufurieta *et al*., 1992). And another set of the pulverized samples was poured in a clean-dried universal bottles and sets for physicochemical analysis.

Experimental Procedures

Physical analysis

Determination of Loss on Ignition (LOI)

The intergranular water (H_2O) was determined as a percentage loss in weight. In each case, between 1 – 15 g of the sample was weighed into a dry clean beaker using mantles pm400 analytical weighing balance and place in an oven at a temperature of 110^{0}C for 1 h. After every 30 min, the sample was brought out from the oven, allowed to cool and weighed. The process was repeated until a constant weight was obtained and the result recorded (Erdogan, 2015; Aroke *et al*., 2013; Aramide *et al*., 2014).

mH Measurement

The pH of the sample was determined with a pH meter as described by (AOAC, 1990). The pH of the samples was determined electronically using a pH meter of ±0.1 percent sensitivity pyrel Unicom model 290 pH meter. In each case, 6 g of the clay was weighed into a clean dry beaker using mettle AT460 Delta Range analytical balance. It was mixed approximately with 60 mL of distilled water, and stirred thoroughly for about 3 – 4 min. The pH was taken after standardizing the pH electrodes with buffer solution of pH 4.0, 7.0 and 10.0, respectively

Determination of plasticity

70g of the clay sample was placed into a container and a little amount of water added to it. The moistened clay was moulded into cylindrical shape using a cylindrical mould. The moulded clay was deformed by dropping on a flat-headed plunger of known weight from a fixed height. The distance travelled was read from the graduated scale. The modulus of plasticity (MOP) for the clay sample was obtained from the expression in equation 1;

$$\text{MOP} = \frac{\text{Original Height}}{\text{Deformation Height}} \text{ ---------- (1)}$$

Also, the percentage making moisture for clay samples were obtained from the expression in equation 2;

$$\% \text{ Making Moisture} = \frac{\text{Wet Weight} - \text{Dry Weight}}{\text{Net Weight}} \times 100 \text{ .....(2)}$$

Determination of Shrinkage

The grinded sample was poured in a basin and moistened with about 390mL of water added to the point wedging. The wedged sample was cast in a brass modes coated with thin film of machine oil to facilitate easy removal when dry. Three-test clay bars of 6.5 × 2.5 × 1.5 cm dimensions were prepared. Two points of 1cm intervals were marked on each of the moulded clay using venire caliper. The bars were allowed to air dry for 24 h and each sample were weighed. The bars were dried in an oven at 110^{0}C for 5 h after which the marked distance was recorded as dry length. The wet-dry shrinkage was obtained as in equation 3:

$$\text{wet} - \text{dry} = \frac{\text{Original Lenght} - \text{Dry Lenght}}{\text{Original Lenght}} \times 100 \text{.....(3)}$$

then, three clay bars were charge individually into an electrical furnace at the temperature of 900, 1000 and 1100^{0}C and fired approximately for 10 h. At each temperature, the sample were removed from the furnace, allowed to cool and each bar observed for colour change, cracks formation and fire weight. These parameters were determined from the formula as in equation 4 and 5:

$$\text{dried} - \text{fired shrinkage} = \frac{\text{Dry Lenght} - \text{Fired lenght}}{\text{Dry Lenght}} \times 100 \text{ ...(4)}$$

$$\text{Percentage Total Shrinkage} = \frac{6 - \text{Fired Lenght}}{6} \times 100 \text{..... (5)}$$

Bulk density determination

This was determined using the method described by Kefas *et al.* (2007). About 5.0 g of the clay sample was filled in a 10 mL measuring cylinder and its bottom was tapped on the laboratory bench for the sample to settle, compact and eliminate air pockets. The volume was noted and the bulk density expressed in g/mL using the formula below in equation 6:

$$\text{Bulk density} = \frac{\text{mass of sample wighed (g)}}{\text{volume of sample in the measuring cylinder (mL)}} \text{.....(6)}$$

Chemical analysis

The chemical analysis were carried out on the sample to determine their chemical composition (% SiO_2, Al_2O_3, K_2O, TiO, CaO, SO_2, Fe_2O_3, and MnO) and mineralogy content (% free silica, kaolinite, feldspar, etc).The analysis was done using the EDX3600B X-ray fluorescence spectrometer (XRF). About 5 g of the clay were analysed. The calibration was done using pure silver sample, the working curve was selected according to the samples and placed in a machine attached with a computer analyser and read out system. The machine was set for 60s and the result was obtained and recorded.

Thermo gravimetric analysis of different clay samples

Thermal stability of a different clay sample was performed to understand the stability of each of the clay structure against elevated temperature gradient. The thermo-gravimetric analysis (TGA) curves (Figs. 1, 2 and 3) of the clay show two well-defined weight loss regions due to the loss of physisorbed water (below 180ºC) and dehydroxylation of coordinated and structural water (above 375ºC) for all the samples.

Thermal analysis procedure

In this study, a Perkin Elmer STA 4000 analyzer at Step-B Lab FUTMinna (Plate X) was used to test the thermal stability of the samples. A known weight (15 mg) of samples was put into a ceramic pan placed in integrated furnace in the equipment. After which the samples were heated from room temperature to 900^{O}C at 10^{O}C/min under nitrogen gas flow at 20 ml/min and Pressure of 2.5 bar. The traces were recorded as weight loss versus temperature for TGA (Osabor *et al.*, 2009; Salawudeen *et al*., 2010).

Statistical analysis

All data were subjected to statistical analysis. Values are reported as mean ± standard error of mean (SEM) while one way ANOVA was used to test for differences between treatment groups. The results were considered significant at p-values of less than 0.05, that is at 95% confidence level ($p<0.05$).

Results and Discussion

Physico-chemical characterization was carried out on Ndia clay deposit using some analytical techniques and the following results were obtained as presented. X-ray

fluorescence analysis (XRF) was performed on the clay samples to know the chemical composition (oxides) present as shown on Table 1 and depicted on Fig. 2. The results shows that silicon dioxide (SiO_2) has the highest content (61.268±1.232%) followed by Al_2O_3 (18.238±0.014%) then Fe_2O_3 (7.607±1.643%) and trace amount of other oxides (SO_3, TiO_2, K_2O, MnO and CaO). The high content of silica (SiO_2) and Alumina (Al_2O_3) in the clay samples is an indication that it is a kaolinite clay. Literature review indicate that the dominance of silica and alumina are often used for floor tile manufacture as well as a potential source for bricks production (Nwajugu and Aneke, 2001; Bergaya *et al*, 2006; Nweke, 2007). This agrees with the fact that the region from which the clay samples were sourced from used more of burnt bricks than cement bricks (Singer and Singer, 1971; Nweke, 2007).

Table 1: Chemical composition of the clay samples

Sample	Al_2O_3	SiO_2	K_2O	CaO	Fe_2O_3	SO3	TiO_2	MnO	LOI	pH
A1	19.08	80.14	5.09	0.23	13.09	0.84	0.89	0.101	50.12	6.70
A2	20.69	82.03	5.30	0.25	11.68	1.15	0.69	0.05	49.87	6.81
A3	25.61	70.64	0.85	0.12	9.24	0.85	0.71	0.04	49.87	6.90
A4	17.00	42.01	0.612	0.15	2.25	0.12	0.21	0.01	49.87	6.85
A5	12.02	31.52	0.374	0.12	1.78	0.12	0.14	0.02	49.83	6.79
Mean	18.24	61.27	2.44	0.17	7.61	0.62	0.526	0.06	49.91	6.81
	±0.014	±1.232	±0.023	±0.143	±1.643	±0.016	±0.015	±0.014	±0.012	±0.015

Table 2: Modulus of plasticity

Sample	Wet Weight(g)	Dry Weight (g)	Deformation height(cm)	Original Height (cm)	Modulus of Plasticity (kgf/cm^3)	% Making moisture
A1	70.67	61.22	5.82	13.26	3.56	21.54
A2	71.98	62.33	5.83	13.30	3.55	20.78
A3	72.55	62.90	5.85	13.31	3.54	20.27
A4	72.90	63.42	5.86	13.55	3.52	20.99
A5	73.50	63.53	5.88	13.56	3.56	20.86
Mean	72.32	62.68	5.85	13.39	3.55	20.89

Table 3: Shows result for Shrinkage test

Temperature (^0C)	Original length (cm)	Dry length (cm)	Fired length (cm)	% fired Shrinkage	% wet dry Shrinkage	% total Shrinkage	Colour
900	6	6.61	5.26	5.70	6.65	12.10	Light red
1000	6	6.64	5.25	7.27	6.8	12.65	More red
1100	6	6.71	5.21	8.76	5.1	12.30	Very red

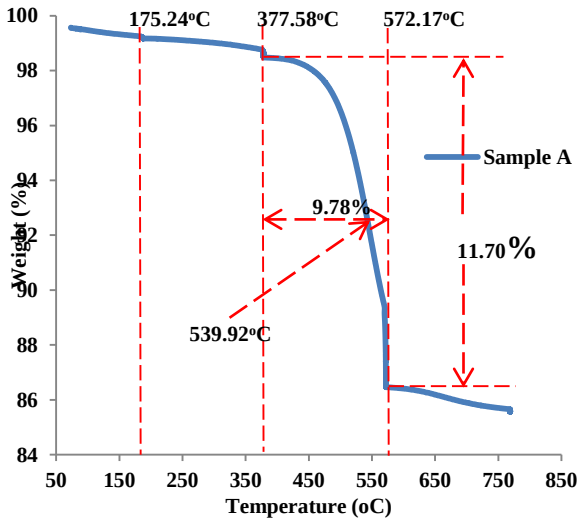

Fig. 1: Thermo Gravimetric Analysis of Clay Sample

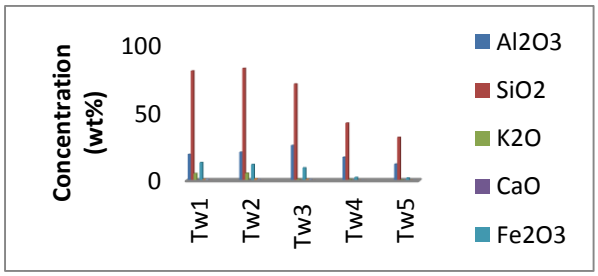

Fig. 2: Bar chart showing chemical composition of the Ndia clay samples (Tw1–Tw5)

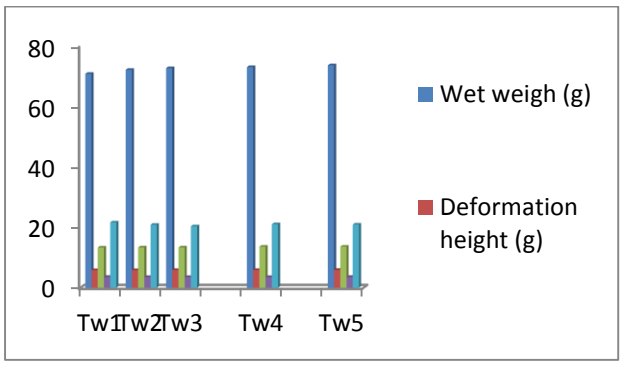

Fig. 3: Modulus of plasticity for Ndia clay

Table 4: Result for Bulk Density

Sample	Bulk Density (g/cm^3)
T_{w1}	1.32
T_{w2}	1.52
T_{w3}	1.56
T_{w4}	1.37
T_{w5}	1.45
Average	1.44

On Table 3, the red colour manifestation of the clay samples after firing is due to the presence of Fe^{3+} ions (Fe_2O_3) in its chemical content. The amount of Fe_2O_3 in the clay sample was found to be 7.607%. This amount though small compared to the amount of silica and alumina shows that on firing the grey color of the clay turns to red. This observation was notice on the shrinkage test whereby the colour deepened as temperature was raised from 900-1100^{o}C and giving % total shrinkage of 12.10% -12.30%. The present of trace amount of other oxides in the clay samples (CaO, K_2O, SO_3, TiO_2, MnO, etc) lower content of K_2O (2.444 ±0.023%), MnO (0.059±0.014%) and CaO (0.171±0.143%) indicates that Ndia clay could be useful as refractory materials. Nweke (2007) had defined refractory material as materials that will retain their physical and chemical nature when subjected to heat at high temperatures. They recommended fire clay, ball clay, kaolin and bentonites as refractory materials. The presence of alkali oxides have been linked to plasticity characteristics of the clay (Nnuka and Enejor, 2001; Bergaya *et al.*, 2006; Olaremu, 2015). The result of modulus on Table 2 and Fig. 3 show modulus of plasticity to be 3.55 kgf/cm^3 on the average which is an indication of good workability nature of the clay (i.e. the clay can be worked into any shape)a .this characteristic makes it useful for many industrial products. Comparison of these result with those of Nkpor, Mbayon and Odukpani clay suggest that the clay can be used as a binder, refractory, flower pots, earthenware etc. (Nwujugu and Aneke, 2001; Osabor *et al.*, 2009; Malu *et al.*, 2013; Folorunso *et al*., 2014). On Table 4, the bulk density of the sample shows that the values (1.32 – 1.56 G/CM^3) which fell within the international accepted standard for clay sample.

The loss on ignition (49.91%) as shown on Table 1 for the clay sample implies that the pores are so closely knit within the clay samples that they tend to hold moisture molecules within the clay complex. The pH was also found to be 6.81. Despite the presence of alkali metal oxides which could have shown the clay to be a basic material, the weakly acidic nature as shown by the pH value could be attributed to the presence of SO_3(0.0619%) which may have dissolved in the moisture to give H_2SO_4 (Nweke, 2007).

From Fig. 1, the TGA of the clay Sample shows a remarkable weight loss of 9.78% at a temperature degradation range of 377.58^{o}C-572.17^{o}C. This degradation temperatures range of the samples is found to be at a very close range which corresponds to the transformation of phase from the crystalline clay particle to the amorphous heated clay as shown in Equation (7).

$$2Al_2Si_2O_5(OH)_4 \rightarrow 2Al_2Si_2O_7 + 4H_2O \quad \ldots\ldots\ldots\ldots (7)$$

Furthermore, the figures showed only one major large endothermic peak at 539.92^{o}C for the Sample corresponding to a weight loss of 11.70% which is in close agreement with literature value for typical clay (Kirk and Othmer, 1997; Salawudeen *et al*., 2010). This observable endothermic peak in the clay samples are due to the liberation of water caused by dehydroxylation of coordinated and structural water molecules present in clay. Thus, 98.30%, of the clay samples were left un-decomposed which is an indication that the clay samples are thermally stable at high temperature.

Conclusion

This study has shown that Ndia clay obtained from Takum Local Government Area of Taraba State was characterized and found to possess a moderate plasticity of 3.55 Kg/cm^2 ,shrinkage value of 12.16%, which make it useful in ceramic industry. The colour characteristics ranged from grey to red on firing. Couple with the physical and chemical properties. Therefore, it can be used in ceramic, refectories and paper industries and also as source of silica for floor tiles. The result for chemical analysis in Table 1 shows a high content of silica (SiO_2, 61.27±1.232%) and alumina (18.24±0.014%) with low amount of alkalis oxides couple with it TGA test (thermally stable at high temperature suggest the clay can be used as source of silica for the production of floor tiles, refractories materials, kilns lining and bricks making. These potentials thus qualify the exploration of this clay deposits for industrial applications by the Taraba State Government for the economic and job creation for the teeming youths.

Conflict of Interest

Authors declare none.

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