

Characterization and Evaluation of Kaolinite Clays from Bara, Kwi, and Wereng in Bauchi and Plateau States for Refractory Applications

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ABSTRACT

The evaluation of physical and chemical properties of clay deposits from Bara, Kwi, and Wereng villages in Bauchi and Plateau States, Northern Nigeria, with a focus on their potential as locally made refractory materials. The chemical composition of the clay was determined using Energy Dispersive X-ray Fluorescence Spectrophotometry (ED-XRF), while physical properties: moisture content (MC), color, pH, tapped density (TD), and refractoriness were determined using standard techniques. Clay bricks was fabricated from the clay samples and fired at intermittent temperature ranging from 150 °C to 600 °C for ten-minute intervals. The analysis of the chemical composition indicated Bara and Wereng clays contain over 70 % of SiO₂ and Al₂O₃, while Kwi clay comprises approximately 60 % of SiO₂ and Al₂O₃. The Physical properties range across the clay samples revealed pH(4.40-5.20), MC(3.304.40 %), TD(0.96-1.11 g/ml), (white to reddish-brown and near-white color), and refractoriness temperature (15261597 °C) for Bara, Kwi and Wereng respectively. The firing of the sample bricks produced resulted in progressive weight losses. The findings unveiled the valuable potential of these clay deposits in refractory production for both domestic and industrial applications thereby cutting down the huge cost of imported refractory material and enhance their performance. Purifications and blend formulations studies of these clay materials can further enhance their refractory potential and properties. These studies lined with the MDGs/SDGs vision 2030 for social, environmental and global economic growth for industrial innovation and infrastructure development and sustainability.

KEYWORDS: Refractory, Characterization, kaolinite, Clay Deposits, Brick Kwi Clay, Alumina.

1. INTRODUCTION

Clay materials are formed from deposited component of the earth composed mainly of layers of fine particle with high characteristics of plasticity at suitable water mixed, turn completely hard when fired.^{24,18} Materials of clay are classified into three groups which include, the kaolinites white, grayishwhite or slightly colored which became more dark, with high plastic properties when wetted with water,^{10,14,20} while the second group is composed majorly of montmorillonite and the intermediate product of the disintegration of mica into kaolin are the third group of clays.²⁰

Hijioke *et al.*⁸ reported several literature that define clay as a natural material used for the manufacture of many industrial products including the refractory materials that has proved to be reliable in the metallurgy processes.⁴ Refractory product have the capacity to resist high temperatures both physically and chemically as such good refractories can withstand temperature variation between 1000 °C to 1500 °C and also serves as better thermal and electrical insulator.⁴ Kaolinites is composed of high content of alumina (up to 39.50 % by weight) among the clay minerals as shown by the chemical formula (Al₂O₃.2SiO₂.2H₂O) but the most suitable for refractory application must have high percentage of alumina (Al₂O₃) as well as very low impurity oxides of fluxes minerals (Na₂O, K₂O etc) and coloring (Fe₂O₃, TiO₂ etc) agents.² Complete chemical analysis of clays unveiled the true mineralogical composition and refractory materials are constructed in varying combinations and shapes depending on their applications.

The increasing demand for refractory products is associated the growth of metallurgical industries which required 80 % of all forms refractories for full operation.⁹ The advancement in the areas of metallurgical processes in Nigeria recently has been the reason for the increasing demand for refractory materials⁴ and a large proportion of it are currently being imported to meet this demand.¹⁷ Following the development and revitalization of the iron and steel industry through the restoration of different inland rolling mills and conceived completion and commissioning of the multibillion-dollar Ajaokuta Steel Complex to produce 1.3 million tonnes of liquid steel, there will be further increasing demand for refractory materials locally.⁵ The Ajaokuta iron steel industry is estimated to require bricks of refractory

Abuja, Nigeria - May 4-7, 2025

materials in volume of over 36,000 tonnes with worth of over sixty million naira for furnace lining only and more than 80 % refractory bricks mostly required are fire clay.¹³

Nigerian four major refineries were reported to have cost more than \$850 million dollars for turnaround maintenance (TAM) operation as from 1997 to 2002 and the main unit in the TAM process operation is the fluid catalytic cracking (FCC) which is made up with large quantity of different grades of refractory channels.¹¹ Omowumi²¹ locally available raw material for the production of different types of refractory products include kaolinite ($Al_2O_3 \cdot 2SiO_2 \cdot 2H_2O$), chromite ($FeCrO_3$), magnesite ($MgCO_3$) etc and additives such as saw dust, graphite and binders. Studies reported by Nnuka and Enejor¹⁹ on the characterization of Nigerian clays showed that the Otukpo clay can accommodate furnace temperature of 1710 °C which comparably can compete favorably with products of refractories imported from foreign countries. Research reported also by Amuda *et al.*⁵ revealed the characterization and evaluation of some clay deposits in Southwest Nigeria to possess good refractory properties with performance results that agreed favorably with standard recommendations and suggest a blend of these clays for good furnace lining and performance.

This present study realized the vital need to further characterize the local clay materials from Plateau and Buachi state axis to explore their potentials to build good and quality refractories which could cut cost, reduced importation and saved foreign exchange rate.¹² Aside metallurgical industries, petrochemicals, chemicals, agrochemicals, oil and ceramic industries used refractory materials for various operational processes, but still yet there is no specialized industry for the production of refractory products in Nigeria, considering the abundant and availability of the clay rocks and other raw materials across the country.¹

Hence, it becomes necessary to the essential need to expand the utilization of local raw content for the production of industrial equipment for application in various industrial operation processes necessitated the desire to study and characterize kaolinite clay samples obtained from Bara, Kwi and Wereng villages in Bauchi and Plateau States, Northern Nigeria. To equally assess and ascertain the suitability of the clay as refractory bricks for furnace linings and other domestics and industrial purposes due to the rich nature of the clays in this region in alumina and silica content.

2. MATERIALS AND METHODS

2.1 Study Area Description

Kwi and Wereng are located in Riyom Local Government Area of Plateau State, North central Nigeria. Riyom L.G.A has its headquarters in the town of Riyom to the North of the area at 9° 38' 00"N 8° 46'00"E. The L.G.A has boundaries with Kaduna and Nasarawa States. Bara is located in Kirfi Local Government Area of Bauchi State, Northeast Nigeria bordering Gombe State in the East. Its headquarters is in the town of Kirfi (or Kirfin kasa). The Northeasterly line of equal latitude and longitude passes through the L.G.A. It is located precisely around latitude 10° 24'N 10° 24'E.

2.2 Sample Collection and Preparation

The Clay samples were collected separately from the sampling sites in Kwi and Wereng villages of Riyom Local Government Area, Plateau State and Bara village of Kirfi Local Government Area, Bauchi State. The sample materials weight of 3 kg was randomly collected at a depth of 500 mm using pitch digger and shovel. The samples were air-dried for 3 days, finely crushed with a laboratory mortar and pestle to achieve homogeneity of particle sizes. Then sieved with a mesh size of 2.0 mm and collected for various physical and chemical analyses.

2.3 Methods and Experimental

The analysis of the physical properties of the clay was carried out at the Department of Chemistry, University of Jos, Nigeria whereas the chemical compositions of the clay were determined at the National Metallurgical Development Centre (NMDC) Jos, Plateau State.

2.4 Determination of Clay Chemical Compositions

The elemental analysis of the raw clay samples was determined using an Energy Dispersive X-ray Fluorescence Spectrophotometer (ED-XRF). The ground samples were filled each in a sample cup as the ED-XRF spectrophotometer was turned on and allowed to stabilize the optics and X-ray tube for some period. The samples were placed in the machine while in operation and ran using the prepared program. The concentration of the elements present in the samples were automatically calculated and displayed. The percentage composition of the various constituents was recorded from the print out.

2.5 Determination of Clay Physical Properties

The clay samples were taken for various physical analysis. The tests conducted were moisture content, pH, color, and tapped density. These tests were conducted using standard test procedures.

2.5.1 Color of the Clay Samples

The colors of the clay samples were determined using the Lovibond Comparator. The device is used to determine the color of liquids. The clay samples were prepared into solutions to unveil all the coloring particles by dispersion and thoroughly shaken in a volumetric flask. The sample solutions each were turned in glass tubes and inserted into the comparator one after the other and compared with series of colored glass disc until their nearest possible color matches were found. The colors for the three samples were recorded.

2.5.2 Moisture Content of the Clay Samples

The percentage moisture content of the clay samples was determined using a thermo-gravimetric approach whereby a sample is heated and the weight loss due to the evaporation of moisture were recorded. Three (3) petri dishes were properly washed and dried in an electric oven for 30 minutes at a temperature of 105 °C and transferred into a desiccator to cool. The dry weight of the petri dishes was taken using an analytical weighing balance and recorded as W_1 . The samples each of weight 1g were transferred into the petri dishes and weighed as W_2 . The petri dishes containing the samples were placed in the oven and heated to a temperature of 110 °C for 16-24 hours. The samples were then cooled in a desiccator. After the cooling process, they were reweighed and recorded as W_3 . To calculate the percentage moisture content of the samples, equation 1 was used.

$$W_2 - W_3$$

$$\% \text{ Moisture Content} = \frac{W_2 - W_3}{W_1} \times 100$$

2.5.3 Clay pH Values

The pH for each of the kaolin clay samples were determined by the use of an electronic pH meter where a glass electrode was inserted into a mixture (suspension) of clay and distilled water to determine the pH which was then noted on a digital display screen and recorded.

2.5.4 Tapped Density

The tapped density of a powder is the ratio of mass of the powder to the volume occupied by the powder after been tapped for a defined period. The tapped density is obtained by mechanically tapping a 250 ml graduated cylinder containing 100g of each sample on a flat surface until a little further volume change is observed after been tapped for fifty (50) times. The tapped density was calculated using the equation 2 below;

$$\text{Tapped Density} (\frac{g}{ml}) = \frac{M}{V_f} \quad 2$$

Where M = the mass of powder in g

V_f = the tapped volume in milliliters

2.6 Refractory Brick Production Process

The method prescribed by Kipsanai¹² was adopted for the bricks production. The grounded and sieved clay samples were mixed to form a thick paste using some quantity of water, then molded to obtain bricks using a wooden mold of dimension 6 cm x 3 cm x 2 cm. The mold was continuously lubricated with water to prevent the clay from sticking to the mold as the bricks were molded by hand. The bricks produced were then air-dried for about 3 days with a mass between 0.80 kg and 1.1 kg, oven dried under temperature of 110 °C for 8 hours.



Fig. 1: Molded bricks.



Fig. 2: Air dried bricks.

2.7 Firing of Clay Bricks at Intermittent Temperatures

The aired and oven dried brick samples were fired in a muffle furnace at intermittent temperatures of 150, 200, 300, 400, 500 and 600 °C. The firing was controlled at steady rate of 10 minutes followed by gradual cooling and weighing of fired bricks to obtain progressive weight losses at various temperatures. The weight losses in fired clay bricks were recorded.



Fig. 3: showing brick in the muffle furnace.



Fig. 4: Fired clay bricks.

2.8 Refractoriness

The prescribed by Shuaib-Babata *et al.*²⁴ and was used to determine the refractoriness of the clay materials. Refractoriness is the capacity of material designed to serve as refractory to withstand high range of temperature conditions under service operations without deforming. The refractoriness was estimated using the Shuen's formula in accordance with Bochraraov and Gensisov.⁷ Equation 3 below;

$$\text{Refractoriness (K)} = 360 + \text{Al}_2\text{O}_3 - \text{RO} / 0.228 \quad \text{---3}$$

Where K = Refractoriness

Al_2O_3 = Percentage alumina content in the clay

RO = Sum of other oxides in the clay excluding SiO_2 and Al_2O_3 (%) The value of 360 and 0.228 are constant.

3. RESULTS AND DISCUSSION

3.1 Chemical Composition of Kaolin Clay Samples

The chemical composition of the clay samples were presented in table 1

Table 1: Chemical Composition (wt %) of Kaolin clay samples

Composition (%)	Kwi	Wereng	Bara
Al_2O_3	24.70	27.80	25.90
SiO_2	38.60	43.70	52.40

P ₂ O ₅	0.58	0.66	0.08
SO ₃	<LOD	0.30	<LOD
K ₂ O	0.40	0.04	1.69
CaO	TiO ₂	0.35	0.43
	V ₂ O ₅	4.12	6.09
	Cr ₂ O ₃	0.21	0.20
	MnO	0.09	0.11
	Fe ₂ O ₃	<LOD	0.03
		18.59	4.79
	NiO	0.01	0.02
	CuO	0.07	0.06
	ZnO	0.03	0.04
	Ga ₂ O ₃	0.04	0.05
	SrO	0.07	0.06
	ZrO ₂	0.81	0.74
	Nb ₂ O ₅	0.10	0.09
	Au	<LOD	0.007
	PbO	88.77	90.02
	Total	0.64	0.49

Al₂O₃ : SiO₂

Key:

% =

Percentage. <LOD = below limit of detection/Loss in ignition (LOI) was not determined

3.1 Chemical Properties

The key indicators for good refractory materials by chemical compositions are high Al₂O₃ content desirable for refractory applications, low Fe₂O₃ content is preferred to minimize fluxing effects and high SiO₂ content can contribute to refractoriness properties. Comparatively, Kwi sample contains; Al₂O₃(24.70 %), relatively high Fe₂O₃(18.59 %) and SiO₂(38.60 %). Wereng sample contains Al₂O₃ (27.80 %) moderate contamination of Fe₂O₃ (9.50 %) and high SiO₂(43.70 %) while Bara sample contains Al₂O₃(25.90 %), low Fe₂O₃(4.79 %) content and high SiO₂(52.40 %). Base on the refractory indicators, Bara samples is more favorable for refractory applications due to low Fe₂O₃ content and high SiO₂ content followed by Wereng sample with moderate refractory potential due to balanced Al₂O₃ and SiO₂ content while the Kwi sample is less favorable due to high Fe₂O₃ content (Fig 5). Therefore, Bara clay sample appears to have the most favorable refractory properties due to its low Fe₂O₃ content and high SiO₂ content.

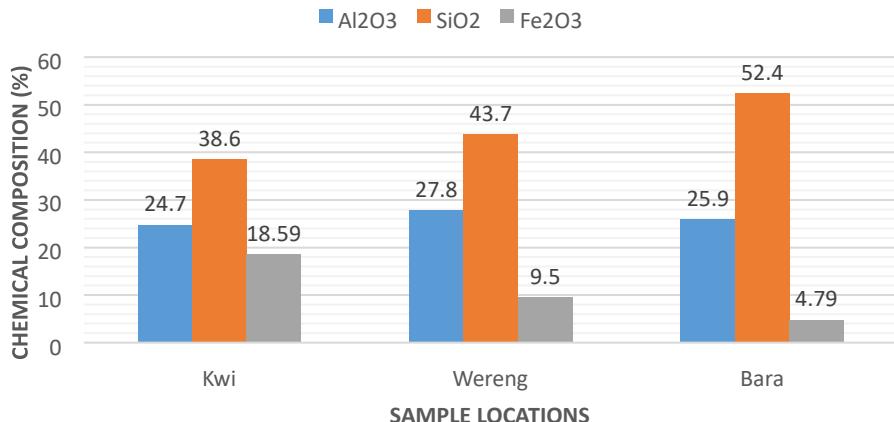


Fig. 5: Chemical Composition of the Clay Samples

The chemical constituent of the clay samples (Table 1) to be principally SiO₂ and Al₂O₃ in which 80 % of the clay material were characterized by eight elements while the remaining were attributed to water, trace elements and organic matter. The result also reflects slight variability of average in values of silica (SiO₂) and alumina (Al₂O₃) contents in Bara and Wereng clays, which constitute 70 % major constituents of the clay. Therefore, the clay samples were classified to belong to the aluminosilicate group. The clay sample from Bara contains high silicate with percentage silica of 52.40 % (Fig. 5) which agree with the standard recommended range of 46-62 % for a good refractory material as reported by Yami and Umaru.²⁷ This implies the clay can be built and designed to fit applications as refractories for linings in melting furnace for low melting temperature metals, heat treatment furnaces, aluminium ladle molds and blast furnaces.

3.2 Physical Properties of Kaolinite Clay Samples

The results of the physical properties of the clay samples are presented in table 2

Table 2: Physical Properties of Kaolinite clay samples

Sample location	Moisture Content (%)	Tapped Density (g/ml)	Color	pH
Bara	3.70±0.24	1.11±0.05	White	5.20
Kwi	4.40±0.33	1.00±0.09	Near White	4.70
Wereng	3.30±0.24	0.96±0.02	Reddish Brown	5.70

Values are expressed as mean \pm SD, n=3 for each group

The physical Properties of the clay bricks revealed the moisture content (MC): Bara (3.70%), Kwi (4.40%) and Wereng (3.30%). Tapped Density (TD): Bara (1.11 g/ml), Kwi (1.00 g/ml) and Wereng (0.96 g/ml) and pH: Bara (5.20), Kwi (4.70), Wereng (5.70) which makes them acidic clays respectively. The refractory Properties (Refractoriness) shows Bara (1597 K), Kwi (1526 K) and Wereng (1575 K) respectively (Fig. 6).

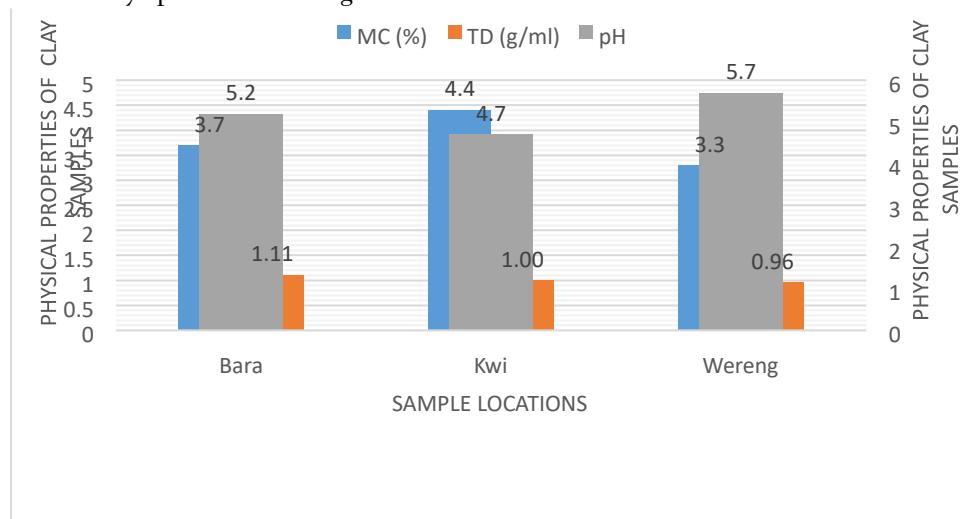


Fig. 6: Physical Properties of Kaolinite clay samples

Bara clay appeared whitish because of traces amount of iron (iii) oxides (4.79 %), Wereng clay contain more iron (9.50 %) with a near whitish color while high iron oxides (18.59 %) in Kwi clay resulted in a tinge of brown-reddish appearance.¹⁶ The process of clay firing alter the color of the resulted clay brick samples slightly from light-brown to dark-brown in Kwi clay due to the presence of high amount of iron oxide and other impurities. The Tapped density for Bara, Kwi and Wereng clays are 1.11 g/ml, 1.0 g/ml and 0.96 g/ml respectively. The Tapped density tells the powder flow ability and compressibility of the clay samples. A more powder flow ability observed in Bara clay followed by Kwi and Wereng the least.

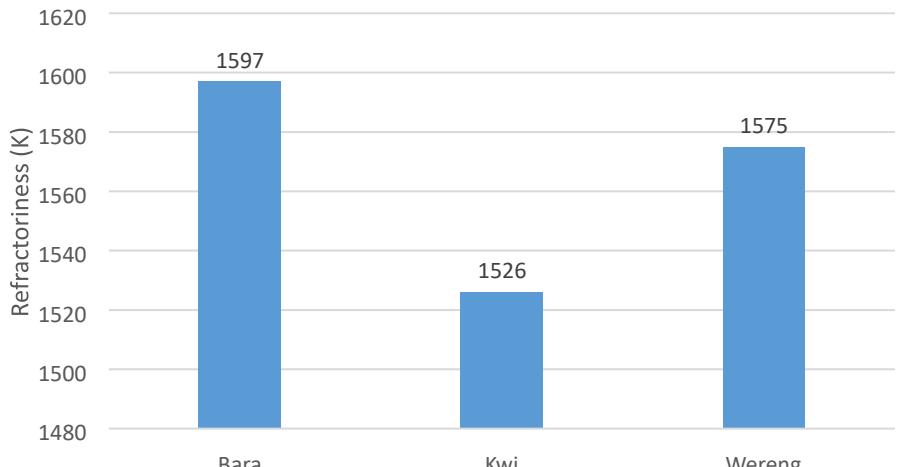
3.3 Refractory Properties of The Clay Brick Samples

The refractory results of the clay brick samples are presented in table 3.

Table 3: Refractoriness Properties of Kaolinite Brick Samples

S/N	Sample location	Refractoriness (K) °C
1	Bara	1597
2	Kwi	1526
3	Wereng	1575

The estimated refractory temperature for Bara clay was 1597 °C, Wereng was 1575 °C and Kwi 1526 °C which falls within standard value (1500-1750 °C) for refractories materials. The high refractoriness in Bara and Wereng clay (Fig. 7) resulted from high alumina contents of 25.90 and 27.80 respectively. The relatively low value of refractoriness in Kwi clay is because of lower content of alumina and silica and the high percentage content of flux materials. Impurities like Fe_2O_3 in aluminosilicate refractory lowers the refractoriness and service limits of bricks.

**Fig.7:** Refractoriness Properties of the Clay Samples (K)

3.4 Effect of Heat on Clay Brick Samples

The influence of heat on the brick samples are shown in table 4.

Table 4: Loss in weight of fired clay bricks at different temperatures

Sample location	Bara	Kwi	Wereng
Temperatures (°C)	Weight (g)	Weight(g)	Weight (g)
100	907.40	718.40	678.10
150	906.50	717.40	676.50
200	905.80	716.10	675.30
300	904.60	714.20	673.40
400	903.40	712.40	671.70
500	902.40	710.30	670.10
600	900.20	706.80	666.00

The progressive loss in weight in fired clay bricks heated at intermittent temperatures of 100 °C, 150 °C, 200 °C, 300 °C, 400 °C, 500 °C, and 600 °C are because of further dehydration and escape of some impurities in the clay as temperature increases. The increase in the firing temperature resulted in the conversion of moisture within the bricks into vapor which diffuse out while creating vacant sites within the clay. The clay particles immediately then migrate and rearranged to occupy the vacant sites, which subsequently results to shrinkage and weight losses. This process consequently reduces porosity and enhances the mechanical strength of the bricks.

3.5 Correlation Relationship between Chemical Composition and Physical Properties

The correlation analysis between chemical composition and refractory properties at $P < 0.05$ revealed percentage Al_2O_3 and SiO_2 in the samples to show positive significant correlation with the refractory properties. While the correlation of physical properties and refractory properties of the clay bricks revealed moisture content and tapped density to show no significant correlation with refractoriness.

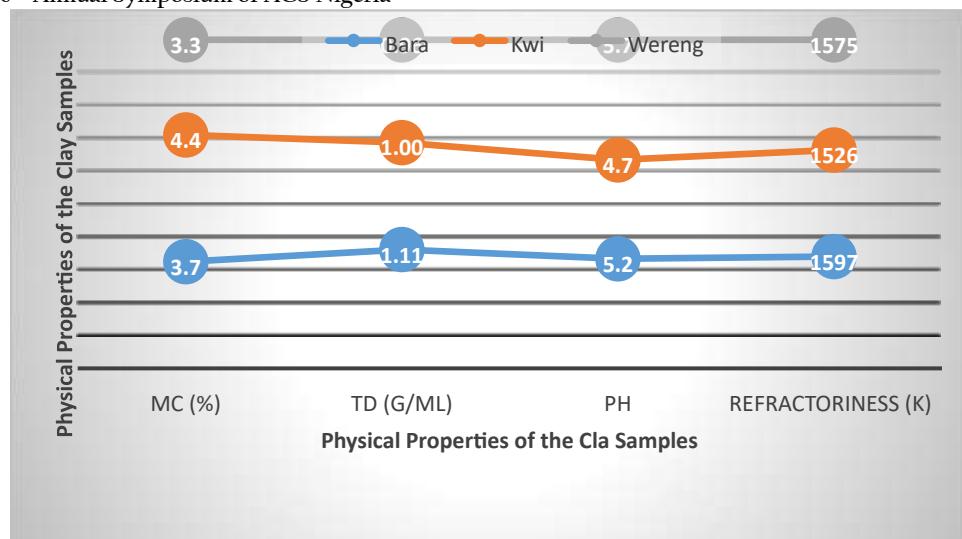


Fig. 8: correlation Between Physical Properties and Refractoriness

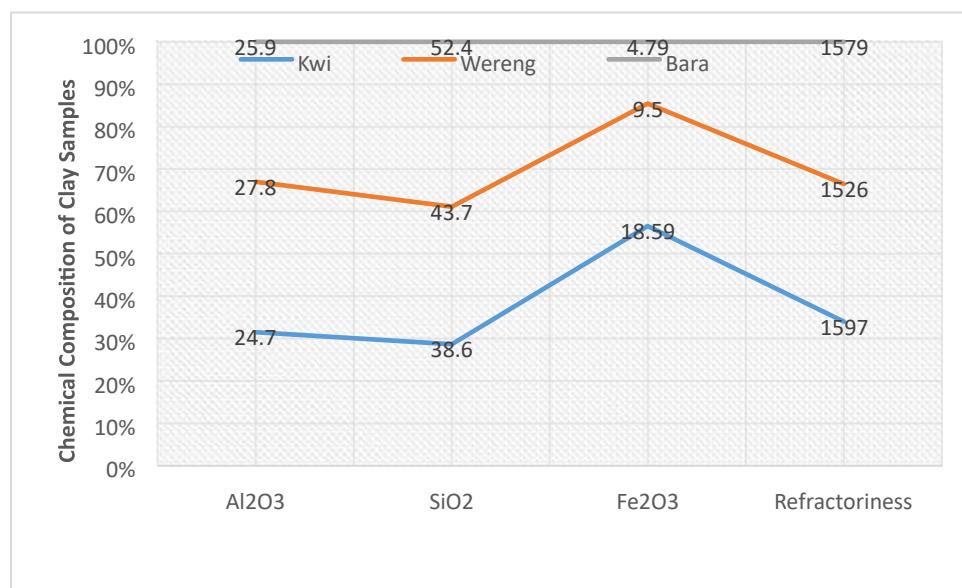


Fig. 9: correlation analysis between Chemical Composition Vs Refractioness

Chemical Composition and Refractory Properties (Fig. 9) - Al_2O_3 and Refractoriness shows positive correlation ($r = 0.53$), SiO_2 and Refractoriness also shows positive correlation ($r = 0.76$) while Fe_2O_3 vs. Refractoriness shows a negative correlation ($r = -0.83$). The correlation between physical properties and refractory properties (Fig. 8) shows moisture content vs. refractoriness to have no significant correlation ($r = -0.21$) and tapped density vs. refractoriness to show a positive correlation ($r = 0.62$) while pH vs. Refractoriness also shows no significant correlation ($r = 0.35$).

These correlations result strongly suggest that higher Al_2O_3 and SiO_2 content are associated with higher refractory properties while higher Fe_2O_3 content is associated with lower refractory properties. However, tapped density also shows a moderate positive correlation with refractoriness.

However, Clay samples from Bara, Kwi and Wereng can be considered as moderate alumina clay that agrees appreciably and consistently with value of 13-30 % alumina reported in a similar study by Ugwuoke²⁶ for good refractory materials. The presence of impurities in aluminosilicate refractory such as TiO_2 , K_2O , P_2O_5 , ZrO_2 lowers the refractory properties and operational durability of the bricks as all the three clay samples have significant amount of such impurities. Studies by Onyeji²² showed that, clays materials with high content of iron could not be used to sources aluminium due to the deleterious

effects of iron on the extraction processes. The clay samples may not be fit for direct application as refractories in furnace linings as it contain high percentage range of iron (4.79-18.59 %) compared to the recommended range of 0.5-2.4 %.²⁴ The elemental composition of the clay samples in table 1 were not in conformity with Sanni's²³ report that suggest refractories clay should contain at least 30 % alumina (Al_2O_3) and less than 1.8 % iron (iii) oxide (Fe_2O_3). This indicates the clay samples are not suitable as refractories materials chemically, except subjected to certain pretreatments and purification processes.

4. CONCLUSION

The chemical characterization revealed the clay samples to contain the required percentages of alumina, silica, metal oxides and other flux minerals that can enhance refractory properties and performance while the evaluation of the clay physical properties revealed the parameters to fall within the range required for the designed and fabrication good refractory materials. The estimated refractoriness values of the clay samples also fall within standard recommended value (1500-1750 °C) for refractories. However, Bara and Weng recorded the higher value of refractories property, which correlated to the high amount of aluminosilicate and other flux mineral content in these clay samples. The progressive weight loss at intermediate temperature changes during firing was due to loss of water vapor and other volatile impurities contained in the clay samples. The process also help to reduce porosity and improve the mechanical properties of the bricks. The results revealed these clay samples to possess the potential to be consider as material for the designed and construction of fire refractory materials for applications in furnace and kiln lining. However, the three clay samples were limited as they contained significantly amount of iron and titanium oxides, which have deleterious effect on the refractory property of the clays. It is therefore, recommended that additives such as purified bauxite (fused alumina) can be controllably added to significantly reduced impurities such as iron and titanium oxides and to enhance the refractory property of the clays. In addition, geological survey of the study areas could also help to map out the substantiality of the deposits and inform the basis to building and designing refractory product markets in Northern Nigeria. This will, in a short run create more jobs opportunities and in a long run, save huge foreign exchange yearly by drastically reducing the importation of similar product materials and revitalized the fortunes for entire region and the country in a whole.

ACKNOWLEDGEMENT

The Authors wish to acknowledge Prof. M.B Dalen (of blessed memory) who supervised this research work, gave guidance and constructive criticism that led to the successful completion of this work.

CONFLICT OF INTERESTS

The authors declare no conflict of interests.

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