EVALUATION AND CHARACTERIZATION OF REFRACTORY PROPERTIES OF SOME CLAY DEPOSITS IN NORTH – CENTRAL NIGERIA

BY

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2006/24205EH

DEPARTMENT OF CHEMICAL ENGINEERING,

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FEDERAL UNIVERSITY OF TECHNOLOGY, MINNA,

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FEDERAL UNIVERSITY OF TECHNOLOGY, MINNA,

NIGER STATE.

IN PARTIAL OF REQUIREMENT FOR THE AWARD OF BACHELOR OF ENGINEERING (B.ENG) DEGREE IN CHEMICAL ENGINEERING.

NOVEMBER,2011

CERTIFICATION

This project is an original work done by **Muhammad Bashir Sambo** with Matriculation Number: **2006/24205EH**, under the supervision of Engr. Musa Umar to meet up with the standard and scope in compliance with the requirements of Chemical Engineering Department, Federal University of Technology, Minna, Niger State, for the award of the degree of Bachelor of Engineering (B. Eng. Hons.) in Chemical Engineering.

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Head of Department

Date

External Supervisor

22/02/2012
Date

DEDICATION

I dedicate this work to my parent Fatima Muhammad Sambo, Alhaji Ibrahim sambo and my siblings; Umar, Abubakar, Maimuna, Usman, Aliyu, Ibrahim and Hafsat.

AKNOWLEDGEMENT

All praise are due to Allah (SWT) the lord of all creations for spearing my life and granting me good health to carry out this work successfully. My profound gratitude to my parents and siblings for being a source of inspiration and courage all through my stay in school. I acknowledge and appreciate my Supervisor Engr. Musa Umar for drilling me in the act of technical and project report writing and also for his advise and counselling. Also, my unreserved appreciation goes to my colleagues and friends: Umar Kure, Ahmed Abdullah, Mustapha Abubakar and Rachael Ndagi. For their contributions and support towards this work

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ABSTRACT

Nigeria is endowed with vast deposits of clay spread across her various regions. Today, almost all the clay requirement of the country in the form of refractory and bentonite are imported from the Great Britain, China and Japan leading to high cost of procurement and consequently result to high cot of finished products. This work is aimed at investigating the refractory properties of locally available clays for adoption in our industries. Tammah, Bukuru, Otaiko and Mekunkele clay were collected from Nassarawa, Plateau, Kogi and Niger state respectively. The properties investigated include Bulk density, apparent porosity, Linear shrinkage, Thermal shock resistance, Cold crushing strength, and loss on Ignition. The Refractoriness was estimated using the Shuen' formula while the chemical composition was determined using the Atomic Absorption Spectrophotometer (AAS) mode 210 VGP. Based on the chemical composition analysis, the clays were found to be Aluminosilicate fireclay and from the result obtained From the physical test, almost all the properties investigated gave results that are acceptable for clay refractory except the cold crushing strength of all samples fall below the minimum specification which could be attributed to inadequate power supply that resulted to inconsistent firing. Findings shows that the Tammah, Bukuru and Mekunkele Clays can be use for refractory purposes such as firebricks, ramming masses, linings of iron and steel making furnaces and ceramic product such as tiles and furnace crucibles.

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CHAPTER ONE

1.0 INTRODUCTION

Clay is a fine textured earth that is plastic when wet but hard and compact when dry, it is used to refer to the finest grain particles in a sediment, soil or rock. clay occurs most abundantly in nature soils, sediments and sedimentary rock (Peter *et al*,2007). Clay is a natural source of many industrial product, one of such product which have proved indispensible in the industries is the refractory material (Ameh and Obasi,2009). Refractories are inorganic compounds mainly of clay origin that are capable of withstanding physical, chemical and corrosive actions without deterioration (http//.PDHonline.org) the main function of a refractory is to withstand and maintain high temperatures and resist the abrasive and corrosive action of molten metal, slag and gasses. Depending upon the application, refractories must resist destructive influences such as abrasion, chemical attack, thermal shock, physical impact, catalytic heat and similar conditions generally at high temperatures (Kumar,2011). Abundant deposits of clay have been reported across major geological belt in the country, some work have been done at different times to evaluate the basic refractory properties in some clay from certain deposits around the country with a view to determine their suitability for adoption as refractory materials for different industrial application (Amuda *et al.*, 2008).

Chukwudi (2008), characterised and evaluated the refractory properties of Nsu clay in Imo state southeast Nigeria and found it suitable for use as ramming mass, ladle bricks and insulation for reactors and boilers. Amuda *et al* (2005), characterised and evaluated the refractory properties of some clay from deposits in southwest Nigeria and found out blending those clay samples in various proportions produced good refractory materials. Abolarin *et al* (2004), investigated clay samples from deposits in Bauchi and Plateau states as local refractory materials for furnace construction, they found out the clay samples could be used as refractory materials because they conform to standard values for refractoriness. Peter *et al* (2007), characterized clay from Mayo-Belwa clay deposit in Adamawa state and found it suitable for use as a source of silica for silica floor tiles and also as a binder in the absence of standard binder. This work is aimed at investigating properties of our local refractory materials and comparing them with existing standards with a view to adopt them for our industrial needs.

1.1 Aim and Objectives

This work is aimed at characterizing and evaluating the refractory properties of some clay deposits in North central geographical region of Nigeria. This aim can be achieved to the realization of the following objectives;

- Determination of physical properties of the clay samples.
- Determination of chemical properties of the clay samples.
- Comparison of results with relevant standard and related literature

1.2 Justification of the work

There are vast deposits of clay spread across every region in Nigeria, although their property differ from site to site on account of geological differences. Ironically, the bulk clay requirement of the country is sourced abroad. Chukwudi (2008) reported that virtually all the refractory requirement of the pyro-metallurgical industry in Nigeria are imported and that the Nigerian metallurgical industries are struggling today as a result of many factors which includes short supply of refractory materials. Amuda *et al* (2005) reported that the four refineries in Nigeria gulped about 850 m United states dollar (USD) for their turn around maintenance (TAM) in the last five years, the critical unit in the TAM unit is the fluid catalytic cracking unit (FCC) this unit is lined with various quantity of enormous grades of refractory linings, these refractory materials are presently sourced abroad. Also, the demand for clay refractory materials is projected to surpass the demand for non-clay refractories world wide by 2015. In view of all these facts, it has become imperative to study and adopt local clay materials as refractory materials in our industries thereby enhancing the local content initiative of the federal government and saving millions of dollars used in clay importation and ultimately boosting the economy.

1.3 Scope of the study

The scope of this study is strictly limited to the physio – chemical characterization of clays from some specific locations within the north central geographical region of Nigeria having abundant clay deposits.

1.4 Problem statement

Nigeria is endowed with vast deposits of clay spread across her various regions. Today, almost all the clay requirement of the country in the form of refractory and bentonite are imported from the Great Britain, China and Japan leading to high cost of procurement and consequently result to high cot of finished products. This doesn't tell well of a country that seeks to become one of the top twenty economies in the world.

CHAPTER TWO

2.0 LITERATURE REVIEW

2.1 History of Refractory

Materials that can withstand very high temperature are known as refractory materials. The main function of a refractory is to withstand and maintain high temperatures and resist the abrasive and corrosive action of molten metal, slag and gasses. depending upon the application, refractories must resist destructive influences such as abrasion, pressure, chemical attack, thermal shock, physical impact, catalytic heat and similar conditions generally at high temperatures (Kumar, 2011)

In the ancient time (especially with the discovery of fire) men have always faced necessity for materials resistance to fire and consequently high temperatures. There have been increases in the form and variety of fire resistant materials with the rapid development of technology in parallel with evolving civilisation. The materials which were amorphous and were used without being baked at first, were later observed to be transformed into materials with high strength and performance in the application after being moulded into shape and baked the later applications have continued to develop in this direction. Consequently, pressed and baked materials with much more strength have been obtained. Together with rapid industrialisation process manufacturing industries which could enable consistency in production of such materials have emerged. Areas used depending on the developing technology, refractory materials is needed in any area where high temperature is needed in any area where high temperatures exist, its area of use covers a wide range from domestic or office stores operating with coal , fuel oil, electricity. Fuel energies that may be deemed to the smallest furnace to such furnace used in heavy industries and capable of reaching very high temperature (http://britanica.com/eb/article/refractory).

2.2 Classification of Refractories

2.2.1 Based On Chemical Composition

Refractories can be classified on the basis of chemical composition which classified them into three categories acidic, basic and neutral refractories (Kumar, 2011).

2.2.1.1 Acidic refractories

Acidic refractories consist of acidic materials like silica (SiO_2). They are not attacked by acidic materials, but easily attacked by basic materials (Kumar,2011). The main raw materials belong to the group RO_2 group e.g Silica (SiO_2), zirconia (ZrO_2), and Alumino-silicate clay ($Al_2O_3.2SiO_2.2H_2O$), (Chukwudi, 2008).

2.2.1.2 Neutral refractories

Neutral refractories are either basic or acidic, and are chemically stable to both acids and bases(Kumar, 2011). The main raw materials belong to the R_2O_5 group, the common examples of these materials are Alumina (Al_2O_3), Chrome (Cr_2O_3) and carbon (C), (http://PDHonline.org)

2.2.1.3 Basic refractories

Basic refractories are used in areas where slag and atmosphere are basic and stable to alkaline materials. They belong to RO group to which Magnesia is very common example include Dolomite and chrome magnesites. Basic were so named because they exhibit resistance to corrosive reactions with chemically basic slag , dust and fumes at elevated temperatures (http://PDHonline.org).

2.2.2 Classification Based on Physical Form

Refractories are classified according to their physical form. These are the shaped and unshaped refractories. The former is commonly known as refractory bricks and the latter as "monolithic"refractories (http://PDHonline.org)

2.2.2.1 Shaped refractories:

Shaped refractories are those, which have fixed shaped when delivered to the user. These are what we call bricks. Brick shapes maybe divided into two: standard shapes and special shapes. Standards shapes have dimension that are conformed to by most refractory manufacturers and are generally applicable to kilns and furnaces of the same type. Special shapes are specifically made for particular kilns and furnaces. This may not be applicable to another furnaces or kiln of the same type. Shaped refractories are almost always machine-pressed, thus, high uniformity in properties is expected. Special shapes are most often hand-molded and are expected to exhibit slight variations in properties (http://PDHonline.org).

2.2.2.2 *Unshaped refractories*

Unshaped refractories are without definite form and are only given shape upon application. These types are better known as monolithic refractories. These are categorized as follows;

Plastic refractories: Plastic refractory is mixtures that is prepared in stiff plastic condition and are delivered in blocks wrapped in polyethylene. During application, the blocks are sliced into pieces and without further preparation, are rammed or poured into place with pneumatic rammer. Plastic are easily rammed to any shape or contour (http://PDHonline.org).

Ramming mixes: Ramming refractory materials are those whose particle sizes are carefully graded to make it workable. These are usually delivered dry and then mixed with a little amount of water just before application. Other ramming mixes are delivered in wet form and are ready for use immediately upon opening. Application is done with pneumatic rammer (http://.PDHonline.org).

Castables: 'Castables' by name implies a material of hydraulic setting in nature. These are the materials that contain cement binder usually aluminates cement, which imparts hydraulic setting properties when mixed with water. Calcium aluminates binder material needs to be stored properly to prevent moisture absorption. Further its strength starts deteriorating after a period of 6 to 12 months. These materials are installed by casting and are also known as refractory concrete (http://PDHonline.org).

Gunning mixes: Gunning mixes are granular refractory materials sprayed on application area using a variety of air placement guns. These are heat setting and are used for patching and maintenance works for kilns and furnaces (http://PDHonline.org).

Fettling mixes: Fettling mixes are also granular refractory materials, similar to gunning mix function, but are applied by shovelling into the furnace needing patching (http://PDHonline.org)

Mortars: Mortars are generally neither classified under refractory brick nor monolithic refractories. These are finely ground refractory materials, which become plastic when mixed with water. These are used to bond the brickwork into solid unit, to provide cushion between the slightly irregular surfaces of the brick, to fill up spaces created by a deformed shell, and to make a wall gas-tight to prevent penetration of slag into the joints (http://PDHonline.org).

2.3 Clay-based Refractories

Most refractories can be classified on the basis of composition as either clay-based or non-clay refractories. There is wide range of non-clay refractories including basic, extra high-Alumina, silica, silicon carbide and zirconia. Most clay-based refractories are processed in a manner similar to other traditional ceramics such as structural clay (stiff-mud processes such as press foaming or extrusion are employed to form the wares, which is subsequently dried and passed through long tunnel kilns for firing), (Chukwudi, 2008). The most important clay mineral for use in refractory is kaolinite. A clay particle does not develop its full strength until it has vitrified. The vitrification range starts at about 900 ^{o}C and extends up to the highest temperature the clay can withstand without melting.

Depending upon the kind of clay minerals and the impurities present the upper temperature limit of the vitrification ranges may vary from below 1000 °C to over 1500 °C. During the vitrification period the porosity of the mass decreases and its strength and hardness increases (Al-Amaireh, 2009). Among the clay-based refractories are fireclay, high-Alumina and mullite (Chukwudi, 2008).

2.3.1 Fireclay

The pillar of the so called clay-based refractories are the so called fireclay materials. These are made from clay containing the aluminosilicate mineral-kaolinite ($Al_2[Si_2O_5][OH]_4$) plus impurities such as alkalis and iron Oxides. The alumina content ranges from 25 to 45 percent depending upon the impurity content and the alumina-to-silicon ratio (Chukwudi, 2008). Fireclay is non-white burning clay, which is free from fluxes such as larger quantities of iron, alkaline earth, alkalis and excess silica. Its main constituent are minerals of the kaolinite group.(Al-Amaireh, 2009). Fireclay are classified as: low-duty, medium-duty, high-duty and super-duty fireclay (http://PDHonline.org).

2.3.1.1 Super-duty fireclay

Super-duty fireclay have good strength and stability of volume at high temperature and an high alumina content of 40 to 44 percent, some super-duty bricks have superior resistance to cracking or spalling when subjected to rapid changes of temperature (http://PDHonline.org).

2.3.1.2 High-duty fireclay

These are used in large quantity and for a wide range of applications because of their greater resistance to thermal shock, high- duty fire clay can often be used with greater economy than medium-duty bricks for the linings of furnaces operated at moderate temperature over a long period of time but subject to frequent shutdowns (http://PDHonline.org).

2.3.1.3 Medium-duty Fireclay

These bricks are appropriate in applications where they are exposed to conditions of moderate severity. Medium duty refractories within their serviceable temperature ranges can withstand abrasion better than many bricks of the high duty class (http://PDHonline.org).

2.3.1.4 Low-duty Fireclay

The low-duty fireclay find application as backing for brick with higher refractoriness and for other services where relatively moderate temperature prevails (http://PDHonline.org).

2.3.2 High Alumina Refractories

High alumina refractories are made from bauxite, a naturally occurring material containing hydroxide (Al [OH]$_3$) and kaolinitic clay (Chukwudi, 2008). The term high alumina refers to refractory bricks having an aluminium content of 47.5 % or higher. The alumina concentration ranges from 45 % to 100 %. The refractoriness of high alumina refractories increases with increase of alumina percentage. The 50 %, 60 %, 70 % and 80 % alumina classes contain their respective alumina content with an allowable range of plus or minus 2.5 %. high alumina bricks are classified by their alumina content according to the following American Society for Testing and Materials (ASTM):

- Mullite brick predominantly contains the mineral phase mullite.
- Chemically-bonded bricks usually phosphate bonded brick in the 75 % to 85 % alumina range.
- Alumina-chrome brick typically from very high purity, high-alumina materials and chromic oxide. At high temperature, alumina and chromia form a solid solution which is highly refractory.
- Alumina-carbide brick high alumina brick (usually bonded by resins) containing a carbonaceous ingredient such as graphite (http://PDHonline.org).

The application of high-alumina clay refractories includes the hearth and shaft of blast furnace, ceramic kilns , glass tanks and crucibles for melting wide range of metals (http://PDHonline.org).

2.2.3 Mullite

Mullite is an Alumino-silicate compound with the specific formula $3Al_2O_3.SiO_3$ and an alumina content of approximately 70 %. It has a melting point of 1850 °C. Various clay are mixed with bauxite in order to achieve this composition. They are the most stable of the aluminosilicate refractories and have excellent resistance to high temperature loading (Chukwudi, 2008).

2.4 Properties of Clay Refractory

Important properties of clay refractories which can be determined most readily are; bulk density, apparent porosity, plasticity and chemical composition. these properties are among those which are used as controls in the manufacturing and quality control process. The chemical composition serves as a basis for classification of refractories. The density , porosity and strength of fired product are influenced by many factors among these are type and quality of raw materials. The size and fit of the particles, moisture content at the time of pressing, temperature and duration of firing, kiln atmosphere and the rate of cooling (http://PDHonline.org).

2.4.1 Bulk Density

It is a useful property for refractories, it defines the material present in a given volume. An increase in bulk density of a given refractory increases its volume stability, its heat capacity as well as its resistance to slag penetration. The standard bulk density for general refractory is within the range 1.7 - 2.1 g/cm^3(Abolarin *et al.*, 2004). Al-Amaireh (2009) determined the bulk density refractory bricks made from fireclay and found it to have lower bulk density of 1.73 g/cm^3 as the binder clay content in the mix drops below 30 %. Amuda *et al* (2005) determined the bulk density of some clay samples from deposits in southwest Nigeria and found their bulk density not to fall within standard value of range 1.7-2.1 g/cm^3 as given by Chester (1973).

2.4.2 Apparent Porosity

The apparent porosity is a measure of the volume of the open pores into which a liquid can penetrate as a percentage of the total volume. This is an important property, a low apparent porosity is desirable since it would prevent easy penetration of the refractory size and continuity of pores will have important influences on refractory behaviour. A large number of small pores is generally preferable to an equivalent number of large pores. In fact, porosity, bulk density and apparent solid density have been termed "vital statistics" of refractory shapes (http://PDHonline.org). Chukwudi (2008) determined the apparent porosity of the Nsu clay and found it to have a value of 22.56 % which is within the acceptable range (10 – 30 %) suggested for refractory clays by Chester (1973).

2.4.3 Linear Shrinkage

This is the water content defined at degree of saturation below which no further soil volume change occurs with further drying (Abolarin *et al.*, 2004). In clay, changes in moisture content are accompanied by changes in volume. Tariful *et al* (1999). Mavakumba et-al determined the linear shrinkage of Mayo-Belwa clay in Adamawa state and found it to have an average shrinkage of 10.5 %. Tariful et al (1999) modified the American Society for Testing and Materials (ASTM) technique for measuring shrinkage of clay using sand instead of wax and mercury as used by the ASTM, they found there is no significant difference between their modified method and the method used by the ASTM.

2.4.4 Thermal Shock Resistance

Thermal shock resistance is the number of cycles (heating at high temperature) clay refractory can withstand before fracture, Amuda *et al* (2005).it is the number of cycles needed to cause conspicuous crack on the clay samples Chukwudi (2008). Ndaliman (2007) determined the thermal shock resistance of ant hill clays under varied proportion of additives and found them to resist spalling up to 24 cycles.

2.4.5 Pyrometric Cone Equivalent (PCE)

The temperature at which a refractory will deform under its own weight is known as the softening temperature, which is indicated by Pyrometric cone equivalent (PCE). refractories due to their chemical complexity, melt progressively over a range of temperature. The Pyrometric cone equivalent indicates only the softening temperature. But in service, the refractory is subjected to load which would deform refractory at a much lower temperature than that indicated by the PCE.

2.4.6 Size

The size and shape of the refractories is a part of the design features. It is an important feature in the design since it affect the stability of any structural accuracy and size is extremely important to enable proper fitting of the refractory shape and to minimise the thickness and joints in construction (http://PDHonline.org)this property (size) has significant influence on the moulding strength as well as permeability of the moulding clay. The fine grained soil has lower permeability than coarse grained particles large particle size leads to a poor surface finish (Abolarin *et al.*, 2004).

2.4.7 Cold Crushing Strength

The cold crushing strength shows the ability of the clay to withstand abrasion and loading (Abolarin *et al.*, 2004). It can be used as a useful indicator to the adequacy of firing and abrasion resistance in consonance with other properties such as bulk density and porosity (http://PDHonline.org). Chester (1973) recommended 5 MPa as the minimum value for refractory clay materials. Al-Amaireh (2009) determined the compressive strength of fireclay refractory bricks and found it to have a value of 300 kg/cm^2 (30 MPa).

2.4.8 Refractoriness

The refractoriness under load test (RUL test) gives an indication of the temperature at which the bricks will collapse, in service conditions with similar load. However, under actual service where the bricks are heated only on one face, the relatively cooler rigid portion of the bricks carries most of the load. Hence the RUL, test gives only an index of the refractory quality, rather than a figure, which can be used in a refractory design. Under service conditions, where the refractory used is heating from all sides such as checkers, partition wall etc. The RUL test data is quite significant (http://PDHonline.org). The refractoriness can also be estimated using the Shuen's formula

$$k = \frac{360 + Al_2O_3 - RO}{0.228} \qquad 2.1$$

Ameh and Obasi (2009) determined the refractoriness of Nsu clay in Imo state using the Shuen's formula and found it to be almost the same as value obtained experimentally by Chukwudi (2008) when he determined the refractoriness of the Nsu clay.

2.4.9 Creep at high temperature:

Creep is a time dependent property, which determines the deformation in a given time and at a given temperature by a material under stress. The criterion of acceptance usually adopted is; that compressive creep under the required conditions of load and temperature shall not exceed 0.3 % in the first 50 hours of the test. This figure has been found to indicate that the creep rate falls by a negligible amount at the end of the stipulated period, and therefore the refractory can be considered safe to use for a much longer time. (http://PDHonline .org)

2.4.10 Volume stability, Expansion and shrinkage at high temperature:

The contraction or expansion of the refractories can take place during service. Such permanent changes in dimensions may be due to; the changes in the allotropic forms which cause a change in specific gravity, a chemical reaction which produces a new material of altered specific gravity. The formation of liquid phase, Sintering reactions It may also happen on account of fluxing with dust and slag or by the action of alkalis on fireclay refractories, to form alkali-alumina silicates, causing expansion and disruption. This is an example, which is generally observed in blast furnaces. While it is desirable that all these changes are effected during manufacturing, it is not possible due to economic reasons, as the process is time dependent. Permanent Linear Change (PLC) on reheating and cooling of the bricks give an indication on the volume stability of the product as well as the adequacy of the processing parameters during manufacture. (http://PDHonline.org).

2.4.11 reversible thermal expansion

Any material when heated expands, and contracts on cooling. The reversible thermal expansion is a reflection on the phase transformations that occur during heating and cooling. The PLC and the reversible thermal expansion are followed in the design of refractory linings for provision of expansion joints. As a general rule, those with a lower thermal expansion co-efficient are less susceptible to thermal spalling. (http://PDHonline .org)

2.4.12 vitrification range

Vitrification is the transition of a substance into a glass. The temperature range of vitrification or glass internation is a very important property in structural products. Vitrification is due to the process of gradual fusion in which some of the more easily melted constituent begging to produce an increasing amount of liquid which makes up the glass bonding materials in the final fired products. Some clay has a short vitrification range, so that the temperature of kiln is closely regulated. The degree of vitrification is regulated by amount shrinkage, and reduction of porosity that can be obtained at a given temperature (Isah, 2010).

CHAPTER THREE

3.0 MATERIALS AND METHOD

Table 3.1 List of Equipments

Equipment	Manufacturer	Source
Extruder	ELE International Limited England	Civil Engineering Laboratory FUT Minna.
Furnace	Tactical 308	Chemistry Laboratory FUT Minna.
Heating Mantle	United National Inventory	Chemistry Laboratory FUT Minna.
Oven	ELE International Limited England	Civil Engineering Laboratory FUT Minna.
Atomic Absorption Spectrophotometer	Mode 210 SVG	Science Laboratory Technology Department Federal Polytechnic Bida Niger state.
Compressive Testing Machine	ELE International Limited England	Civil Engineering Laboratory FUT Minna.
Beakers and Desiccators	Pyrex England	Chemistry Laboratory FUT Minna.

3.1 Sample Collection

The basic raw materials used in this work are clay procured from four different locations within the north central geographical region of Nigeria. These deposits are Tammah (Nassarawa state), Otaiko (Kogi state), Bukuru (Plateau state) and Mekunkele (Niger state) and the clay are thus called Tammah, Otaiko, Bukuru and Mekunkele clays respectively.

3.2 Sample preparation

The clays from the various deposits were crushed and ground to fine grains, The samples were moulded with adequate amount of water and then moulded into test laboratory sizes of rods of length 8.5 cm after which they were air dried for twenty four hours, the linear dried dimension of each sample was measured and recorded after air drying overnight. The clay rods were oven dried at a temperature of 110 ^{0}C for 24 hours. The rods made from this samples were then fired in an electric muffle furnace at 1000 ^{0}C for 8 hours. The firing was done at a slow rate to avoid the cracking of the rods. The rods were cooled to room temperature in desiccators thereby making the rods ready for testing.

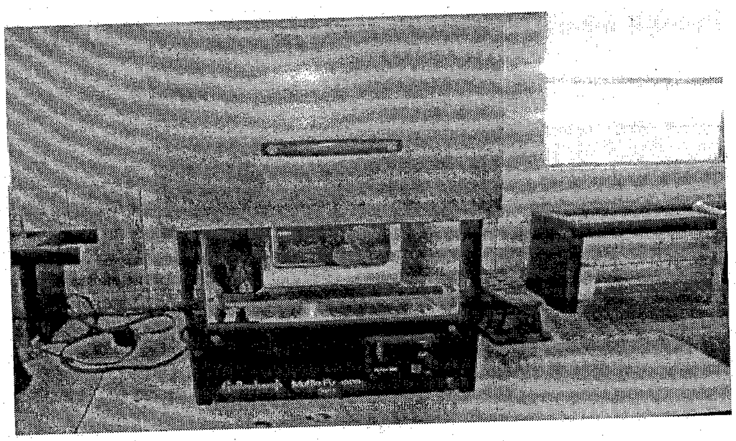

Plate I; Firing Clay Samples

3.3 Experimental Procedures.

3.3.1 Determination of bulk density

Procedure: The weights of the test samples were measured and recorded as the dry weights (D) of the samples after which the samples were individually transferred to a beaker and heated for thirty minutes to assist in releasing trapped air. The samples were allowed to cool inside the water after which soaked weights (W_S) for each sample was measured, the samples were then suspended in water and the suspended weights (S_S) were measured and recorded. The bulk density was calculated from;

$$B_D = \frac{D\rho_w}{W_s - S_s} \qquad 3.1$$

Where D is the dried weight, W_S soaked weight and S_S suspended weight all in gramme (g).

3.3.2 Determination of thermal shock resistance

Procedure: The test samples were inserted into a furnace that is kept at 1100 ^{0}C. This temperature was maintained for 10 minutes. The samples were removed one after the other and then cooled in water for five minutes. the specimens were returned to the furnace for a further 10 minutes. The process was continued until conspicuous cracks appear on the samples. The number of cycles withstand before cracks appear was recorded for each sample.

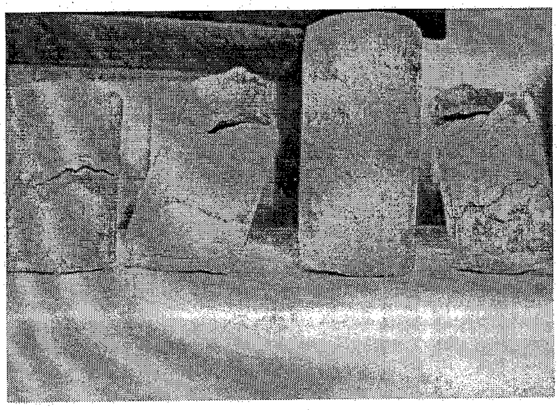

Plate II: Cracked Clay Sample Rods

3.3.3 Determination of cold crushing strength

Procedure: The samples were placed on a compressive testing machine and load applied axially by turning the hand wheel at a uniform rate until failure occurs, using a well calibrated scale, the cold crushing strength is determined using the formula;

$$CCS = \frac{maximum\ load}{cross\ sectional\ area} \left(\frac{KN}{m^2}\right) \qquad 3.2$$

3.3.4 Determination of linear shrinkage

Procedure: the length of the cylindrical mould was measured as the original length of each of the sample. The samples were air dried for twenty four hours. The linear dried dimensions were measured after air drying overnight. The linear shrinkage was determined using the relationship;

$$LS = \frac{\Delta L}{L} \times 100 \qquad 3.3$$

Where ΔL is change in length of the rod and L is the original length of the test rod.

3.3.5 Determination of the apparent porosity

Procedure; The dried weights of the samples were weighed and recorded , the samples were each placed inside beakers of boiling water and heated for twenty minutes . the samples were allowed to cool while still in water. After which the weight of the soaked sample (Ws) was taken. The samples were removed from water and cleaned up and weighted again in air to know the saturated weight (Wss). Apparent porosity was determined using the formula:

$$\text{Apparent porosity} = \frac{W_{ss}-W_a}{W_{ss}-W_s} \qquad 3.4$$

where Wa = weight of the sample dried in the air. Ws = weight of the soaked sample .Wss = saturated weight of the sample in the air.

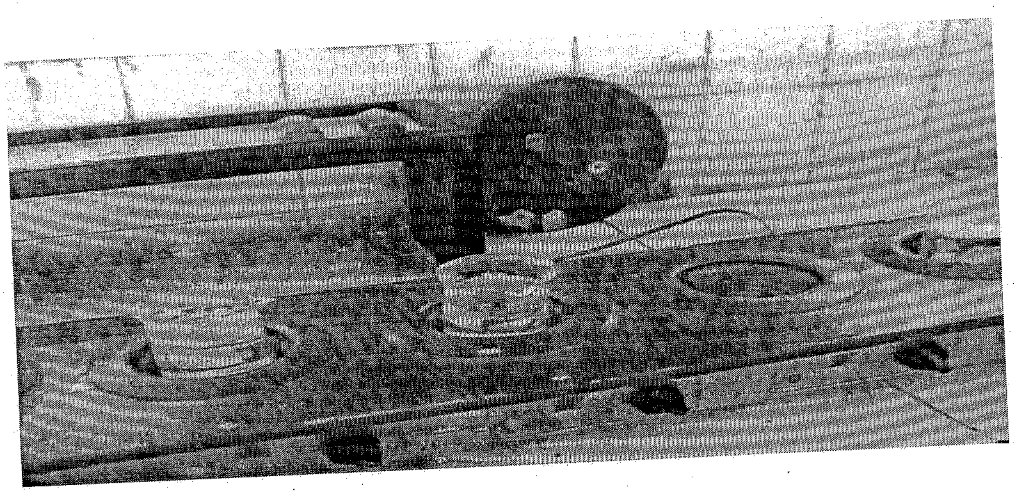

Plate III: Boiling water to determine apparent porosity

3.3.6 Determination of Chemical composition

Procedure; 10 g of each of the raw clay samples was dissolved in 150 ml of water. Each of the dissolved sample was transferred into plastic bottles and shaken by a flask shaker for 45 min. The samples were filtered and the filtrates were taken for compositional analysis using the atomic absorption spectrophotometer mode 210 VGP situated at the science laboratory technology department of the federal polytechnic Bida Niger state.

CHAPTER FOUR

4.0 RESULTS AND DISCUSSIONS

4.1 Results

The following tables show results of the tests carried out on the samples

Table 4.1 Chemical Composition of Samples

Clay sample	Oxides					
	Al_2O_3	SiO_2	Fe_2O_3	MgO	CaO	Na_2O
Tammah	27.480	56.430	0.000	0.106	0.003	0.643
Otaiko	22.310	54.200	0.042	0.158	0.013	1.252
Bukuru	26.830	47.800	0.000	0.025	0.009	0.241
Mekunkele	30.240	49.710	0.000	0.128	0,000	0.232

Table 4.2 Refractory Properties of Clay Samples

Properties	Tammah	Otaiko	Bukuru	Mekunkele
Apparent Porosity (%)	30.69	30.75	15.32	28.09
Bulk Density (g/cm^3)	3.10	1.96	1.98	2.19
Cold Crushing Strength (KN/m^2)	11214.95	1601.42	13725.49	13445.73
Linear Shrinkage (%)	7.41	4.37	7.76	9.41
Loss on Ignition (%)	4.75	7.85	2.37	3.07
Refractoriness (^0C)	1696.18	1661.60	1694.10	1710.00
Thermal Shock Resistance (cycles)	22	17	20	28

4.2 Discussion of Results

Table 4.1 show the result of the chemical compositions of the Clay samples. The silica content of the clay samples are 56.43, 54.2, 49.80 and 47.80 % and the aluminium content are 27.48, 22.31, 26.83 and 30.24 % for Tammah, Otaiko, Bukuru and Mekunkele samples respectively, this shows the clay belong to the aluminosilicate family and the Tammah, Bukuru and Mekunkele clays belong to the fireclay group as their aluminium content falls within the classification for fireclay (25 % – 45 %) given by Chester (1975).

The presence of alkalis and iron oxide denote the presence of impurities in Clays (Chukwudi,2008). Table 4.1 denote Tammah, Bukuru and Mekunkele Clay samples to have no trace of iron Oxide but the Otaiko Clay sample have an iron Oxide content of 0.042 %. Furthermore, the calcium content of the Clay samples are 0.003, 0.013, 0.009 % for Tammah, Otaiko and Bukuru clay samples respectively While the Mekunkele clay sample does not have trace of calcium Oxide. The result also show the Magnesium contents to be 0.108, 0.158, 0.025 and 0.128 % respectively for the Tammah, Otaiko, Bukuru and Mekunkele clay samples. The sodium content in the clay samples are 0.643, 1.252, 0.241 and 0.232 % respectively for Tammah, Otaiko, Bukuru and Mekunkele clay samples. These values of the alkali and iron Oxide denote the clay samples to have low composition of impurities which means clays from these deposits can be processed as refractories without chemical purifications.

The standard bulk density for general refractory as reported by *Abolarin et al* (2004) is within the range 1.7-2.1 g/cm^3. As shown on table 4.2 Otaiko and Bukuru clays fall within the standard range with bulk density of 1.96 g/cm^3 and 1.98 g/cm^3 respectively. The Mekunkele sample have bulk density 2.19 g/cm^3 which is slightly above the standard range recommended by (*Abolarin et al*,2004) but is less than 2.26 g/cm^3 obtained for Mayo – Belwa Clay by Peter *et al* (2007) and below 2.5 g/cm^3 reported for Nafuta Clay by Ameh and Obasi (2009). The Tammah sample have bulk density of 3.10 g/cm^3 which is outside the standard range recommended by Chester (1975). But is still within the acceptable range of 3.5 % recommended by Waing (2009). An increase in bulk density of a given refractory increases its volume stability, heat capacity and resistance to slag penetration (Waing *et al,*2008). Hence, the results of the bulk density show that the clays are suitable for use as linings of furnace walls.

Lower apparent porosity denote good heat conductivity (Isah,2010). Table 4.2 show the apparent porosity of the samples to be 30.69, 30.75, 15.32 and 28.09 % for Tammah, Bukuru, Otaiko and Mekunkele respectively Mekunkele and Bukuru sample porosity are consistent with the standard of 10 % - 30 % recommended by Chester (1973) while the Tammah and Otaiko sample have apparent porosity slightly above the standard range recommended by Chester but are less than 38.74 % obtained by Ameh and Obasi (2009) for Nafuta Clay. This means Bukuru and Mekunkele samples have better thermal conductivity and therefore suitable in making insulated fire bricks.

The result of the linear drying shrinkage in table 4.4 shows the clay samples linear shrinkage as 7.41, 4.37, 7.76 and 9.41 % for Tammah, Otaiko, Bukuru and Mekunkele samples respectively. Chester (1973) recommended that standard linear shrinkage to fall between 7 – 10 %. Tammah, Bukuru and Mekunkele samples are consistent with the values recommended by Chester (1973) which means they don't expand or shrink excessively while the Otaiko sample fall below the standard range recommended by Chester (1973) but is above 4.25 % obtained for Mayo – Belwa clay and above 2.7 % reported by *Abolarin et al* (2004) for Alkaleri Clay. It is crucial that refractories should not shrink or expand excessively otherwise cracking and collapse could occur where they are used as furnace walls Okafor (2007). This result show that the clay samples are suitable for use in making firebricks, because Shrinkage is the property of clay that is important for brick making (peter *et al.*,2007)

High cold crushing strength indicates good resistance to abrasion and more resistance to impact. from table 4.5, Bukuru, tammah and mekunkele sample show fair cold crushing strength of 13725.49.KN/m^2, 11214.95 $KN/m^2$and 13445.73 KN/m^2 respectively while the otaiko sample have the least value of cold crushing strength of 1601.42 KN/m^2. The minimum value for cold crushing strength recommended by De Bassy (1972) is 15000 KN/m^2. All the samples fall below the minimum value, this may be as a result of inconsistent firing due to fluctuation in power supply.

Thermal shock resistance determine the ability of a substance to withstand heating and cooling process (*Abolarin et al*,2004). The result for linear shrinkage depicted on table 4.2 Bukuru, Mekunkele and Tammah samples were found to have good thermal shock resistance with 20, 28 and 22 cycles respectively which falls within the standard range of 20 – 30 cycles recommended by Chester (1973), the Otaiko sample fall outside this range with 17 cycles Which makes it to have fair thermal shock resistance when compared with 8 cycles reported by Onyeji (2010) for Nyikangbe clay.

Refractoriness is the temperature at which all the properties of a refractory fail. Table 4.2 shows the refractoriness of the samples to be 1696.18, 1661.60, 1694.10 and1710 °C for Tammah, Otaiko, Bukuru and Mekunkele samples respectively. The results are consistent with the standard 1500 °C – 1700 °C recommended by Chester (1973). This imply that the clay samples have good vitrification temperature and therefore can be used as ramming mixes, ladles, furnace walls and insulation bricks. The refractoriness of the clay samples could not be determined experimentally because test samples were fired to limited temperature 850 – 1200 °C but were estimated using the Shuen's formula as used by Ameh and Obasi (2009) in determining the refractoriness of Nsu clay and found it to be 1671 °C which was about the same value obtained by Chukwudi (2008) when he determine the refractoriness of Nsu clay experimentally to be 1683 °C.

CHAPTER FIVE

5.0 CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

The clays from the four deposits were analysed to be aluminosilicate fireclay. It was shown in this work that on the basis of the physio – chemical characteristics of these fireclay deposits, they can be successfully used for refractory purposes like firebricks, ramming masses, linings of iron and steel making furnaces and ceramic product such as tiles and furnace crucibles. almost all the properties investigated gave results that are acceptable for refractory clay except the cold crushing strength which all fall below the minimum which could be attributed to inadequate power supply that resulted to inconsistent firing.

5.2 Recommendation

- Further work on the production of firebricks should be carried out on the mekunkele, bukuru and tammah samples.
- Also, work on production of floor tiles with the Mekunkele clay should be carried out.
- Refractory properties of the clay samples can be enhanced by the use of additives such as asbestos, rice husk, graphite and coconut shell.
- There is need for geological survey to determine the extent of the four clay deposits. Also the respective state governments should channel resources towards harnessing the potential of the Tammah, Bukuru and Mekunkele clay deposits respectively so as to promote the local content initiative and thereby boosting the economic base of the various states.

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APPENDIX

Apparent porosity AP = $\frac{W_{ss}-W_a}{W_{ss}-W_s}$

Wa = weight of the sample dried in the air.

Ws = weight of the soaked

Wss = saturated weight of the sample in the air

$AP_T = \frac{166.30-145}{166.3-96.90} = 30.69\ \%$

$AP_O = \frac{164.80-146.50}{164.80-105.30} = 30.75\ \%$

$AP_B = \frac{158.0-145.8}{158.0-78.40} = 15.32\ \%$

$AP_M = \frac{150.50-134.80}{150.50-94.60} = 28.09\ \%$

Table 1 Result of Apparent Porosity of Clay Samples

Clay sample	Soaked weight(g)	Saturated weight(g)	Weight in air(g)	Apparent porosity(%)
Tammah	96.90	166.30	145.00	30.69
Otaiko	105.30	164.80	146.50	30.75
Bukuru	78.40	158.00	145.80	15.32
Mekunkele	94.60	150.50	134.80	28.09

Bulk Density $B_D = \frac{D\rho_w}{w_s-S_s}$

D = dry weight

W_S = soaked weight

S_S = Suspended weight

$$B_{DT} = \frac{145 \times 1.0}{158.3 - 112.6} = 3.10\ \%$$

$$B_{DO} = \frac{146.5 \times 1.0}{163.4 - 88.6} = 1.96\ \%$$

$$B_{DB} = \frac{145.8 \times 1.0}{167.4 - 93.8} = 1.98\ \%$$

$$B_{DM} = \frac{124.9 \times 1.0}{144.4 - 87.4} = 2.19\ \%$$

Table 2 Result of Bulk Density of Clay Sample

Clay sample	Dry weight (g)	Soaked weight (g)	Suspended weight (g)	Bulkdensity (g/cm^3)
Tammah	145.0	158.3	112.6	3.10
Otaiko	146.5	163.4	88.6	1.96
Bukuru	145.8	167.4	93.8	1.98
Mekunkele	124.9	144.4	87.4	2.19

Cold Crushing strength (CCS)

$$CCS = \frac{maximum\ load}{cross\ sectional\ area}\ (KN/m^2)$$

$$CCS_T = \frac{12}{1.07 \times 10^{-3}} = 11214.95\ KN/m^2$$

$$CCS_O = \frac{9}{5.62 \times 10^{-3}} = 1601.42\ KN/m^2$$

$$CCS_B = \frac{14}{1.02 \times 10^{-3}} = 13725.49\ KN/m^2$$

$$CCS_M = \frac{16}{1.19 \times 10^{-3}} = 13445.73\ KN/m^2$$

Table 3 Result of Cold Crushing Strength

Clay sample	Maximum load (KN)	Cross-sectional area (m^2)	Cold crushing strength (KN/m^2)
Tammah	12	1.07×10^{-3}	11214.95
Otaiko	9	5.62×10^{-3}	1601.42
Bukuru	14	1.02×10^{-3}	13725.49
Mekunkele	16	1.19×10^{-3}	13445.73

Linear Shrinkage $LS = \frac{\Delta L}{L} \times 100$

$$LS_T = \frac{8.5-7.87}{8.5} = 7.41\%$$

$$LT_O = \frac{8.5-8.13}{8.5} = 4.37\ \%$$

$$LT_B = \frac{8.5-7.84}{8.5} = 7.76\ \%$$

$$LT_M = \frac{8.5-7.6}{8.5} = 10.5\ \%$$

Table 4 Result of Linear Shrinkage of Clay Samples

Clay sample	Original length(g)	Final length(g)	Linear shrinkage(%)
Tammah	8.5	7.87	7.41
Otaiko	8.5	8.13	4.37
Bukuru	8.5	7.84	7.76
Mekunkele	8.5	7.6	10.5

Refractoriness $k = \frac{360 + Al_2O_3 - RO}{0.228}$

$$K_T = \frac{360+27.48-0.752}{0.228} 1696.18\ ^\circ C$$

$$K_O = \frac{360+22.31-1.423}{0.228} = 1661.6\ ^\circ C$$

$$K_B = \frac{360+26.83-0.275}{0.228} = 1694.10\ ^\circ C$$

$$K_M = \frac{360+30.24-0.36}{0.228} = 1710.00\ ^\circ C$$

Table 5 Result Refractoriness of Clay Samples

Clay sample	**Refractoriness (^oC)**
Tammah	1696.18
Otaiko	1661.60
Bukuru	1694.10
Mekunkele	1710.00

DECLARATION

I Muhammad Bashir Sambo declare that this project is solely the result of my work and has never been submitted for any degree. The literature cited have been duly acknowledged in the reference.

uhammad Bashir Sambo

tudent Name | Sign | Date