

**ANALYSIS AND CHARACTERIZATION OF NYIKANGBE CLAY,
CHANCHAGA LOCAL GOVERNMENT AREA, NIGER STATE.**

BY

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75%

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THE DEPARTMENT OF CHEMICAL ENGINEERING,

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NIGER STATE.

IN PARTIAL FULFILMENT OF REQUIREMENT FOR THE AWARD OF

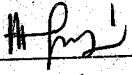
BACHELOR OF ENGINEERING (B.ENG) DEGREE IN CHEMICAL

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DECEMBER, 2009

DECLARATION

leniyi Adewale Amos hereby declare that this project is solely the result of my work and never been submitted for any degree. The literature sited has been duly acknowledged in reference.



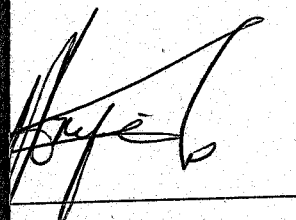
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CERTIFICATION

This research project is an original work done by Adeniyi Adewale Amos with Matriculation Number: 2005/21685EH, under the supervision of Engr Onyeji L.I. to meet 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.



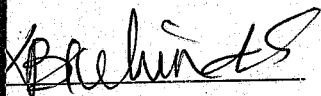
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DEDICATION

This project is dedicated to Almighty God, the only wise God our Saviour. May glory, dominion and power be given to Him for his protection, provision, and guidance throughout my university days and in the cause of this project work. I also dedicate this work to my father, Late Pa M.A Adeniyi of blessed memory, my mother and my brothers for their moral and financial support towards my academic advancement.

AKNOWLEDGEMENT

God be the glory, who gives me the privilege and divine grace to accomplish my academic pursuit. My profound gratitude goes to my supervisor, Engr. Onyeji, L.I. not only for his vast knowledge and wealth of experience but also for his encouragement and identified assistance which led to the successful completion of this project.

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ABTRACT

Characterization and analysis of Nyikangbe clay deposit have been carried out with the view of finding its possible industrial applications. The chemical analysis was carried out using X-ray Fluorescence Spectrometer. Physical property tests such as firing shrinkage, porosity, cold crushing strength, bulk density, thermal shock resistance and refractoriness were carried out using standard techniques.

The result of chemical analysis indicates that the clay is composed of silica (SiO_2)-54.03 %, alumina (Al_2O_3)-26.84 %, Iron oxide (Fe_2O_3)-11.93 %, Calcium oxide (CaO)-1.33 %, potassium oxide (K_2O)-3.60 %, Titanium oxide (TiO_2)-1.85 % and other oxides as trace. The result of the physical tests conducted are as follows: Porosity-6.34 %, cold crushing strength-180.65 kg/cm^2 , bulk density-2.35 g/cm^3 , thermal shock resistance-8 cycles, firing shrinkage-20 %, with an estimated refractoriness of 1400 °C.

The clay is kaolinite with free quartz present. The deposit therefore could be used for the production of ceramics, basic refractory bricks for lining of furnaces for certain non-ferrous metals, lining mortar and kilns. Also because of its attractive colour the clay could be used in paint, chalk and paper making industries.

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CHAPTER ONE

1.0

INTRODUCTION

Clay is a complex mixture, whose composition extremely varies depending on their geographical locations. It is a natural substance occurring in great abundance in nature which is constantly being formed on the earth surface as a result of rock weathering. Clay is a widely distributed abundant material resource of major industrial importance for enormous variety of uses (Apiam, 1985).

Everyone knows that common substance called clay, which is so easily mixed into paste with water. But everyone does not consider perhaps what an important part in agriculture and industry this familiar substance performed. All soils that are of value for the production of vegetables contain clay. The principal element of this substance---alumina--- is necessary to the development of plants; and its presence is necessary to retain the humidity of soil that is indispensable to vegetable life. To indicate the importance of clay in the industrial world, it is only necessary to say that tiles, bricks, pottery, from the coarsest to the finest Sevres are almost exclusively formed from this substance (Kent, 2004).

In common with many geological terms, the term "clay" is ambiguous and has multiple meaning:

- (a). A group of fine – grained mineral- i.e., the clay mineral.
- (b). A particle size smaller than silt.
- (c). A type of rock- i.e., a sedimentary deposit of fine- grained materials – usually composed largely of clay mineral. (Patterson et.al., 1983).

Clay also includes fine – grained deposits of non – aluminosilicates such as shale and some argillaceous soil (Bates et al, 1987). Clays have a number of minerals, when most clays are wet they become "plastic" meaning they can be formed and molded into shapes. When they are fired i.e. exposed to very high temperatures, the water is driven off and they become as hard as stone. As a result, nearly all civilization have use some form of clay for different applications, from bricks to pottery to tablets for business transaction, recording etc.

Because of their small particles sizes and variables degree of crystal perfection, it is not surprising that clay minerals proved extremely difficult to characterize adequately prior to the development of modern analytical techniques. The popular adoption in the early 1950s of the x-ray powder diffractometer, for clay studies helped to solve some of the problems of identification. Improvements in electron microscopy, electron diffraction and oblique texture electron diffraction, infra-red and DTA equipments, the development of nuclear and isotope technology of high speed electrocomputers of mossbauer aided all in the accumulation of factual information on clays. (Balley, 1980).

Clays are rarely present as pure mixtures of different clay types, one group or type normally being dominant. The physical properties of clays, which determine their commercial values depend on whether or not the various types are admixed types of clay, are present and their percentage. Important physical properties are pore size distribution, specific area, pore volume, plasticity e.t.c. Industry usually classifies clay by the properties which determine its usage. These types of classification include ball clay, fuller's earth pottery clay, fire or refractory clay, high purity kaolin and several others (Williams et al, 1951).

- (a) **KAOLIN CLAY:** It is a fine white clay consisting chiefly of the mineral kaolinite. It has its application paper in paper industry as filler and coating material. It is also used for fine pottery and porcelain.
- (b) **BALL CLAY:** This contains kaolinite and certain micas and has strong binding properties. It is used in making pottery (usually called potter's clay).
- (c) **FIRE CLAY:** is basically kaolinite with some iron oxides, magnesia, and alkaline. It can resist high temperature. It is used for making firebricks which is used to line surfaces, kilns, and crucibles.
- (d) **COMMON CLAY:** is used for bricks, clay pipes, clay floor and wall tiles and other building materials. It contains more impurities than fire clay, and does not have as great resistance to heat.
- (e) **BENTONITE:** It consists largely of montmorillonite. Some types that contain sodium swell when mixed with water. It found its application in metal industry used as molds for

casting metals and in materials drilling. Mud that are used in drilling for petroleum.

Bentonite that expands when mixed with water is used in engineering work to seal leaks.

- (f) **FULLERS EARTH:** is composed of montmorillonite and is high in magnesia. It is used as filters from oil and grease. Some are also used in pet litter, pesticides and other products.

The important physical properties of clay are plasticity, colour, clay strength, drying and firing shrinkages, bulk density, apparent porosity etc. The percentages of the minerals oxides (i.e. chemical composition) is also important property that ultimately determine the areas of its applications and uses.

Nigeria has appreciable distribution of industries engaged in metals and process industries, hence, the need for raw materials to support their growth. Clay products such as ceramics wares, burnt bricks, roofing floors tiles are cheaper and durable building materials than cement particularly in Nigeria. There are vast deposit of clay spread across every region in Nigeria, though their properties differ from site to site on account of geological differences. The present economic state imposes the need for sourcing for local materials to meet up the increasing demands. (Haruna et al, 2006).

Presently, at Nyikangbe, Minna, Niger State, there is a clay deposit. Most porters in Minna take their raw materials from this deposit. Unfortunately, the extent of the deposit and the characteristics/properties of this clay are not known. Thus, this project is aimed at analyzing and characterizing this clay to ascertain its chemical composition and mechanical /physical properties.

1.1 Aims and Objectives

This project is aimed at:

1. Determining the chemical characteristics of Nyikangbe Clay.
2. Determining the physical characteristics of the Nyikangbe clay.
3. Determining the nature /type of clay and thus its applications/uses.

1.2 Approach and Methodology

This research work is to be carried out using X-ray Fluorescence Spectrometer (XRFs) to determine the chemical composition of the clay. The physical properties such as porosity, cold crushing strength, bulk density e.t.c. are also determined using standard techniques.

1.3 Scope of Work

The scope covers the analysis of chemical composition and determination of physical properties such as bulk density, porosity, firing shrinkage etc and thereby determines the industrial application of the clay.

CHAPTER TWO

2.0 LITERATURE REVIEW

2.1 Origin of Clay

Clays and clay minerals have been mined since the Stone Age. Today they are among the most important minerals used by manufacturing and environmental industries.

The term "clay" is applied both to materials having particle size of less than 2 micrometers and to the family of minerals that has similar compositions and common crystals structural characteristics (Olorunsogbon, 2007). Clay minerals have wide range of particle sizes from 10's of angstroms to millimeters. Thus, clay may be composed of mixtures of finer grained clay minerals and clay-sized crystals of other minerals such as quartz, carbonate, and metal oxides clay and clay minerals are found on or near the surface of the earth. The origin of clay can be attributed to either two geological processes via sedimentation and weathering process. (Raymond et al, 1990).

The formation of clay by the weathering process is determined by the nature of the parent rock, climate topography, vegetation and the time period during which these factors have operated and taken place. Clay formed as a result of weathering process are termed residual clay and are usually deposited at the site of weathering. They accumulate where intense chemical weathering occurs. Soluble products are usually leached downward into the soil and there is relatively little transportation of solid products. Hot and humid regions of low relief in most part of Northern Nigeria favours the formation of residual clay and vegetation cover help to prevent significant amount of run off which carry away minerals. Formerly, clay was incorrectly thought to be particles of quartz, feldspar, mica etc but now with the use of X-ray in analyzing clay, the specific composition of clay are known. Clay minerals are mostly newly formed from the soluble products of primary and secondary minerals (Raymond, 1990).

There are two types of clays that are recognized, the silicate clays of temperate regions and the iron and aluminum hydroxide clays found in tropics and semi tropics. The great agricultural

regions of the world are dominated in a large degree by clays of a siliceous nature. All clay particles are crystalline and not amorphous as was originally supposed. Each clay particle regardless of its individual shape is made up of sheet like molecules or units, held loosely together. Clay particles will also show considerable variation in size. The plate like molecules themselves has a lamellar organization, their sheets of atoms being two or three in number. These units are quite definite, usually changing in size only by lateral extension. A clay particle might be visualized by comparing it with a piece of mica as the flakes of the latter present the plate-like molecules or units. Clay particles because of their fineness of division must expose a large amount of external surfaces as well, the sum of which usually greatly exceeds that of a superficial character.(www.essortment.com).

It has been shown that clay particles are composed of two distinct parts, the inner, porous and enormously large insoluble acidoid or micelle and the outer and more or less dissociated swarm of cations with variable amounts of water of hydration. Since these absorbed cations are usually rather easily displaced, they are spoken of as exchangeable ions. The replacement called ionic exchange, or more commonly base exchange, is one of the most important phenomena. Calcium and magnesium are the absorbed metallic cations held in the largest amounts by the siliceous clays of most natural soils. Since so much of the total calcium is replaceable, its activity is assured. The main concern, therefore, is the amount present. Thus, we use the practice of liming. With potash the total amount is often ample, but the proportion active is exceedingly small.(www.essortment.com)

Two groups of clay are commonly recognized, the kaolinite and the montmorillonite. The molecules of the kaolin are thought to be composed of two sheets or plates, one of silica and the one of alumina. The second group, the montmorillonite is composed molecularly of two silica sheets one of alumina. The molecules of these clays are less firmly linked together than those of the kaolin group and are usually further apart. In discussing the mineralogical nature of silicate clay, it must not be forgotten that other minerals besides the ones mentioned are present, either as mere accessories or as an important part of silicon, iron and aluminum should be mentioned.

While these probably occur but sparingly in temperate-region soils, the silicate clays often contain a larger admixture of colloidal iron and aluminum oxides. (www.essortment.com).

During formation, the removal of some of the soluble products by leaching of the soil solution will reduce clay formation and alter the kind of clay formed. Soils of regions that have moist climate favour the formation of clay ; due to poor drainage, the water logged and primary minerals dissolves in it, which then crystallizes to clay. This hot humid tropical soil therefore tends to be high in percentage clay, even to depth of 5-20m (Raymond, 1990).

2.2 The Nature of Clay

Majority of clays are crystalline in nature i.e. they are of definite repeating arrangement of atoms which they are composed. They are made mostly of planes of oxygen atoms, with silicon and aluminum atoms holding the oxygen together by ionic bonding which is the attraction of positive and negative charge atoms. The three aluminum atoms or others, depending upon the clay make up layer. One clay particle is composed of many layers stacked like a deck of cards. A clay particle is called a micelle. A few clays have the oxygen and other atom and less regular oriented and are called amorphous material (Raymond, 1990).

2.3 The charge on clay

The charge on clay comes from ionizable hydrogen ions and from isomorphous substitution. Ionizable hydrogen ions are hydrogen from hydroxyl ions on clay-ionized surfaces. The Al-OH or Si-OH portion of the clay ionizes the H^+ and leave the unneutralized negative charge on oxygen Al-O⁻ or Si-O⁻. The extent of ionized hydrogen depends on solution's PH. More ionization occurs in more alkaline (base) solution (Kim, 1993).

Noticed that most substitution are by ions with lower charge (less positive) than one being replace since the total negative charge from the ions (oxygen) remains unchanged. The lower positive charge because of substitution result in an excess negative charge at the location in the structure. This excess negative charge on the crystal lattice caused by isomorphous substitution are called cation exchange site. This site attracts and holds cations somewhat loosely. Other cations in the solution can compete with and even replace originally absorbed cations. (Can exchange with absorbed ions). The total amount of negative charge is

referred to as cations exchange capacity (CEC). The cations exchange sites hold and held mostly (Ca^{2+} , NH_4 , Zn^{2+}) occur on some of the exchange sites. (Raymond, 1990).

2.4 Clay minerals

They are the major components of clay material and occur in extremely small particles which are essentially crystalline and limited in number. They are hydrous aluminum silicates. Alkalis and alkaline earths are present as principal constituents in some clay minerals, magnesium or iron or both substitute wholly or in part for aluminum. Chemical constituents of clay minerals vary not only in amounts but also in the way in which they combined (Hamza, 2007).

Most clay minerals are formed as a result of weathering of some pre-existing rock. The bulk of clay present in sedimentary rocks was derived by the weathering of silicate minerals composing igneous rock. Some clay minerals are hydrothermal in origin (dickite, rectorite, hallosite), whereas others are formed from the alteration of other types of clay (example nontronite). Nine clay minerals (plus rectorite) have been reported in Arkansas, kaolinite-serpentine group, antigorite, dickite, halloysite, kaolinite, nacrite, illite, smectite group, berdelite, montmorillonite, nontronite. (Hamza, 2007).

The term "clay minerals" is most commonly used to denote a family of hydrous alumino-silicates (most especially phyllosilicates). Most clay minerals around in nature are with particle sizes $<4\mu\text{m}$ range. They are chemically and structurally similar to other phyllosilicates, such as the true and brittle micas. There are other materials of geological and biological importance that are clay sized; however, they are not "clay minerals" by the above definition. These other clay-sized minerals and materials include other silicates such as the hydrous sulfates, hydroxides, oxyhydroxides, hydrous oxides, amorphous compounds, organic compounds, prokaryotes, and viruses. Because of their intimate association with clay minerals, they are included into the domain of clay mineralogy.

If we look at the volume of material at the earth's surface we see that clay minerals constitute about 16 % of its total. 20 km is considered to the surface because it is the region from which we extract natural resources (and dump our waste.....). The diagram below graphically explains how the value of 16 % is obtained:

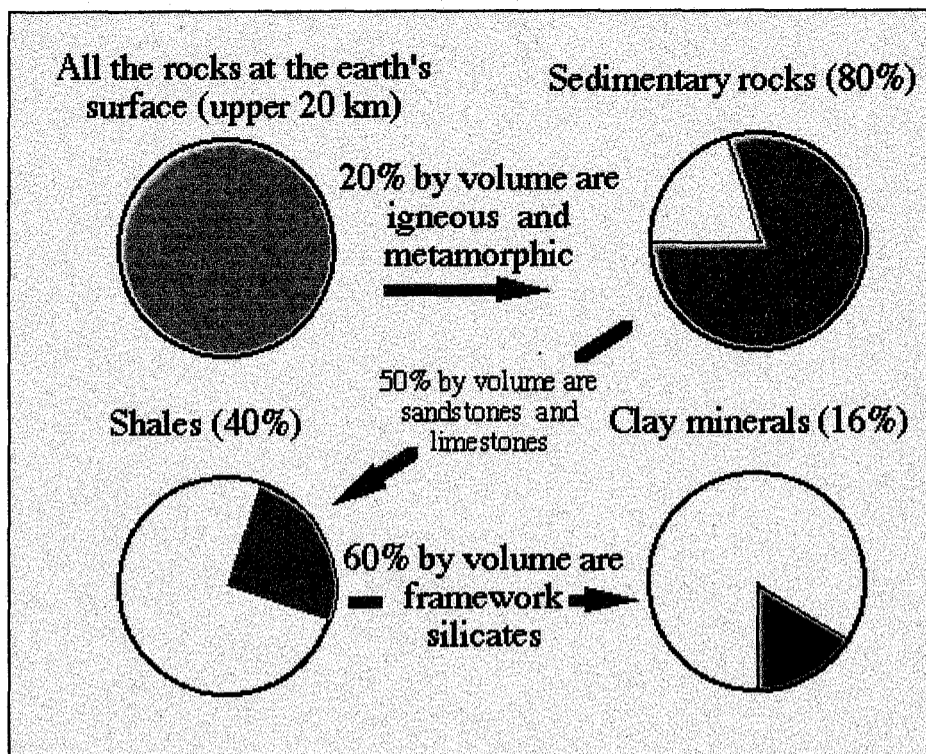


Fig 1: Percentage composition of clay mineral on the earth surface. (www.gly.uga.edu)

Minerals contents of the clay are classified as follows:

1. Kaolinite-white mineral consisting of hydrous silicate of aluminium that constitutes the principal mineral in kaolin.
2. Montmorillonite- a soft water clayey water absorbent mineral that is a hydrous magnesium-aluminium silicate mineral.
3. Attapulgite – is a kind of crystalloid hydrous magnesium-aluminum silicate mineral.
4. Illite- any group of clay minerals having essentially the crystal structure of muscovite.

These minerals contain various proportion of aluminum silica, usually combined with water to form hydrous aluminum silicates. Some of the minerals also contains calcium, sodium, magnesium, iron, or other elements. (www.huppages.com).

2.5 Classification of Clay

Clays are most present as pure mixture of different clay types, one group or type normally being dominant. Complete satisfactory classification is difficult because clay is often composed of clay materials of such poor crystalline that the individual components cannot be adequately characterized. Also, there seems to be continuous gradation between some of the types. The following classification has proved (Mc GrawHill, 1982).

Industry usually classifies clay by the properties which determine its usage. These types of classification includes ball clay, fuller's earth pottery clay, fire or refractory clay, high purity kaolin and several others (Hamza, 2007).

2.5.1 Bentonite

The principal clay mineral composing bentonite is smectite. Smectite is often used as a group name encompassing a number of very fine grained minerals which possess certain ion-exchange ability which determines the clay physical expression and colloidal properties. Sodium rich bentonite are valuable for well-drilling. (Brenner, 1908).

Bentonite is classified as fine grained primary clay. Although chemically like clay, technically, it swell excessively turning to gel. It also has very high shrinkage and stickiness properties. It is clay of high plasticity and chiefly composed of montmorillonite. The plastic sizes are largely in the colloidal range which undoubtedly contributed to its very high plasticity exceeding that of ball clay. It is even possible to get some degree of plasticity by mixing one-fifth as much bentonite in place of ball clay. Its ceramic significance is in increasing plasticity and its high iron oxide content (3-4 %) limits its usage in the ceramic industries. (Hamza, 2007).

2.5.2 High Purity Kaolin or China Clay

This is the main primary clay, generally being white and most refractory due to the minimum amount of contaminants that might change those properties. Kaolin has numerous industrial application and new uses continue to be discovered. It is unique industrial minerals due to several properties, including chemical inertness over wide range of acid-alkaline condition, white colour, in paper-filling application, softness and abrasive properties, and low conductivity.

Some uses of kaolin require very high rigid specification including particle size distribution, colour, brightness, and viscosity in manufacture of cement, paint and ceramics. (Miser, 1913).

The sedimentary kaolin has much greater plasticity than residual kaolin and some are practically as plastic as ball clays. They are used extensively in white wares, porcelain. Due to high purity and composition of kaolin, they are most of all the clays with softening points

range of 1700 °C to 1850 °C. Kaolin clay exhibits distinct physical and chemical characteristics which may differ from other clay minerals.

(a) Physical Properties of Kaolin Clay:

1. It has fine and tiny particle size about 0.002 m.
2. It is whitish in nature and appearance.
3. Kaolin clay is colourless and can be in lump or powdered form.

(b) Chemical Properties of Kaolin clay:

1. Kaolin has little or no surface absorbed water in it. Therefore it has low water retention ability.
2. When kaolin is heated above 8000 °C, the structure ruptures and can not be reconstructed by dehydration.
3. Kaolin is partially soluble in dilute acid.
4. Kaolin is chemically inert and as such provides a better resistance to chemical attack (Miser, 1913).

2.5.3 Ball Clays

Ball clays are good quality clays used mostly in pottery but also added to other clays to improve their plasticity. Ball clays are finest particle-sized clays, most sticky and are characterized by high rate of shrinkage due in part to their high organic content which burns out. The small particle size makes them excellent in suspensions. They have low iron content, usually fired to a pale off white colour (Brenner, 1908).

2.5.4 Fire Clays

Fire clays are all clays (excluding bentonite and ball clays) that are used to make items resistant to extreme heat. These products are called refractory products. Nearly all (81 %) of fired clay are used to make refractory products. A poorly defined categorization, generally referring to non-kaolin but with slightly more iron content and large particle size.

2.5.5 Refractory Clays

Refractory clays exhibit range of high melting temperature which makes them useful whenever high temperature application exists. Refractory bricks line the roasting and processing plants and ceramics.

2.6 Properties of Clay

The characterization common to all minerals derived from their chemical composition, layered structure and size. Clay minerals have the ability to exchange ions, relate to the surface clay particle or take up within the structure. This mineral property of clay that causes ions in solution to be fixed on clay surface or within internal site applies to all ions, including organic molecules like particles (Hamza, 2007).

Most clay become plastic when mixed with varying proportion of water. Plasticity of a material can be defined as the ability of the material to undergo permanent deformation in any direction without rupture under a stress beyond that of elastic yielding. Clays range from those which are very plastic called fat clay to those who are barely plastic called lean clay. The type of clay minerals, particle size and shape, organic matter, soluble salts, adsorbed amount and type of non-clay minerals are also known to affect the plastic properties of clay (McGraw Hill, 1982).

The following properties are considered in clay classification:

- 1. Strength:** Green strength and dry strength properties are very important because most structural clay products are handled at least once and must be strong enough to maintain shape. Green strength is the strength of the clay in the wet, plastic state. Dry strength is the strength after it has been dried. Dry and green strength are closely related and are affected largely by the same variable. Dry strength is dependent on the degree of the colloidal fraction. The presence of small amount of montmorillonite, which is of very fine particle size and highly dehydrated, generally increases the high strength. (McGraw Hill, 1982).
- 2. Shrinkage:** Both firing and drying shrinkage are important clay properties used for structural clay products. Shrinkage is the lost in volume of clay when dried or when it is fired. Drying shrinkage is dependent on the water content, the character of the clay mineral and the particle

size of the constituents. Drying shrinkage is high in most very plastic clays and tends to produce cracking and warping. It is low in sandy clays of plasticity and tends to produce a weak porous body. Montmorillonite in relatively large amount (10 % - 25 %) causes excessive shrinking, cracking and low drying. Firing and shrinking depends on the volatile material present, the types of crystalline phase changes that take place during firing and dehydration characteristics of clay minerals.(Mc GrawHill, 1982).

3. Vitrification range: Vitrification is the transition of a substance into a glass. The temperature range of vitrification or glass formation 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 begins to produce an increasing amount of liquid which makes up the glass bonding materials in the final fired products. Some clays have a short vitrification range, so that the temperature of kiln must be closely regulated. Illites montmorillonite and chlorites all have lower vitrification temperature. The degree of vitrification is regulated by amount of shrinkage, and reduction of porosity that can be obtained at a given temperature.

4. Colour: Colour is important in most structural clay products, particularly, the maintenance of uniform color. The colour of products is influenced by the rate of oxidation of iron, the state of the division of the minerals, the firing temperature and the degree identification, the proportion of alumina, the lime and magnesia in the clay material, and the composition of the fired glass during the burning operation.(McGraw Hill, 1982).

Some other properties of clay are as follows:

(I). Plasticity: Most clays become plastic when mixed with varying proportions of water to undergo permanent deformation in any direction under a stress beyond that of elastic yielding.

(II). Flow Characteristics: Clay-water mixtures show a number of types of flow, depending on soluble additions made to the water. Although some clay suspensions may appear to flow like liquids, they rarely behave exactly like homogeneous liquids which would show Newtonian viscous flow: dilatant, thixotropic flow, and shear hardening or negative thixotropy.

(III). Permeability: The ability of water to pass through a layer of clay depends on the way the particles are packed together, and on the water film absorbed around in between the particles. The permeability of clay is a major factor in the drying characteristics of clay. (Journals of Metallurgical and Materials Engineering, Volume 4, No: 2, September, 2009)

2.7 Uses of Clay

All types of clay are used in the agricultural, pottery, and chemical industry etc. but in general, the clay that is used is considered to be relatively high grades.

- They are sources of kaolin used for the production of alum.
- Clays are used for making conduit tiles and sewer pipes. These types of clay contain large proportion of kaolinite.
- Clays are used in the production of refractory materials as liners in kiln, crucibles for melting materials.
- They are used in industry as filters/extruders.
- Clay is used in the production of paint because of its adhesive nature.
- Clay is used in pharmaceutical companies as coating minerals on tablets e.g. very pure white kaolin.
- They are used for the production of brick and other construction materials.
- The cement industry uses large quantities of impure clays for providing alumina and silica as charger for the cement. Kiln clay of this nature is found where cement is manufactured. (McGraw Hill, 1982).

2.8 Kaolin Clay Deposit in Nigeria

The use of kaolin dates back to the third century BC in China. Today, it is mined and used in significant quantities for numerous industrial uses. Its most important use is in paper production, where it is used as a coating material. In addition, it is used in great quantities in the paint, rubber, plastic, ceramics, chemicals, pharmaceutical and cosmetics industries. (Grim, 1968).

The extensive occurrence of clay has been established all over the country. Sources of important clay deposits in Nigeria are Abeokuta, Ibamaje and Ayetorin in Ogun State, Kankha in Kastina State, Tsanyawa and Minjibir in Kano, Ozubulu, Anambra State, Giru Kaoje in Sokoto State, Ikoya- Ondo State to mention but a few. Nigeria has an estimated reserve of three billions tones of good kaolin deposits discovered in about 25 states and Abuja, FCT. Kaolin occurs extensively in cretaceous, tertiary and recent sediments of the major Nigeria sediments basins. Good deposits

occur in the south in Umahia, Uturru, Okigwe, Agbaghara, Nsu, Awom amma, ihube, ekeebi, Okijaand Ozubulu Ekulu in Abia, Imo and Enugu States respectively.

In the south-south region of Nigeria, extensive deposits of good kaolin clay has been reported within the Benin area at Ignoria Kli, Ossiomo, River Valley, Ameki formation in the Ika, Aniocha, Asaba and Utulu-uku in Edo and Delta State respectively. Kaolin clay also occurs extensively in Cross Rivers and Akwa-Ibon States.

Also, in the middle belt region of Nigeria, kaolin occurs in large deposits in Lokoja and its environs (Obajana) in Kogi State. Deposits are found in Kutugi and Mekunkele in Niger State. Kaolin is also found in Kwara State, Abuja. It is found in Kwali, Bwari, and Gboko in Benue State.

In addition, there are kaolin deposits in Northern Nigeria. It occurs in large quantity in Kankara, Bauchi State. Bajoga and its environs in Gombe State. It also occurs in Gwando, Kano, Jigawa, Borno, Yobe, Sokoto, Kebbi and Adamawa states. Also, in Western Nigeria, a wide distribution of kaolin deposits occur in Abeokuta province in Ogun State particularly in Osiele, Bamijo river valley near Ayetoro, Elefun Hill, Tontogangan and Ibadan area in Oyo State. The kaolin found in all these locations are same; only a slight difference in colour change as those found in the Southern are mainly brownish and creamy in nature. Only a few are white due to soil properties. Those found in the north are mostly white and ash in colour. (Olorunbun, 2007).

2.10 Clay Processing

2.1.1 Effect of Impurity on Clay

There are impurities in clay. However, other foreign materials coexisting with clay mineral actually tend to jeopardize those attractive characteristics to such an extent that removal or control of quantity of such materials becomes necessary. Generally, the effect of impurities in clay depends upon:

- Their nature and behaviour when the clay is used.
- Their occurring proportions.
- The shape and size of the clay and impurities.

- The effect of interaction:
 - Maximum temperature attained.
 - The duration of heating.
 - The effect of any other substance which is present.

The natural clay contain a wide variety of impurities, most of which must be removed in order to make a commercial useful clay products. The coarse impurities are generally micro-size impurities such as anatase, limonite, and pyrite, illite, organic matter etc. (Hamza, 2007).

2.1.2 Purification of Clays

Otherwise known as beneficiation, this is the process of separating the clay from undesired impurities. The coarse impurities, generally quartz muscovite and heavy minerals are rather easily separated by settling or screening. Partial or complete removal of non-clay and other undesirable materials (impurities) in an economical ways has been a subject of intensive research and process equipment development in the industry over the past three decades. This has enabled the production of clay products of remarkable purity.

2.1.3 Processing of Kaolin Clay

The kaolin clay is processed in dry and in some cases wet. The dry process usually consists of crushing, drying and pulverizing. The clay is crushed to fist size or smaller, and dried usually in rotary driers. After drying it is pulverize to a specific mesh size, such as 90 % retain on a 200-mesh screen. In other cases, the material may have to be pulverized to 99.9 % finer than 25,325 mesh. The material is shipped in bulk or in bags. All clays are processed using this method in kaolin; the material is slurred in water at the mine face at about 35-40 % solids with the aid of a dispersant. The slurry is pumped to a degritting station, where the very coarse impurities are removed; and then through pipe lines to the processing plant. The general processing steps are particle size fraction, that is impurities dewatering by filtering, drying and pulverization. Fractionation is done by centrifuging or gravity settling, dewatering by centrifuges, filter press or rotary vacuum drum filters.

Drying is done by rotor, apron, drum or spray driers, most of the kaolin is shipped dry bulk rail but about 20 % is bagged and shipped by rail or trunk. Some kaolin are shipped in slurry form tank, and solid at 70 % in can. (McGraw Hill, 1982).

CHAPTER THREE

3.0 Equipments/Materials and Method

3.1 Materials and Equipments Used

The following equipments materials were used to carry out the analysis of the clay:

1. Clay samples from Nyikangbe clay deposit.
2. Lithium tetraborate bonder ($\text{Li}_2\text{B}_4\text{O}_7$).
3. Water.
3. Mercury.
4. Tercramics Densometer.
5. Electric furnace.
6. Weighing balance.
7. Vernier caliper.
8. Mortal and pestle.
9. Crucible tongues.
10. Colour chat.
11. Hydraulic press.
12. Dessicator.
13. Beaker.

3.2 Methodology

The experimental analysis carried out in the course of this research work includes the determination of:

1. Apparent porosity.
2. Bulk density.
3. Refractoriness of the clay.
4. Firing shrinkage.
5. Cold crushing strength.
6. Chemical composition.

3.2.1 Determination of Apparent Porosity of the Clay

The sample was cut into cube form of 2.5*2.5 cm. It was dried in an oven at 110°C to a constant weight (D) with accuracy of 0.1g. The dried sample was suspended in distilled water such that the sample did not touch the bottom or sides of the container (300 ml beaker).

The sample was boiled for two hours while still suspended in water cooled to room temperature and its soaked weight (S) was noted. The sample was removed from the water and water was wiped off from its surface by lightly blotting with a wet towel and its suspended weight (W) in air was determined. The apparent porosity (P) was then calculated by the formulae:

$$P = (V_i/V) * 100 \dots\dots\dots (1)$$

Where V = Actual volume of open pore of the specimen = (W-D)

V_i = External volume of the sample = (W-S)

P can be expressed as follows:

$$P = (W-D)/(W-S) * 100\% \dots\dots\dots (2)$$

3.2.2 Determination of Bulk Density of the Clay

The sample was formed into a cylindrical rod shape of 2.5cm*1.5cm. The weight of the sample was measured. 1000g of clean mercury was poured into 100ml beaker and place on the balance pan centrally under the saddle. The balance was adjusted until the gramme scale reads zero. The saddle was lowered into the mercury by the means of the hand wheel and lock with the bridge locking screw. The micrometer was adjusted until the pointer touches the surface of the mercury. The balance was read which is represented by W_2 in the formula below... The saddle was raised and the specimen removed.

The volume of the sample is given by the formula:

$$\text{Volume} = W_2 d \dots\dots\dots (3)$$

Where d = the density of the mercury

The table below shows the density of mercury at different temperature. The temperature of mercury used in the experiment was 23 °C, the corresponding value of density was used for the calculation.

Table1. Density of mercury at different temperature

Temperature (°C)	Density(g/cm ³)
18	13.5508
19	13.5484
20	13.5459
21	13.5435
22	13.5410
23	13.5386
24	13.5361
25	13.5337
26	13.5312
27	13.5288
28	13.5263

The bulk density is calculated from the expression:

$$\text{Bulk density} = W_1d/W_2 \dots\dots\dots (4)$$

The value of d at temperature of 23 °C was used for the calculation.

3.2.3 Determination of the Refractoriness of the Clay

The clay sample was formed into simple cones of 1.27 cm base and a perpendicular vertical length of 3.81 cm. The cones were mounted on refractory base along with several other standard cones of the same dimensions and standard composition. The cones were heated at a rate of 5 °C/min in an oxidizing atmosphere in the furnace until the test cone squatted (bends) over its own weight. After cooling the test cone was compared with the standard cones. The test material was said to have the pyrometric cone equivalent of the standard cone whose behaviour most resembled the test cone.

3.2.4 Determination of the Thermal Shock Resistance of the Clay

The test pieces of refractory bricks were thoroughly dried and placed in the cold furnace and heated at a rate of 5 °C/min up to 1200 °C, the testing temperature was kept for 30 minutes with a pair of tongs previously warmed in the furnace for short time. The test pieces were placed on cold fire bricks in a position free from draught. After the test pieces had been cooled in this way for 10 minutes. The test pieces were replaced in the furnace for a further 10 minutes and the cycle was repeated until failure occurred.

3.2.4 Determination of Firing Shrinkage of the Clay

The clay sample was moistened with water to the point of wedging (which moistened clay materials remain packed into a ball in hand until intensional vibration causes the mixture to flow). The wedge sample was cast into brass molds. The clay bar was prepared whose original/initial length was marked on the sample. The final length after the clay had been fired was recorded. The firing shrinkage was then calculated by the expression below:

$$\text{Firing Shrinkage (F}_s\text{)} = (A-B)*100/A \dots\dots\dots (5)$$

Where A = Initial/ original length.

B = Final length.

3.2.5 Determination of the Cold Crushing Strength of the Clay

A cube was made from the refractory brick; the length and the breadth of the sample were measured. A cardboard sheet not exceeding 0.63cm in thickness was placed between the platens of the hydraulic press and the bearing. The faces of the test piece were placed centrally on the platen. The load was applied until the test piece failed to support the load. The maximum recorded load was taken as the crushing load.

The load cold crushing strength is calculated by the formula:

$$\text{CCS} = M/A \dots\dots\dots (6)$$

Where, M = mass of the sample, A = Surface area.

3.2.6 Determination of Clay Colour.

The colours of the raw clay and at higher temperature were determined using colour chart.

3.2.7 Determination of Chemical Composition of the clay

The chemical composition of the clay was determined using X-ray fluorescence spectrometer (XFS). It is a non-destructive analytical techniques used to identify and determine the concentration of element present in solid, powdered and liquid samples. XRF is capable of measuring all elements, from beryllium (Be) to Uranium (U).

(a) Sample presentation: The sample was grounded and sieved to 75 Nm particle size. 4 g of the sieved were intimately mixed with 1 g of Lithium tetraborate binder ($\text{Li}_2\text{B}_4\text{O}_7$) and pressed in a mould under a pressure of 10-15 ton/n² to a pellet. The pressed pellets were dried at 110 °C for 30 minutes in an oven to get rid of absorbed moisture and were finally stored in a desiccator for analysis.

(a) Analysis: The spectrometer was switched on and allowed to warm up and also gained in order to stabilize the optics and the X – ray tube. It was then calibrated to determine the expected elements present in the sample.

The sample was run using the prepared program (calibration) and the elements concentrations present in the samples were calculated and displayed after applying automatic statistics to the results by the spectrometer.

CHAPTER FOUR

4.0 Result and Discussion of Result

4.1 Result

The results from the experimental analysis are given below:

Table 2. Result of Chemical Composition of Nyikangbe Clay.

Element	Oxide	Oxide composition (%)
Aluminium	Al_2O_3	21.80
Silicon	SiO_2	54.03
Potassium	K_2O	3.60
Calcium	CaO	1.33
Titanium	TiO_2	1.85
Vanadium	V_2O_5	0.09
Chromium	Cr_2O_3	0.04
Manganese	MnO	0.13
Iron	Fe_2O_3	11.93
Nickel	NiO	0.007
Copper	CuO	0.05
Zinc	ZnO	0.02
Rubidium	Rb_2O	0.03
Strontium	SrO	0.06
Zirconium	ZrO_2	0.17

Table 3. Chemical composition of Nyikangbe clay as compared to Grim Shaw and composition specification for other industrial clays.

Constituent	Nyikangbe clay	Grim Shaw	Agriculture	Ceramics	Paper	Paint	Fertilizer	Refractory Bricks
SiO ₂	54.03	40-60	49-68	60.50	45.0-45.8	45.3-47.9	46.07	51.70
Al ₂ O ₃	21.80	25-45	37-65	26.50	33.5-36.1	37.9-38.4	38.07	25-44
Fe ₂ O ₃	11.93	1-5	0.88	0.5-1.2	0.3-0.6	13.4-13.7	0.33	0.5-2.4
CaO	1.33	2.5	0.03	0.18-3.0	0.03-0.60	0.03-0.60	0.33	0.10-0.20

The tables below show the result obtained on physical tests conducted on the clay:

Table 4. Result of the Bulk Density of the Clay

S/N	W ₁ (g)	W ₂ (g)	Bulk Density (g/cm ³)	Average Bulk Density (g/cm ³)
A	13.58	78.00	2.357	2.35
B	18.31	105.69	2.345	

Table 5. Result of the Apparent Porosity of the Clay

S/N	Constant weight (D)	Weight of sieve in water (W _w)	Weight of sieve + sample (W _{ws})	S	W	V _i (W-S)	V (W-D)	Porosity (%)	Average Porosity (%)
1	18.3546	8.1082	19.3040	11.1958	18.8546	7.6588	0.5000	7.20	6.34
2	13.6145	8.0839	16.3279	8.2440	13.9256	5.6816	0.3111	5.48	

Table 6. Result of the Firing Shrinkage of the Clay

Initial length (A)	Final length (B)	A-B	Firing Shrinkage
10 cm	8 cm	2 cm	20 %

Table 7. Result of the cold crushing Strength of the clay

S/N	Length (cm)	Breadth (cm)	Area (cm ²)	Force (KN)	Mass (Kg)	CCS (kg/cm ²)	CCS Average (kg/cm ²)
A.	2.3	2.0	4.6	8.8	880	191.30	180.65
B.	2.5	2.4	6.0	10.2	1020	170.00	

Table 8. Result of the Refractoriness and Thermal Shock Resistance

Refractoriness	Thermal Shock Resistance
1400 °C	8 Circles

Table 9. Result of the Colour Change

Raw sample (Green)	Sample at 1200 °C	Sample at 1300 °C
Moderate Yellow	Dark yellow orange	Moderate Brown

4.2 Discussion of Result

The chemical composition of the clay is shown in table 2. The results show a high silica (SiO_2) content of 54.03 % and moderate alumina (Al_2O_3) content of 21.80 %. The results suggest that Nyikangbe clay is kaolinite with free quartz present. Iron oxide is also present in appreciable amount (11.93 %). This result suggests that the clay can not be used as a source of aluminum since the iron oxide will likely have a deleterious effect on the process of extraction. Other oxides are in minor quantities.

From the results the percentage of the oxides present in the clay do not sum up to 100 % but to a total of 95.14 %. This wide discrepancy may have resulted due to the present of impurity in the clay. Also it may be due to traces of other materials too infinitesimal for detection which is expected to have been removed during pretreatment.

The firing shrinkage result is shown in table 6 to be 20 %. This will be useful properties of the clay applicable in ceramic industry. Measures have to be taken to avoid cracking as a result of high shrinkage. Also, the colour of the clay varied from moderate yellow to moderate brown on firing as shown in the table 9. This could be attributed to higher iron oxide content present. The ferrous iron impacted brown color on the fire samples due to conversion from ferrous to ferric compound. The color variation is considered usable for the manufacture of flower pot and earth wares. Also, it can be used in paint, chalk and ceramics producing industries.

The refractoriness of the clay is estimated to be about $1400\text{ }^\circ\text{C}$, though a little bit low, but can be used in processing refractory bricks needed for lining of the wall of furnaces for smelting of certain non-ferrous metal (low melting point $< 1300\text{ }^\circ\text{C}$) like copper, Zinc,

Aluminum etc. It can also be used as soaking pit kilns, ladles, ovens and heat-treatment furnace of materials of low melting points.

The clay cold crushing strength is estimated to be 180.65 kg/cm^2 . The high content of K_2O and CaO enhance the clay bond strength. Therefore, the clay can withstand high load when used as supports.

Also, porosity of the clay is estimated to be 6.34 % and bulk density is 2.35 g/cm^3 . Shrinkage, porosity and bulk density are inter-related quantities. The more the materials shrinks, the less porous it is, hence, the denser. The low porosity of the clay makes it a good heat conductor which has its application in making oven smelting furnace and ceramics. Moreover, the thermal shock resistance of the clay is 8 cycles before it cracks.

CHAPTER FIVE

5.0 CONCLUSION AND RECOMMENDATION

5.1 Conclusion

The X-ray fluorescence result shows that Nyikangbe clay has high silica content and moderate aluminum oxide content. The physical characteristics were found using standard techniques. The clay was found to have good cold crushing strength of 180.65 kg/cm^2 , the firing shrinkage of 20 %, the colour characteristics ranged from moderate yellow to moderate brown on firing, the refractoriness of $1400 \text{ }^\circ\text{C}$, thermal shock resistance of 8 cycles, porosity of 6.34 % and bulk density of 2.35 g/cm^3 .

Nyikangbe clay is suggested to be kaolinite with free quartz present which has its usefulness with specified industrial application. The clay is therefore suitable for use ceramic, paint and chalk manufacturing industries. The clay may also be used in the production of refractory bricks for lining of walls of furnaces of certain non-ferrous metal production, kiln, soaking pits, ovens, ladles and heat-treatment furnaces.

5.2 Recommendation

- If this clay deposit is exploited and properly harnessed it provides internal sourcing for raw materials which will go long way to reduce over dependence on importation of materials used in ceramic, chalk and paint industries.

- Geological survey to determine the extent of the deposit should be carried out.

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APPENDIX

1. Apparent Porosity.

$P = V_i/V$ where,

$V_i =$ Actual volume of open pore of the sample = $W-D$.

$V =$ external volume of the sample = $W-S$.

$W =$ Weight of the soaked sample suspended in air

$S =$ Weight of fired clay in water

$W_{wl} =$ Weight of the sieve in water.

$W_{ws1} =$ Weight of the sieve in water and the sample.

$S_1 = W_{ws} - W_w = 19.3040 - 8.1082 = 11.1958g$.

$P_1 = (W_1 - D_1) * 100\% / (W_1 - S_1) = (18.8546 - 18.3032) * 100\% / (18.8546 - 11.1958) = 7.20\%$

$P_2 = (W_2 - D_2) * 100\% / (W_2 - S_2) = (13.9256 - 13.6141) * 100\% / (13.9256 - 8.2440) = 5.48\%$

$P = (P_1 + P_2) / 2 = (7.20 + 5.48) / 2 = 6.34\%$

2. Bulk Density.

$d_B = W_d/W$ where

$W =$ Weight of the sample.

$d =$ Density of mercury at $23^\circ C$

$W_m =$ Weight of the sample when immersed in mercury.

$d_{B1} = W_1 d / W_{m1} = 13.58 * 13.5386 / 78.00 = 2.357g/cm^3$

$d_{B2} = W_2 d / W_{m2} = 18.31 * 13.5386 / 105.69 = 2.345g/cm^3$

$d_B = (d_{B1} + d_{B2}) / 2 = 2.35g/cm^3$

3. Firing Shrinkage (F_S)

$F_S (\%) = (A - B) * 100\% / A$, where,

$A =$ Initial length before firing the sample.

B = Final length after firing the sample.

$$F_s = (10-8) * 100\% / 10 = 20\%$$

4. Cold Crushing Strength (C.C.S)

M = Mass of the sample in kg.

F = Force in KN.

g = Acceleration due to gravity = 10m/s^2

A = Surface area of the sample.

L = Length of the sample.

B = Breadth of the sample.

$$M(\text{kg}) = 1000F/g$$

$$A = L * B$$

$$M_1 = 1000F_1/g = 1000 * 8.8/g = 880\text{kg}$$

$$A_1 = L_1B_1 = 2.3\text{cm} * 2.0\text{cm} = 4.6\text{cm}^2$$

$$\text{C.C.S}_1 = 880/4.6 = 191.30\text{kg/cm}^2$$

$$M_2 = 1000 F_2/g = 1000 * 10.2/10 = 1020\text{kg}$$

$$A_2 = L_2B_2 = 2.5\text{cm} * 2.4\text{cm} = 6.0\text{cm}^2$$

$$\text{C.C.S}_2 = 1020/6.0 = 170\text{kg/cm}^2$$

$$\text{C.C.S} = (\text{C.C.S}_1 + \text{C.C.S}_2) / 2 = (191.30 + 170.00) / 2 = 180.65\text{kg/cm}^2$$

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LETTER REF. NO.

NMDC/TECH/179/313

DATE 4th December, 2009

CERTIFICATE OF ANALYSIS

CLIENT: Adeniyi Adewale Amos
Federal University of Technology,
Minna.

SAMPLE: Clay

JOB: Physical and Chemical Test

TEST RESULT:

Physical Test

S/NO	TEST CARRIED OUT					
	Firing Shrinkage (%)	C.C.S. (kg/cm ²)	Refractoriness (O°)	Thermal Shock Resistance (Cycles)	Porosity (%)	Bulk Density (g/cm ³)
1	20%	180.65	1400°C	8 cycles	6.34%	2.35

Chemical Test

S/N	Sample	%	%	%	%	%	%	%	%
		Al ₂ O ₃	SiO ₂	K ₂ O	CaO	TiO ₂	V ₂ O ₅	Cr ₂ O ₃	MnO
1	Nyikangbe Clay	21.80	54.03	3.60	1.33	1.85	0.09	0.04	0.13
		%	%	%	%	%	%	%	%
		Fe ₂ O ₃	NiO	CuO	ZnO	Rb ₂ O	SrO	ZrO ₂	
		11.93	0.007	0.05	0.02	0.03	0.06	0.17	

Key: % = Percentage

NAME OF VERIFYING OFFICER:

J. OBIYI NWOSU

SIGNATURE:

J. OBIYI

DATE:

4th DECEMBER, 2009