EFFECT OF ADDITIVES ON REFRACTORY PROPERTIES OF SOME SELECTED CLAY DEPOSITS IN NIGER STATE, NIGERIA

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ABSTRACT

In metallurgical industries the quality of refractory clay depends largely on the refractory properties of clay and proper selection of clay affect these properties which include Porosities, Compressive strength, Linear shrinkage, Thermal shock resistance, and refractoriness. This gives need on the improvement on some properties of selected Nigerian clays. Clay collected from Bida, Chanchaga, Kpakungu and Maikunkele areas of Niger State were analysed to determine the chemical composition of the clays samples and raw clay were beneficiated and mixed with additives which include ash of Sawdust, Rice husk and Cornhusk in a ratio of 5, 10, 15, 20, 25 and 30 % weight to produce clay bricks which were subjected to tests to determine the effect of the additives on the refractory properties of the clay samples. The result revealed Bida clay increase in porosity from 16.64 % to 35 %, Chanchaga clay increase in porosity from 9.98 % to 38.38 %, Kpakungu clay increase in porosity from 8.12 % to 33.37 % and Maikunkele clay increase in porosity from 10.50 % to 35 %. Compressive strength of the clay samples were reduced Bida clay reduced in compressive strength from 51.58 N/m^2 to 8.63 N/m^2, Chanchaga clay reduced in compressive strength from 65.85 N/m^2 to 29.11 N/m^2, Kpakungu clay reduced in compressive strength from 63.59 N/m^2 to 20.55 N/m^2, Maikunkele clay samples reduced in compressive strength from 59.06 N/m^2 to 18.52 N/m^2. The refractory temperature of Bida, Chanchaga, Kpakungu and Maikunkele clay samples were increased from 1410°C to 1480°C, 1580°C to 1620°C, 1550°C to 1570°C and 1650°C to 1690°C at 5%wt of additives. Conclusively it was revealed that the additives has a great influence on improving some refractory properties of the clay samples and encourage consumption of local refractory clay in metallurgical industries.

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CHAPTER ONE

1.0 INTRODUCTION

1.1 Background to the Study

Clay is a complex inorganic mixture, whose composition varies widely depending on the geographical location. It is a natural substance occurring in great abundance in nature, being constantly formed on the earth's surface as a result of rock weathering. Clay is composed of silica (SiO_2), Alumina (Al_2O_3) and water (H_2O) with appreciable concentration of oxides of iron, alkali and alkaline earth. Some of the commonest and important applications of clays are in the manufacture of paper, paint, plastics, ink, roofing sheets, pottery, bricks, ceramics, floor tiles and rubber. Clays also find various applications in the manufacture of cement, fertilizers and insecticides. They are used in advanced chemical processing because of their reactivity and catalytic activity. Clays are also utilized in pharmaceuticals and food processing industries. Some of these applications require the processing or the blending with other materials so as to improve on some desired characteristics of the finished product (Umaru *et al.,* 2012).

Clay is grounded in mills, mixed with water to make it plastic and then moulded either by hand or machine to shape and size of brick. Hand moulded brick are pressed by hand in a sanded wood mould dried and fired, have a sandy texture, irregular shape and colour. Machine made bricks, are either hydraulically pressed in steel moulds or extruded as a continuous batch of clay. The moulded brick is baked to dry out the water and burned at a high temperature so that part of the clay fuses the whole mass of the brick into a hard durable unit. Clay is suitable for brick making because of its wide variation in composition and also possible to burn over a wide range of temperature sufficient to fuse the material

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into a durable mass. Clay Bricks may be classified into common clays and refractories clay depending upon area of application. Refractories bricks are made from selected clay that has been carefully prepared, heavily moulded, thoroughly burned and safely capable of carrying much heavier loads. These types of bricks are exceptionally hard, dense, low porosity and absorb very little water.

Clay based refractories is the oldest and most common of the refractory used for construction of high temperature equipment such as furnaces, kilns and crucibles. They are used to construct high temperature equipment because they have high thermal characteristics and capable of holding molten or solid metals without having undesirable chemical reactions with them (Adamu *et al*., 2018). With the development of iron and steel industry which make use of refractory materials, there is need enhance the quality of clay used as refractory materials to meet the growing demand for them. Since government is proposing on bringing Ajaokuta steel complex to production which will require 36,000 metric tons of refractory bricks worth a lot of money annually for lining purposes (Atanda *et al*., 2012). Earlier research works on some Nigerian clay deposits for refractory purpose showed the need for improving the physical properties of clay to meet the suitability for intending purposes.

1.2 Statement of the Problem

Clays of Bida, Chanchaga, Kpakungu and Zungeru were evaluated by Okoroafor, (2001). In the recommendations presented therein, Okoroafor stated that further work should be conducted to improve the Thermal shock resistance, Porosities and general Refractoriness of Bida clay, Chanchaga clay, and kpakungu clay. Although, the clays exhibited good refractory properties but the porosity test conducted shows that none of the clays fall within the recommended values of between 20 % – 30 % for dense fire bricks according to (Chester, 1973). The clay of Kpakungu also shows low thermal shock resistance. It became necessary to look into the refractory properties of all the clays and improve them before recommending for refractory production. The refractory properties of clay have a greater effect on the final quality of refractory production.

1.3 Significance of the Study

In Nigeria, at present-day there is a standard shift from large dependence on importation to local production of materials with virtual advantage. Refractories are not isolated from these items. The recent move by the federal government to bring around the iron and steel industry is another motivation for an investigation of this nature. At the Ajaokuta steel complex, the basic oxygen furnace and other furnaces in the plant will surely need effective refractory requirement. A challenge has been made to raise the overall efficiency of clay brick by adding other suitable materials along with clay in the manufacturing process. Thus, improving the quality of final steel product, reduce the cost of production and increase the overall efficiency of clay refractory of the selected clay sample and encourage the use of local clay as refractory.

1.4 Aim and Objectives of the Study

The aim of this research is to investigate the effect of additives on the refractory properties of Bida, Chanchaga, Kpakungu and Maikunkele clays using saw dust ash, rice husk ash and corn husk ash as additives.

The aim will be achieved through the following objectives:

(i) Determination and calculation of the sieve analysis parameters and data of Maikunkele, Chanchaga, Kpakungu, Bida Clays.

(ii) Characterisation and determination of the physio-chemical compositions of Maikunkele, Chanchaga, Kpakungu, Bida Clays

(iii) Beneficiation of clay and production of clay bars for refractory tests

(iv) Determination of the refractory properties of Maikunkele, Chanchaga, Kpakungu, Bida Clays such as; linear shrinkage, bulk density, apparent porosity, water absorption, compressive strength, thermal shock resistance and refractoriness.

1.5 Scope of the Study

The scope of the investigation of the clay properties involves collection of clay samples from dug holes and surface exposure from respective areas in Niger State, chemical analysis, physical and mechanical properties done by Okoroafor (2001) were considered, with addition of materials and chemicals to the clay samples firing test for moulded samples of clay at different temperature in order to determine linear shrinkage, porosity, water absorption, thermal shock resistance, compressive strength, and refractoriness.

CHAPTER 2

2.0 LITERATURE REVIEW

2.1 Clay formation and origin

Clay may be define as those sand particles fewer than 20 microns in diameter which fail to settle at the rate of 25mm/min, when suspended in water. Average clay particles are in collodial size. Clay binders are classified under fire clay, bentonite, illinite, limonite, kaolinite (Kutelu *et al.,* 2014).

Clays are formed by deposit of the finest particles of weathered rocks transported by flowing water, glacier or other means. A weathered rock not transported from its parental rock is likely to contain fragment and reaction product characteristics of the parental rock, with such clays known as residual or primary clays. Whereas decomposed fragment which are transported far distance from their original rock subjected to complex sorting and remixing may remove fragment and reaction product characteristics of the original rock. This type of clay is called secondary clays or sedimentary clays (Okoroafor, 2001).

To the engineers, clay is a material of colloidal size and is either hydrophilic (swells in water) or hydrophobic (not swell in water). Clay consists of a mixture of minerals derived from the product of weathering and break down of rocks. The constituent grains are formed at different stages in the history of the Clay deposit (Okoroafor, 2001). Main constituent of transported clays are clay mineral, quartz, mica, feldspar and carbonaceous matter which can greatly affect their physical properties. The chemical components of clay are silica, alumina, iron compounds, alkali component, magnesium compounds, zinc compounds, calcium compounds and others.

2.1.1 Chemical analysis of clay

Chemically all clay minerals are hydrous silicate which on heating lose all their moisture content and yields refractory material.

Pure kaolines contain basic Oxides in following percentage

Al_2O_3 – 39.50%

SiO_2 _ 46.60%

H_2O – 13.90% (Okoroafor, 2001).

After calcining the percentage of these oxides are: -

Al_2O_3 – 46.00%

SiO_2 _ 54.00% (Okoroafor, 2001).

Apart from these principal components, clays in the examine conditions contain certain amount of impurities which are evenly distributed, up to 1,300 to 1,350°C quartz is inert which plays part of non-plastic filler. At 1,350°C and above quartz becomes an active flexing agent. Another common impurity of clay is pyrites, Marcasite (Feg_2), siderite ($FeCo_3$) and Iron Oxide. These could cause spalling, swelling, and sometimes stains on the surface of the bricks at fairly high temperature. Hence Iron Oxide are often said to cause appreciable deterioration of the refractory properties of clay (Abolarin *et al.,* 2004).

Flexing agent such as CaO, MgO, Na_2O and K_2O Calcite ($CaCo_3$), Gypsum ($CaSo_4$), rutile (TiO_2) and organic matter of vegetable origin are also found in clays (Arowolo, 2000).

2.1.2 Classification of clays

A Clay material differs in composition and properties which makes it impracticable to classify them accurately and in great detail. There are seven major clay groups they are as follows

i. Kandite (Kaolin) group: - this group include kaolinites, Dickite, Narcite, Halloysite, and metahalloysite.
ii. Illite group: - this group include illite phengite, hydromica, glauconite, brammallite, and celadonite.
iii. Smectite group: - this group include montmorillite, nontronite, hectorite, sapionite, sauconite and beidellite.
iv. Vermiculite
v. Chlorite group: - this group include penning, clinochlore, prochlorite.
vi. Palygorskite group: - include sapiolite and attapulgite.
vii. Allophane :- Amorphous (Okoroafor, 2001)

most mineral describe as clay, the clay mineral are the predominant and essential constituent, and this should always be in mind though in some commercially useful bricks and time clays the total clay mineral May be only 30 – 35 % of the whole material, and in some workable clay it may be less (Arowolo, 2000).

2.1.2.1 *Industrial clays*

Industrial clays are well known and widely used clays which fall under the kaolin group. They include China clays, Ball clays, Halloysite- Bearing clays Fire clays, bricks etc.

2.1.2.1 *China Clays*

China Clays are residual clays formed by break down of primary minerals in crystalline rocks and Consist of kaolinite mainly. Impurities in China Clays include magnesium, calcium, quartz, mica, and some traces of Iron. Feldspar decomposition result in the loss off its alkali, silica and grains water of constitution. China Clays are worked to produce dried bricks and pottery.

Pure kaolin or China Clays $Al_2O_3.2SiO_2.2H_2O$ has the composition by weight of Aluminia 39.56%, Silica 46.54%, and water 13.90%. Commercial kaolin contain between 80-90% of the clay mineral, contaminated mainly with quartz and fine grained mica or hydrous mica but seldomly with appreciable portions of feldspar. As the particles size distribution of all the minerals present is similar, it is difficult to deduce a mineralogical analysis with much degree of accuracy. Pure kaolin or China Clays is white burning and has a refractoriness of about 1,785°C, although if much impurities is present the softening temperature will be reduced (Okoroafor, 2001).

2.1.2.3 *Ball clays*

These are white burning plastic clays that can be added to China clay to improve their properties. Ball clays have fine grain size lesser than that of China Clays as one of its main features. This is the main reason for their high plasticity. Ball clays are sedimentary in character, invariably dark in colour, due to associated lignite but readily burns away to produce

The mineralogy is dominantly kaolinite with variable amount of quartz illite and micas. Chemically it is richer in silica and low in alumina (Al_2O_3) other materials like alkali, iron oxide and carbonaceous matter are present. Comparatively, Ball clays have larger percentage of impurities than purified China Clays and so have lower refractoriness. They tend to shrink appreciably on firing and produce a denser body (Okoroafor, 2001).

Results of chemical analysis show that ball clays are richer in silica than China Clays. The chemical composition of ball clays usually falls within the following range shown in Table 2.1.

Table 2.1: Chemical composition of ball clays

Silica	43% - 60%
Alumina	25% - 35%
Loss on ignition	7% - 15%
Iron Oxide	Less than 2%
Lime and Magesia	Less than 1%
Potash and soda	Less than 3%

(Abifarin, 1999)

The main uses of ball clays are to increase the plasticity of other white-burning clays, flint, in the manufacture of white water bodies. They are also used in the manufacture of abrasive wheels, insulator, and moulding sands. They can be used as filters in the rubber, paint and plastic industries.

2.1.2.4 Halloysite – bearing clays

These are clay that resemble the China clay in appearance and some properties, but are more plastic and produce a denser fired body. Halloysite has a similar origin to kaolinite but hydrothermal agents were more acidic and it may have contained sulphate or chloride radicle. The fact that Alumite ($K_2O.3AL_2O_3$. $4SO_3.6H_2O$) is commonly associated with Halloysite clays supports this theory (Arowolo, 2000).

2.1.2.5 Fire clays

These are clays with high refractoriness and although there is no definite stipulated lower limit temperature, a softening point of 1500°C is usually regarded as minimum and one greater than 1600°C is usually required. Fire clay consist of numerous hydrous alumina silicate minerals which on heating break down to several constituent that form Millite $3Al_2O_3.2SiO_2$ which is the only compound of silica and alumina stable at high temperature (Arowolo, 2000).

They are the most extensively used refractory material due to it cheapness and the nature of the minerals present in them which has great influence on the property of furnace bricks have been the subject of much research for many years. Fire clays is used extensively in iron and steel industry, non-ferrous metallurgy, glass industry, pottery, kilns, cement industry and many others.

Fire clays have low proportion of Iron, calcium and alkali's to enable the clays withstand high temperature. These elements act as flexing element (Okoroafor, 2001).

They also have high alumina content. Considerable variation in chemical composition is found in fireclays from different sources the range of composition is given in Table 2.2.

Table 2.2: Chemical composition of fire clay

Composition	%
Silica	40-80
Alumina	10-40
Loss on Ignition	5 - 14
Iron Oxide	1 - 5
Lime	Less than 5
Magnesia	Less than 5
Alkalies	Sample may have up to 4

(Abifarin, 1999)

In general the higher the Aluminia content of a fire clay the greater the refractoriness. (Okoroafor 2001).

2.1.3 Refractory clay

Refractories are heat resistant materials that can withstand high temperature without rapid physical and chemical deterioration. Refractory clay can also withstand other exerted strains such as abrasion, thermal shock, impact and high level of load at elevated temperature.

The various combination of conditions in which refractories are used, make it necessary to manufacture a range of refractory material with different properties. The range refractories incorporate fired, chemically and carbonaceous bonded materials that are made in varying combinations and shapes for diverse applications.

Refractories are porous multi component and heterogeneous material contained of thermally stable mineral aggregates, a binder phase and additives.

Refractories find function in the following

i. As a major material used by the metallurgical industries in the internal linings of furnace for melting
ii. In vessels for holding and transporting metal and slag
iii. In furnace for heating before further processing
iv. In stack or flues through which hot gases are conducted

2.1.3.1 Properties of refractory clays

A good refractory should process the following properties (Okoroafor, 2001).

i. High refractoriness: - this is the ability of refractory material to withstand high temperature.
ii. High Resistance to thermal fatigue: - this is the ability of refractory material to withstand sudden changes in in temperature.
iii. High Resistance to abrasion: - this is the ability of refractory material to resist the rubbing action of material coming in contact with.
iv. High Resistance to corrosion: - this is the ability of refractory material to withstand chemical action of molten metal, slag erosion, hot gases that come in contact with.
v. High Refractoriness under load: - this is the ability of refractory material to resist cracks, deformation and subsequent crumbling under the prevailing condition of pressure and temperature.
vi. Low porosity and permeability: - this is the ability of refractory material to be impermeable to gases and liquids as possible.

vii. Low Thermal Conductivity: - this is the ability of refractory material to conserve heat in the furnace.

viii. Low Coefficient of thermal Expansion: - this is the ability of refractory material to undergo structural changes on heating and cooling without disintegration.

2.1.4 Classification of refractory

Refractories are classified on the bases off their chemical mineralogical compositions, metallurgical reaction behaviour and their Found application.

2.1.4.1 Classification base on chemical mineralogical compositions

Refractories can be classified base on their chemical mineralogical compositions as follows (Okoroafor, 2001).

i. **Silica refractories** :- silica refractories covers all silica refractories having Aluminia content less than 1.5 %, titania less than 0.2 %, iron oxide less than 2.5 %, calcium oxide less than 4 %, SiO_2 at least 93 % also their flux factor which is the percentage of total Alkalis is less. Silica brick are silica refractories which have found extensive use in the iron and steel melting furnace. In addition to its high fusion point, resistance to thermal shock and their high refractoriness. It finds use in glass making and steel industry.

ii. **Insulating refractories: -** They are characterized by low thermal conductivity, low heat capacity, low density and high porosity.

iii. **Alumina refractory: -** these are alumina silica refractories with 50 % or more alumina concentration range from 55 – 80 % and some metal oxide and silica up to 5%. The refractoriness of high Alumina refractory increases with increase in Alumina percentage. The 50 %, 60 %, 70 % and 80 % Alumina classes contain their respective alumina content with allowable range of plus and minus 2.5 %. High Alumina bricks are classified by their alumina content according to ASTM C-24 (1985).

a. Mullite Brick :- predominantly contains the mineral phase mullite

b. Chemically Bonded Brick: - usually phosphate bonded brick in the 75 % to 85 % alumina range. An alumina Orthophosphate bond can be formed at relatively low temperatures.

c. Alumina – Chrome Brick: - they are formed from very high purity, high alumina materials and chronic oxide. At high temperature Alumina and chromia form a solid solution, which is highly refractory.

d. Alumina Carbon Brick: - these are high alumina brick usually bonded by a resin containing a carbonaceous graphite. High Alumina Carbon Brick finds application in hearth and shaft of blast furnaces, ceramics kilns glass tanks and crucibles for melting a wide range of metals.

iv. **Special refractories: -** these include zircon, silica carbide refractories, fusion cast product, chronic oxide, carbon refractories and ultra-high alumina refractories.

a) Zircon Refractories: - zirconium dioxide (ZrO_2) is a polymorphic material. There are certain difficulties in its usage and fabrication as a refractory material. It is essential to stabilize it before application as a refractory. This is achieved by incorporating small quantities of calcium, magnesium, and cerium oxide. Its properties depend mainly on degree of stabilization and quantity of stabilizer as well as quantity of original raw materials. Zirconia refractories have very high strength at room temperature, which is maintained up to temperature as high as 1500°C (Olaiya *et al.,* 2015).

They are, therefore, useful as high temperature construction materials for furnace and kilns. The thermal conductivity of zirconium dioxide is found to be much lower than most other refractories sand the material is therefore used as high temperature insulating refractory. Since zirconia exhibit very low thermal loss and does not readily react with liquids metals, it is particularly useful for making refractory crucibles and other vessels for metallurgical purposes.

v. **Dolomite refractory: -** Dolomite mineral is a double carbonate of calcium and magnesium ($CaCO_3$. $MgCO_3$)

Theoretically dolomite contains 30.41 % CaO, 21.87 %MgO and 47.72 % CO_2.

Dolomite used for the manufacture of refractories should be a mixture of equimolecular proportion of $CaCO_3$ 54.35 % and $MgCO_3$ 45.65 %. Dolomite refractory should be hard and compact with uniform texture containing low percentage of Iron, silica, alumina as they adversely affect the refractories of Dolomite refractories. A limited number of dolomite deposits exist in the world with satisfactory uniformity, purity, and calcining behavior to be processed into high purity refractory at a reasonable cost. High purity refractory is greater than 97% CaO + MgO.

Dolomite refractories are found to be most compatible materials with the cement kiln clinker as they provide excellent coating stability, very good resistance to thermal shock and attack in varying kiln operating conditions. These refractories with zirconia enrichment are used for crack arresting (Olaiya *et al.,* 2015).

vi. **Carbon refractories: -** carbon refractories are light grey in colour and give a ringing sound when struck. They have high refractoriness that can attain any temperature and show no deformation under load of 2kg/cm^2 at 1750°C. Carbon refractory has a porosity of 20 – 25 % and a specific gravity varying from 1.5 to 1.9. They are not attacked by slags as they are not generally wetter by molten metal and gasses. They are used for the construction of furnace used in iron and steel industry.

2.1.4.2 Classification on the bases of applications

Refractories are also classified on the bases of their applications these are super duty, high duty, medium duty, low duty and also semi silica. These classes covers the range from approximately 18 % to 44 % Alumina and from about 50 % to 80 % silica (Olaiya *et al.,* 2015).

Super duty: - these refractories have good strength and stability of volume at high temperature and an alumina content of 40 % to 44 %. Some super duty refractories have superior resistance to cracking or spalling when subjected to rapid change of temperature. They are mostly used in steel industries.

High duty: - these refractories are used in large quantities and for a wide range of application because of their greater resistance to thermal shock, they find applications in cement, iron and steel industries.

Medium duty: - these refractories are appropriate in applications where they are exposed to conditions of moderate severity. They are used in making kilns furnace bricks and refinery.

Low duty: - low duty refractories find application in fire place, bakery oven and distillery.

Table 2.3 shows that as the quantity of impurities increases as the amount of Al_2O_3 decreases, the melting point of refractory brick decreases.

Table 2.3: Chemical composition of refractory base on application

Bricks	$\%SiO_2$	$\%Al_2O_3$	Other constituent	PCE (°C)
Super Duty	49–53	40–44	5 – 7	1745 – 1760
High Duty	50–80	35–40	5 – 9	1690 – 1745
Medium Duty	60–70	26–36	5 – 9	1640 – 1680
Siliceous	65–80	18–30	3 – 8	1620 – 1680
Low Duty	60–70	23–33	6 – 10	1520 – 1595

(Olaiya, 2015)

2.1.4.3 Classification on the bases of metallurgical reaction

Refractories can be classified based on their metallurgical reaction such as basic refractories, acidic refractories, and neutral refractories.

1) Basic Refractories

These are refractories which when subjected to high temperature exhibit the properties of base. Such properties include the following:

i. Turning red litmus blue

ii. Reaction with acids to produce salt and water

iii. Reaction with ammonium salts to generate ammonia gas.

Basic Refractories are used in areas where slag and atmosphere are basic. They are stable to alkaline materials but react with acids. Their main raw material belongs to RO group. Magnesia (MgO) is a very common example. Other examples include Dolomite and chrome-magnesite.

Basic Refractories were so named because they exhibit resistance to corrosive reactions with chemically basic slag, dust and fumes at elevated temperatures. Basic refractories are produced from a composition of dead burned Magnesite, Dolomite, and Chrome-ore or compatible mixture of Magnesite, Dolomite and Chrome-ore.

Composition of basic refractories

The principal raw materials used in the production of basic refractories are Magnesite, Dolomite, chrome-ore, spinel and carbon. Basic refractory bricks such as magnesia chrome and magnesia-spinel are made of synthetic magnesia clinker or dead burned natural Magnesite in combination with chrome-ore and preheated spinel clinker or with alumina coating material that form spinel during firing of the brick.

2) Acidic Refractories

These are refractories which when subjected to high temperature exhibit the properties of an acid such properties include the following

i. Turns blue litmus red
ii. React with base to produce salt and water
iii. React with carbonate to liberate carbon dioxide

These refractories found applications where slag and atmosphere are acidic. They are stable to acidic but attacked by alkalis. The main raw material of acidic refractories belongs to the RO_2 group such as Silica SiO_2, zirconia ZrO_2, and alumino-silicate clay (Al_2O_3 $.2SiO_2.2H_2O$). According to (Madugu, 2016) these refractories vary widely in their physical, chemical and mineralogical characteristics depending on the nature and proportion of silica and Alumina present in them.

3) **Neutral Refractories**

These are refractories that are used in area where slag and atmosphere are either basic or acidic and are chemically stable to both acids and bases. Their main raw material belongs but not confined to R_2O_3 group. The common example of these materials are alumina Al_2O_3, Chrome Cr_2O_3 and carbon. (Adamu *et al.,* 2018) revealed that alumino-silicate refractories are sometimes classified as neutral refractories but when exposed to basic slag they exhibit acidic reaction.

2.1.4.4 Classification of refractories based on method of production

The existing methods of manufacturing of refractories are as follows

a. Dry press process
b. Fused cart
c. Hand moulded
d. Formed (Normal, Fired, or chemical bonded)
e. Informed (Monolithic – plastic, Ramming mass, Gunning, Castable, Spraying)

2.1.4.5 Classification of refractories based on physical form

Refractories can also be 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 (Madugu, 2016).

1) Shaped Refractories

These are refractories that have fixed shape when delivered to the user and are referred to as bricks. Bricks shape may be divided into standard and special shape. Standard shape refractories have kilns and furnace of the same type. Special shapes are specifically made for particular kilns and furnace. This may not be applicable to another furnace or kiln of the same type. Shaped Refractories are almost always machine pressed this account for high uniformity in their properties. Special shapes are often hand moulded (Madugu, 2016).

2) Unshaped Refractories

Unshaped refractories are almost without definite form and are only given shape upon application. These types are known as monolithic refractories. These are categorized plastic refractory, Ramming mixes, castables, mortars, running and settling mixes.

Plastic Refractory: - These are mixtures prepared in stiff plastic condition and are delivered in blocks wrapped in polythene. During application, the blocks are sliced into pieces and without further preparation, are rammed o poured into place with pneumatic rammer. Plastics are easily rammed to any shape or contour.

Ramming mixes: - These are refractories whose particles size are carefully graded to make it workable. They 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.

Castables:- Castables by name implies a material of hydraulic setting in nature. These are materials containing cement binder usually Aluminates cement, which imparts hydraulic setting properties when mixed with water.

Calcium – Aluminates binder material needs to be properly stored to prevent moisture absorption. Further, its strength begins to deteriorate after a period of 6 to 12 months. These materials are installed by casting and also known as refractory concretes.

Gunning mixes: - These 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 furnace.

Fettling mixes: - These are also granular refractory materials, similar to gunning mix function but are applied by shovelling into furnace needing patching.

Mortars: - These are neither classified under refractory brick nor monolithic refractories. They are finely ground refractory material, which becomes plastic when mixed with water. They are used to bond the brick work into solid unit, to provide cushion between slightly irregular surfaces of the brick, to fill up spaces created by a deformed shell, and to make a wall gas tight to prevent slag penetration into the joints.

2.1.5 Method of manufacturing of refractories

Refractories are manufactured from naturally occurring materials in the earth crust such include clay, Quartzite, Magnesite, Chromite, Bauxite, etc. Although none of these naturally occurring materials is suitable for use as refractory directly without undergoing certain treatment and manufacturing processes. Steps involve in the production of refractories from naturally occurring materials is shown in Figure 2.1.

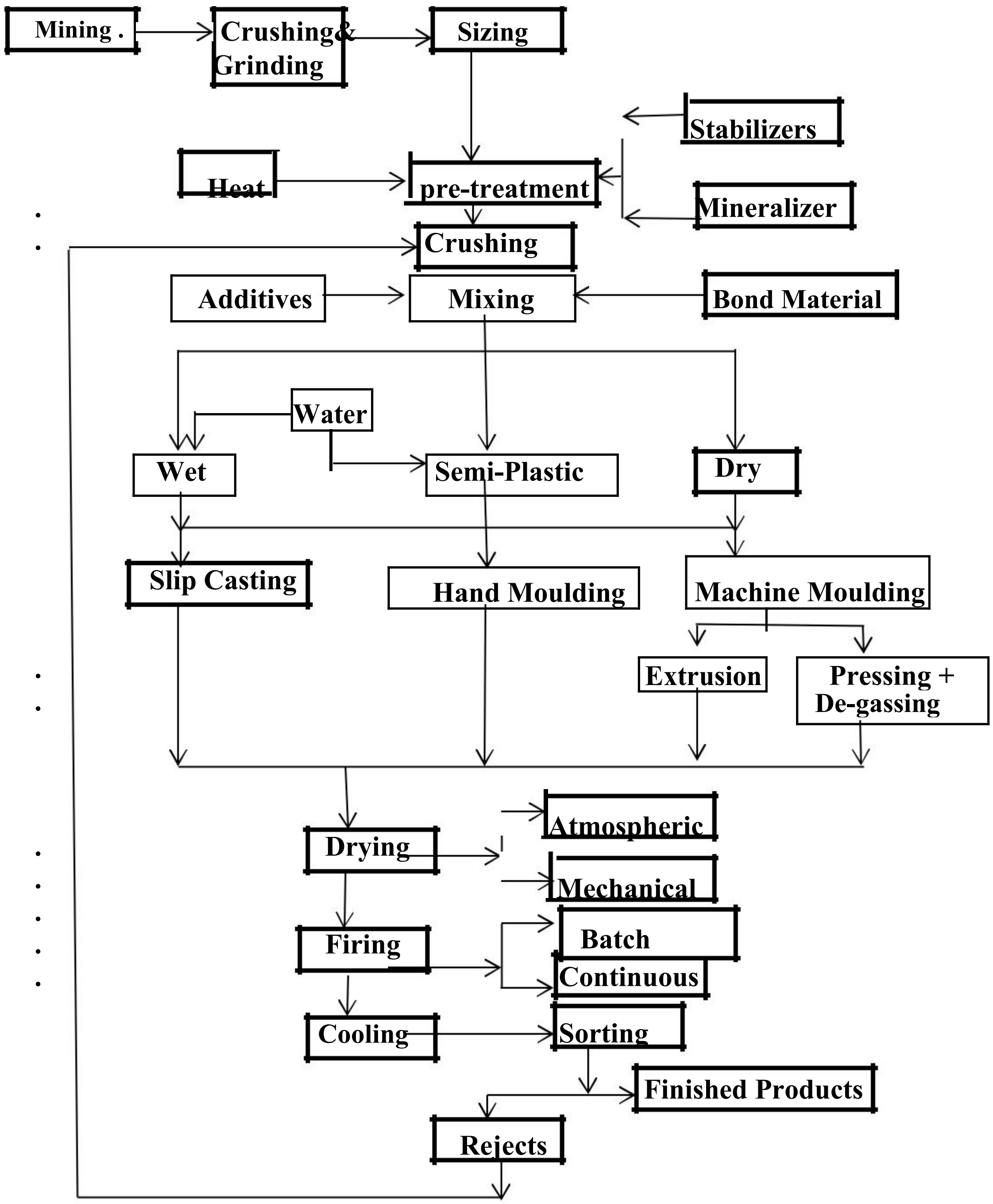

Figure 2.1: Flow Chart for Production of Refractory Bricks **(**modified**)** (Olaiya *et al.,* 2015).

2.1.5.1 *Mining and crushing*

Raw materials are dug from I identified location where it's deposited which is large enough to support small or medium scale production. With the use of various type of crushers such as hammer mill, ball mill, pulverizer, and press the materials are grounded to obtain proper size and ensure that the ratio of coarse to fine particles is even.

2.1.5.2 *Pre-treatment*

Primary pre-treatment given to these raw materials is firing or calculation at elevated temperature for considerable period of time to enable complete mineral conversation and hence stabilizes the materials. Firing temperature for various refractories are shown in Table 2.4.

Table 2.4: Firing Temperature for Various Refractories

Refractory Type	Firing Temperature (°C)
Fire Clay Bricks	1250 – 1400
High Alumina Bricks	1450 – 1550
Silica Bricks	1450 – 1510
Chrome Bricks	1450 – 1650
Dolomite Bricks	1450 – 1750
Magnesite Bricks	1450 – 1760
Direst Bonded Baric Bricks	1650 – 1760
Silicon Carbide Bricks	1370 – 1510

(Olaiya *et al.,* 2015)

2.1.5.3 *Mixing*

The raw material for refractory grounded and sieved is mixed with binding material to evenly distributed plastic state which will facilitate easy moulding. Mixing is usually carried out in pug mills for even distribution of the fine and coarse grains in the whole mass, pre calculated amount of water, additives, binding material and stabilizers are added and the mass is thoroughly mixed to ensure uniform composition of product, this mixture is allowed to remain as such for a day or more to ensure increased plasticity for easy moulding.

Mixing can be dry, semi plastic or wet. Up to 20% of water is used in wet mixing which is usually used for hand moulding. Semi plastic mixture has lower percentage of water to achieve semi state of plasticity. Dry mixture includes non-plastic basic mixes and clay mixes containing less than 5% of water. In this type of mixing spraying of water is used to achieve proper mixing. Due to high moulding pressure, semi plastic and dry mixture is machine pressed.

2.1.5.4 *Moulding*

Moulding is done manually with the use of Hand or mechanically with use of press or by extrusion. Moulding with hand is successful with wet mixtures containing about 20% of water and it is carried out in a wooden box, it is relatively cheaper than machine on a mobbing basis. Machine moulding is of practical application and a cheaper process for mass production of standard shapes. Hand moulded refractories have lower density and strength values than the mechanical moulded refractories. Machine moulded refractories have higher strength and density. Machine moulding can be used for semi plastic mixture using moderate moulding pressure.

Extrusion is usually used to get the rough or irregular shape of approximate dimensions which are subsequently pressed to shapes. Machine moulding of dry mixture requires a pressure of about 1000kg/cm^2 or more (Adamu *et al.,* 2018). Power pressing is used for moulding refractories involving dry compacting or slightly dry refractory powder mixture in metallic dies using sufficient higher pressure to produce strong and dense refractory shapes.

2.1.5.5 *Drying*

Drying is done at a slow rate to avoid voids and high shrinkage. Drying of moulded refractory increases its green strength by removing moisture and thus making them safe for subsequent handling. Drying is usually carried out under shade (not in direct sunlight) on large drying floor. Floor heated by waste heat from kilns are also used where refractories are laid out in arrays. Drying is carried out in predetermined set of conditions and drying rate is kept at minimum to avoid higher shrinkage and crack formation.

For faster drying of refractory of constant shape and size tunnel kilns are used where raw refractories are stacked in hours or placed on belt moving through a tunnel against steam of hot air.

2.1.5.6 *Firing or burning*

This involves burning of the moulded brick in kilns (down draught kiln or tunnel kiln) to remove water of hydration, vitrification and development of stable mineral forms. Shrinkage in volume of up to 30% occurs during burning. Burning or firing of refractories which follows their drying, facilitates development of stable mineral forms in them and high crushing strength of finished products.

2.1.5.7 *Cooling, sorting and storage*

The burnt bricks are cooled preferably with the oven or rotary kiln where the firing was done. On cooling, the burnt bricks are sorted. The good finished bricks are packaged while the rejects are crunched and grounded for recycling.

The refractory bricks must also be subjected to some laboratory test to certify that they are acceptable quality standard. Such test are listed below:

i. Refractoriness (Pyrometric Cone Equivalent PCE)
ii. Refractoriness Under Load (RUL)
iii. Apparent porosity
iv. Thermal shock resistance
v. Cold crushing strength
vi. Linear shrinkage
vii. Bulk density
viii. Permeability to air

2.2 Review of Related Works

A lot of research has been carried out on the conversion of clays for industrial uses and other applications.(Mohammed, 2009) reported in his work on refractory properties of termite hills under varied proportions of additives that over 80% of the total refractory materials are being consumed by the metallurgical industries for the construction and maintenance of furnaces Kiln, reactors, vessels and boilers, while the remaining 20% are being used in the non-metallurgical industries as cement, glass and hardware.

Worral and Ryan, (1975) carried out research work on the effect of adding deflocculant to clays maintained at constant density. They found that the main effect of adding deflocculant such as 5% sodium oxide and 5% sodium carbonate to the clay alter the yield value.

Aniyi and Adewara, (1986) investigated the refractory property of kankara clay and found that the clays are suitable for the production of refractory bricks possessing good shrinkage. The thermal conductivity value was 0.48w/m°C, refractoriness under load of 2kg/cm^3 was found to be 1405°C which was relatively low when compared with 1750°C for imported alumina fire bricks. He also discovered that kankara fire bricks softened and forms a glassy phase on the surface when heated above 1400°C in addition these bricks were porous with an apparent porosity of 38.07%.

Aderibigbe and Chukwuogo, (1984) carried out research work on clay deposit in Nigeria (Bus, Ukpor, Ozubulu, Enugu, Onibode, Odun, Oshiele, Ifon, Okpeke, Werrom, Sabon Gida, Alkaleri, Kankara, and Giro) revealed that the basic constituent of all clays were alumina (Al_2O_3) and silica ($2SiO_2$). In general, the alumina content were found to vary between 19.30% for Ozubulu clay and 39.30% for Onibode clay, while the silica contents were over 40% in all samples except that of Sabon Gida.

Aniyi, (1985) carried out a research work on the properties of kankara clay and discovered that, kankara clay found in katsina state was suitable for the production of refractory bricks with respect to adequate strength and thermal shock resistance values. However, they showed weakness in low refractoriness and high porosity which is responsible for low thermal conductivity properties. The refractoriness under load values was 1450°C. Addition of sawdust, asbestos and graphite to refractory clay give an improvement in the refractory properties. However, the bricks showed a sharp degradation in the crushing strength beyond an optimum limiting value of 15,000kn/m^2 in respect of either of them.

Hassan *et al.,* (1994) evaluated the refractory properties of some Nigeria clay (Oshiele, Onibode, and Bauchi clays). They discovered that the thermal spalling resistance of Oshiele and Bauchi bricks made of clay at 1300°C was low. Bauchi recorded 6cycles while Oshiele recorded 18 complete cycles. Bauchi clays have the highest apparent porosity value of 33.33% which was found to be slightly outside the normal range of 30% for dense refractory bricks. They concluded that Onibode clay produced the fire bricks with best overall refractory properties. Oshiele and Bauchi clays produced refractory bricks with inferior properties with respect to low shrinkage valuesand low bulk density. It was recommended that further investigation to reduce the shrinkage value of onibode clay and to improve the apparent porosity of Bauchi clays.

Aniyi and Adewara, (1986) of Metallurgical Engineering Department, Ahmadu Bello University, Zaria wrote on the refractory properties of kankara clay they recommended the clay for production of refractory fire bricks since it processes good cold shrinkage and thermal conductivity. However, they noted the high apparent porosity of 38.07% and softening temperature of 1400°C as its major disadvantage.

Refractories are mineral and chemical-based materials with very high heat-resisting properties, which make them ideal for use in the construction of oven and furnace walls, ceilings, and associated elements of iron and steel industry blast furnaces, glass manufacturing tanks, cement kilns, hot stoves, ceramic kilns, open hearth furnaces, nonferrous metallurgical furnaces, and steam boilers. Refractory materials can be made from clay refractory and non-clay refractory. Non clay refractory are been made of alumina, zirconia, silicon carbide, chromium, magnesite, graphite and other less common materials, but the cost of these non-clay refractory is much higher than that of fire clay (Atanda *et al.,* 2012).

Refractories have been an essential element in heat engineering plants since the 1960s, where they were successfully used to improve performance and energy efficiency. This is because refractories are chemically and physically stable at high temperatures depending on the operating environment. Refractories need to be resistant to thermal shock and be chemically inert. Good fireclay refractories should always have 24% - 26% plasticity and the shrinkage after firing should be within 6% - 8% maximum. A good fireclay refractory should also not contain more than 25% Fe_2O_3 (Atanda, *et al.,* 2012).

Under actual conditions of operation, refractories are subjected to mechanical loads. For this reason, refractory materials are assessed by their ability to withstand loads at high temperatures (Moorkah and Abolarin, 2005).

Sanni, (2005) defined fire clays as clays with high refractoriness (resistance to high temperature) and possess the capability of maintaining both physical and chemical identity at high temperatures (resistant to fusion and softening at elevated working temperatures) the clays used for furnace linings in metallurgical industries are classified as refractory clays. However, the degree of refractoriness and plasticity of any clay material is often influenced by the amount of the impurities contained in them. Moreover, the ability of selected refractory clay to withstand high temperature and resist physical and chemical corrosion determines the quality and the suitability of such material for use as furnace lining.

Clays when fired, lose their chemically bonded water and plasticity at about 500°C, thus acquiring higher mechanical strength between 950 °C and 1350 °C as the firing temperatures progresses (Al-Amaireh, 2009).

In recuperators where maximum heat transfer is desired to take place, the utilization of refractories with high thermal conductivities is highly desired. (Chesti, 1986) defined refractories as materials that are "hard to fuse" and classified them as materials that can withstand high temperatures, resist the action of corrosive liquids and withstand the thermal stresses imposed by dust-laden currents of hot gases or vapors without losing their insulating properties and wall rigidity.

A material is refractory in nature if it has a very high melting point in addition to its physical, chemical mechanical and thermal properties that make it suitable for use in furnaces, kilns, reactors and other high temperature vessels as reported by (Solomon, 2002). In his investigation concluded that the properties of refractory clay samples compare favourable with imported fire clay refractories.

Experimental investigations by Olphemvan, (1963) on the theological properties of four different clays (Oza, Ewu, Ere, and Owe) in formal bender state of Nigeria revealed that the clays have very low amount of flying or alkali oxides (0.30% – 1.39%). In addition, the alumina content of the clay deposits at Ere and Owe were fairly high and that Ewu clay was very plastic and also most of it particles were perhaps of colliding dimension.

Experimental analysis on the refractory properties of local clay material for furnace building by Obi, (1995) revealed that all the clay samples were classified as low alumina with kaolinic nature. The physiochemical analysis of the samples investigated showed that the clay can be used to produce fire bricks with good cold crushing strength with excellent resistance to thermal shock and intermidiate porosity values. It was observed that other properties like refractoriness under load linear shrinkage, resistance to neither slag attack for two samples nor fit into any of the imported bricks therefore limit their application.

Investigation carried out by Hussani, (1997) on the refractory properties of bricks produced from five Nigerian clays revealed that All the samples were found to possess good refractoriness (PCE) with excellent firing shrinkage values that fall within the 3 – 5% range of the imported bricks. The thermal shock resistance also found to be very good ranging from 26^{+} to 30^{+} cycles. The pyrometric cone equivalents (PCE) were high ranging from 1,650 – 1,780°C which showed that they could be used for making refractory bricks of medium temperature furnace. Furthermore, almost all the porosity of the samples clay falls within acceptable range of 16% - 30%.

Olusola, (1998) Investigated the properties of Zungeru clay material for high temperature applications. He discovered the firing shrinkage value to be 9.18%, apparent porosity 22.0%, bulk density 1.948g/cm3, permeability 80.4 .And specific gravity 2.82. These values fall within acceptable standard for dire clays. The thermal shock resistance was found to be 15 cycles and the refractoriness exceeds 1,200°C. The refractoriness showed that it can be used for medium temperature furnace.

Arowolo, (2000) In its work investigation of local clays for the production of electric cooker plate and electrical insulator revealed that sample clay changed from dark brown to reddish brown after firing which compared internationally, the clay having moisture content of about 7% and water of plasticity content 40%. This was found to be good for mouldability of porcelain high tension insulator and cooker base. The sample clay having thermal shock resistance of 30^{+} and 20^{+} cycles at a temperature of 1200°C, Apparent porosity of 20% at 1200°C, the clay sample shows permeability of 33.15 and refractoriness temperature range of 1200°C – 1500°C and it is recommended for use in furnace lining.

Onyejaka, (1994) in her report investigated the refractory properties of Ukpor clay. The clay was seen to possess good refractory temperature of 1700°C with a low shrinkage value of 6.8%. The alkali content, porosity and water absorption capacity was found to be good enough for refractory purposes. In spite of her inability to conduct Pyrometric Cone Equivalent test and refractoriness under load, she recommended the clay for refractory purposes.

The test conducted by Zubairu, (1997) on the bricks made from kauranndole, kujama (Borno), jamkasa- malaki and Maraban Rido clays in kaduna state revealed that the mouldability of the bricks with average water content 13.00% by weight was found good enough for all the four clay samples. All the clay samples show very low shrinkage values ranging from 2.17 – 4.15% with Kujama (Borno) clay having having the highest shrinkage value.

The test conducted by Agha, (1998) on the bricks from kauranndole, kujama (Borno), jankasa – Malali, and Marban Rido clays in kaduna state of Nigeria revealed that the mouldability of the bricks with average water content of 13.00% by weight was found good enough for all the four clay samples. All the clays samples showed very low shrinkage values, Kujama and kauranndole clays show porosity values of 21.7% and 19.90% respectively compared with 20-30% for typical fire clay porosity. Clays bricks of Kauranndole showed appreciable value of 19.621 and 14.285 KN/m2 of forming direction. This is good compared with fire clay bricks. The clay samples from Borno and kauranndole are capable of being used for furnace bricks of which the temperature limit would not exceed 1400°C.

According to Omotoyinbo and Oluwole, (2008) in their work titled Working properties of some selected refractory clay deposit in South western Nigeria it was reported that the clay samples contain Aluminium oxide and silica as major constituent making them suitable for Aluminium-Silicate refractory material. It was also reported that two of the samples collected indicated high refractoriness due to high content of Aluminium oxide. The cold crushing strength and thermal shock resistance of the two samples as well as porosity of all the batch samples are high while the linear shrinkage values are low. They concluded that the two samples mentioned above are suitable for furnace lining in non-ferrous metal processing.

Lawal *et al.,* (2008) in their work Characterization of the refractory property of Osiele clay, revealed that the clay was silicious alimino- silicate with low content of Iron III Oxide. The water absorption, bulk density and apparent porosity decreased with the firing temperature, whereas the total shrinkage increased as the firing temperature increased. It was concluded that Osiele clay can be used as refractory material for the lining of furnace particularly for operating temperature in the pre-1100°C range.

Loto and Omotosho (1990) Investigated on the suitability of Igbokoda clay in Ondo State as a binder for synthetic moulding sand, it was discovered that this clay was mainly kaolinite and has good binding properties. While they added additives such as cassava flour, sodium carbornate and coal dust to enhance the mechanical properties for binding purposes. Addition of cassava flour, sodium carbonate, and coal dust as additives on Igbokoda clay was discovered to have improved the mechanical properties of the clay for binding purposes. The bricks produced during the experiment were sound and without any defect.

Akinbode, (1996) carried out investigation on the properties of termite hills as a refractory material for furnace lining. In his report, he observed that the refractory properties of termite hill material which include porosity, density, dimensional change and permeability are very similar to known refractory materials.

Folaranmi, (2009) Investigated the effect of additives (sawdust and ashes) on the thermal conductivity of clay, result obtained showed that with sawdust addition the clay was suitable as oven materials as well as good insulator.

Abolarin *et al.,* (2004) studied the characteristics of Nigerian clays and discovered that the Barkin Ladi and Alkaleri clay samples were suitable for construction of furnace and furnace lining.

Madugu, (2016) worked on the refractory properties of kankara clay. The clay was reported suitable for the production of castables when mixed with about 5% fondu cement. The castables were reported to be capable of being used as refractory insulator.

Abdullahi and Mahadi, (2008), in their work titled comparative study of some refractory properties of some selected clay deposit in Biu and Hawul Local government area of Borno state, revealed that the highest temperature obtained were for Gula and Kwayabura 1400°C and those for Hema and Mangada 1300°C but fire clay refractory should have refractoriness in the range of 1500°C – 1700°C (Chester,1973; Chesti, 1986) which implies that the usage of the clay deposit in the two LGAs is restricted to non-ferrous materials processing.

According to Chukwudi, (2008), in his work Characterization and evaluation of the refractory property of Nsu clay deposit in Imo state reported that on the basis of physio chemical characteristics of this kaolinitic fire clay deposit, it can successfully be processed for use as refractory materials such as ladle bricks and ramming mass.

Clays from five major towns in Baruten Local Government Area, Kwara State, Nigeria were examined by Yaru *et al.,* (2018) using ASTM C-484(2019) guidelines to determine their suitability for refractory applications. The clay samples were classified as Alumino-Silicate refractories due to high values of Al_2O_3 and SiO_2. The results showed apparent porosity (19.4% - 25.6%), bulk density (1.83-1.90 g/cm^3), cold crushing strength (38.7-56.1 N/mm^2), linear shrinkage (4.4% – 9.3%), clay contents (52.71% - 67.83%), moisture content (17.0% - 23.6%), permeability (68-82 cmsec-1), plasticity (16.7% - 30.4%), refractoriness (>1300°C) and Thermal Shock Resistance (23$^+$ - 25$^+$ cycles) for the clay samples, which were measurable with the established standards for fireclays, refractory clays/brick lining or alumina-silicates and kaolin. They concluded the natural clays could suitably replace imported clays in some refractory applications.

Olaiya *et al.,* (2015) in there Experimental Investigation and Suitability of Alagutan Dolomite Deposit as Refractory Raw Materials revealed that dolomite samples from Igbeti (Oyo state) and Alaguntan (Kwara state) are suitable for lining of furnace where materials requiring basic environment can be melted because of high percentage of calcium oxide and magnesium oxide in there composition. This is consequent to the fact that their chemical composition and physical properties are comparable to the values obtained by earlier researchers on dolomite refractories in other parts of the world. However, the maximum operating temperature of such furnaces should not exceed 1778°C. It was also revealed that the chemical composition from suspected deposit at Ijoko, Ibamajo, and Onibode are acidic due to high percentage of silicon oxide and aluminium oxide in their composition. Further investigation on their refractory properties show refractoriness of 1778°C for each of them. The bulk density of Igbeti and Alaguntan was found to be 2.84g/cm^3 and 2.76g/cm^3, apparent porosity of 19.28% and 22.74%, permeability test of 88mm/s and 102mm/s, linear shrinkage of 0.2 for both and 23$^+$ cycles was obtained for Igbeti sample while Alaguntan gave 18$^+$ cycles. He concluded that the refractory produced of dolomite from Igbeti and Alaguntan deposit are suitable for furnace lining where basic environment is required.

The performance evaluation of refractory bricks produced from locally sourced clay materials from Oghara, Ekpan, Ubeji and Jeddo communities in Delta State, Nigeria have been studied .The results showed that these local clays investigated are suitable to produce locally available refractory bricks which was found to compare favourably with the imported refractory bricks. Refractory material produced from Oghara clay was found to be the one with the best properties of all the clay investigated from Delta State, Nigeria. It had a bulk density (1.74g/cm^3), porosity (31.44%), cold compressive strength (100kg/cm^2), shrinkage (5%), and loss of ignition (12.18%), respectively. Its properties were found to be comparable with the imported castable refractory brick with bulk density (2.60g/cm^3), porosity (35.21%), cold compressive strength (325kg/cm^2), and shrinkage (7.74%), loss of ignition (5-53%), respectively. They concluded that the local clay deposit at Oghara is considered suitable for exploitation and would be used for the wall linings of our high temperature equipment in Warri Refining and Petrochemical Company (WRPC) and other local industries in Nigeria (Osarenmwinda and Chukwuemeka, 2014).

Ezeofor *et al.,* (2018) investigate the industrial potential of Ebiaji clay the result shows the clay is alumino silicate that can be used in paper industry, ceramics, production of refractory bricks and tiles but require additives to obtain desired properties. Moreover, Rabiu *et al.,* (2019) said beneficiated dolomite can be used as additives in moulding sand casting.

Government, researchers and engineers trying to introduce the clay bricks with the value addition of natural agro waste materials. With introduction of groundnut shell ash to clay samples to increase the physical properties of the clay, the average density of clay bricks decreases with increase in the groundnut shell ash percentage, the decrease behavior can be attributed to the coating of the clay with ground nut shell ash, that result to larger particles with larger voids and less density of the baking temperature, density increase with increase with temperature to certain limit. According to his statement clay bricks increase from 1800kg/m^3 to 2200kg/m^3 while increasing baking temperature from 900°c to 1100°c respectively which is decreasing from 2200kg/m^3 while increasing the baking temperature from 1100°c to 1200°c the effect depends on the chemical and physical behavior of the clay materials. Not only that this effect is unlike related to normal clay brick due to the mixing of ground nut shell ash. The formation of pores could be ascribe to the presence of unstable organic compound in the ground nut shell ash that burn off during the firing process however, the reduction in density is a useful outcome that revealed the potential use of the fired clay brick as light weight building material (Fayomi *et al.,* 2011).

According to Nuhu and Abdullahi (2008), the impurities in fire clays are limonite, pyrites, quartz, calcites, ferrous carbonates and some organic compounds. The organic impurities impart plasticity to the clays while impurities such as quartz and iron reduce their refractoriness. However, refractory clay material obtained from a single site cannot possess all the required properties that will make it a perfect refractory material, hence, it becomes imperative to select clays based on the physical, chemical, and thermal, analysis of samples. The selected refractory clay will have to be beneficiated with refractory clay material from other sites and be properly blended with other additives to improve their physical, thermal, and chemical properties of the final product.

According to Ndaliman, (2001) Termite hills can be used to produce insulating refractory when 25% additives (corn husk and sawdust) are used. However, low values of refractoriness 1200°C were recorded.

In yet another investigation which sought to increase the refractoriness, Ndaliman, (2000) used 25% each of graphite powder and asbestos with termite hill material. It was found that the refractoriness of 1900°C for graphite powder and 1600°C for asbestos were obtained in addition to improvement recorded in other properties.

The effect of cowdung and graphite on the properties of local refractory clays investigated by (Lawal *et al.,* 2005) revealed, cowdung reduced the refractoriness, bulk density, compressive strength and thermal shock resistance of all the clays examined in the range of 5% to 25%. However, it increased both the porosity and permeability to air. All the clay possessed reasonable refractoriness which range from 1500°C to 1700°C. Addition of graphite to the clays in the range of 15 to 25% generally improved their refractoriness and bulk density while thermal shock resistance reduced alongside compressive strength.

Akinbode, (1996) carried out an investigation on the properties of termite hill as refractory material for furnace lining. In his report he observed that the refractory properties of termite hill possess a close relationship between porosity, density, dimensional change, and permeability of the known refractory material for furnace lining. The thermal shock resistance and the cold crushing strength were found to be low when compared with other refractory material. The refractory property of the termite hill revealed that it can be used for lining of furnace operating at temperature below 1500°C. He however reiterated that other properties could be improve if additives like sawdust asbestos graphite, rice husk ashes, corn husk ashes, and sugar cane bagasse are added to the termite hill. In addition clay or organic binders could be used to improve the refractoriness property of the termite hill.

Falodun *et al.,* (2017) carried investigation on an improvement on the refractory properties of Ikere clay by the addition of high alumina cement and silica sand (SiO_2) with the use of Atomic Absorption Spectrophotometer to determine the physico-chemical composition of the clay. Samples containing mixture of different quantities of alumina cement (5, 10, 15, 20, 25 and 30 wt.%), fixed silica sand content of 10 wt.% and a small quantity of water to make them plastic were prepared. Tests of apparent porosity, bulk density, linear shrinkage, thermal shock resistance, compressive strength and refractoriness were carried out on the fired samples. The refractory properties investigated were greatly improved by the addition of silica sand and cement additions. The result revealed that the refractoriness increase from 1450 °C to 1600 °C, while compressive strength increased from 4856.50 N/mm^2 to 6522.49 N/mm^2 at 15wt% alumina cement before a downward trend was observed above 15% alumina cement. However linear shrinkage, bulk density and apparent porosity of the samples were desirably reduced by the addition of alumina cement. For all the properties tested they concluded that 10% silica sand and 10 –15% alumina cement gave the desired requirement for the production of a refractory material suitable for refractory applications.

Hassan, (1985) investigated the effect of some additives like sawdust, graphite, and asbestos on kankara clay. He discovered that a good thermal insulating fire clay bricks could be produced from this clay by addition of graphite and sawdust (15%). It was also found that the density of the fire clay brick with adequate strength at operating temperature could be made by mixing the clay with up to 25% asbestos.

Odo and Mba, (2008), in their work titled Effect of Agricultural waste ash additives on refractory properties of a blend of two Nigerian clays, it was discovered that addition of ash residues from agricultural waste gave an improvement in the apparent porosity and water absorption of the blended clays. The values of transverse strength and density were slightly reduced as a compensation for increased porosity in the ash ceramic composite bodies.

Investigation into Refractory properties of termite hills with varied proportion of additives by (Ndalima, 2006), revealed that the clay of termite hill indicated high silica content while other constituent are normal as obtained in clay refractories. It was also reported that properties such as refractoriness and linear shrinkage remains the same as the proportion of the additives were increased. However, significant improvements were obtained because of the additives. They further revealed that thermal shock resistance and specific gravity decreased gradually, as additives were increased, while the density, porosity, and permeability varied in reverse. They concluded that it was possible to select percentage composition that gives compromised values of certain properties.

Okoroafor, (2001) evaluated the sutability of some Nigeria clay for producing furnace bricks (Kpakungu, Chanchaga, Zungeru and Bida clays) he recommended the clays for producing furnace bricks having refractoriness temperature range from1470°C - 1680°C and possesses good cold shrinkage and thermal conductivity. However, he noted all the clay to having their greatest apparent porosity at sintering temperature of 900°C and recommended on more research to improve on the clays porosity.

Clay of Obe characterized by Ugwuoke *et al*., (2017) shows the clay belongs to alumino silicate and the physical analysis depict the clay has properties within the acceptable range for international standard for refractory production.

The shrinkage properties of the Ipetumodu and Awo clays compared with standard shrinkage property. It was observed that the shrinkage properties of both mixes were comparable with standard shrinkage at about 20-25% cement content and decreasing until 40% cement content. Too much shrinkage of refractories will lead to spalling the thermal cycling test for the clay mixes. The clay cement mixes with high shrinkage properties were susceptible to thermal shock. Thus increasing cement content increased resistance to thermal shock and hence thermal spalling, the apparent porosity decreasing with increasing cement content. While compressive strength increasing with increasing cement content, apparent porosity and cold crushing strength are superimposed on each other as porosity is decreasing cold crushing strength increases. And as apparent porosity is decreasing, resistance to thermal shock is increasing, while porosity decreased, thermal conductivity increased and with increasing the cold crushing strength, resistance to thermal shock increased while the shrinkage and thermal conductivities superimposed on each other as shrinkage decreased, thermal conductivity increased. Shrinkage and cold crushing strength properties are superimposed on each other as shrinkage decreased, crushing strength increased. The shrinkage and porosity properties of the moulding sand with increasing cement content shows that the porosity decreased with decreasing shrinkage. While the shrinkage and thermal shock resistance properties vary with Increasing cement content. It shows that as shrinkage decreased, thermal shock resistance increased. This is also known as resistance to thermal spalling (Atanda *et al.,* 2012).

Sarmad *et al.*, (2018) in their work improving the thermal and physical properties of fire clay refractory by added magnesia, shows that that porosity of clay increases and density decreases with the proportion of magnesia oxide added where such increase and decrease affect the thermal properties of the refractory.

Clay samples from Sokoto state prepared using standard methods were tested for properties such as apparent porosity, bulk density, cold crushing strength, thermal shock resistance, firing shrinkage, and refractoriness. The result of the chemical composition analysis shows that the, the clay is rich in Oxides of Silica (SiO_3), Aluminum (Al_2O_3), and Iron (Fe_2O_3) with other oxides in trace amount. The overall experimental analysis carried out, shows that all the samples are siliceous in nature and of the alumino-silicate refractories that are classified as kaolinitic fireclay with appreciable and reasonable values of the refractory properties that are comparable to the standards. On the basis of the physiochemical characteristics of this kaolinitic fireclay deposit, it can successfully be processed for use in the paper industry as filter, in furnace lining and in the development of improved wood burning stoves in addition to usual pottery activities (Aliyu *et al.,* 2013).

Clays samples of Ifon, Ipetumodu, and iseyin investigated by Aramide *et al.,* (2014), Clays of Ifon contains low kaolinite (5.63%) which could not be used for making high temperature caliber refractories except with the addition of some additive that will improve on their refractory properties because of their high content of feldspars which favor liquid phase formation and densification at low temperature but it could be processed for the production of feldspar for glass and iron making industries. Clays of Ipetunmodu is considered to be appropriate for the production the refractory composite having appropriate content of both kaolinite (23.74% kaolinite) and feldspars (26.12% microcline and 11.28% plagioclase/albite) which is necessary for producing mullite fibers in ceramic matrix at a temperature of around 1400°C. Clay of Iseyin, which contains very low feldspars (3.00% microcline and 3.08% plagioclase/albite) and high content of kaolinite, was considered suitable for further processing for making high temperature caliber refractories.

The sample compositions containing between 20 - 30% Portland cement and 70 - 80% clay of either Awo or Ipetumodu would serve well for heat treatment furnaces due to their light weight, moderate porosity, minimum shrinkage and medium strength with additions of non-linear expansivity materials to increase thermal shock resistance (Atanda *et al.,* 2012).

The relatively low porosity values of Ado and Isan clay compared to Ikere, Ilawe and Ire clay could be due to presence of low content of combustible materials in them. The presence of pores in clay affects the strength by reducing the cross sectional area expose to an applied load, hence samples Ado and Isan clay when used for furnace lining are expected to be able to support the weight of materials charged into the furnace better than sample Ilawe, Ikere and Ire. Because of decrease in number of pores created in them, they are capable of having good resistance to the penetration of molten slags, metal and flue gases when used as furnace lining (Oshuoha *et al.,* 2014).

Titiladunayo and Fapetu, (2011) in their work selection of appropriate clay for furnace lining in a pyrolysis process(Ikere Ekiti, Fagbohun Ekiti, Isan Ekiti clays), discovered Ikere Ekiti clay to be less dense, contain more porosity and possesses low iron content 2.07% with higher refractoriness with fusion temperature above 1500°C, Fagbohun Ekiti clay was said to be suitable for medium thermal application in furnace kilns and stoves while Isan Ekiti clay could be beneficiated to improve on their physical and chemical properties of the clay.

The proportion of elemental Composition of iron in clays determines the thermal conductivity potential of such materials; hence Sanni, (2005) suggested that any fire clay to be used in refractories should have at least 30% Al_2O_3 and less than 1.8% Fe_2O_3. The proportional increase in the ratio of Al_2O_3 in samples will undoubtedly improve clay refractoriness, whereas a progressive reduction in Fe_2O_3 content of samples will perhaps lower their thermal conductivity in that order. This probably suggests why Ikere clay would be regarded as having better insulating property than the samples from other locations (Titiladunayo and Fapetu, 2011).

The organic impurities impart plasticity to the clays while impurities such as quartz and iron reduce their refractoriness. However, refractory clay material obtained from a single site cannot possess all the required properties that will make it a perfect refractory material, hence, it becomes imperative to select clays based on the physical, chemical, and thermal, analysis of samples. The selected refractory clay will have to be beneficiated with refractory clay material from other sites and be properly blended with other additives to improve their physical, thermal, and chemical properties of the final product (Nuhu and Abdullahi, 2008).

High-temperature ceramic coatings are seldom used in refractory applications, since typically the refractory is too rapidly consumed or worn away in use. However, in certain applications, the life of a refractory metal or ceramic part may be extended by applying a high-density refractory material to the surface by flame or plasma spraying (Masin, 2013).

The shifts in metallurgical process and the aggravation of operating conditions by the use of oxygen have also caused changes in the qualities to be satisfied by refractories. The development of refractory products and of the lining technology has had to be adapted to these changed requirements (Moorkah and Abolarin, 2005). Investigation into Eha- Ndiagu clay deposit reveals the clay to be good with industrial potentials but with the incorporation of additives that will help to obtain the desired properties (Iloabachie *et al.,* 2020).

Thermal conductivity decreases in refractory materials as its porosity increases with the pores acting as non-heat conducting media. Porous refractories have air entrapped in them. Moreover, the porosity of refractory materials is determined by the amount of air entrapped in its pores and consequently a measure of its insulating quality. Hence, when the porosity of refractory material is high; its thermal conductivity will be low and vice versa. Therefore, the refractories used in melting furnaces, are made to have low thermal conductivities, ensure minimal heat loss and maximum heat retention, and to guarantee large temperature variation within the thermal envelope with maximum energy conversion efficiencies (Titiladunayo and Fapetu, 2011).

Omotoyinbo *et al.,* (1997) gave factors that are known to affect porosity to include composition, size and shape of clay particles others include ramming pressure, and reaction occurring on firing. According to Shuaib and Mudiare, (2017) porosity determines the resistance of the material to penetration of molten slags, metal, and flue gases.

CHAPTER THREE

3.0 MATERIALS AND METHODS

3.1 Materials

The materials required for the investigation are listed below. However the chemicals used are sourced locally off shelf from Pamlac Chemicals shop Minna, Niger State.

Table 3.1: Materials and reagents

SN	MATERIALS AND REAGENTS	SOURCE
1	Distilled water	Pamlac chemicals
2	Clays	Maikunkele, Bida, Kpakungu and Chanchaga
3	Rice husk ash	Tungagoro, Minna, Niger State.
4	Saw dust ash	Shango sawmill, Minna, Niger state.
5	Corn husk ash	Kadna, Minna, Niger State.

Table 3.2: Equipment and apparatus

SN	EQUIPMENT	SPECIFICATION	MANUFACTURER
1	Magnetic stirrer	Model 300	Gallenkamp, England
2	Beaker	0.250ml	Pyrex, England
3	Funnel	Plastic	Pyrex, England
4	Filter paper	Medium	Beijing, China
5	Vials	50ml	Interlab, sethi
6	Round bottom flask	Pyrex	Pyrex, England
7	Measuring cylinder	50 – 250 ml	Gallenkamp, England
8	Furnace	MD010	Daiki Scientific co. Korea
9	Oven	Memmert 600	Gallenkamp, England
10	Weighing Balance	Modiel i2006	Citizen, japan
11	Firing Kiln	0-250 ml	Pyre, Hungary
12	Electronic weighing balance	Pro SP202	Ohaus Corp, Pine Brook NJUSA
13	Pair of tongs		Gallenkamp, England

3.2 Methods

In analyzing the clays the following methods were adopted for each process.

3.2.1 Sieve analysis of selected clay samples

A mechanical vibrator having several sieves stacked in order from the smallest to the largest starting at the bottom with the pan below the smallest sieve was used for the sieve analysis using the ASTM standard testing procedure ASTM (1985). Clay samples of 500g after being washed and dried was introduced into the sieve, vibrated and separated into discrete size ranges for about 15 minutes. This vibration allows irregularly shaped particles to reorient as the sample fall through the sieve. At the end of the 15 minutes vibration, the mass retained on each sieve and in the pan was weighed. This process was repeated for all clay samples used. Equations (3.1) to (3.3) was used to determine the values of the cumulative mass retained, percentage cumulative retained and percentage passing respectively.

$$C_{MR} = I_{MR} + C_{MRn+1} \tag{3.1}$$

$$C_{PR} = \frac{CMR}{\quad} 100 \tag{3.2}$$

$$P_P = 100 - C_{PR} \tag{3.3}$$

Where:

C_{MR} = Cummulative mass retained

I_{MR} = Individual mass retained or Weight of soil retained

C_{PR} = Cummulative percent retained

P_P = Percent passing

M = Mass of sample

3.2.2 Characterisation and determination of physio-chemical composition of selected clays

Samples of the four clay materials form Bida, Chanchaga, Kpakungu and Maikunkele were analyzed to determine their chemical constituents. The chemical analysis was done using Xray-fluorescence (XRF) analysis machine.

Consequently, samples were loaded in the XRF machine model (XRF Bruker S4 Pioneer) for elemental analysis. The machine was operated at maximum voltage and current 60 KV and 1 mA respectively to generate the X-rays which will energize the sample for a particular time (specifically, 10 minutes) with X-ray tube of rhodium anode and scintillation detector of current 40 mA and voltage 40 mV (Shehu *et al.,* 2017).

3.2.3 Raw material beneficiation and production of clay bars for refractory test

The raw clay samples of 500 g were air dried before processing. The raw dry clay samples were crushed in a mortar to small grain sizes. The samples were soaked in a plastic container of water and allowed to soak for three days. The clay was dispersed in excess water in a pretreated plastic container and stirred vigorously to ensure proper dissolution. Mineral constituents, soluble alkalis and dead organic matter were removed by washing during the soaking. This treatment was done since the presence of alkali oxides (Na_2O and K_2O) tend to retard mullite formation, hence lowers refractoriness and strength of clay brick. The dissolved clay was then filtered through a 0.425 mm mesh sieve to get rid of unwanted particles and plant materials. The filtrate was filtered further by the use of a mesh sieve of size 0.18 mm in order to obtain finer particles. The filtrate was allowed to settle for three days after which excess water was decanted off. The clay slip obtained was sun dried for 2 days and then dried in an oven at 100°C.

The processed dried clay was pulverized and then passed again through a 0.18 mm mesh sieve. Each of the clay samples was mixed with water and molded using different mould shapes and sizes that suited the respective tests they were to be used for. Bida, Chanchaga, Kpakungu and Maikunkele clays were blended with saw dust ash (SDA), rice husk ash (RHA) and corn husk ash (CHA) in different ratios as shown in Table 3.3 to develop different test samples.

Table 3.3: Different additive ratio of clay mixture

Clay /additives	M_1		M_2		M_3		M_4		M_5		M_6		M_7	
	Wt(g)	(%)	Wt(g)	(%)	Wt(g)	(%)	Wt(g)	(%)	Wt(g)	(%)	Wt(g)	(%)	Wt(g)	(%)
Clay	250		237.5		225		212.5		200		187.5		175	
RHA	-	-	5.00	2	7.50	3	12.5	5.0	15	6	20	8	25	10
CHA	-	-	3.75	1.5	10.0	4	12.5	5.0	17.5	7	20	8	25	10
SDA	-	-	3.75	1.5	7.50	3	12.5	5.0	17.5	7	22.5	9	25	10
TOTAL	250	0	250	5	250	10	250	15	250	20	250	25	250	30

Where:- RHA is Rice husk ash, CHA is Corn husk ash, SDA is Sawdust ash, Mwt(g) is mass of clay sample & (%) is percentage of added additives.

Each of the samples (250 g) was mixed with 35% (0.087 Liters) of water to make the clay plastic for moulding. The clay was then molded into three different shapes using pop moulds with the application of powder lubricant to the surface of the moulds to prevent the test pieces from sticking to the surface. The moulds were used for forming the shapes. An improvised wooden material was prepared which was used for transmitting the moulding pressure of 2 MPa to the mould when the required quantity of plastic molding mass was put into the mould. After pressing, a suitable wooden plunger was used to extrude out the green brick from the mould. The test samples were sun dried, oven dried to 110°C and finally fired to 1150°C before testing for the respective properties.

Showing on Plate I-IX are some clay samples and production process of the refractory bricks.

Plate I: Bida clay

Plate II: Chanchaga clay

Plate III: Kpakungu clay

Plate IV: Maikunkele clay

Plate V: Wet clay samples

Plate VI: Forming of brick in mould

Plate VII: Sun drying of some brick samples produced

Plate VIII: Preparation of oven for drying

Plate IX: Sample of oven dry brick produced

3.2.4 Determination of moisture content

In order to determine the moisture content of the clay, samples were weighed using a weighing balance and the value of the wet sample was recorder. The sample was then placed in an oven for 24 hours at 110°C. After the expiration of 24 hours, the sample was brought out and the weight of the oven-dried clay taken. The value of the moisture content was determined using equation (3.4).

$$= \frac{1 - 2}{1} \; 100\% \qquad (3.4)$$

Where:

W_1 = Weight of wet sample

W_2 = Weight of dry sample

3.2.5 Determination of refractory properties

The properties of the pure clays and clays with different additive ratio such as linear shrinkage, bulk density, apparent porosity, water absorption, compressive strength, thermal shock resistance and refractoriness were carried according to (ASTM, 1985).

3.2.5.1 *Linear shrinkage*

The dimensional changes in length were taken and the results were used to determine the fired shrinkage after firing at 950°C to 1150°C in Daiki scientific furnace (MD010). The linear shrinkage was determined using Equation (3.5) (ASTM, 1985).

$$\text{FIred Shrinkage} = \frac{L_d - L_f}{L_d} \times 100\% \qquad (3.5)$$

Where:

L_d = Dry length and L_f = Fired length

3.2.5.2 *Bulk density*

Bulk density was determined using a direct volume measurement method. This method exploits the relative density of a substance multiplied by the density of water to obtain the required bulk density. Equation (3.6) was used to obtain the bulk density in *g/cm³* (ASTM, 1985).

$$\text{Bulk density} = \frac{W_d}{W_s - W_{sp}} \times \text{Density of water} \qquad (3.6)$$

Where W_s = soaked weight, W_d = dry weight and W_{sp} = suspended weight

3.2.5.3 *Apparent porosity*

The boiling method was used for this test at 1000°C for 2 hours. The test pieces were subjected to a two-hour boiling followed by an additional four hour water soaking and then weighed W_s. The soaked piece was then suspended from the beam of a balance in a vessel of water so arranged that the test piece under consideration was completely immersed in the water without touching the side of the vessel. The suspended specimen in water weighed as W_{sp}. Porosity was then calculated as a function of the specimen's weight difference between soaked weight and dry weight to specimen's weight difference between soaked weight and suspended immersed weight. The results were obtained using Equation (3.7) (ASTM, 1985).

$$\text{Porosity (P)} = \frac{W_s - W_d}{W_s - W_{sp}} \times 100\% \qquad (3.7)$$

Where:

W_s = soaked weight, W_d = dry weight, W_{sp} = suspended weight

3.2.5.4 *Water Absorption*

The boiling method was used for this test at 100°C for 2 hours. The test pieces were subjected to a two-hour boiling followed by additional four hour water soaking. Water absorption was calculated as a function of the specimen's weight difference prior to and after water submersion. The water absorption was computed using Equation (3.8) (ASTM, 1985).

$$\text{Water absorbtion} = \frac{W_s - W_d}{W_d} \times 100\% \qquad (3.8)$$

Where:

W_s = soaked weight after boiling at 1000°C for 2 hours and W_d = dry weight

3.2.5.5 *Compressive strength*

The compressive strength of each brick was determined in accordance with the Specification of the Standard Organization of Nigeria (SON) as contained in Test for Compressive Strength of Solid Bricks using the Testing Machine. A 40 mm square platen was used on the compressive testing machine. Three test cubes were preconditioned by immersion in cold water at room temperature (29°C ± 2°C) for 24 hours, removed and all traces of water wiped off, and then stored under moist conditions for 24 hours prior to testing. Each test piece was centrally positioned between the platens of the testing machine, and the load was gradually increased until failure (Agbede & Joel, 2011). Mathematically, compressive strength can be determined using Equation (3.9) (Ogunsemi *et al., 2018*).

$$\text{Compressive strength} = \frac{\text{Maximum failure load x Proving ring factor}}{\text{Area sample}} \ (N/\ ^2) \qquad (3.9)$$

3.2.5.6 *Thermal shock resistance*

This test was carried out with the help of an electrical furnace (Thermodyne 46200) heated at the rate of 5 °C/min. The thermal shock resistance was determined by prism spalling test method according to ASTM C- 484 (2019) standard in which the spalling resistance was measured by the number of thermal cycles (heating, cooling and testing for failure). The test pieces of refractory bricks were thoroughly dried and placed in the cold furnace and heated at the rate of 5°C/minute until the furnace temperature got to 1200°C. The samples were then removed one after the other using a pair of tongs and cooled in air for 10 minutes, and then observed for cracks. In the absence of cracks (or fracture), the bricks were put back into the furnace and reheated for a further period of 10 minutes and then cooled for another 10 minutes. This process or cycle of heating, cooling and observing for cracks was repeated until cracks were observed. The number of complete cycles that produced visible cracks in each specimen was noted. This constituted the thermal shock (spalling) resistance.

3.2.5.7 *Refractoriness*

The refractoriness or softening point was determined using the method of pyrometric cone equivalence (PCE) in accordance with ASTM C24-79 (1985) the test pieces were mounted on the refractory plaque along with some standard cone whose softening points are slightly above or below those expected of the test cones. The plaque was then inserted into the electric furnace. The temperature was raised at the rate of 5°C per minute during which softening of Orton cone occurred along with the specimen test cone. The temperature was further raised up to 1400°C and samples that with-stood the temperature were soaked in the furnace at that same temperature for 5 and 6 hours for the clay samples, until the tips of the test cones had bent over the level with the base. Then the plaque bearing the specimens was removed from the furnace and the test cones examined when cold. The test cones were then compared with the standard cones and the test materials were said to have the pyrometric cone equivalent (PCE) of the standard cone that it resembled most in bending behavior. The refractoriness of each test cone is the number of the standard pyrometric cone that has bent over to a similar extent as the test cone. The temperature corresponding to the cone number was read off from the ASTM Orton series. The minimum PCE for intermediate and high are 24 and 27 which corresponds to the following fusing temperatures 1400°C (5hrs), 1400°C (6hrs) respectively. The soaked time in minutes was converted to equivalent temperature value by dividing with a factor of two (Chima *et al.,* 2017).

Showing on Plate X is the Preparation for Compressive strength test.

Plate X: Preparation for Compressive strength test

CHAPTER FOUR

4.0 RESULTS AND DISCUSSION

4.1 Sieve Analysis

The result of the sieve analysis for Bida, Chanchaga, Kpakungu and Maikunkele Clays are presented in Tables 4.1, 4.2, 4.3 and 4.4 respectively.

Table 4.1: Sieve analysis result of Bida clay

Sieve size(mm)	Weight of clay Retained (g)	Cumulative mass Retained (g)	% Cumulative Retained (g)	% Passing
100	12	12	0.23	99.77
90	14	26	0.50	99.5
75	15	41	0.79	99.21
63	18	59	1.12	98.88
50	211	270	5.12	94.88
37.5	781	1051	19.95	80.05
25	950	2001	37.99	62.01
19	933	2934	55.71	44.29
16	1338	4272	81.11	18.89
12.5	895	5167	98.10	1.9
Pan	100	5267	100	0

Table 4.2: Sieve analysis result of Chanchaga clay

Sieve size(mm)	Weight of clay Retained (g)	Cumulative mass Retained (g)	% Cumulative Retained (g)	% Passing
100	26.57	26.57	2.31	97.69
90	73.02	99.59	8.66	91.34
75	122.82	222.41	19.34	80.66
63	136.28	358.69	31.19	68.81
50	99.13	457.82	39.81	60.19
37.5	102.12	559.94	48.69	51.31
25	108.44	668.38	58.12	41.88
19	70.38	738.76	64.24	35.76
16	112.6	851.36	74.03	25.97
12.5	162.25	1013.61	88.14	11.86
Pan	136.39	1150	100	0

Table 4.3: Sieve analysis result of Kpakugu clay

Sieve size(mm)	Weight of clay Retained (g)	Cumulative mass Retained (g)	% Cumulative Retained (g)	% Passing
100	16.10	16.10	1.4	98.60
90	64.06	80.16	6.97	93.03
75	133.05	213.21	18.54	81.46
63	97.87	311.08	27.054	72.95
50	137.54	448.62	39.01	60.99
37.5	109.94	558.56	48.57	51.43
25	131.22	689.78	59.98	40.02
19	123.39	813.17	70.71	29.29
16	110.17	923.34	80.29	19.71
12.5	127.07	1050.41	91.34	8.66
Pan	99.59	1150	100	0

Table 4.4: Sieve analysis result of Maikunkele clay

Sieve size(mm)	Weight of clay Retained (g)	Cumulative mass Retained (g)	% Cumulative Retained (g)	% Passing
100	64	64	1.22	99.77
90	192	256	4.9	99.5
75	470	726	13.7	99.21
63	693	1419	26.79	98.88
50	497	1916	36.16	94.88
37.5	510	2426	45.78	80.05
25	584	3010	56.81	62.01
19	688	3698	69.79	44.29
16	287	3985	75.20	18.89
12.5	1215	5200	98.11	1.9
Pan	100	5300	100	0

The selected clay samples were sieved in a mechanical vibrator having several sieve attached in order from the largest to the smallest (100, 90, 75, 63, 50, 37.5, 25, 19, 16 and 12.5) for a period of 15 minutes. The parentage passing of clay decreases with the decrease in the size of the sieve while the weight of clay samples retained and the cumulative mass retained increases with the decrease in the sieve size. Sieved clay samples have sizes finer than 400μm this shows that clay samples are not too fine. Smith (2006) claim that the finer the clay particles the greater its plasticity index.

4.2 Chemical Composition

The chemical composition analysis of Bida, Chanchaga, Kpakungu and Maikunkele clay as done using X-ray fluorescence (XRF) is presented in Table 4.5. This shows that the mined clays are rich in oxides of Alumina. It could be seen that there are oxides of iron, calcium, and titanium. From the result, it is evident that SiO_2 is the major component with 44.67, 49.45, 52.58 and 54.59wt%, followed by Alumina which has 33.01, 28.58, 27.32 and 29.50wt% respectively for Bida, Chanchaga, Kpakungu and Maikunkele clays respectively. Atta *et al*., (2007) and Kovo (2010) reported that raw clay is expected to have a Silica/Alumina ratio of 2 to 1. As can be seen in Table 4.5, the SiO_2/Al_2O_3 ratio of Bida, Chanchaga, Kpakungu and Maikunkele mined clay is 1.546 which is within the theoretical value.

Table 4.5: Chemical composition of selected clay samples

Oxide(s)	Bida(%)	Chanchaga(%)	Kpakungu(%)	Maikunkele(%)
SiO_2	44.67	49.45	52.58	54.59
Al_2O_3	33.01	28.58	27.32	29.50
Fe_2O_3	2.1	6.50	4.50	4.50
TiO_2	0.3	0.25	0.10	0.02
CaO	1.90	2.59	2.90	2.10
MgO	1.1	2.41	2.30	1.90
K_2O	2.4	0.45	0.55	0.48
Na_2O	0.3	0.10	0.12	0.04

The selected clay sample were generally found to be Siliceous due to having high proportion of silica and the presence of Alumina made the clay samples to be within the class of Alumina Silicate refractory clays which is in line with the view of Shuaib and Mudiare (2017).

4.3 Moisture Content

The value of the moisture content for Bida, Chanchaga, Kpakungu and Maikunkele clays are as presented in Table 4.6, where it was observed that Chanchaga clay had the highest value of 19.81 and Maikunkele clay has the lowest value of 10.09.

Table 4.6: Moisture content of selected clay samples

Location	**Moisture content (%)**
Bida	17.81
Chanchaga	19.18
Kpakungu	15.98
Maikunkele	10.09

Ademila and Adebanjo (2017) said the moisture content is important in clay used for industrial application. It is a function of the void ratios and specific gravities of the clay sample. Ojo, *et al.,* (2017) suggested a specific level of not lower than 8% moisture content where clays are easily workable and at which mould clay will crack. The moisture content of clay samples shown in Table 4.6 conforms to the generally accepted standard.

4.4 Linear Shrinkage

Results of the linear shrinkage are shown in Appendix A Table A1 and Figure 4.1

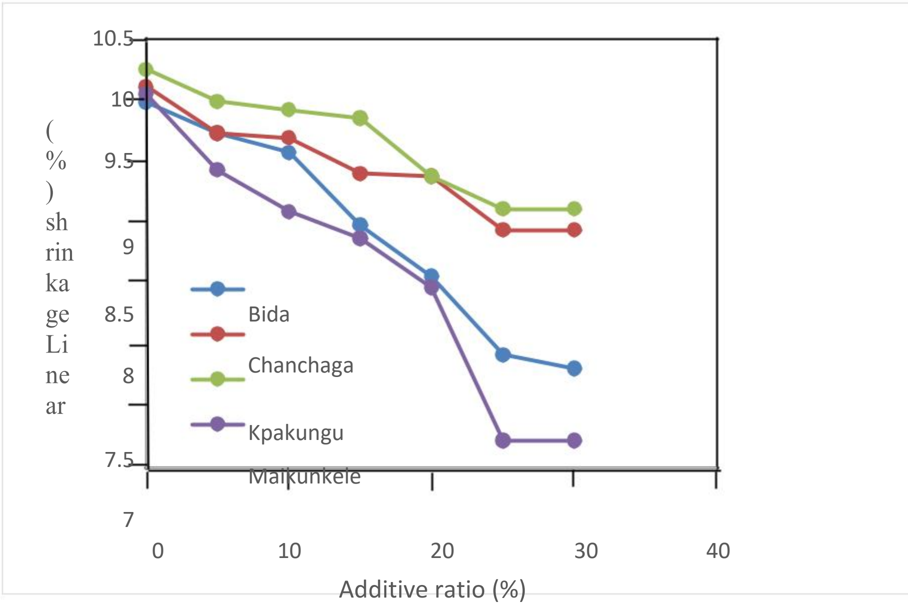

Figure 4.1: Effect of additives on linear shrinkage at a temperature of 1150^{o}C

The results for shrinkage is presented in Table A1, it indicate that shrinkage occurred in the sample fired in the range of 8.31 to 9.98% for Bida clay, 9.21 to 10.11% for Chanchaga clay, 9.38 to 10.25% for Kpakungu clay and 9.32 to 10.05% for Maikunkele clay respectively. The percentage of shrinkage decrease with increase in the amounts of additive for most cases. The linear shrinkage decreased from 8.31% for the natural Bida clay sample to a minimum value of 5.8% at 30% additive ratio and at a temperature of 950°C. While clay samples of Chanchaga, Kpakungu and Maikunkele reduced from 9.21 to 6.41%, 9.38 to 7.12% and 9.32 to 6.58% respectively at temperature of 950°C and addictive ratio of 30%. (Omowumi, 2001) reported a recommended range of 4 to 10% for fireclays while (Abolarin *et al.,* 2004) stated that lower values were more desirable as this implies that the clay is less vulnerable to volume change. The four clay samples employed in this study however has a comparatively linear shrinkage values within range for normal kaolin

($Al_2O_3.2SiO_2.2H_2O$) which is between 7 and 10 %. (Chester, 1973) advised a linear shrinkage range of 7-10 % for refractory clays.

The effect of additives on linear shrinkage of the selected clay samples is shown in figure 4.1. It was observed that for all the clay samples, the linear shrinkage reduced with increase in the percentage of additive added. Bida clay reduced from 9.98 to 7.81%, Chanchaga clay from 10.11 to 8.94%, Kpakungu clay from 10.25 to 9.11% and Maikunkele clay from 10.05 to 7.22% as the additive increased from 0 to 30%. Maikunkele clay had the least linear shrinkage of 7.22% at additive ratio of 30% while Kpakungu clay had the highest linear shrinkage of 9.11% at same conditions. (Abolarin *et al.,* 2004) pointed out that lower values were more desirable as this means the clay is less susceptible to volume change.

4.5 Bulk Density

Result of the density are shown in Appendix A Table A2 and Figure 4.2

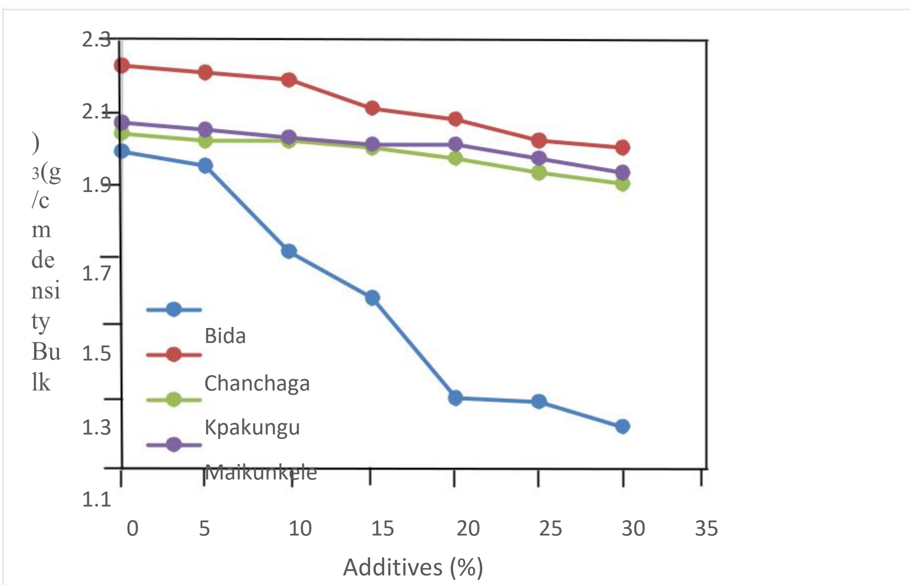

Figure 4.2: Effect of additives on bulk density at a temperature of 1150^{O}C

The results of the bulk density for Bida, Chanchaga, Kpakungu and Maikunkele clay are shown in Table A2. In this study, the bulk density of fired test clays was inversely proportional to the quantity of additives added. The bulk densities of the clay samples decreased with increase in the amounts of additives from 0%wt to 30%wt, but the bulk densities of specimens increased when firing temperature rise. The results shows that bulk density for Bida clay ranged from 1.77 g/cm^3 at 0% additive ratio and a temperature of 950°C to 1.22 g/cm^3 with additive ratio of 30% and temperature of 1150°C, Chanchaga clay ranged from 2.19 g/cm^3 at 0% additive ratio and a temperature of 950°C to 2.00 g/cm^3 with additive ratio of 30% and temperature of 1150°C, Kpakungu clay ranged from 2.00 g/cm^3 at 0% additive ratio and a temperature of 950°C to 1.90 g/cm^3 with additive ratio of 30% and temperature of 1150^{o}C and Maikunkele clay ranged from 2.02 g/cm^3 at 0% additive ratio and a temperature of 950°C to 1.93 g/cm^3 with additive ratio of 30% and temperature of 1150°C.

It was observed that Bida clay had the lowest bulk density of 1.77 g/cm^3 and Maikunkele clay had the highest bulk density of 2.19 g/cm^3. This can be attributed to low percentage of porosity of the sample (Aliyu *et al*., 2013) and also to some extent, the mineral composition of the clay (Umaru *et al.,* 2012). It was reported that specific gravity of clay by (Mazen, 2009) is indirectly proportional to the Al_2O_3 content in the raw material. This can be seen by the results in table of chemical composition and bulk density. Bida clay which has the highest Alumina content has the lowest bulk density. The relationship between bulk density, apparent porosity and linear shrinkage was studied by (Onyeji, 2010) and reported that shrinkage is less in denser clays because they are less porous. Hence Bida clay with its resultant low bulk density is more likely to shrink.

Figure 4.2 shows the effect of additives on the bulk density value of Bida, Chanchaga, Kpakungu and Maikunkele clay samples with Bida clay have the least bulk density of 1.22

g/cm^3 at 30% additive ratio followed by Kpakungu clay with a bulk density of 1.90 g/cm^3. Maikunkele and Chanchaga clays had bulk density values of 1.93 and 2.00 g/cm^3 respectively.

4.6 Apparent Porosity

Results of Apparent porosity are shown in Appendix A Table A3 and Figure 4.3

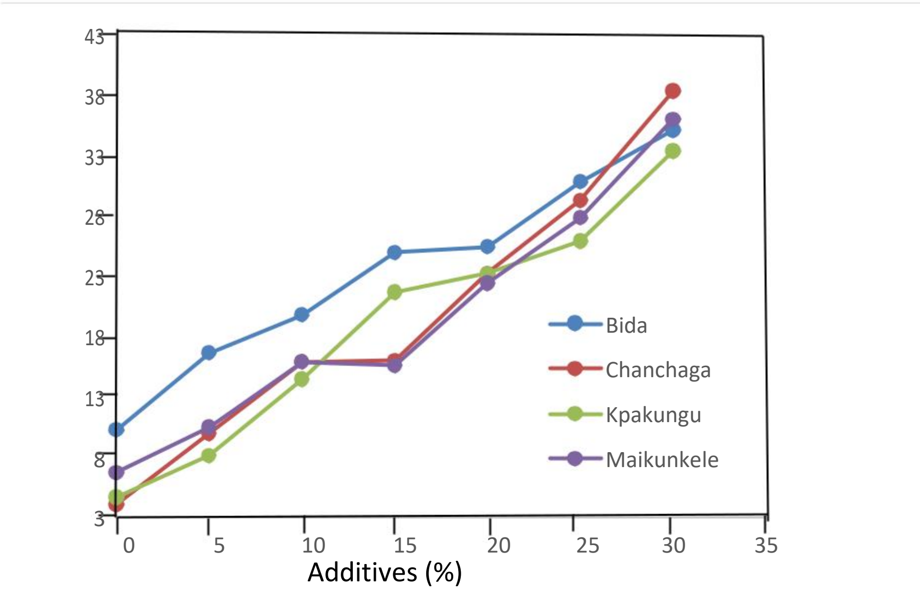

Figure 4.3: Effect of additives on apparent porosity at 1150^{o}C

The values of the apparent porosity of the clay samples from Bida, Chanchaga, Kpakungu and Maikunkele in percentages were also determined as shown in Table A3. Bida clay has the highest porosity of 51.63% against 48.40% for Chanchaga clay, 51.17% for Kpakungu clay and 45.83% for Maikunkele clay at 30% additive ratio and 950°C respectively. The porosity of refractory clay material is directly related to the air pockets contained in it, hence, the higher the porosity of the clay material, the higher its insulating properties. All the four samples gave apparent porosity values of which is within the acceptable range (10-30%) suggested for refractory clays (Chester, 1973) at different additive ratios and temperatures. At additive ratio of 0 to 15% and temperature of 1150°C, Bida clay apparent porosity was 10.24, 16.64, 19.81 and 24.96 which were all within acceptable range. As reported by (Mazen, 2009), Maikunkele clay with additive ratio of 10 to 20% and temperature of 950°C to 1150°C were within the international values of 15 to 25% except for the value of 27.82% at additive ratio of 20% and temperature of 950°C which was higher. Kpakungu and Chanchaga clay showed acceptable porosity values as well. This implies that the clay samples have common major compositions in terms of oxides, which constitutes the highest percentage of the clay mineral compositions .However, the variation in values of the porosity account for the variation of clay types.

It was further observed that the apparent porosity of all the clay samples increased with increase in additive ratio, this is equivalent to the values and proportionality reported by (Etukudoh *et al.,* 2016). One of the major requirements for a good insulating refractory is that it must be highly porous (Manukaji, 2013). The presence of pores helps the insulating characteristics of the fired body by serving as air spaces to trap heat and prevent flow or conduction. The apparent porosity showed an increase as the percentage of additives increased because the additives burnt off at elevated temperatures inducing porosity. Therefore the more the additives were added the more the pores created (Folorunso *et al.,* 2015).

From the values of apparent porosity expressed in Figure 4.3, it was seen that porosity of the clay samples increased with increased in the amount of additives added. Chanchaga clay with an initial apparent porosity value of 4.05 increased to 38.33 when the additive was raised from 0 to 30%. Bida clay which had the highest initial value of 10.24 at zero additive added increased to 35.13 when 30% of the additive was added. Maikunkele and Kpakungu clays had initial and final values of 6.72, 35.99 and 4.67, 33.37 respectively.

4.7 Water Absorption

The result of Water Absorption are shown in Appendix A Table A4 and Figure 4.4

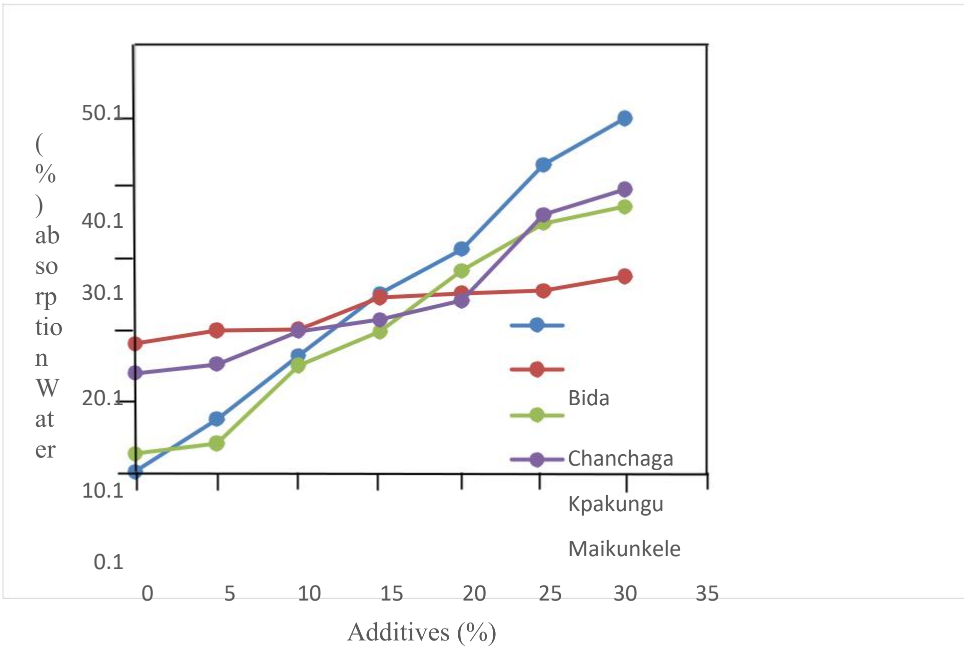

Figure 4.4: Effect of additives on water absorption at 1150°C

Table A4 shows the effect of firing temperature and additive ratio on the water absorption of Bida, Chachanga, Kpakungu and Maikunkele clay. It was observed that similar trend as the apparent porosity was obtained in which a decrease in water absorption of all the samples with increase in firing temperature was obtained. This is because the water absorption is dependent on the pores and is directly proportional to the apparent porosity of the fired body. Since the pores are responsible for water uptake, the decrease in water absorption is attributed to a decrease in porosity of the clay body with increase in firing temperature. Similar to the case of apparent porosity, the water absorption showed an

increase with increase in the percentage of additive ratio in the fired body, also attributed to increase in porosity due to the burning of more of the additives.

Water absorption is an important factor for the durability of clay bricks. When water infiltrates bricks, it decreases the durability of bricks. Thus, the internal structure of bricks must be dense enough to void the intrusion of water. To increase density and decrease water absorption of bricks, the firing temperature must be raised. In this study, the amount of additives added into the samples that were fired at lower temperature (950°C) increased the water absorption rate in a linear manner. On the contrary, when testing the samples with higher amounts additives by firing at a high temperature (1150°C), the water absorption of fired samples decreased. According to Table 4.6, the water absorption of the samples after firing at the temperatures between 950 and 1150 °C is in the range of 0.31–72.84%. The standard criteria of TIS 77-2545 determined that good quality bricks should not have water absorption rate higher than 20%.

As observed in Figure 4.4 the values of water absorption increased as the amount of additives increased with Chanchaga clay having a minimal increase of 24.61 to 25.5% when 15 to 25% of additives was added has the lowest water absorption of 27.48%. Bida clay showed a linear increment of 0.31 to 49.5% from zero to 30% additive being the highest value observed. Kpakungu and Maikunkele clays had values of 37.2 and 39.6% respectively at 30% additive.

4.8 Compressive Strength

Results of Compressive Strength are shown in Appendix A Table A5 and Figure 4.5

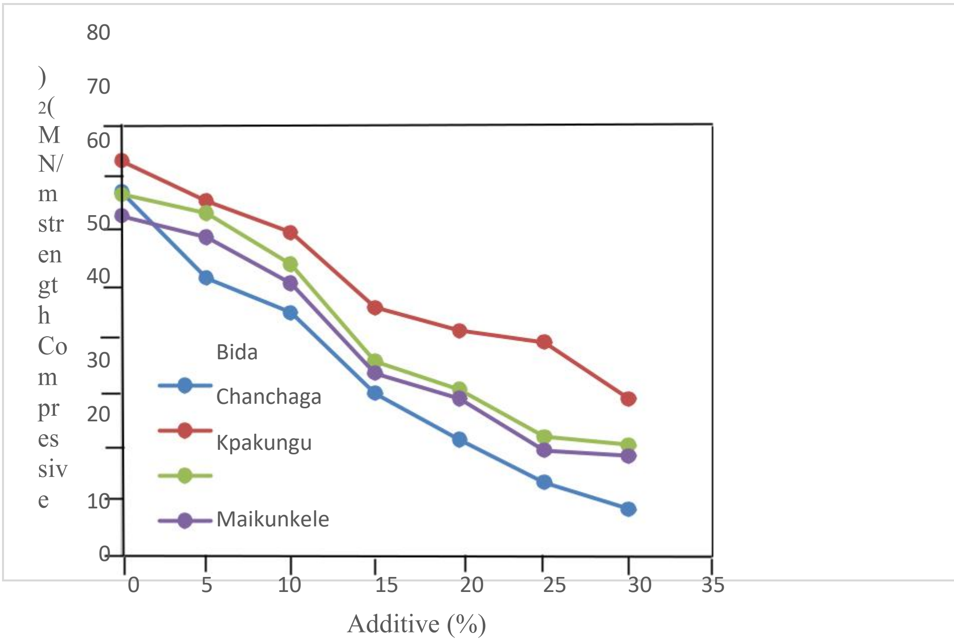

Figure 4.5: Effect of additive on compressive strength at 1150^{o}C

In this study the result indicated that the strength of the samples greatly depended on the amount of additives and the firing temperature. The results of compressive strength as shown in Table A5 indicated that the compressive strength of fired samples increased when the firing temperature rise. An increase in compressive strength was due to a decrease in porosity and an increasing temperature. The results revealed that the compressive strength was 67.46, 73.32, 67.06 and 63.09 MN/m^2 at 0% additive ratio for Bida, Chanchaga, Kpakungu and Maikunkele clays respectively and at a temperature of 1150°C. When the additives varied from 5 to 30% and firing temperatures from 950°C to 1150°C the compressive strength decreased with increasing additives ratio from 7.33 to 1.73%, 25.23 to 9.11%, 16.67 to 2.23% and 11.60 to 1.80% for Bida, Chanchaga, Kpakungu and Maikunkele clay repectively, but increased with increasing temperature from 7.33 to

51.58% for Bida, 25.23 to 65.85% for Chanchaga, 16.37 to 63.59% for Kpakungu and 11.60 to 59.06 for Maikunkele clays at 5% additive ratio. It was further observed that at 30% additive ratio the values increased from 1.73 to 8.63% for Bida clay, 9.11 to 29.11 for Chanchaga clay, 2.23 to 20.55% for Kpakungu clay and 1.80 to 18.52% for Maikunkele clay.

The four clays investigated, only Bida clay had a value in less of 18 MN/m^2 at 8.63 MN/m^2, which falls short of the 26.5 MN/m2 reported by (Ameh & Obasi, 2009) for Nsu clay. For Maikunkele clay, the value obtained was 18.52 MN/m^2 while Kpakungu clay produced a value of 19.27 MN/m^2 and Chanchaga clay produced a value of 29.11 MN/m^2 which meet the criteria of the TIS 77-2545 that defined values no lower than 17 MN/m^2. In the Table A5, it was found that the compressive strengths of clay brick at 0-5% additives and 1150°C are very high (67.46-73.32 MN/m^2). Thus, the phase of Mullite is created, and the particles sizes are moved closer together (Lawanwadeekul and Bunma, 2015). Generally, in traditional ceramic system, the strength properties decrease as the porosity increases (Sutcu and Akkurt, 2009).

Figure 4.5 shows that Bida clay has the least compressive strength of 8.63 MN/m^2 followed by Maikunkele, Kpakungu and Chanchaga clays with values of 18.52, 20.55 and 29.11 MN/m^2 respectively at a temperature of 1150^{o}C and 30% additive.

4.9 Thermal Shock Resistance

The result of Thermal shock resistance are shown in Appendix A Table A6 and Figure 4.6

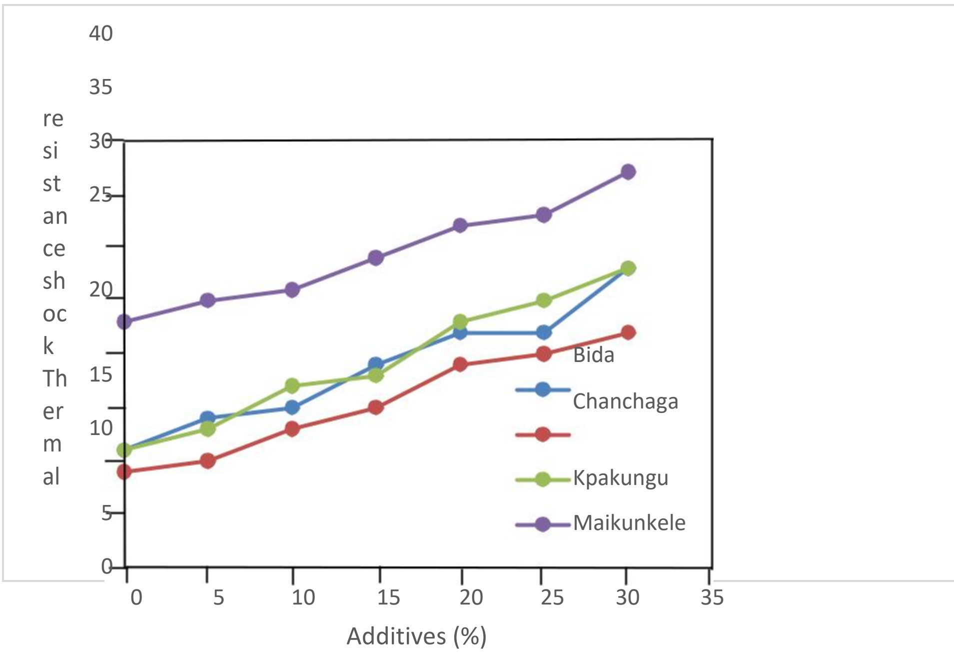

Figure 4.6: Effect of additives on thermal shock resistance at 1150^{o}C

Thermal shock resistance for all the clay samples obtained gave the number of cycle to be less than 20^+ cycles at 0%wt additives ratio and temperature range of 950°C – 1150°C except for Maikunkele clay which had 33^+ cycles at 950°C. The value obtained for Bida Chanchanga and Kpakungu clay samples was lower than the Values reported by (Mazen, 2009), (Ndaliman, 2006) who reported above 20^+ cycles and up to 18^+ cycles respectively. Thermal shock resistance of the clay samples increases with increase in additives ratio and decrease with rise in temperature. Bida clay show 36^+ cycles at 950°C and 28^+ cycles at 1150°C. Maikunkele bricks show 56^+ cycles at 950°C and 1150°C Kpakungu bricks show 42^+ cycles at 950°C and 28^+ cycles at 1150°C While Chanchanga clay bricks shows 31^+ cycles at 950°C and 22^+ cycles at 1150°C all at an additive ratio of 30%wt which gives an excellent thermal shock resistance.

The excellent thermal shock resistance exhibited by all the clay samples can be attributed to the insulating property due to uniformly distributed pore at high ratio of additives that burns off at high temperature. This made the bricks samples to absolve and release heat at lower rate and shielding the bricks from the relative effect of high thermal shock.

Figure 4.6 further shows the effect of additive ratio on thermal shock resistance as it was observed that the cycles of Bida and Kpakungu clay had the same values of 28^+ at additive ratio of 30% and temperature of 1150^oC and the same values of 11^+ cycles at zero additive. Meanwhile, Chanchaga and Maikunkele clays had values of 22^+ and 37^+ cycles respectively with Maikunkele clay having the highest value.

4.10 Refractoriness

Result of the Refractoriness is shown in Table 4.7

Table 4.7: Refractoriness of selected clay samples at 0% to 30% additive ratio

LOCATION	0%	5%	10%	15%	20%	25%	30%
Bida	1410	1480	1480	1480	1480	1480	1480
Chanchaga	1580	1620	1620	1620	1620	1620	1620
Kpakungu	1550	1570	1570	1570	1570	1570	1570
Maikunkele	1650	1690	1690	1690	1690	1690	1690

From the results of the refractoriness test as shown in Table 4.7, it was noted that for the clays without additives, the softening point occurred at 1410°C, 158°C, 1550°C and 1650°C for Bida, Chanchaga, Kpakungu and Maikunkele clay samples respectively while for those blended with the additives ratios of 5%wt to 30%wt were 1480°C, 1620°C, 1570°C and 1690°C for Bida, Chanchaga, Kpakungu and Maikunkele clay samples respectively. This suggests an enhancement in the refractoriness value which may be traced to the presence of useful oxides found in the additives. The oxides of alumina and phosphorus though in trace quantity have improved the refractoriness of the composite clay materials to the value required for good refractory material. It was also found that combination of two or more additives yielded better refractoriness value than single one which was done in previous research works (Izwan *et al.,* 2011).

The refractoriness of the selected clay samples increased when additives were introduced to the clays at 5% to values of 1410, 1580, 1550, and 1650 to 1480, 1620, 1570 and 1690 for Bida, Chanchaga, Kpakungu and Maikunkele clay samples respectively and remained constant from the 5% additive to 30%.

CHAPTER FIVE

5.0 CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

In this research, the improvement of some physical properties of selected Nigerian clays of Bida, Chanchaga, Kpakungu and Maikunkele was studied and the effects of rice husk ash, corn husks ash and sawdust ash additives investigated at varying temperatures. Sieve analysis was carried out to assess the particle size distribution or gradation of the clay samples, the chemical composition of the clay was determined using X-ray flourences and the moisture content of the clay samples was ascertained. The refractory properties of the clay such as linear shrinkage, bulk density, apparent porosity, water absorption, compressive strength and refractoriness was determined and the effect of additives at different ratio and temperatures observed. The following conclusions were drawn based on this.

(i) The clay samples crushed and sieved in a mechanical vibrator for a period of 15minutes for each of the clay samples. Particles size is finer than 400µm for all the clay samples

(ii) The chemical composition result shows the clay samples contain Al_2O_3 and SiO as major constituent and making them suitable as Alumino–Silicate refractory materials.

(iii) Clay samples were beneficiated, mixed with various proportions of additives and clay bricks were produced.

(iv) Kpakungu clay having higher value of linear shrinkage of 10.25 and 9.11% at 0 and 5% additive and temperature of 1150^{O}C is susceptible to volume change as lower values are more desirable. Bulk density values for Bida, Chanchaga, Kpakungu and Maikunkele clays at 0 and 5% additive and temperature of 1150^{O}C was determined to be 1.99 and 1.22, 2.23 and 2.0, 2.04 and 1.90, 2.07 and 1.93 respectively. The low bulk density can be attributed to some extent of mineral composition. The Values of apparent porosity and water absorption were greatly improved .The experimental result shows that at maximum temperature of 1150^{O}C, compressive strength was determined to be 67.46 and 8.63, 73.32 and 29.11, 67.06 and 20.55, 63.09 and 18.52 at 0 and 5% additive ratio for Bida, Chanchaga, Kpakungu and Maikunkele clay samples. While the minimum and maximum result of the thermal shock resistance was determined to be 11 and 28, 9 and 22, 11 and 28, 33 and 37 at same conditions. The refractoriness of the Bida, Chanchaga, Kpakungu and Maikunkele clay samples were determined to increase from 1410 to 1480^{O}C, 1580 to 1620^{O}C, 1550 to 1570^{O}C, 1650 to 1690^{O}C from zero to 5% additive and remained constant up to 30% additive.

5.2 Recommendations

1. Further research work should investigate the effect of these additives on a blended composite of clay in order to determine the influence it will exact on the refractory properties of the clay samples.

2. Further research work should investigate the effect additives other than agricultural ash residue on Bida, Chanchaga, Kpakungu and maikunkele clays.

5.3 Contribution to Knowledge

It has been revealed that ash of agricultural waste residue has a great influence in improving some refractory properties of clay samples and encourage consumption of local refractory clay in metallurgical industries. The result revealed Bida clay increase in porosity from 16.64% to 35%, Chanchaga clay increase in porosity from 9.98% to 38.38%, Kpakungu clay increase in porosity from 8.12% to 33.37% and Maikunkele clay increase in porosity from 10.50% to 35%. Compressive strength of the clay samples were reduced Bida clay reduced in compressive strength from 51.58N/m^2 to 8.63N/m^2, Chanchaga clay reduced in compressive strength from 65.85%N/m^2 to 29.11N/m^2, Kpakungu clay reduced in compressive strength from 63.59N/m^2 to 20.55N/m^2, Maikunkele clay samples reduced in compressive strength from 59.06N/m^2 to 18.52N/m^2. The refractory temperature of Bida, Chanchaga, Kpakungu and Maikunkele clay samples were increased from 1410°C to 1480°C, 1580°C to 1620°C, 1550°C to 1570°C and 1650°C to 1690°C at 5%wt of additives.

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APPENDIX A

Experimental Result for the Refractory Properties of the Clay Samples

Table A1: Linear shrinkage (%) result of selected clay samples at 0% to 30% additive ratio

Locations	Temperature (°C)	0%	5%	10%	15%	20%	25%	30%
Bida								
	950	8.31	7.21	6.81	6.67	6.43	5.68	5.68
	1000	8.80	7.42	6.98	6.64	6.43	5.79	5.79
	1050	9.05	7.89	7.56	7.28	6.81	5.98	5.85
	1100	9.85	8.69	7.97	7.68	6.85	6.24	6.83
	1150	9.98	9.73	9.57	8.98	8.56	7.92	7.81
Chanchaga								
	950	9.21	8.96	8.56	8.03	7.35	6.55	6.41
	1000	9.65	9.01	8.88	8.33	7.69	6.77	6.77
	1050	9.84	9.68	9.56	9.14	8.99	7.28	7.89
	1100	10.01	9.78	9.61	9.23	9.43	8.87	8.51
	1150	10.11	9.73	9.69	9.40	9.38	8.94	8.94
Kpakungu								
	950	9.38	8.93	8.51	7.70	7.53	7.15	7.12
	1000	9.52	9.46	9.28	9.01	8.85	8.28	8.12
	1050	9.87	9.79	9.62	9.26	8.99	8.62	8.62
	1100	9.96	9.80	9.71	9.34	9.03	8.86	8.80
	1150	10.25	9.99	9.92	9.85	9.38	9.11	9.11
Maikunkele								
	950	9.32	8.01	7.70	7.57	7.38	6.58	6.58
	1000	9.80	8.12	7.96	7.55	7.32	6.65	6.65
	1050	9.83	8.69	8.66	8.09	7.70	6.70	6.70
	1100	9.97	9.33	8.94	8.55	7.94	6.84	6.83
	1150	10.05	9.43	9.09	8.87	8.47	7.22	7.22

Table A2: Bulk density (g/cm^3) of selected clay samples at 0% to 30% additive ratio

Locations	Temperature (°C)	0%	5%	10%	15%	20%	25%	30%
Bida								
	950	1.77	1.61	1.52	1.32	1.27	1.02	0.97
	1000	1.80	1.77	1.48	1.18	1.24	1.08	1.01
	1050	1.82	1.73	1.57	1.38	1.28	1.12	1.13
	1100	1.85	1.84	1.67	1.45	1.30	1.17	1.19
	1150	1.99	1.95	1.71	1.58	1.30	1.29	1.22
Chanchaga								
	950	2.19	2.11	1.89	1.89	1.77	1.69	1.53
	1000	2.19	2.13	1.90	1.92	1.85	1.73	1.71
	1050	2.21	2.18	2.09	2.01	1.95	1.91	1.83
	1100	2.21	2.18	2.12	2.08	2.01	1.94	1.89
	1150	2.23	2.21	2.19	2.11	2.08	2.02	2.00
Kpakungu								
	950	2.00	1.98	1.93	1.93	1.86	1.80	1.76
	1000	2.01	1.99	1.95	1.94	1.91	1.88	1.81
	1050	2.01	2.00	1.97	1.94	1.92	1.89	1.84
	1100	2.02	2.00	2.01	1.98	1.95	1.91	1.86
	1150	2.04	2.02	2.02	2.00	1.97	1.93	1.90
Maikunkele								
	950	2.02	2.01	1.98	1.95	1.86	1.83	1.80
	1000	2.02	2.01	2.01	1.97	1.91	1.90	1.84
	1050	2.04	2.02	2.01	1.99	1.94	1.90	1.87
	1100	2.04	2.02	2.02	2.00	1.98	1.95	1.90
	1150	2.07	2.05	2.03	2.01	2.01	1.97	1.93

Table A3: Apparent porosity (%) of selected clay samples at 0% to 30% additive ratio

Locations	Temperature (°C)	0%	5%	10%	15%	20%	25%	30%
Bida								
	950	27.16	30.12	32.17	33.82	37.43	44.07	51.63
	1000	23.82	29.63	31.95	30.77	34.57	38.51	43.71
	1050	20.14	21.70	22.53	27.98	32.73	35.09	40.74
	1100	17.28	20.28	20.24	26.96	34.65	33.90	41.13
	1150	10.24	16.64	19.81	24.96	35.43	30.82	35.13
Chanchaga								
	950	19.00	23.28	26.23	28.34	30.87	46.68	48.40
	1000	14.12	17.59	19.59	22.76	26.90	33.25	40.76
	1050	9.74	12.78	18.97	19.78	25.95	29.49	37.79
	1100	10.01	12.97	16.38	16.49	22.67	31.12	35.20
	1150	4.05	09.98	15.91	16.01	23.23	29.28	38.33
Kpakungu								
	950	22.65	26.18	29.64	30.32	35.47	45.87	51.17
	1000	19.38	25.07	30.60	33.58	37.34	40.76	49.61
	1050	9.22	13.20	18.87	28.14	27.45	31.43	35.68
	1100	7.86	11.28	16.79	23.50	26.89	30.36	35.07
	1150	4.67	8.12	14.47	21.68	23.22	25.90	33.37
Maikunkele								
	950	18.11	20.18	23.59	25.93	27.82	43.69	45.83
	1000	12.87	15.98	17.85	20.13	24.75	31.60	38.98
	1050	09.00	11.48	17.55	18.17	24.79	28.18	36.48
	1100	10.54	13.89	14.44	16.12	24.36	30.11	34.28
	1150	6.72	10.50	15.91	15.58	22.41	27.85	35.99

Table A4: Water absorption (%) result of selected clay samples at 0% to 30% additive ratio

Properties	Temperature (°C)	0%	5%	10%	15%	20%	25%	30%
Bida								
	950	18.11	21.07	30.47	31.94	51.85	62.00	72.84
	1000	12.84	25.34	36.49	47.33	41.72	53.02	61.09
	1050	8.27	20.18	27.00	36.57	42.23	55.19	58.11
	1100	3.45	10.43	19.29	30.32	36.68	47.52	51.30
	1150	0.31	7.64	16.46	25.09	31.32	43.06	49.50
Chanchaga								
	950	16.16	17.59	15.37	21.40	22.48	19.70	19.04
	1000	17.37	18.00	16.84	21.99	23.47	21.64	20.46
	1050	17.77	20.55	19.69	22.85	24.95	23.80	18.47
	1100	17.28	19.91	20.88	2453	25.00	25.41	17.36
	1150	18.15	20.01	20.20	24.61	25.20	25.50	17.48
Kpakungu								
	950	18.22	19.95	31.75	34.11	51.14	59.50	67.05
	1000	18.79	21.75	34.67	40.17	47.63	57.23	60.13
	1050	8.76	10.06	21.96	34.22	36.55	42.44	47.32
	1100	7.84	8.67	17.42	23.54	29.13	37.37	38.07
	1150	2.79	4.22	15.11	19.77	28.31	34.95	37.20
Maikunkele								
	950	18.45	19.98	26.21	26.25	31.95	58.96	57.10
	1000	16.76	19.70	22.17	24.35	31.22	58.04	55.35
	1050	15.89	16.53	18.47	23.45	31.16	47.03	47.54
	1100	15.23	16.86	21.67	18.68	25.19	36.18	35.86
	1150	14.02	15.30	19.89	21.47	24.12	36.10	39.60

Table A5: Compressive strength (N/m^2) of selected clay samples at 0% to 30% additive ratio

Location	Temperature (°C)	0%	5%	10%	15%	20%	25%	30%
Bida								
	950	19.87	7.33	4.43	4.50	2.80	2.30	1.73
	1000	23.45	11.28	9.72	6.13	5.07	4.18	3.74
	1050	25.94	18.50	17.60	10.96	10.44	6.52	5.50
	1100	56.61	40.83	38.51	25.33	13.18	9.46	7.58
	1150	67.46	51.58	45.09	30.16	21.56	13.59	8.63
Chanchaga								
	950	29.42	25.23	21.55	19.08	15.34	11.40	9.11
	1000	34.07	29.48	25.76	22.88	19.49	14.00	11.12
	1050	48.22	35.10	29.29	26.60	22.28	20.02	15.78
	1100	61.45	53.74	48.27	42.85	37.94	32.18	27.68
	1150	73.32	65.85	59.99	46.07	41.79	39.69	29.11
Kpakungu								
	950	21.78	16.37	12.67	6.97	4.70	2.73	2.23
	1000	27.68	23.94	21.51	18.19	12.99	7.75	5.28
	1050	31.47	25.04	20.91	19.95	14.29	8.03	7.26
	1100	55.55	51.86	42.55	35.89	29.35	26.82	19.27
	1150	67.06	63.59	54.09	36.07	30.88	22.04	20.55
Maikunkele								
	950	13.45	11.60	9.57	6.37	4.33	2.63	1.80
	1000	23.70	18.55	17.49	10.81	6.70	5.38	3.96
	1050	28.49	20.28	17.92	16.43	11.85	8.68	6.22
	1100	54.78	48.43	40.61	30.90	27.40	20.83	17.57
	1150	63.09	59.06	50.55	33.91	29.18	19.58	18.52

Table A6: Thermal shock resistance ($^{+}$cycle) of selected clay samples at 0% to 30% additive ratio

Location	Temperature (°C)	0%	5%	10%	15%	20%	25%	30%
Bida								
	950	18	21	22	26	31	33	36
	1000	17	18	20	23	28	29	32
	1050	15	16	18	23	26	27	31
	1100	14	17	20	21	23	24	29
	1150	11	14	15	19	22	22	28
Chanchaga								
	950	19	23	23	26	27	31	31
	1000	16	19	19	23	24	27	27
	1050	15	16	16	19	20	22	25
	1100	11	12	14	17	20	22	24
	1150	9	10	13	15	19	20	22
Kpakungu								
	950	17	22	24	28	33	37	42
	1000	15	16	22	27	31	34	39
	1050	13	16	20	24	29	32	36
	1100	13	14	18	21	26	27	30
	1150	11	13	17	18	23	25	28
Maikunkele								
	950	33	35	38	42	46	49	56
	1000	30	34	37	39	44	49	55
	1050	30	33	34	37	43	48	52
	1100	28	30	33	36	39	41	44
	1150	23	25	26	29	32	33	37