

**PRODUCTION OF INSULATING REFRACTORY BRICKS
FROM KANKARA KAOLIN USING ACHA (*Digitaria Exilis*)
HUSK AS ALTERNATIVE PORE-FORMER**

BY

CHRISTOPHER HARUNA MADUGU

**GLASS TECHNOLOGY SECTION
DEPARTMENT OF INDUSTRIAL DESIGN
AHMADU BELLO UNIVERSITY,
ZARIA, NIGERIA.**

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FORMER

BY

Christopher Haruna MADUGU

B.Sc Glass Technology, 1997 (ABU, Zaria)

M.Sc/Env.Design/45743/2012-2013

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JANUARY, 2016

DECLARATION

I declare that the work in the dissertation entitled “Production of Insulating refractory Bricks from Kankara Kaolin using Acha (*Digitaria Exilis*) Husk as Alternative Pore-former” has been carried out by me in the Department of Industrial Design (Glass Technology Section), under the supervision of Dr. E.A. Ali and Dr. A.D. Garkida. The information derived from the literature has been duly acknowledged in the text and a list of references provided. No part of this dissertation was previously presented for another degree at any other University.

MADUGU, Christopher Haruna
Name of Student

Signature

Date

CERTIFICATION

This project dissertation entitled “PRODUCTION OF INSULATING REFRACTORY BRICKS FROM KANKARA KAOLIN USING ACHA (*Digitaria Exilis*) HUSK AS ALTERNATIVE PORE-FORMER” by CHRISTOPHER MADUGU HARUNA meets the regulations governing the award of the degree of Master of Science of Ahmadu Bello University, and is approved for its contribution to knowledge and literary presentation.

Dr. E. A. Ali
Chairman, Supervisory Committee
Department of Industrial Design
Ahmadu Bello University, Zaria

Date

Dr. A. D. Garkida
Member, Supervisory Committee
Department of Industrial Design
Ahmadu Bello University, Zaria

Date

Dr. C. V. Alkali
Head of Department
Department of Industrial Design
Ahmadu Bello University, Zaria

Date

Prof. Kabiru Bala
Dean, School of Postgraduate Studies
Ahmadu Bello University, Zaria

Date

DEDICATION

To My Late Brother: Idris Haruna

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ABSTRACT

The exploration of the potentiality of Acha (*Digitaria Exilis*) husk (AH) as an alternative pore-former in insulating bricks was investigated. X-ray fluorescence (XRF) analysis was conducted for both beneficiated and un-beneficiated kaolin sample. Fourier Transform Infra-Red (FTIR) and XRF analysis was conducted for five AH samples sourced from different locations. The beneficiated kaolin was calcined at 1280°C and milled into six different grain sizes, which were further collapsed into two coarse: fine grog ratios. Batch formulation of the dried AH, grog ratios and beneficiated kaolin was made using Andreason's Parameter. Two variables were investigated. AH wt% varied between 1-10% in each five Coarse: fine grog ratio that is, 70:30, 66:34, 60:40, 55:45 and 50:50. Insulating bricks were produced by semi-dry mixing and hydraulic press. Samples were dried and fired in a kerosene fueled Kiln at 1280°C. The results showed that, sinterability of the beneficiated kaolin for drying and firing shrinkage were 6% and 7.4% respectively. Atterberg plasticity index (API) result was 18% classified as intermediate plasticity. Particle size distribution (PSD) result showed that, more than 71% of the kaolin sample passed through mesh 200 (75µm). XRF analysis results of kaolin sample showed that, 31.60% Al₂O₃, 54.80% SiO₂ and 0.87% Fe₂O₃ were within tolerable limits required for refractory materials while the AH results showed, 6.67% Al₂O₃ in addition to other oxides, a major advantage because it enhances the refractoriness of the bricks. FTIR analysis showed high percentage of organic materials in the AH, which are highly combustible, enhancing their insulating properties. Results obtained from the property tests of insulating bricks showed that, total shrinkage increased with increased in AH contents. Coarse: fine grog ratios 50:50 and 60:40 results ranged from 1.0% - 2.1% compared with Indian Standard Testing Methods (ISTM, 2014), which ranged from 0.5% - 1.5%. Apparent porosity test results ranged between 20% - 75% for Coarse: fine ratios 50:50 and 60:40, meeting the acceptable range of international standard 20 - 80% and ISTM ranged between 45 - 70%. Bulk density generally decreased with increase in AH. It ranged between 0.9g/cm³ - 1.58g/cm³ for grog ratios 50:50, 55:45 and 60:40 compared with ISTM 0.65g/cm³ - 1.60g/cm³. Thermal conductivity decreased with increased AH content and values obtained ranged between 0.005 - 0.32 W/m.k, the minimum is outside ISTM values 0.23 - 0.70 W/m.k. the maximum is within. Cold Crushing Strength (CCS) results showed that higher the coarse grog size, lower the CCS while high fine grog size resulted to high CCS. Also as AH admixture increased, CCS reduced. Thermal shock resistance results of the ten samples showed no crack or warpage after passing through 30 cycles and rated excellent thermal shock. It was concluded that, insulating bricks with good physical, mechanical and thermal properties were noted in Coarse: fine grog ratios, 66:34, 55:45 and 50:50, maximum AH content 6%. Bricks No 10, 11, 12, 14 and 19 gave optimal results. From these products, AH produced uniformly distributed micro-porosity in the solid bricks. The AH is of circular morphology and fine enough to allow better binding and relatively high bulk density. This conferred structural strength to the insulating bricks which can be used where compressive strength is required. Further investigation of potential properties of AH to ascertain its thermal integrity is recommended.

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LIST OF ABBREVIATIONS

| | | |
|----------------|---|--|
| AH | - | Acha Husk |
| AP | - | Apparent Porosity |
| API | - | Atterberg Plasticity Index |
| ASTM | - | American Society for Testing and Materials |
| BD | - | Bulk Density |
| CCS | - | Cold Crushing Strength |
| DTA | - | Differential Thermal Analysis |
| KK | - | Kankara Koalin |
| LDSd | - | Linear Drying Shrinkage on the dried basis |
| LDSF | - | Linear Firing Shrinkage on the as formed basis |
| LFS | - | Linear Firing Shrinkage |
| LL | - | Liquid Limit |
| LOI | - | Loss on Ignition |
| MOR | - | Modulus of Rupture |
| NA | - | Not Available |
| P.C.E | - | Pyrometric Cone Equivalent |
| P ₁ | - | Plasticity Index |
| P | - | Maximum Load |
| L _s | - | Length of the Sample |
| B | - | Average Breath of sample |
| d | - | Average Depth (height) of Sample |
| PDS | - | Particle Size Distribution |

| | | |
|-------|---|--|
| RMRDC | - | Raw Materials Research and Development Council |
| PFmax | - | Maximum Packing Fraction |
| PFc | - | Coarse Packing Fraction |
| PFm | - | Medium Packing Fraction |
| PFf | - | Fine Packing Fraction |
| RUL | - | Refractoriness Under Load |
| XRD | - | X-Ray Diffraction |
| XRF | - | X-Ray Fluorescence |
| FTIR | - | Fourier Transform Infra-Red |
| BS | - | British Standard |
| NGSA | - | Nigeria Geological Survey Agency |
| EDXRF | - | Energy Dispersive X-Ray Florescence |
| P.V.C | - | Polyvinylchloride |
| BIA | - | British Industry Association |
| R & D | - | Research and Development |
| ISTM | - | Indian Standard Testing Methods |

DEFINITION OF OPERATIONAL TERMS

- I. **Acha**-Is an old African small brown cereal crop.
- II. **Acha husk**-The brown outer covering surrounding the acha grain.
- III. **Batch**-The amount of beneficiated Kankara kaolin (Binder), Grog, Acha (*Digitaria Exilis*) husk and water used for the bricks production.
- IV. **Beneficiation**-Synonymous to upgrading. Is the improvement of the quality of raw materials by reducing the impurities to barest minimum commonly achieved by washing, sedimentation, decantation, solution, sieving and chemical treatment.
- V. **Brick**-Formed by mixing calcined clay with lower water content and molded to required shape and size by compaction and burnt.
- VI. **Calcining**-Pre-heating kaolin material to change and improve its properties.
- VII. **Characterization**- The determination of the quantitative and qualitative content of materials.
- VIII. **Crushing/Milling**-Size reduction hard materials (calcined kaolin) into required particles sizes.
- IX. **Grading**-Separating crushed materials into known grain or particle sizes.
- X. **Grog**- Pre-fired Kaolin
- XI. **Porosity**- The ratio of open spaces to solid body of the bricks
- XII. **Refractory**-Any inorganic, non-metallic material used for the construction of high temperature furnace, kiln, incinerator and so forth.
- XIII. **Thermal insulation**-A heat insulator placed between a hotter region and cooler region in order to reduce heat flow from hotter cooler region.

CHAPTER ONE

INTRODUCTION

1.1 Background of the Study

Refractory is defined technically as an inorganic, non-metallic material used for construction of high temperature equipment e.g. furnaces, kilns and crucibles. They are used to construct high temperature equipment because they themselves can withstand high temperature (Ahmed, 2014).

Refractory could be classified into two categories depending on the porosity namely, dense and porous refractory. These refractories were used in contact with hot liquid metal and gases. Porous refractory constitute one of refractory groups which are most commonly used today for their heat insulation in industrial applications. They are lightweight refractory, with much lower thermal conductivity and heat capacity than other refractory. Porosity is usually created by adding combustible materials to the raw materials mixture. During firing, the combustible materials burns, and leaves a large fraction of pores within the fired body. Different types of pore-formers such as sawdust, Rice husk, and organic foams, or granular materials and so forth are commonly used to obtain decreased density or to produce porous bodies in the insulating materials (Gupta, 2004).

The contents of these agro wastes that create porosity such as Sawdust and rice-husk depend on soil chemistry, climatic conditions and even geographic localization (Aramide, 2012). More so, the pore characteristics of the products vary with agro waste particle size (Watari, 2006). This study will focus on the experimentation with AH for production of semi dry-pressed, low temperature insulating refractory bricks. AH could be a better alternative organic material locally

available, finer than rice husk. Kaolin deposit that has been investigated to be good for refractory products from past research will be used. Example of such kaolin is Kankara kaolin deposit in Katsina state.

Many thousands of years ago, man tamed fire. The use of fire necessitated the use of refractory materials that will withstand high temperatures without melting. The Egyptians were the first to melt iron in vessels and furnaces, which were simply a hollow of earth filled with iron ore and charcoal. Radical advancement in steel making technology was brought about by the invention of the Bessemer converter in 1856, a steel vessel lined with refractory materials which have grown from craft to an applied science (Hloben, 2000).

The rapid increase in population world over and the economic growth has led to an increase in demand for energy. Energy is an expensive commodity a major requirement in metallurgical extraction processes, rising use of glass production. The cost in satisfying this energy requirement therefore requires very high energy conservation. Additionally, high mechanical strength is required to resist load, impact, abrasion, corrosion and erosion in refractory materials (Hloben, 2000). Traditionally, pores have been avoided in ceramic and refractory products to increase the crack resistance, but in last decades an increasing number of applications that require porous ceramic and refractory have emerged, in the areas of kilns, furnace lining, which have applications in many industrial areas. The presence of pores in a material can hinder itself all sorts of useful properties that the corresponding bulk materials would have not. To save energy and obtain acceptable furnace shell temperatures, insulating materials are normally used as a backup lining for furnaces and kilns.

The enormous volume of unused organic waste (rice husk, saw dust, Acha husk, etc), by their nature, creates disposal and environmental degradation problems that can potentially result in large scale air and water pollution. According to Kumar *et al*, (2012), many research studies have been conducted using rice husk and saw dust as insulation raw materials, result showed that they produced good insulating bricks. Other parameters that make Insulating raw materials (agro waste) better insulators includes: soil chemistry, climatic conditions and particles size.

Africa is richly blessed with natural resources which could make her develop her capacity for High-tech and value added manufacturing activities to fully participate in world trade. It is rewarding to utilize these local raw materials to develop our society (Kumar *et al*, 2012). This particular research intends to utilize available raw materials such as Kaolin and AH at generating refractory insulating materials. AH is finer, having the right morphology, available and of low or no cost processing into useful products rather than dumping them where future inhabitants are at risk of health problems. This initiative will reduce the dependence on importation of refractory bricks.

1.2 Statement of the Problem

Furnaces, kilns, fore-hearts and so forth, like those used for melting, heat treatment and regeneration demand maximum heat conservation, maximize heat efficiencies and minimize fuel consumption. Insulating bricks are therefore a special class of materials produced to have porous structure with air pockets (Gupta, 2008). Researchers have used rice-husks, saw-dust, waste paper, hard wood chips as pore formers with kaolin to produce insulating bricks (Gupta *et al*, 2008 and Kumar *et al*, 2012). However, the utilization of AH for production of insulating bricks has not either been exploited or no available literature to the researcher to that effect. Despite its

abundance as the oldest African cereal crop it has not been utilized as a pore former. “Acha” grain is surrounded by an outer protective covering (husk, hull or glume) similar to that of rice from which AH is obtained. It also constitutes environmental hazard (Shamle *et al*, 2014). Therefore, the problem of the study is to explore the potentiality of AH as an alternative combustible raw material for insulating bricks, which does not require technical processing and grading.

1.3 Aim of the Study

The aim of this study was to produce thermal Insulating bricks from Kankara Kaolin using AH as pore former.

1.4 Objectives of the Study

The objectives of this study were to:

1. source and beneficiate Kankara Kaolin and Acha Husk.
2. characterize the raw materials.
3. formulate different compositions from the raw materials.
4. produce thermal insulating firebricks.
5. characterize the properties of products.

1.5 Delimitation

The study was delimited to Kankara Kaolin and AH from Kenyi, Kagarko Local Government, Kaduna State.

1.6 Research Questions

1. How will the materials be sourced?
2. What were the effects of beneficiating and calcining the raw materials?
3. What were the chemical contents of the raw materials and their effects?
4. Which formulation was the best?
5. Was available production conditions gave products similar to those produced under conventional conditions?

1.7 Significance of the Study

1. The success of the research will introduce an alternative raw material for insulating fire bricks.
2. Cost of processing AH is quite low compared to Rice husks and Saw dust.
3. The outcome of the research will add to the local raw materials for insulating bricks.
4. The research report will contribute to the number of literatures available for research purposes.
5. The research proposes a reduction in environmental degradation by utilization of the dumped waste.

1.8 Justifications for the Study

1. Cost of energy too high.
2. High-tech required for processing other combustible materials (Rice husk, Saw dust, Straw etc.)
3. Unavailable literature on AH.
4. Health hazards.

1.9 Limitations

1. Ceramic materials processing and production requires modern standard equipment or machines which are obviously either non-functional or unavailable for example, kaolin processing Plant, hydraulic press, oven- furnace, industrial furnace and so forth, consequently impeding expected outcome of results.
2. None concentration of some of these available equipment in a single laboratory led to movements of research materials from one location to another for necessary test or production processes. This tampers with the qualities of products.

1.10. Scope of the Study

The research covered the physical and chemical characterization of the clay sample from Kankara and AH to determine their plasticity index particle size distribution elemental oxide composition. The research involved beneficiation of Kankara kaolin using wet method, production of grog (calculation, size reduction and sieving using the beneficiated Kankara kaolin), production of the refractory bricks and property test were conducted.

CHAPTER TWO

REVIEW OF RELATED LITERATURE

2.1 Introduction

This chapter discusses on refractories, classification, thermal insulation, insulating materials, kaolin and its processing, batch formulation (Andreason's Parameter Equation), manufacturing of thermal insulating firebricks and property test.

2.2 Historical Perspective of Refractory

Refractories have double origin. They owe their form to the ancient industry of brick making and their substance to medieval alchemists who discovered the utility of fire resistant clays. The art of brick making first took place in the molds of ancient Egyptian artisans. According to scholars, this probably took place during the first Egyptian Dynasty, about 3200-2660 BC, when semi-dried mud bricks were used for construction of shrines and tombs. Fired clay bricks were first made in Chaldea (Ahmed and Green, 2000).

Medieval alchemists first used clay as a refractory material to line retorts, crucibles and furnaces. It was not until the middle of the 18th century that fire bricks were made in England by common brick making methods of special fire clays then available. The joining of these two crafts signaled the birth of the refractory industry (Ahmed and Green, 2000).

Egyptians were the first to melt iron in vessels and furnaces, which were simply a hollow of earth filled with iron ore and charcoal. Radical advancement in steel making technology was brought about by the invention of the Bessemer converter in 1856, a steel vessel lined with refractory materials have now grown from craft to an applied science (Hloben, 2000).

2.3 Classification of Refractory

A refractory is a material that retains its shape and chemical identity when subjected to high temperature and is used in applications that require extreme resistance to heat (Ahmed, 2014). Generally, refractories are classified into chemicominalogical, special applications, refractoriness and method of production.

2.3.1 Chemicomineralogical classification

This is based on the chemical and mineralogical composition of the refractory. Refractories are classified as acid, neutral and basic based on their chemical reaction with slag, fluxes and furnace gases. Acid refractories are attacked by basic slag, they include: Aluminium-silicate refractories (fireclay, high alumina, kyanite, silimanite, bauxite, diaspore, mullite, andulusite etc.). Silica refractories (mainly silica and very low in alkalis, metallic oxides, natural rock, ganister, quartzite, etc.)

Basic refractories are attacked by acid slag, they include: Alumina (Al_2O_3) refractories manufactured from burnt bauxite, electrocast bauxite, or electro fused corundum. Dolomite refractories produced by calcining natural dolomite which is mainly a mixture of lime and magnesia. Magnesite refractories produced dead burnt magnesite (MgO). Chrome-magnesite and Magnesite-chrome refractories which are calcined magnesite and chromium in various proportions.

Neutral refractories are not attacked either by acid slag or basic slag, they include: chromite, carbon, artificially produced refractories like silicon carbide, zirconium carbide, titanium carbide, etc. metals used as refractory like thorium, tungsten, zirconium, platinum, nickel, iron, molybdenum, copper, vanadium, tantalum and so forth (Gupta, 2008).

2.3.2 Classification based on special applications

Insulating refractories are suitable for minimizing heat losses and thus achieving heat conservation in the furnaces. They have high porosity, low thermal conductivity and high thermal insulation properties and are produced from asbestos, fireclay, kieselguhr, foam ceramics, ceramic fibre, wool, slagwool, vermiculite and so forth. Special refractories are expensive materials used for making crucibles and furnaces for special/experimental purposes where cost of refractory is no consideration. They include pure oxides (e.g. magnesia, silica, alumina, beryllia, thoria etc.) borides, nitrides, silicides, carbides and so forth. Others include: Sialons, zircon, carborundum, alundum. Cermet refractories are combination of ceramic material (e.g. oxide, nitride, carbide, etc.) and metallic/metallic alloys materials. It has the combination of good properties of both metallic and ceramic materials e.g. high strength and resistance to high temperature. Cermets are used for nuclear reactors, missiles and space crafts and so forth (Gupta, 2008).

2.3.3 Classification based on refractoriness

Refractoriness is the resistance to fusion of the refractory under a steady rising temperature in the absence of disturbing conditions. Fire-clay refractories vary depending upon the fire-clay used for its manufacture. There are three grades of fire-clay:

- 1st grade having softening temperature between 1770°C and 1790°C
- 2nd grade having softening temperature between 1670°C and 1770°C
- 3rd grade having softening temperature between 1580°C and 1670°C

Wide variation in the refractoriness of the fire-clay bricks varying from Pyrometric cone equivalent (PCE) values of Orton 31 to 34 and is dependent on the type of clay used, their

alumina content and the type of grog used. The maximum safe working temperature for fire-clay bricks and other refractories is 1545°C (Gupta, 2008).

2.3.4 Classification based on method of production

Some of the methods of production of refractories include: Dry Pressed, Semi dry, Wet pressed, Fused Cast or electro cast, Hand moulded, Formed and Unformed. In dry pressing, the batch material usually contains about 20 - 50% grog, in the case of fireclay, mixed with relatively small amount of moisture about 5- 8%. The batch is charged into steel moulds and compacted mechanically or hydraulically applied pressure of about 200kg/cm² or more.

These are dried and fired to the desired temperature. Lower moisture contents in mixtures require high pressure and higher moisture contents mixes increases the slumping chances at the time of release from the mould.

Extrusion is a method of production in which the material is passed through a pug mill, where it is mixed with a rather greater amount of water and is then extruded through an auger machine, often under a partial vacuum, cut into lumps of desired size and then repressed. This process is used for production of small quantities of special shapes (Chesti, 1994).

Slip casting is another method of moulding refractories of hollow and other special shapes from very fine powders. In slip casting, a colloidal suspension is poured into a plaster of Paris of a uniform layer of clay on the walls of the mould. When the desired thickness of clay is deposited, which depends on the time allowed for deposition, the mixture in suspension is poured out of the mould resulting to hollow refractory with irregular internal contours.

Powder pressing is another forming process extensively used, besides other purposes, for refractories which involve compacting dry or slightly damp refractory powder mixtures in metallic dies using sufficiently high pressures to produce dense and strong refractory shapes. More recently processes like reactive hot pressing and isostatic pressing have been used for moulding some typical refractories (Chesti, 1994).

2.4 Properties of Refractory

Refractories are mechanically strong and heat resistant to withstand rapid temperature change and corrosion and erosion by molten metal, glass melt, slag and hot gas.

A refractory must possess some required properties for it to be accepted in service. These requirements however vary according to the type of refractory application which generally include; resistance to high temperature, bearing load, fluxes, corrosive fluids like slag, thermal shocks due to rapid change in temperature (also called spalling resistance) and so forth. Other properties are strength, specific gravity, thermal and electrical conductivity (Gupta, 2004). Some are discussed below; Drying and firing shrinkage, bulk density, apparent porosity, modulus of rupture, cold crushing strength and so forth.

2.4.1 Drying shrinkage: Shrinkages are more commonly expressed in linear form. In expressing drying linear shrinkages, two basis on which the calculation is made. If L_{fd} is the dimension of the dried unfired sample, percentage linear drying shrinkage on the as-formed basis is given as (ASTM C179-14):

$$\%LD_{sf} = \frac{L_{fd} - L_d}{L_{fd}} \times 100 \dots\dots\dots(1)$$

Percentage linear drying shrinkage on the dried basis is

$$\%LDSd = \frac{L_f - L_d}{L_d} \times 100 \dots\dots\dots(2)$$

The greater the amount of water presents in the formed sample, the greater the drying shrinkage.

2.4.2 Firing Shrinkage

Firing Shrinkage of refractory is normally based on the dried unfired dimension. It is of more importance to ceramist than drying shrinkage. If L_f is the dimension of the fired, percentage linear firing shrinkage is given as (ASTM C179-14):

$$\%LFS = \frac{L_d - L_f}{L_d} \times 100 \dots\dots\dots(3)$$

2.4.3 Porosity

It is the percentage relationship between the volume of the pore spaces on a refractory material and the total volume of the material. Porosity can be apparent or true. Apparent porosity is the percentage relationship between the volume of the open spaced and the total volume of the material. It is given as (ASTM C20-00 2015):

$$ApparentPorosity = \%Pa = \frac{W - D}{W - S} \times 100 \dots\dots\dots(4)$$

Where:

D= Constant Weight of the dry sample

S= Weight of the sample suspended in water.

W= Weight of sample in air including the moisture in its open pores.

True Porosity is the percentage relationship between the volume of the combined open and closed pore spaces and the total volume of the refractory (Chesti, 1994).

2.4.4 Bulk density (BD)

The BD of a refractory indicates whether the refractory was well fired and thus the degree of densification. It is determined by dividing the test brick mass by the exterior volume thus (ASTM C20-00 2015);

$$\text{Bulk Density} = \frac{\text{Dry weight of sample}}{\text{Saturated Weight in air} - \text{Suspended weight in water}} \dots\dots\dots(5)$$

2.4.5 Modulus of rupture (MOR)

Cold MOR is a measure of the breaking strength of a refractory at room temperature, while hot MOR measures the breaking strength at high temperatures (Chesters, 1973). MOR shows maximum bending load a material can support. This test is generally carried out for rectangular and cylindrical specimens. The loading system may also be a three point or four point loading system.

MOR for a three point loading systems for a rectangular bar is given by (ASTM C133-97 2015):

$$MOR = \frac{3PL_s}{2bd^2} \dots\dots\dots(6)$$

Where;

P= Maximum load, N

Ls= Length of the sample, m

b= Average breadth of sample, m.

d= Average depth (height) of sample, m.

2.4.6 Cold crushing strength (CCS)

Refractories must be able to withstand the structural load coming over the dense and fine grained refractories which generally possess good crushing strength whereas porous and coarse grained refractories have poor crushing strength (ASTM C133-97 2015)

$$\text{Cold Crushing Strength} = \frac{\text{load Applied (kg)}}{\text{Area (cm}^2\text{)}} \dots\dots\dots(7)$$

2.4.7 Thermal conductivity

Thermal conductivity increases with the decrease of porosity. Porous refractories have air entrapped in their pores and this acts as a non-heat conducting material. The amount of the entrapped air increases with the porosity of the refractory and hence its thermal conductivity decreases. Refractories used in melting furnaces, Kilns and so forth, should have low thermal conductivity to ensure least heat losses and maximum heat efficiencies whereas in recuperators where maximum heat transfer is desired to take place, refractories with high thermal conductivities are used. Insulating refractories have low thermal conductivities.

2.4.8 Thermal shock resistance

Thermal Shock resistance is the ability of a refractory to withstand wide variations in temperature without failure. It is also referred to as spalling resistance. Refractories subjected to cycles of heating and cooling must not easily spall or crack and crumble to pieces. Refractories with low coefficient of thermal expansion and coarse grains have greater resistance to sudden changes in temperature (Chesti, 1994).

2.4.9 Refractoriness or Fusion point

It is the temperature at which the refractory fuses. It should be much higher than the operating temperature of the refractory. Fusion point decreases when refractory is under load and this is called refractoriness under load, (RUL).

2.4.10 Abrasion resistance

A refractory is subjected to wear when it comes in contact with a moving charge as in case of Blast furnace, descending charge materials result in abrasion/wearing away of the refractory

lining material. Abrasion resistance of refractory is increased by using a dense (low porous), fine grained and wear resistant refractory lining.

2.4.11 Erosion resistance

Erosion (Damage due to mechanical action) occurs when metal or gas carrying dust and slag particles hit against the refractory lining resulting in 'Chipping off of particles from the refractory. Splashing of slag erodes the refractory lining whereas slag particles entrained by flame and gases erode particularly the arches and bends of the structure.

2.4.12 Electrical conductivity

For electrical furnaces, the electrical conductivity of refractory material should be low. Except graphite, all other refractories are bad conductors of electricity. The electrical resistivity of the refractory material drops rapidly with rise in temperature (Gupta, 2008).

2.5 Alumino-Silicates

Alumino-Silicates group of refractories comprise essentially of two basic oxides Al_2O_3 and SiO_2 associated with impurity oxides: TiO_2 , Fe_2O_3 , MgO , CaO , Na_2O , K_2O , etc, to the tune of about 2-5% when put together

The Alumino-Silicates group is a large group of refractory materials encompassing kaolin, fireclay, kyanite, andalusite, silimanite, bauxite-based, corundum, mullite, alumina, silica as well as other alumina and silica based minerals. It represents the commonest and cheapest group of refractories and their materials, especially when fireclay and or kaolin are/is used (Sullayman. 2005).

2.5.1 Kaolin

Kaolin is a clay mineral that has a wide application in the industry depending on its purity. Kaolinite clay with formula $\text{Si}_2\text{Al}_2\text{O}_5(\text{OH})_4$ is the major mineral component of kaolin, which usually contain quartz and mica and also, less frequently, feldspar, illite, montmorillonite, illiminite, anatase, heamatite, bauxite, zircon, rutile, kyanite, silimanite, graphite, attapulgate and halloysite (Ajayi and Adefila, 2012).

Clay is an alumino-silicates when moist it is soft, when dried it is hard, when fired it becomes very strong (baked). It is the centre of major ceramic materials. Clay is formed as a result of weathering of several kinds of rock in a process that takes thousands and millions of years as a result of the actions of agents such as wind, rain, glacier, and freezing. In addition to the weathering process, clay deposits may be formed in a place (Primary Clay) as residual deposits in soil for example, Kankara kaolin. However, thick deposits usually are formed as a result of secondary sedimentation deposition process after they have been eroded and transported from their original location of formation (Secondary clay) for example, Onibode clay (Ahmed, 2014).

2.5.2 Structure of kaolin

The theoretical pure clay is usually formed in site and it has the chemical formula $\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$. Kaolinites structure is composed of silicate sheet bonded to aluminium oxide/hydroxide layers called gibbsite layers (Sullayman.2005).

The structure of kaolinite was first suggested in general outlines by Pauling in 1930 and was worked out in some detail by Gruner and later revised by other workers. The structure is composed of a single silica tetrahedral sheet joined to a single alumina octahedral sheet combined in a unit so that the tips of the silica tetrahedrons and one of the layers of the

octahedral sheet form a common layer. Apical oxygen atoms from the tetrahedral sheets replaced hydroxyl ions (OH⁻) on the octahedral sheet and thereby articulate the sheets to form a 1:1 layer. Thus, Kaolinite minerals are composed of structurally asymmetric layers with oxygen planes exposed on one side of a layer and hydroxyls on the other (Nurudeen, 2010).

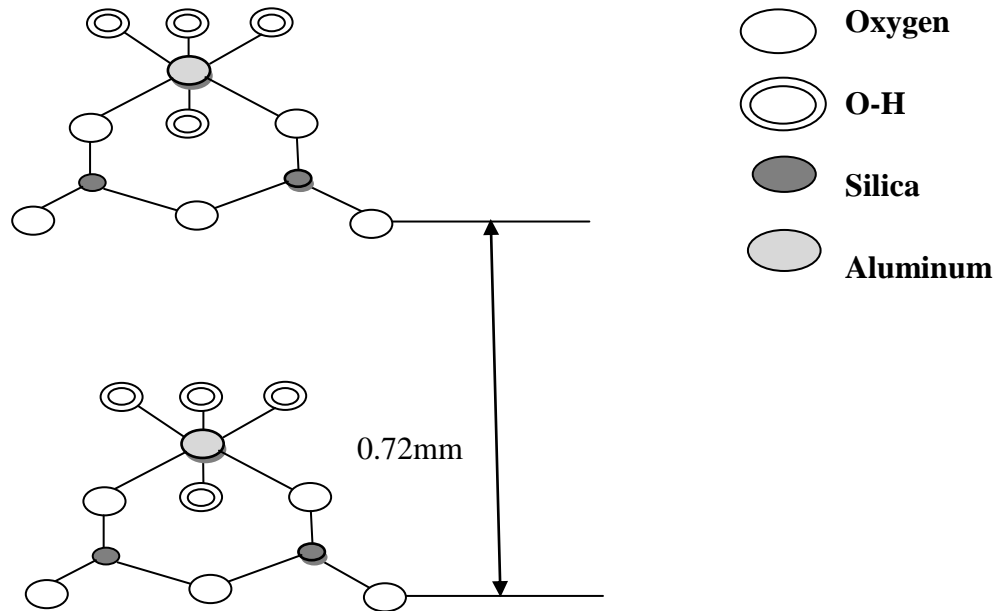


Figure 2.1: Projected Representation of the Structure of Kaolinite (Source: Nurudeen, 2012)

In the layer common to the octahedral and tetrahedral groups, $\frac{2}{3}$ of the atoms are shared by the SiO₂ and Al₂O₃ and then they become O instead of OH. Only $\frac{2}{3}$ of the possible positions of aluminum are filled and there are three possible planes of regular population of the octahedral layer with aluminum. Two aluminium atoms are separated by an OH⁻ above and below making a hexagonal distribution in a single plane in the centre of the octahedral sheet. The OH is placed such that each OH⁻ is directly below the perforation of the hexagonal net of oxygen's in the

tetrahedral sheet. The structure of kaolinite mineral is shown in Figure 2.1 above, with the charge distribution in the layers thus:

Table 2.1 Charge Distribution of Kaolinite Material

| Specie | Charge |
|-----------------------|--|
| $6O^{2-}$ | 12^{-} |
| $4Si^{4+}$ | 16^{+} |
| $4O^{2-} + 2(OH)^{-}$ | 10^{-} (layers common to tetrahedral sheets) |
| $4Al^{3+}$ | 12^{+} |
| $6(OH)^{-}$ | 6^{-} |

(Source: Nurudeen, 2010)

2.5.3 Some deposits of kaolin in Nigeria

The bulk of kaolinite deposits in Nigeria are either of sedimentary or residual origin. Kaolin has wide spread occurrence having been founded in significant quantities in the south-west, North-East, South-West and South-South zones of the country (RMRDC, 2004).

The most extensively investigated clay deposits that mark the Nigeria landscape are the well-known Kankara kaolin deposits in Katsina state, Warram, Onibode, Majohota, clay deposits in Nigeria. Earlier research efforts in Nigeria by others according to (Abdullahi, 2009) are; Bauchi clay, Ikebedi clay, Ikere clay, Eguaben clay, Uloshi clay and so forth, indicated that, most of the Nigerian kaolin compared favourably with the foreign kaolin in terms of quality. Kankara kaolin

for instance, is a good quality China clay that appears even superior to the famous Cornish Chinaclay in Britain. Kankara kaolin contains less iron oxide and other impurities and is of higher clay content by LOI value.

Most of these kaolins have qualities that meet the requirements for refractory clay demand for the iron making shop, that is minimum alumina content of 32% and Fe_2O_3 content of 2.5% and minimum refractoriness of 1730°C (Sullayman, 2005).

2.5.4 Uses of kaolin

- i. Paper:** The largest single users of kaolin is the paper industry which used approximately 1,200,000 tons in 1958, because kaolin is used, paper products print better and are made white and smoother. Kaolin used as filler in the industries of the sheet adds ink receptivity and opacity to the paper sheet. Kaolin used to coat the surface of the paper sheet makes possible sharp photographic illustrations and bright printed colors. Kaolin constitutes nearly one-third of greatest value to the paper industry are whiteness, low viscosity, non-abrasiveness, controlled particle sizes and flat hexagonal plates.
- ii. Rubber:** Kaolin is used as filler in many rubber goods. It adds strength, abrasion resistance and rigidity to both natural and synthetic rubber products. In general, most rubber products extrude more easily after kaolin fillers is added. The major reason that kaolin is used in rubber compounds is its whiteness and low cost. Although kaolin costs less than most other rubber pigments, it has excellent functional properties.
- iii. Ceramics:** Kaolin is used for ceramic white ware products, insulators and refractories. In white-wares, kaolin adds accurate control of molding properties and adds dry fired strength, dimensional stability and a smooth surface finish to the ware. The excellent

dielectric properties and chemical inertness of kaolin makes it well suitable for porcelain electrical insulators. In refractory applications, the dimensional stability, high fusion point and low water content, along with high green strength, makes kaolin an important constituent.

- iv. **Paint:** Kaolin is used in paint because it is chemically inert and insoluble in the paint system, has a high covering power, gives the paint desirable flow properties and low in cost.
- v. **Plastics:** The addition of kaolin to thermosetting and thermoplastic mixes gives smoother surfaces, a more attractive finish, good dimensional stability and high resistance to chemical attack. In addition the flat hexagonal kaolin plastics had the reinforcing fibers and give the mix flow ability to simplify the molding of complex shapes (Ahmed, 2004).

2.6 Fireclay

Fireclay are highly alumina refractories from "Alumino-silicate" group of refractories comprising essentially of two basic oxides Al_2O_3 and SiO_2 associated with impurity oxides TiO_2 , Fe_2O_3 , MgO , CaO , Na_2O , K_2O , and so forth, to the tune of about 2 to 5% when put together. Fireclays are naturally occurring earthly minerals having the chemical compositions given by the formula $\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$. Fireclays may be plastic, semi-plastic or non-plastic. Flint clays are very hard and non-plastic. Their plasticity is increased by the addition of clays to them. Plastic clays may be less refractory and hence their refractoriness is increased by the addition of more refractory clay and thus desired properties of the fireclay are achieved by mixing different clays in proper proportions.

A clay article does not develop its full strength until it has vitrified. The vitrification range of clay starts at about 900°C and extends up to the highest temperature the clay can without melting. Depending upon the kind of clay minerals and the impurities present, the upper temperature limit of the vitrification ranges may vary from below 1000°C to over 1500°C. During the vitrification period, the porosity of the mass decreases and its strength and hardness increases (Garkida, 1998).

The most important equilibrium diagram for the clay technologist is that of the binary system: $\text{SiO}_2\text{-Al}_2\text{O}_3$. It expresses the melting behavior of clay and of clay silica mixes and indicates the reactions taking place during firing. There are three stable crystalline compounds in the system. There are cristobalite at high temperature (1470°C) form of silica, corundum Al_2O_3 (1840°C) and mullite $3\text{Al}_2\text{O}_3\cdot 2\text{SiO}_2$ -(1595°C). Thus, in mixes containing more than 55% alumina, SiO_2 acts as a flux and reduces the softening point of the mixture. If the alumina content exceeds that of the eutectic point mixtures of increasing refractories can be prepared by raising the content of alumina in the mix (Nurudeen, 2010).

2.6.1 High alumina refractories

High alumina refractories have gained importance as they can give better service under severe conditions than fireclay refractories because of their good volume stability, high resistance to abrasion and erosion, high resistance to thermal shock and creep, high refractoriness, greater load bearing capacity and resistance to slag attack. Their content may vary from 45% to as high as 95% or more. As sillimanite, such high alumina containing raw materials like bauxite, bauxitic clays, kyanite, andalusite, and even chemically produced alumina are used for the manufacture of

high alumina refractories. Chemical compositions of some high alumina materials are given in Table 2.2

Table 2.2: Alumina Content of Alumino-Silicate Refractory

| Alumino-Silicate Refractories | Alumina content, wt% |
|--------------------------------------|-----------------------------|
| Fireclay | 25 – 45 |
| Sillimanite | 45 – 65 |
| Mullite | 65 – 75 |
| Bauxite-based | 75 – 90 |
| Corundum | 90 – 100 |

(Source: Ahmed, 1986)

2.7 Thermal Insulating Bodies

A thermal insulator is a poor conductor of heat and has a low thermal conductivity. Insulation is used in buildings, kilns, ovens, furnaces and in other manufacturing processes to prevent heat loss or heat gain. Although its primary purpose is an economic one, it also provides more accurate control of process temperatures and protection of personnel. It prevents condensation on cold surfaces and the resulting corrosion. Such materials are porous, containing large number of dormant air cells (Fatai, 2012).

The insulating firebrick is a class of brick, which consist of highly porous kaolin and combustible organic material. They are light weight, low in thermal conductivity, and yet sufficiently resistant to temperature to be used successfully on the hot side of the furnace wall, thus permitting thin walls of low thermal conductivity and low heat content. The low heat

content is particularly valuable in saving fuel and time on heating up, allows rapid changes in temperature to be made, and permits rapid cooling (Ugheoke et al, 2014).

2.7.1. The principle of thermal insulation

The principle of thermal insulation by the incorporation of air spaces in solid materials was known long before it was understood. Heat transfer occurs by conduction, convection and radiation. Air being a poorer heat conductor than solid materials, the larger the proportion of air in a brick the less heat conducted through it. Convection requires air movement and is prevented if the air spaces are small. Radiation depends on the temperature difference between opposite solid faces. The smaller the air trap the smaller the temperature difference and hence, the less radiation.

The three factors have different relative importance at different temperatures. Fine-pored materials show a linear increase of transmission with temperature, whereas for coarse pored ones, the curve starts at lower transmission and is linear at first but thereafter rises much more steeply. This shows that at low temperatures transmission is due to conduction and the lighter the material the better the insulation. At high temperatures, radiation is the chief method of transmission and fine-pored materials are better insulators (Ahmed and Green, 2000). The pore structure of an insulating brick must therefore be adapted to the anticipated temperature of application.

2.7.2. Thermal insulating materials

Raw materials required for the production of insulating bricks could be categorized into five namely, aggregates, volatiles, binders, combustibles and fire bloomers.

- i. Aggregate:** These include vermiculite, diatomaceous, anorthite (Kaolin and Gypsum), mica and so forth. They introduced low density ($< 0.5\text{g/cm}^3$), high strength, opened porosity up to 85%. They may show volume expansion, flakes and produce air space, with operational temperatures $1100^\circ\text{C} - 1260^\circ\text{C}$ (Chesters, 1973).
- ii. Combustibles:** These include rice husk, saw dust, kiesel-guhr, hardwood chips, starch, Naphthalene, paper, coke, coal, straw and so forth. They burn out during initial stage of firing and create pores in the bricks. The size and shape of pore former controls the pore morphology in insulation bricks. They can be applied for both low and high temperature operations (Ogle, 2003).
- iii. Volatiles:** These include powdered Naphthalene, Ammonium Chloride, Sulphate, Calcium Phosphate and so forth. They evaporate during initial stage of firing and create pores in the bricks.
- iv. Binders:** These include plastic clay, ethyl cellulose, starch and molasses, silicone, sodium silicates and so forth. They are used in manufacturing paste insulation brick. They improve the strength properties of felts. Silicone withstands high temperatures of up to 1150°C even under prolonged use (Gupta, 2008).
- v. Fire bloating:** A number of the more impure clays and shale used for brick making give off gases during firing. If the firing is rapid, this may occur after some fusion and glass formation has occurred. If the viscosity of the glass is sufficiently high, gases become trapped and the whole body expands (Chesti, 1994).

2.7.3. Suitability of the combustible raw materials

The addition of combustible materials has found the most widespread use, primarily because they are widely available agricultural wastes which creates disposal problem due to less

commercial interest. Burning these agro-wastes in ambient atmosphere leaves a residual ash. Also, handling and transportation of these agro-wastes is problematic due to its low density. These agro-wastes ash is a great environmental threat causing damage to land and surrounding area where it is dumped. Bricks made using these agro-wastes (rice husk, sawdust, etc.) develop plenty of pores during heat treatment due to burning out of organic material. The more the percentage of agricultural waste-husk in a brick, the more porous would be the brick and better thermal insulation. Presence of entrapped air in pores has thermal insulating characteristics, thus makes the porous fire brick structure suitable for back-up insulation (Ajayi and Adefila, 2012).

More so, the availability and relative low cost of sourcing and processing compared to other raw materials is an advantage. One of the most important features of combustible materials is nature of the inevitable ash that remains. Coal, coke, anthracite and so forth, have a residue of iron oxide which colours a clean burning brick and acts as a flux, particularly as the reducing conditions prevailing, while the combustible burns out will enable ferrous silicate to form. Sawdust, straw, chaff and so forth, generally contains alkalis which although they do not decolourize the product, have a serious fluxing effect. The refractoriness of a refractory insulator made porous by the addition of combustible material must always therefore be less than that of the original body (Ahmed and Green, 2000).

2.8 Processing of Raw Materials (Kaolin & AH)

2.8.1 Beneficiation of Kaolin

Kaolin is a clay mineral that has a wide application in the industry, depending on its purity. The quality of kaolin mined around the world is depleting especially with depth and rate of mining.

Consequently, the usability of this mineral is threatened by the presence of some inherent impurities. Beneficiation enhances kaolin applications (Ajayi and Adefila, 2012).

The crude kaolin usually requires some purification to separate various impurities present. One of the methods is sedimentation, aimed at separation of minerals on size basis. The method is effective for the separation of kaolin from sand, silt and quartz.

Another method of purifying kaolin is by wet sieving in which impurities are separated on size basis. This method is sometimes preferred to sedimentation techniques because it is much faster to accomplish. Wet sieving is achieved by soaking clay in water, blunged and sieved through the appropriate sieves (Ahmed, 1986). The slip so formed is poured into plaster bats to further extract water to harden. The plastic kaolin is made into rough slaps which are dried in ambient air for two weeks. The slaps are Oven dried for 24 hours at 110°C then calcined at 1260°C-1300°C (Chesti, 1994).

2.8.2 Particles size distribution of kaolin aggregates

The Particles size distribution of kaolin is very important in assessing its plasticity, cohesion and sinterability. If clay particles of the same sizes are used, there will be more spaces between the particles when mixed, which allows easy penetration of thermal shock through the spaces. If different particle sizes are used, the smaller particles would tend to occupy the spaces between the bigger particles. This reduces permeability and increases the resistance to thermal shock. Low porosity can be attained by using a close packing grading such that the interstices between the largest particles are filled by a smaller grade (Nurudeen, 2010).

Several theories exist on grain size packing. The three most used are:

1. The Ainsworth's theory
2. The Andreason's theory

3. Furnas theory

The Ainsworth's theory is based on the utilization of some specified grog particle sizes leaving out others that are not within the range. This leads to wastage of those particles sizes not within the range specified. In the Furnas theory, smaller particles introduced and distributed in the interstices of packed larger particles will reduce the porosity and pore sizes.

When the large particles in a packing are in contact, the theoretical maximum Packing Fraction PF_{max} for a mixture of coarse medium and fine particles is;

$$PF_{max} = PF_c + (1 - PF_c) PF_m + (1 - PF_c)(1 - PF_m)PF_f$$

Where PF_c, PF_m, PF_f are the packing factors for coarse, medium and fine particles respectively. The weight fraction f_i^w of each size at PF_{max} is:

$$f_i^w = \frac{W_i}{W_{total}} \dots\dots\dots(8)$$

Where W_i is the weight of each size

and $W_{total} = \sum W_i$ (Nurudeen, 2010).

Andreason's theory is however more favored because it allows the usage of all particle size in the distribution. The Andreason's equation for optimum dry packing is given as:

$$P = 100(d/D)^n \dots\dots\dots(9)$$

Where, P = Proportion of the fraction

d = Average Particle size/diameter of fractions

D = Maximum particle size in the distribution

n = Andreason's distribution parameter = ($n = \geq 0.1 - \leq 1$)

(Ahmed, 2014)

For a very good composition, a value of n from 0.7 to 0.9 is better and for most refractories, $n = 0.7$ gives good results (Nurudeen, 2010).

Andreason's parameter is adopted in this study for grain size grading owing to its advantages in helping to attain standard refractory properties and maximizing the utility of all particle grain sizes. Theoretically, Coarse, Medium and Fine fractions are usually in the preferred ratio of 66:25:9 respectively. But for specific user requirement of a refractory product, are tenable for adjustment of grog ratios (Ahmed, 1986), while working on "Phosphate bonded fireclay castables" adjusted the above ratio to 66:34 of coarse and medium fractions by adding the fine fractions to the medium fractions. (Sullayman, 2005), adopted the following percentages as a range of grog fraction. They were 40%-55% for coarse fraction, 20%-33% for medium and 22% -30% for fine fractions.

Low porosity can be attained by using a "closed packing grading" such that interstices between the largest particles are filled by a smaller grade. Thus there is some control in the ultimate porosity and strength, for maximum number of contact will also be made with the closest packing.

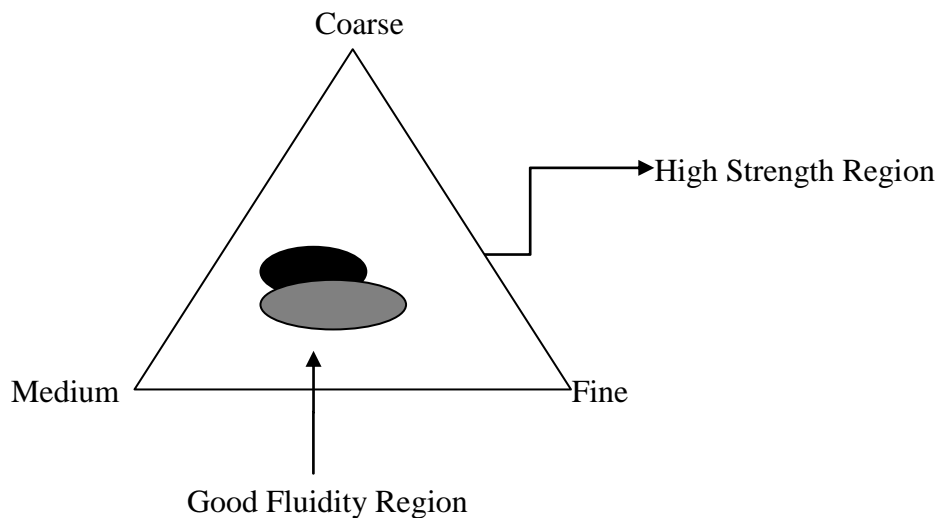


Figure 2.2: Schematic Prediction of Flow Ability and Strength According to Particle Size (Source: Abdullahi, 2009).

Particle sizes are classified into: Fine, Medium and Coarse. With various formulations in the triangle, different flow rheology and strength results. Particles finer than 0.045mm are classified fine, medium particles are between 0.045mm-1mm and particles more than 1mm are denoted as coarse (Abdullahi, 2009).

2.8.3 Combustible additive processing and content

The technological trend towards waste utilization and cost reduction in industrial processing has attracted the use of AH value added material. The most commonly used Agro waste raw materials for the production of insulating refractories are sawdust, rice husk, sugar cane waste, paper and so forth, for producing insulating fireclay products. The agro-waste is screened to be sure that there existed no grains. Then ground and stored in container (Ugheoke *et al*, 2014). The amount of combustible additive added to the composite greatly affects the properties of the insulating bricks. The two properties of importance here are thermal conductivity and transverse strength.

As the amount of additive is increased, the porosity of the light weight material increases. Essentially, when all other conditions are fixed the more pores present and the less solid the lower will be the thermal conductivity. The insulating effect is principally the result of achieving series of air spaces between an alternate series of solid boundaries. The heat storage capacity is determined by the amount of solid. Also, the strength of the material suffers greatly as the amount of combustible additive is increased. The content of some of these agro-wastes (rice husk, etc,) depends on variety of the agro-waste, soil chemistry, climatic conditions and even geographic location (Kumar *et al*, 2012).

The research work is adopting the combustible (agro-waste) class of insulating raw material AH, because of its availability, cheaper method of processing, environmental threat and a potential insulating brick raw material for its fine nature which is capable of producing uniform micro pores and its circular morphology which enhances binding. What is not available in literature (to the researcher's knowledge) is the mixing ratio of AH required to produce insulating bricks. This study will also formulate batch compositions to determine the optimum products.

2.9 Refractory Binders

The sole purpose of a refractory binder is to get materials together in the green state. At elevated temperature binders then either dehydrate, volatilize or sinter leaving behind a refractory material. there are a number of binders which are commonly used for refractory applications, the commonest of which is high alumina cement. They are always selected with the environment of the final application in mind (Nurudeen, 2010).The types of bonds formed by the various refractory binders are summarized in Table 2.3.

Table 2.3: Bond Formed by Refractory Binders.

| Type of Binder | Nature of Bond Formed | Material |
|-------------------|-----------------------|---|
| Cement | Hydraulic bond | Calcium aluminate cement |
| Activated alumina | Hydraulic bond | Pseudo beohmite |
| Colloids | Hydraulic bond | Colloidal alumina, colloidal silica |
| Phosphate binder | Chemical bond | Aluminum phosphate |
| Silicate binder | Chemical bond | Sodium or potassium silicate |
| Resin binder | Organic bond | Phenolic resin |
| Adhesive binder | Adhesive bond | Dextrin, starch, PVA, pitch, Arabic gum, pitch etc |
| Ceramic binder | Ceramic bond | All refractory materials after their heat up to high temperature. |

(Source: Nurudeen, 2010).

2.9.1 Binders used for fireclay brick production

The most commonly used binder for fireclay bricks is plastic clay. However, some of these clays may add impurities and fluxing agents into the batch thereby affecting the final refractory properties of the brick. It is preferred that beneficiated kaolin be used as the clay binder, although ball clays could also be used.

For dry pressing method, clay slip or paste is used as the binder. Trough or Lancaster mixer can be used directly. When using damp or dry clay, it is better to mix the grog and water first and then add the clay. True dry pressing make no use of the natural plasticity that could be developed in any clay by water that is present. By suitable additions of lubricant and binders, dry pressed body can be produced from any raw material that will become hard on firing, whether they possess any plasticity or not. Water soluble binders such as starch fulfill the function of

producing a high plastic strength at the mixer (supplementing the clay) in order to facilitate granulating. They must therefore be used with a plasticizer or a second binder which will function in the absence of water.

Most of the binders used for firebrick products with dry-pressed method are clays. But a very high pressure of forming is required for the pressing and a sufficiently strong steel mould is required. Low pressures can be used for forming fire clay bricks using semi-dry or almost semi-wet method with a moderately strong steel mold (Nurudeen, 2010).

Al-amaireh, (2009), developed fireclay bricks from local Jordanian clays using different Coarse: fine, grog/clay ratios of 70:30, 60:40, 50:50. Chesti, (1994) also stated that “the higher the grog ratio in a brick, the denser the brick. This research will adopt the use of beneficiated Kankara kaolin and water glass (Na_2SiO_3) as binders.

2.10 Fabrication Process of Insulating Bricks

Grog is pre-fired kaolin to about $1,300^\circ\text{C}$, so that no further contraction in volume takes place. Grog addition to firebricks is mainly as an anti-shrinkage element in the form of angular particles of various sizes to achieve better interlocking of grains. The main advantages of grog addition to firebrick include enhances strength of fired refractories, greater spalling resistance, increased specific gravity and decreased apparent porosity, increased production rate due to necessity of less addition of water to get a workable plasticity and lesser time required for drying the green refractory.

Grog addition ranges from 20% to 90% in the clay mixture depending upon the properties of the raw clays and the desired properties of finished products (Gupta, 2008).

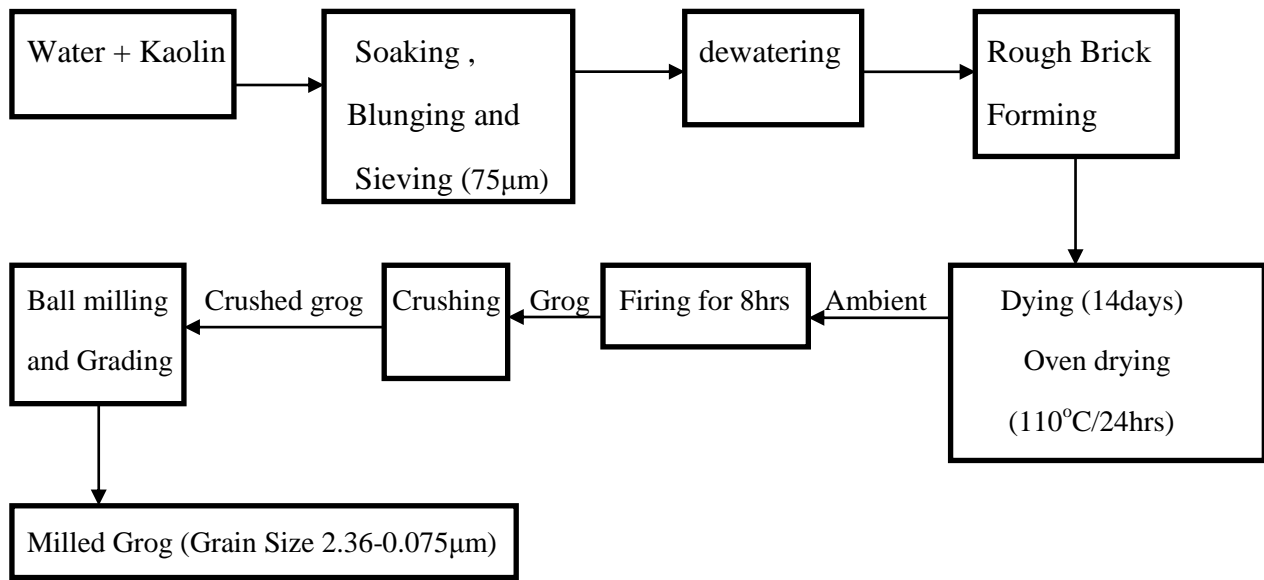


Figure 2.3: Flow Diagram for the Various Stages in the Preparation of the Grog (Source: Gupta, 2008).

2.10.1. Mixing grog with additives

Milled grog materials are graded into required sizes, mixed with the binding material and the combustible additive and pre-calculated amount of water, to ensure a product of uniform composition and distribution of fine and coarse grain, plastic bond materials and so forth, semi-dry mixing and Hydraulic Press machine is adopted.

Moulding is done mechanically by applying pressure or by hand, in steel or wooden mould. Machine moulding has more practical applications and is cheaper for mass production of standard bricks. Machine moulding of dry mixture requires a pressure of about 1000 kg/cm^2 or more (Gupta, 2008).

Drying is done at a slow rate to avoid voids formation, high shrinkage and increased green strength of the bricks by extracting moisture which made for safer subsequent handling. Brick

are burnt in Kiln to remove water of hydration, vitrification and development of stable mineral forms. Shrinkage in volume up to 30% occurs during burning.

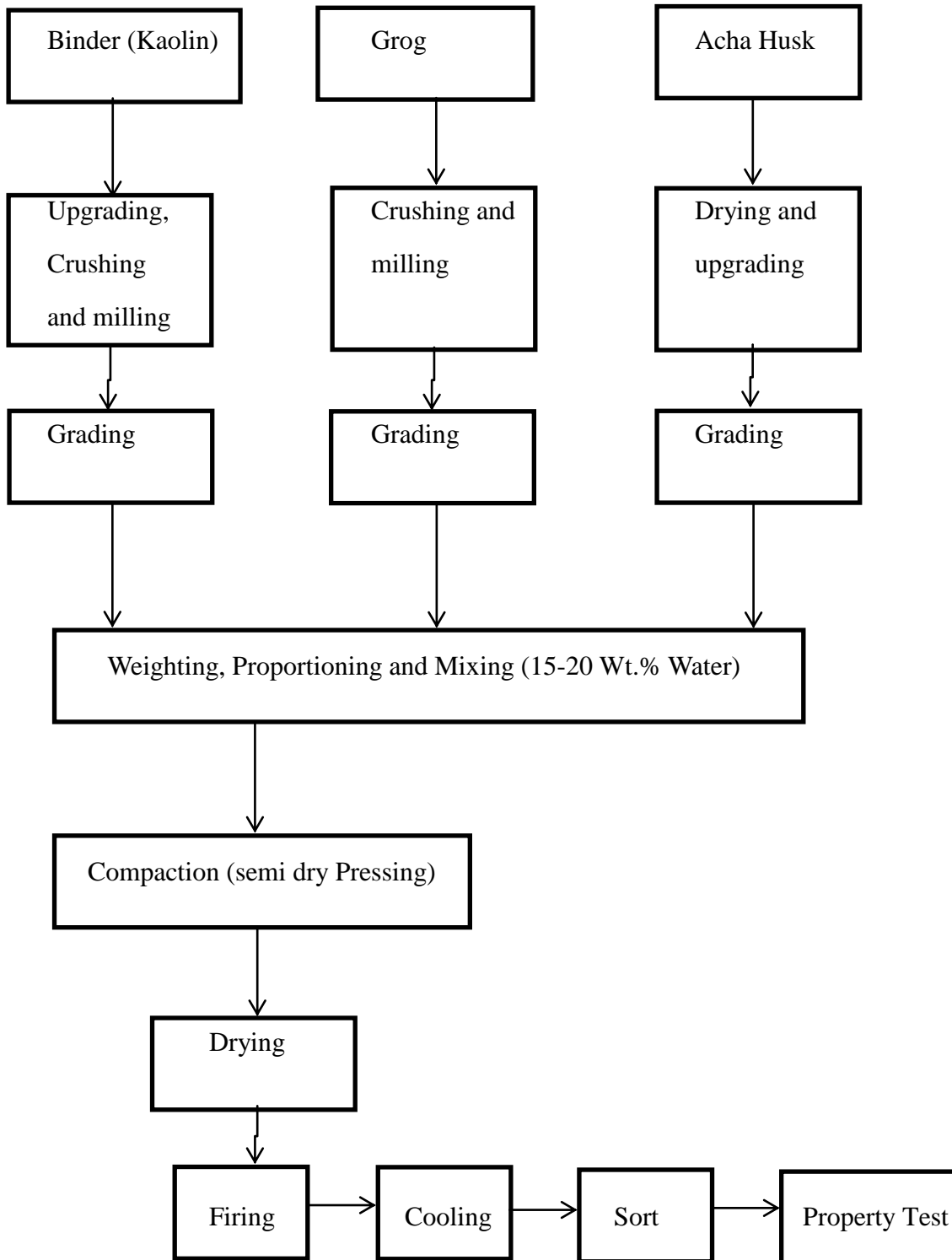


Figure 2.4: Flow Diagram of the Various Stages of Brick Production
(Source: Ahmed and Green: 2000).

2.11 Quality Assurance of Insulating Firebricks

Testing of refractories is necessary because of many variables involved in the manufacture of refractories (Such as multi component raw material, fine, coarse through ultrafine particle sizing, contaminants possibilities, mixing efficiency, firing time and temperature, and many more), testing is necessary to ensure uniform, consistent production. In addition, tests are needed to compare “equivalent” products from different manufactures, to confirm that products meet or exceed user’s specification to determine the effects of service and for research and development among other purposes (Ahmed, 2014).

In this research endeavor, the following tests will be conducted: Physical properties to determine porosity, density and so forth, mechanical properties to determine cold crushing strength; thermal properties to determine thermal conductivity, thermal shock, refractories; Fourier Transform Infrared (FTIR) to determine the functional groups in AH and X-ray fluorescence (XRF) to analyze chemical and elemental constituent of minerals.

CHAPTER THREE

MATERIALS AND METHODS

3.1. Introduction

This study adopts experimental approach from raw materials sourcing, beneficiation of Kankara kaolin and AH, grog preparation, reduction by crushing, milling and grading, particles fractionalization, screening of AH, mixing, bricks making, drying and firing of the bricks and property test and analyses.

3.2. Materials

The materials used for this research include:

- i. **Kankara kaolin:** The colour was off-white with both pink and brown patches. It was used both as the principal raw material and binder. Sourced from Kankara Katsina State.
- ii. **AH:** Light brown covering of Acha grain serves as pore-former raw material sourced from Kenyi, Kaduna State.
- iii. **Water glass:** Used as binder. Purchased from Onitsha.
- iv. **Water:** Normal tap water was used as the liquid vehicle for beneficiation of the kaolin, mixing purposes, bulk density and porosity tests.
- v. **Lubricating oil:** Engine oil was used to lubricate the internal surface of the mould for easy discharge of formed brick.

3.3 Equipment

Several instruments and equipment were used in this research. Some of which include:

- i. **Blunger:** Where the soaked crude kaolin was washed, disintegrated, dispersed and sieved.
- ii. **Jaw crusher:** Used to crush calcined kaolin.
- iii. **Kerosine fired kiln:** Used for heating materials to very high temperature.
- iv. **Muffle furnace,** Model NYC-12, Serial No. 070620901
- v. **Salters spring balance** (ML 25): Used for suspended weighing of bricks.
- vi. **Weighing balance:** Used for high precision measurement.
- vii. **Hydraulic press machine:** Used for compaction of bricks.
- viii. **Venier calliper:** Used for measurements.
- ix. **Tongs.**

3.4 Sourcing of the Raw Materials

The Kaolin sample was obtained from the Kankara kaolin deposit, Katsina State through pit mining. The AH was procured from threshing huts of producers in Kenyi, Kagarko Local Government, Kaduna State.

3.5 Characterization of Kankara Kaolin

Testing the behaviour or nature of raw materials is very important in determining the properties of refractory products and their quality controls for research and development, establishing criteria for and evaluating compliance with specifications and providing data for design purposes (Gupta, 2008)

Kaolin sample was characterized with respect to loss on ignition (LOI), particle size distribution, plasticity, sinterability and chemical compositions.

3.5.1. Atterberg plasticity index

Atterberg plasticity index (API) is the numerical difference between the moisture contents over which the kaolin/clays will behave plastically.

The test includes the determination of the liquid limits, plastic limits and plastic index for the soil.

3.5.2 Liquid limits

The kaolin sample for liquid limit (LL) test was air dried and 200g of the material passed through BS No 4 sieve (425µm), were mixed thoroughly with water to form a homogeneous paste on flat plates. A portion of the paste was then placed in the cup of the casangrande apparatus, leveled off parallel to the base and divided by drawing the grooving tool along the diameter through the center of the hinge. The device cup was lifted up and dropped by turning the crank on the LL device until the two parts of the clay come into contact at the bottom of the groove. The number of blows at which that occurs was recorded and a little quantity of the soil was taken and its moisture contents determined. If the number of blows exceeded 25, more water was added. When the consistency of the mixture was such that after 25 blows, the groove was closed at a distance of 1.25cm, the percentage of water corresponding to this mixture was taken as LL. The number of blows was used for scaling and plotting the “Liquid and Plastic limit instrumentation graph” on the x-axis, while the moisture content values were recorded on the y-axis of the graph.

The LL for the Kaolin was therefore, determined using the formula (Atterberg Limit Test, 2003):

$$\%LL = \frac{\text{wt of moisture}}{\text{wt of dry sample}} \times 100 \dots\dots\dots(10)$$

Five levels of blows i.e. 49, 26, 22, 19 and 13 were recorded for moisture contents of 46.70, 48.50, 49.2, 50.70 and 59.20 respectively. This was due to the fact that samples had to be tested and plotted on at least four points with four numerical values each (for blow points and moisture contents).

3.5.3 Plastic limit

A portion of kaolin mixes used for the LL test was retained for the determination of plastic limits (PL). The ball of the kaolin admixture was molded between the fingers and rolled between the palms of the hand until it dried sufficiently (even though the kaolin was already relatively drier than the ones used for LL).

The samples were then divided into approximately four equal parts. Each of the parts was rolled into threads between the first finger and the thumb. The thread was then rolled between the tips of the fingers until the thread is reduced to about 3mm. Refer to Plates Ia–e for illustrations.

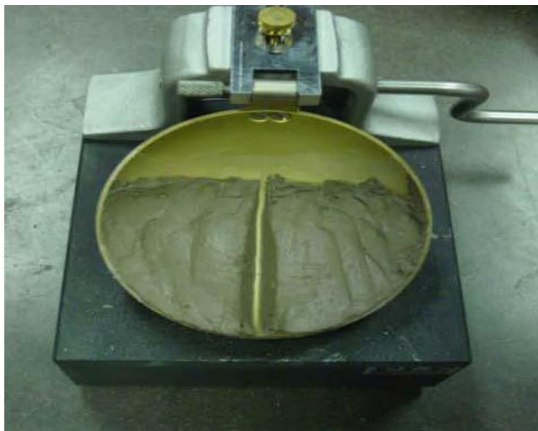
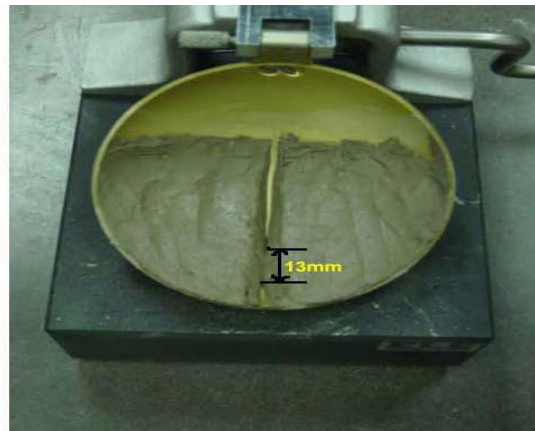


Plate Ia: Liquid Limit Test



Plate; Ib

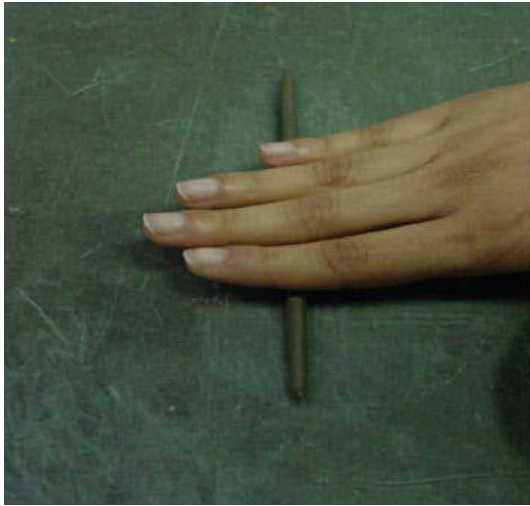


Plate Ic: Plastic Limit Test



Plate; Id

(Source: Atterberg Limit Test, 2003)

The crumbled soil was then put in the moisture container and the moisture content determined. Three samples of crumbled kaolin were placed in an oven and heated to a temperature of 110°C for 24hrs. After which, the dry weight of the sample were measured and recorded. The weight of the empty containers were also measured and recorded. To obtain the %PL for the kaolin, the three weighs of moisture in the three columns of the graph were each calculated against the dry weight in % that is,

$$\%PL = \frac{\text{wt of moisture}}{\text{wt of dry sample}} \times 100 \dots\dots\dots(11)$$

The moisture content was plotted on the graph with a chosen scale of 20(ASTM D4318 – 10, 2013).

To obtain the weight of dried sample, the weight of the empty container was subtracted from the total weight of dry sample and the container, that, $(D_w + W_c) - W_c = D_w$. Alternatively,

subtracted from the total weight of the wet sample (W_s) and weight of container (W_c) to arrive at weight of moisture (W_m).that is, $(W_s + W_c) - W_m$. The plastic limits of the samples were calculated. The average weights of the two calibrations served the PL.

3.5.4 Plastic index

The plastic index (PI) of the kaolin mix is the difference between the LLs and their corresponding PL which is calculated as:

$$PI = LL - PL$$

Where:

LL = Liquid Limit

PL = Plastic Limit

PI = Plastic Index.

3.5.5 Particle size distribution

The particle size distributions of the kaolin were determined by wet sieving. Fifty grams of dried clay was weighed into a weighed dried Tyler sieve and washed with water to screen out all fractions finer than the sieve opening used. The sieved and retained coarse materials were dried in a Gallen Kamp Oven, type at 110°C to constant weight to determine the percentage of the coarse material. The procedure was carried out with Tyler sieve of mesh numbers 7, 14, 25, 36, 52, 72, 100, 200 and <200.

Based on the particle size distribution curves the optimum sieve size for screening off the coarse impurities was found to be 200 mesh (sieve opening of $75\mu\text{m}$) for all clays (Ahmed, 1986).

Hence, the fractions of kaolin less than 75 μ m were taken as the beneficiated samples. Subsequent characterization was done for both the whole and beneficiated samples.

3.5.6 Hydrometer analysis

The suspension of pretreated kaolin passed through the sieve 52 and 75 μ m (200mm) was transferred into a 1000ml sedimentation cylinder without losing any clay. 25mls of a dispersant solution (sodium hexametaphosphate) was added. Distilled water was placed to hold the hydrometer in between the readings. The sedimentation was allowed to sediment overnight. Thereafter, the sedimentation cylinder was firmly sealed and vigorously shaken by applying about 60 end- over-end cycles within a period of 2minutes. As soon as it was placed at the upright position, the time clock was started (zero time for that sample).

The hydrometer was steadily inserted and allowed to float freely. Readings of the hydrometer at the top of the meniscus level were taken at the following times from zero seconds 10seconds, 15seconds, 30seconds, 1minute, 2minutes, 4minutes, 8minutes, 15minutes, 30minutes, 1hour, 2hours, 4hours, 6hours, 12hours and 24hours. The hydrometer was slowly removed each time and rinsed in distilled water and thermometer readings were recorded.

Percentage fine than each determined size was plotted against the corresponding particle diameter on the same sheet as that used for sieve analysis. Refer to Figure 3.1 for hydrometer apparatus set up.

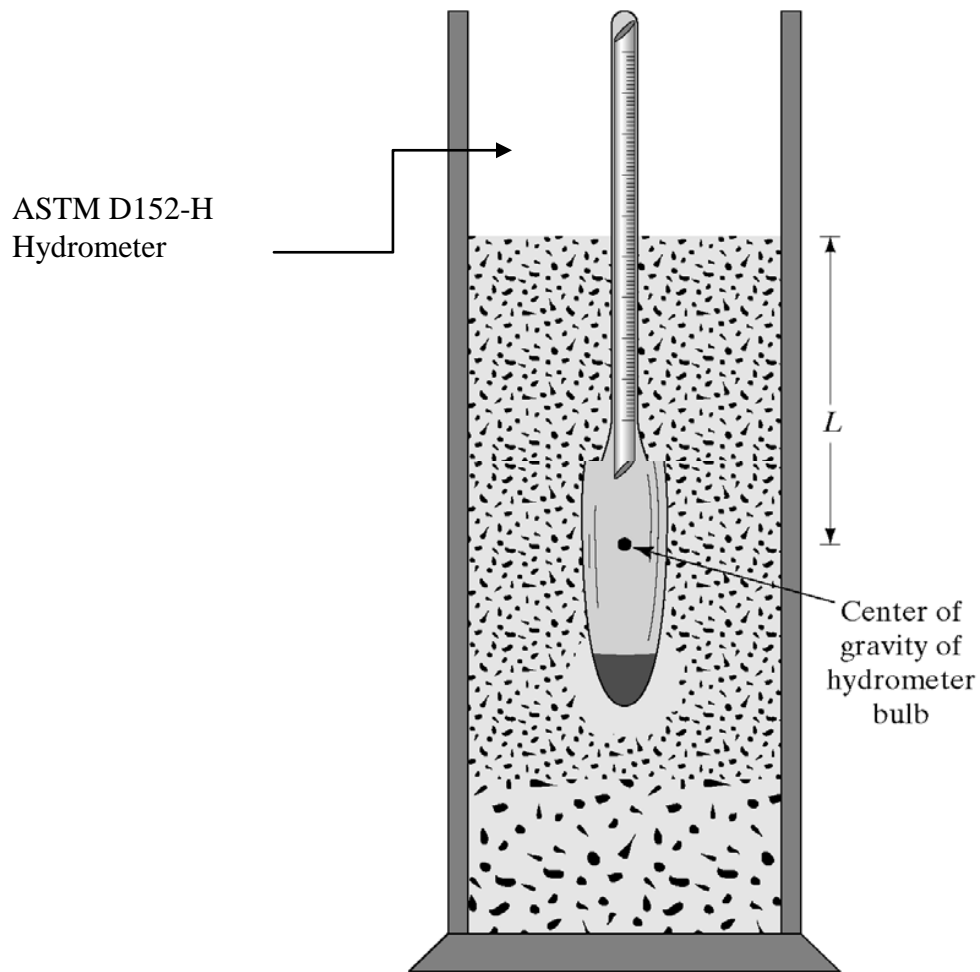


Figure 3.1: Hydrometer Analysis Apparatus

3.5.7 Sinterability

Sintering is the tendency of alumino-silicate minerals or other refractory materials to decrease or shrink in size due to drying or firing schedules. This phenomenon is often referred to as drying and firing shrinkage.

3.5.8 Drying shrinkage test

This refers to the percentage decrease in the original dimensions of refractory materials while plastic, compared to the dimensions obtained after drying. This is expressed as follows (ASTM C179-14):

$$\%DS = \frac{WL - DL}{WL} \times 100 \dots\dots\dots(12)$$

Where WL = Wet length

DL = Dry length

DS = Dry shrinkage

Ten plastic clay test bars were prepared measuring 15cm each; with an indented 10cm line along its lengths. The test bars were allowed to dry under ambient condition for seven days. They were then dried in Gallen Kamp oven, Type – at 110°C for 24 hours. The indented lines on the bars were measured again after drying and the new lengths due to shrinkage, colour change and cracks were noted.

3.5.9 Firing shrinkage test

The dried samples were fired in a kerosene fired kiln to about 1200°C and soaked at that temperature for 8hours. After firing, the bars were observed for colour change, cracks and shrinkage. Shrinkage values were evaluated using the formula (ASTM C179-14):

$$\%FS = \frac{DL - FL}{DL} \times 100 \dots\dots\dots(13)$$

Where:

DL = Dry length

FL = Firing length

FS = Firing Shrinkage

3.5.10 Chemical analysis of samples

Chemical analysis of Kankara kaolin sample and AH were carried out to ascertain and determine their elemental oxide compositions. X-ray fluorescence (XRF) Spectrometry is a method of quantitative analysis that uses X-ray energy (Chesti, 1994). XRF tests chemical and elemental constituents of materials. It detects and quantifies elements from atomic number 11-92. It is regarded as having the “advantage of high level of chemical specificity and reliability of it is elemental presence”. This study utilized the XRF instrument at Nigeria Geological Survey Agency (NGSA) Kaduna for it raw materials analysis. (NGSA, 2015)

3.5.11 Sample (kaolin) preparation

The samples were pulverized using arget pulverizing machine (Planetary Micro mill pulverisette – 7). The ground samples were ensured to pass through 150 micro mesh sieves to ensure homogeneity of the sample. 5g of the pulverized sample was weighed into a beaker, 1g of binding aid (starch soluble). The mixture was mixed to ensure homogeneity, which was pressed under high pressure (6 “tons”) to produced pellets; labeled and packaged for analysis.

3.5.12 Sample preparation of AH

The sieved samples were fired to ash at 700°C using a Carbolite furnace (1300 series). The ash samples were then analyzed directly using “Minipal 4” XRF machine for the determination of their oxides compositions.

3.5.13 Procedure of the analysis

Energy Dispersive X-ray fluorescence (EDXRF) Spectrometer of Model “Minipal 4” was used for the analysis, (DY NO: 1055). The pellets were carefully placed in the respective measuring positions on a sample charger of the machine. The following condition sets were made as the machine was switched on: elemental composition determination, nature of the samples to be analyzed as press powder (pellet), the current used as 20kv for the trace elements/rare earth metals, selected filters Ag/Al – thin for the trace elements

The selection of filters was guided by a given periodic table used for elemental analysis. Time of measurement for each sample was 100 seconds and the medium used was air throughout. The machine was then calibrated by the machines gain control, after which the respective samples were measured by clicking the respective positions of the sample charger (NGSA, 2015).

3.5.14 Principle of XRF

The origin of X-rays is from the loss associated with the interaction of high energy electron with atoms. Electrons from x-ray tubes move towards the electronic field of the electrons in the various shells of the atoms of the targeted material. The incident electrons are decelerated and lose energy. Incident high energy electrons penetrate the outer orbital of the atoms and collide with an electron in the inner orbital. These inner electrons may be completely removed, leaving

the atom in an unstable state. Electron rearrangement to restore stability takes place, leading to the release of energy in form of x-rays. X-rays generated in this way have discrete wavelength which is related to the atomic number of the atoms producing them. They are called 'CHARACTERISTIC X-RAYS'. The detection and measurement of the characteristic x-rays are the basis of x-ray spectrometry (NGSA, 2015)

3.6. Beneficiation of Kankara Kaolin

Raw Kankara kaolin (163kg) was weighed into three aluminium containers and soaked in tap water for 24 hours. It was poured into a blunger, where it was vigorously stirred to break up the small lumps and passed through sieve manually. The slurry was sieved through Tyler Mesh 200 (75 μ m) aperture size, to get rid of coarse particles of +75 μ m. The beneficiated kaolin slurry was soaked for four (4) days to enhanced plasticity and sedimentation. The water was decanted from the slurry and poured to another container to rid-off sediment fine silica, while the materials were poured into plaster dry-mould to achieve high percentage extraction of water within 48 hours.

3.7. Preparation of the Grog/Chamotte

The dewatered kaolin was worked into rectangular mass of stiff consistency and worked into rectangular rough bricks. The bricks were dried in ambient air for fourteen (14) days and electric oven dried at 110°C for 24 hours. After drying schedules, the bricks were loaded in kerosene fueled kiln and pre-heated slowly for 2 hours full firing was done at temperature of about 1280°C under oxidized atmosphere.

The kiln was left to cool down within 26 hours. Crushing of the calcined bricks was done using jaw crusher at the Ceramic Section, Industrial Design Department, Ahmadu Bello University Zaria.



Plate II: Calcined Kaolin (Grog)

The materials were further ball milled into six different particle sizes. The aim is to meet the industrial standard by which maximum grain size of the grog should range between +1.00m – 2.36mm (coarse), and coarse to fine ratio of the grog is 55: 45 (Chesti, 1994).

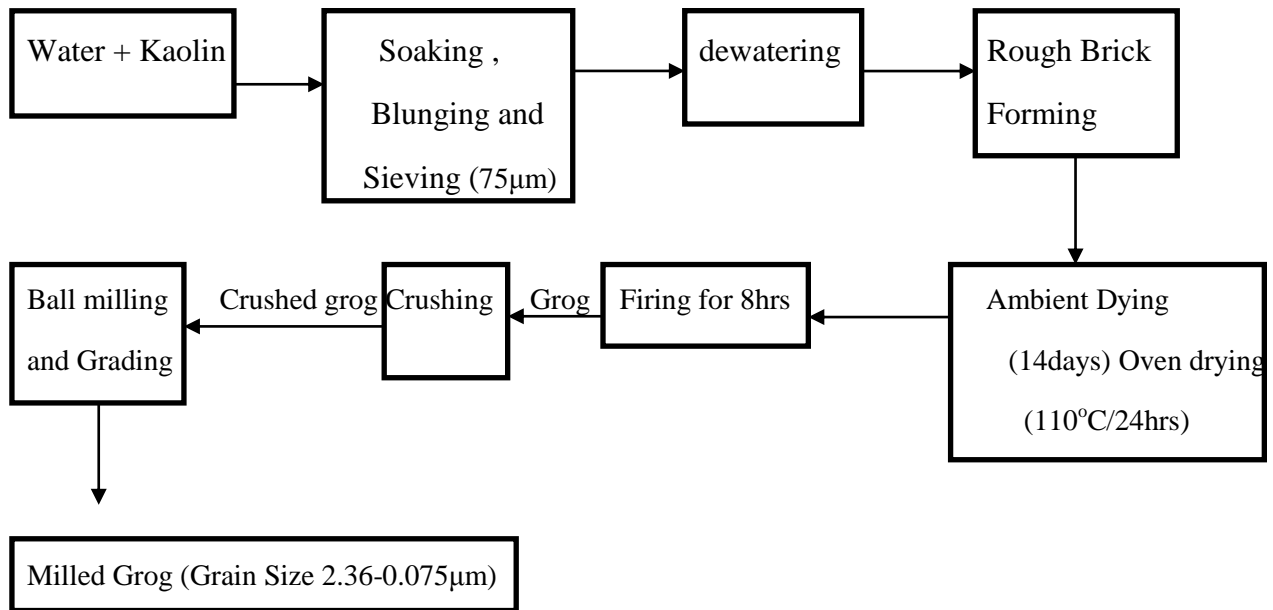


Figure 3.2: Flow Chart for Grog/Chamotte Production

3.8. Acha Husk Preparation

The combustible material (AH) was screened using Tyler Mesh -250µm+150µm (Ahmed and Green, 2000). It was dried and stored in P.V.C bags before utilization.

3.9. Bond Material Preparation

The beneficiated Kankara kaolin was being used as binder. This choice was due to the plasticity index of the kaolin and its purity. In addition 5wt% (29.60cm³) of Na₂SiO₃ was added per brick batch for enhanced binding of the matrix.

3.10 Selection of Batch Composition and Calculations Using Andreason's Parameter Equation.

In choosing the composition of the batches, the following factors were considered:

- i. The size fraction of the grog.
- ii. The proportion of the different fractions of grog, and
- iii. The size fractions of the combustible additives.

(Ahmed and Green, 2000) worked on development of insulating firebricks used particle size range (1.18mm-0.50mm) and $-75\mu\text{m}$ for coarse and fine respectively. While Sullayman, (2005) and Abdullahi, (2009) worked on utilization of local raw materials for the production of dense Alumino-silicates refractory bricks; and Characterization of Ahoko Kaolin for its refractory's properties respectively, used particle size range (-2.36mm +1.18mm) as Coarse, (-1.18mm +0.600mm) as Medium, (-0.600mm +0.300mm), (-0.300mm + 0.150mm) (-0.150mm + 0.075mm) and (-0.075mm) as Fine particles. Based on these factors enumerated above, this research adopted (-2.36mm + 1.18mm) as coarse, (-1.18mm + 0.600mm) as coarse grain size, (-0.600mm + 0.300mm) as medium and (-0.300mm + 0.150mm),(- 0.150mm + 0.075mm) and (0.075mm)as fine for porous bricks.

3.10.1 Mathematical calculations of Andreason's Parameter

Andreason's parameter represents the ratios of the three particles size classification in refractory composition, such as, Coarse, Medium and Fine grain fractions necessary for the desired packing. The different size fractions of the grog include:

- i. $-2.36\text{mm} + 1.18\text{mm}$

- ii. -1.18mm + 0.600mm
- iii. -0.600mm + 0.300mm
- iv. -0.300mm + 0.150mm
- v. -0.150mm + 0.075mm
- vi. < 0.075mm

Andreason's Parameter Equitation:

$$P=100(d/D)^n$$

Where:

P= Weight % fraction or proportion of the fraction

d= Average diameter (Mean) of fraction

D= Maximum particle size in the system

Andreason's Parameter (For Refractories n= 0.7) (Ahmed, 2014)

Table 3.1: Total Proportion of the Six Grog Fraction

| Grog Fraction | Proportion |
|----------------------|-------------------|
| P1 | 81.76 |
| P2 | 50.53 |
| P3 | 31.35 |
| P4 | 19.30 |
| P5 | 11.92 |
| P6 | 5.51 |
| Total | 200.37 |

Therefore, the sum total of batch aggregate particles classification (%) is shown in the table below:

Table 3.2 Batch Aggregate Particle Classification (%)

| Grog fraction | Proportion |
|----------------------|-------------------|
| P1 | 40.80 |
| P2 | 25.22 |
| P3 | 15.65 |
| P4 | 9.63 |
| P5 | 5.95 |
| P6 | 2.75 |
| Total | 100.00 |

Table 3.3 Total Grog Fractions Obtained by Weight (Kg) after Sieving

| Grog fraction | Sieve sizes | Grog particle weight(Kg) | % Grog fraction based on total weight (56.06Kg) |
|----------------------|--------------------|---------------------------------|--|
| P1 | -2.36 +1.18mm | 8.95 | 15.95 |
| P2 | -1.18 + 0.600mm | 22.94 | 40.92 |
| P3 | -0.600 + 0.300mm | 12.44 | 22.19 |
| P4 | -0.300 + 0.150mm | 5.64 | 10.06 |
| P5 | -0.150 + 0.075mm | 3.84 | 6.85 |
| P6 | -0.076mm | 2.26 | 4.03 |
| | Total | 56.06 | 100.00 |

The total amount of grog prepared = 56.06kg. The specific grog fractions obtained using the percentage values from Table 3.3

Table 3.4: Grog Fractions by Weight % each Fraction

| Grog Fraction | Weight (Kg) |
|----------------------|--------------------|
| P1 | 22.87 |
| P2 | 14.14 |
| P3 | 8.77 |
| P4 | 5.40 |
| P5 | 3.34 |
| P6 | 1.54 |
| Total | 56.06 |

Table 3.5: Grog Fractions Required Per Bricks

| Grog Fraction | Weight (Kg) |
|----------------------|--------------------|
| P1 | 93.796 |
| P2 | 255.876 |
| P3 | 138.756 |
| P4 | 62.906 |
| P5 | 42.834 |
| P6 | 25.199 |
| Total | 624.995 |

Five grog compositions designated A, B, C, D and E were formulated. The coarse and fine size ratios were used to determine the most suitable ratio(s) for insulating bricks. They include ratios, 70:30, 66:34, 60:40, 55:45 adopted from (Ahmed, 1986; Chesti, 1994, Ahmed and Green, 2000) and ratio 50:50 being experimented.

AH particle size $-230\mu\text{m} + 150\mu\text{m}$ was adopted & used from (Ahmed and Green, 2000). The AH compositions were varied in each set of the batches to determine its effect. Two parameters were studied i.e. different coarse: fine grog ratios and the AH% weight variation.

Table 3.6: Batch Composition for each Ratio

| Coarse/Fine (Ratio) | Weigh % of Coarse(g) | Weigh % of Fine(g) | 10 × Weigh % Coarse | | 10 × Weigh % Fine | |
|------------------------|-------------------------|-----------------------|------------------------|-------|----------------------|-------|
| | | | g | Kg | g | Kg |
| 70:30 | 437.717 | 187.593 | 4377.17 | 4.311 | 1875.93 | 1.876 |
| 66:34 | 412.705 | 212.604 | 4127.05 | 4.128 | 212.04 | 2.126 |
| 60:40 | 375.186 | 250.124 | 3751.86 | 3.752 | 2501.24 | 2.501 |
| 55:45 | 343.921 | 281.389 | 3439.21 | 3.439 | 2813.89 | 2.814 |
| 50:50 | 312.655 | 312.655 | 3126.55 | 3.127 | 3126.55 | 3.127 |

Table 3.7: Compositions of Bricks for the Sets of Batch.

| Sample (Set) | Weight percentage | | | | Translated to actual weight | | | |
|-----------------|-------------------|---------------|------------|------------|-----------------------------|---------------|-----------|--------------|
| | Grog Wt% | Kaolin wt% | AH (AH) | Total % | Grog (g) | Kaolin (g) | AH (g) | Total (g) |
| 1 | 89 | 10 | 01 | 100 | 556.25 | 62.5 | 6.25 | 625 |
| 2 | 88 | 10 | 02 | 100 | 550 | 62.5 | 12.50 | 625 |
| 3 | 87 | 10 | 03 | 100 | 543.75 | 62.5 | 18.75 | 625 |
| 4 | 86 | 10 | 04 | 100 | 537.50 | 62.5 | 25 | 625 |
| 5 | 85 | 10 | 05 | 100 | 531.25 | 62.5 | 31.25 | 625 |
| 6 | 84 | 10 | 06 | 100 | 525 | 62.5 | 37.50 | 625 |
| 7 | 83 | 10 | 07 | 100 | 518.75 | 62.5 | 43.75 | 625 |
| 8 | 82 | 10 | 08 | 100 | 512.50 | 62.5 | 50 | 625 |
| 9 | 81 | 10 | 09 | 100 | 506.25 | 62.5 | 56.25 | 625 |
| 10 | 80 | 10 | 10 | 100 | 500 | 62.5 | 62.50 | 625 |

3.11. Calculating for Bond Material

The raw kaolin from Kankara deposit was adopted as bond material for this research. This choice is as a result of the availability, low cost, purity due to its tolerable iron oxide content and its plasticity index-value as supported by available literature and results from API.

Bond material of about 10wt% of the total grog quantities is recommended (Chesti, 1994). Hence, 10wt% binder of the total weight of grog fractions per a laboratory size refractory brick was computed in each case for the fifty (50) bricks produced.

Total quantity of grog computed per brick = 0.625 kg i.e. 625g

∴ 10wt% of total grog quantity represents the quantity of raw clay used as binder.

$$\text{i.e. } \frac{10}{100} \times 625 = 62.52g$$

3.11.1 Calculating for AH

The combustible additive content of between 1wt% and 10wt% were used per set of the five different grog proportion ratios (70:30, 66:34, 60:40, 55:45 and 50:50).

3.11.2 Moisture content

Semi Dry Press Method of refractory brick production was adopted for this research. Various literatures have recommended a minimum of between 5wt% water (Gupta, 2004) and a minimum of 10wt% moisture per weight of particles or kaolin used.

About 20wt% of water was used for particle cohesion and the required ball-in-hand consistency. Under a clenched of fist, because 10wt% and 15wt% moisture could not give the required particle cohesion.

Thus, about 20wt% moisture was used for batching weight of grog per brick + Binder (Kaolin) + Husk = 625g

$$\therefore 20\text{wt}\% \text{ moisture content} = \frac{20}{100} \times 625 = 125\text{g} \cong 125\text{mls moisture}$$

3.12. Production of Insulating Refractory Bricks

3.12.1 Semi-dry mixing of the batch

Weighing of the size fractions for the five different ratios was done firstly-coarse and fine, secondly the proportion of the different fractions of grog, binders (Kaolin), Sodium Silicate and different wt% of the combustible additives were thoroughly mixed using spatula and later hand to obtain uniformity. Finally, the required weight percentage of water was added in each of the batch per brick and properly mixed to attain some level of particle cohesion for a ball-in-hand consistency, indicative of adequate moisture content.

3.12.2 Packaging and storage for ageing

The batches for each of the 50 bricks were separately secured in polythene bags and stored in a plastic bucket. The materials were kept for two weeks, which enabled gain in plasticity of the kaolin (binder).

3.12.3 Mould design and fabrication

Three (3) mild steel moulds, A, B, and C were designed and fabricated. Moulds A and B were fabricated at the Mechanical Engineering Workshop, Ahmadu Bello University, Zaria. Mould C was fabricated at Jimoh's Workshop Samaru Zaria.

I. Mould A

The mould is an assembly of a single component with a plunger and bottom plate. Figures 3.3a and 3.3b are the orthographic projections and isometric view respectively.

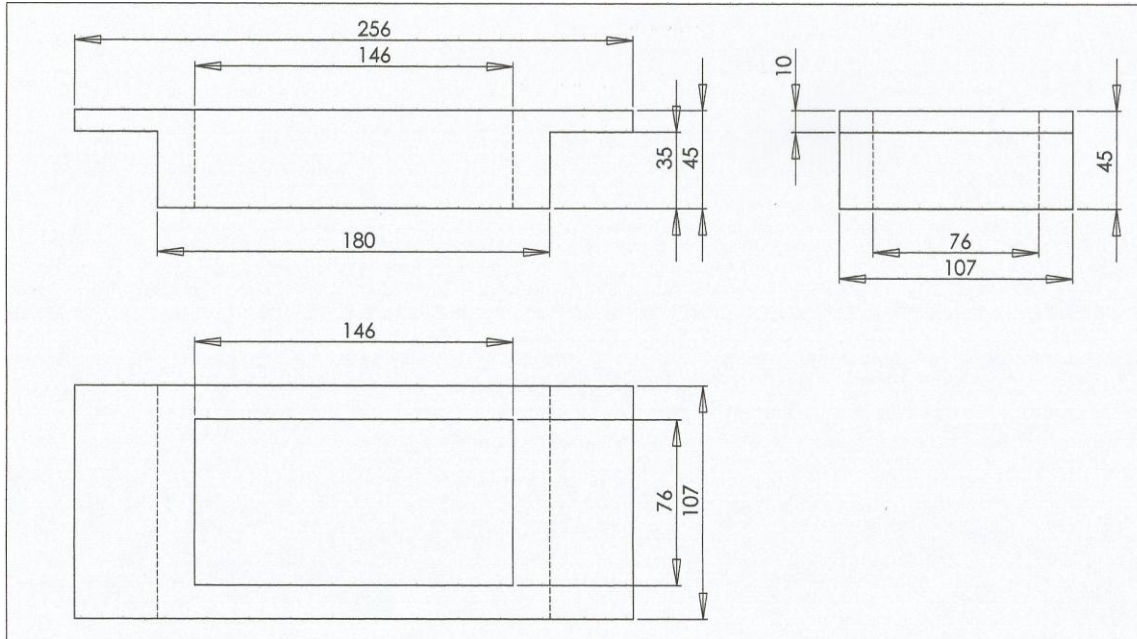


Figure 3.3a: Orthographic Projections of Mould A

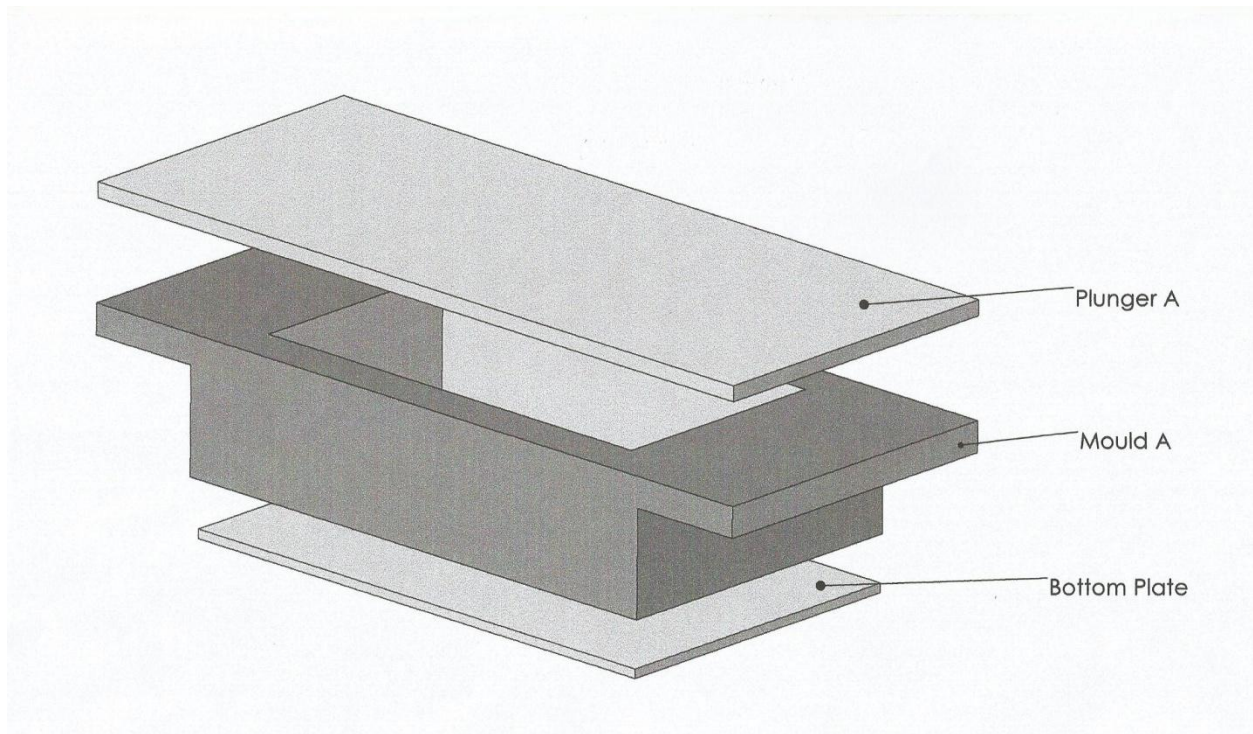


Figure 3.3b: Assembled Mould-A

Problem associated with Mould A, was non-release of the brick even when engine oil was applied on the internal surface of the Mould. This led to the design and fabrication of Mould-B.

II. Mould B

The mould is an assembly of two components joined by four bolts (two on each side), to enable coupling and decoupling during brick production. It also has a plunger and bottom plate. Figures 3.3c, 3.3d and 3.3e are the orthographic projections and isometric view respectively.

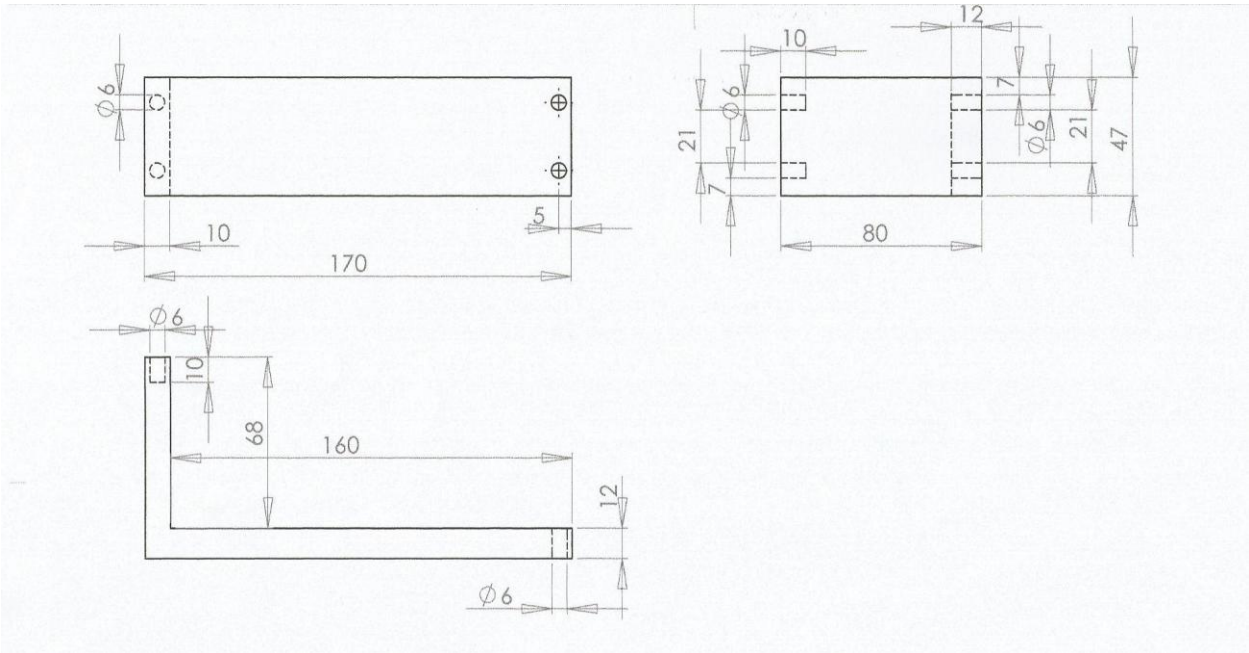


Figure 3.3c: Orthographic Projections of Mould B Side-1

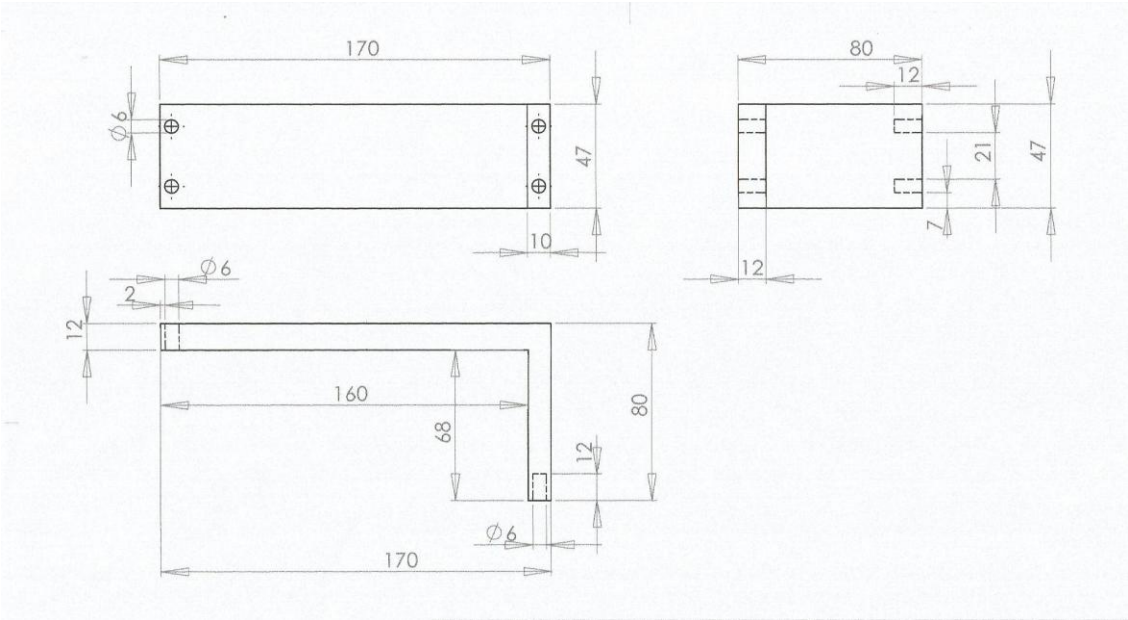


Figure 3.3d: Orthographic Projections of Mould B Side-2

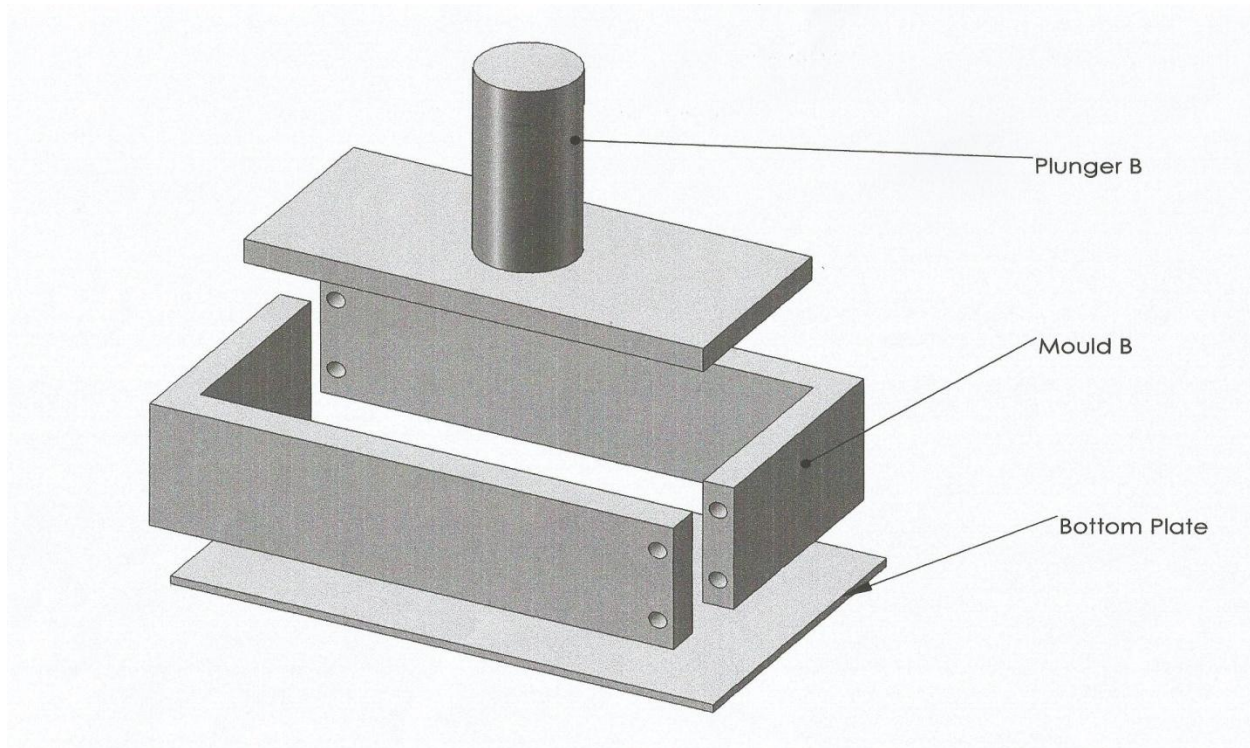


Figure 3.3e: Assembled Mould-B

Problems associated with Mould-B, are the longer time taken to screw and unscrew the bolts, each time batch charging and brick discharge is done. It took about 25 - 30minutes to produce one brick. More so, the brick formed was being stressed in the process of bolting and de-bolting, consequently, the shape of the brick gets deformed.

III. Mould - C

The Mould is an assembly of two components with two hinge-way device on both sides of the mould instead of bolts. The mould is firmly held by two fasteners easily inserted into each hinge. Figures 3.3f, 3.3g and 3.3h are the orthographic projections and isometric view respectively.

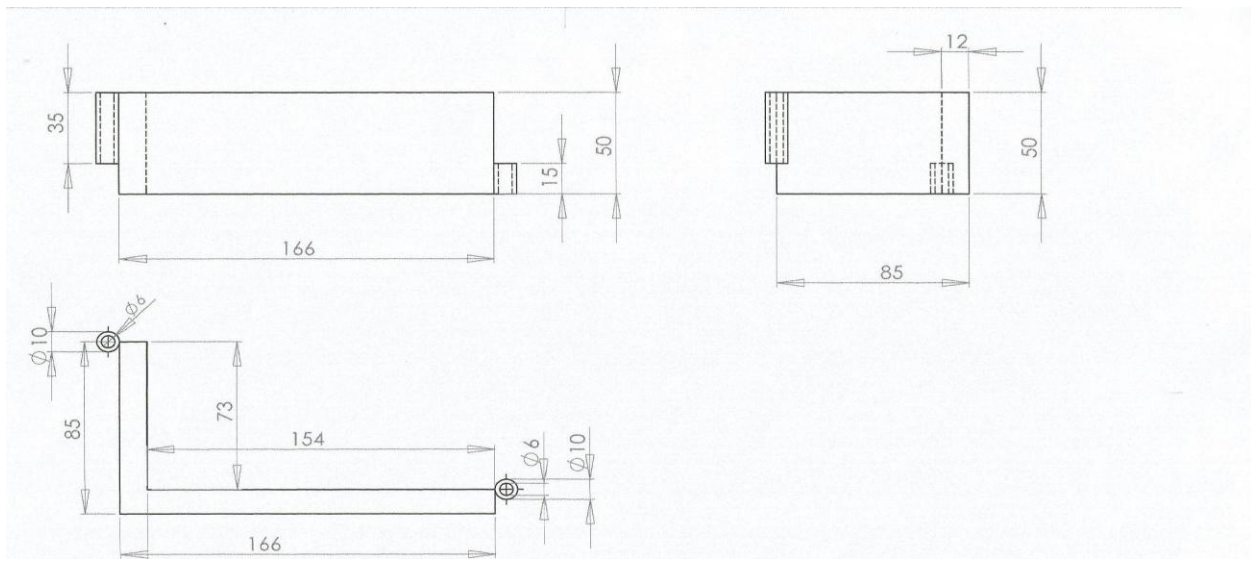


Figure 3.3f: Orthographic Projections of Mould-C Side-1

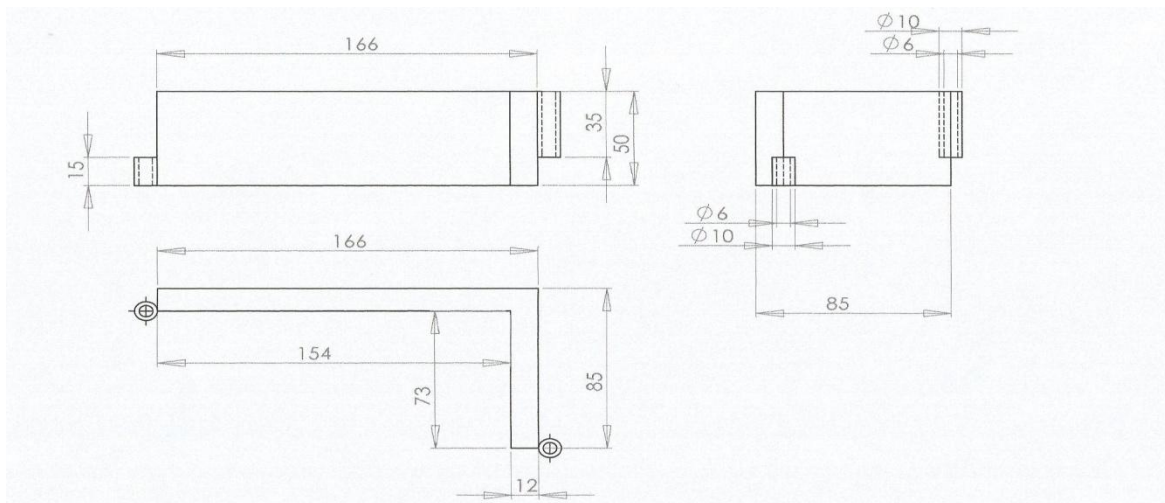


Figure 3.3g: Orthographic Projections of Mould-C Side-2

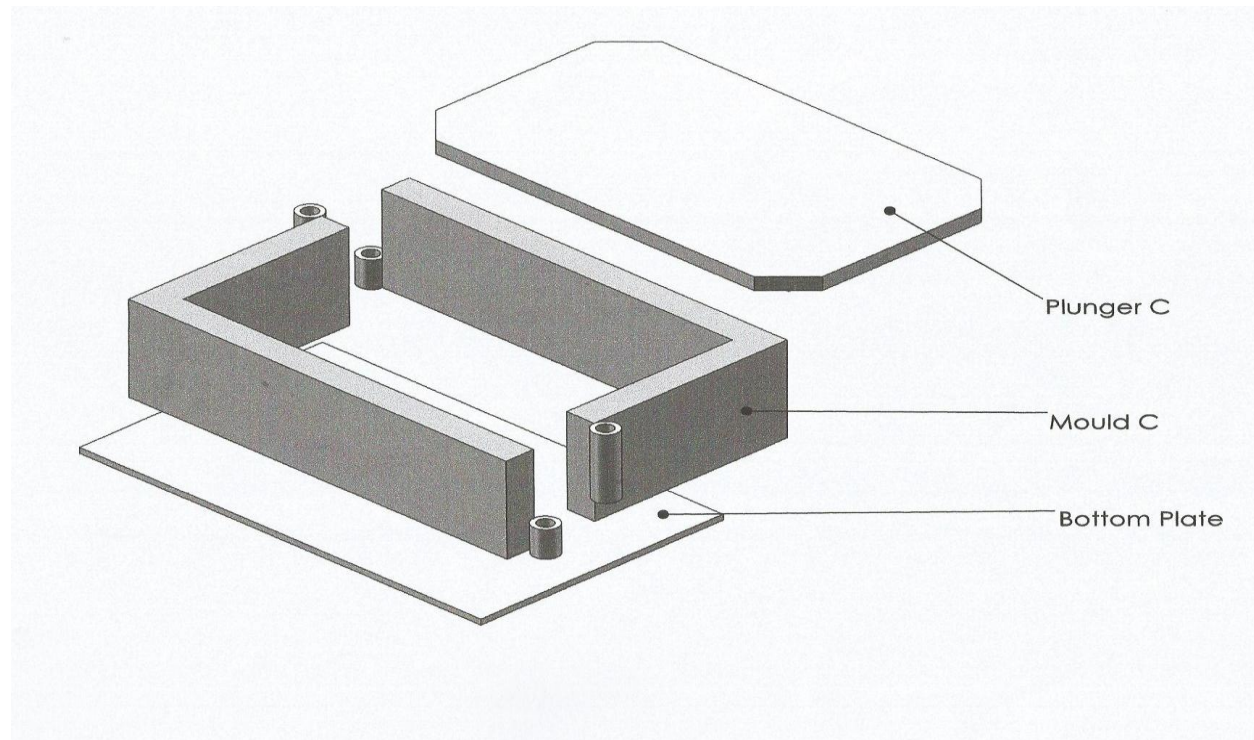


Figure 3.3h: Assembled Mould-C

The mould does not stress the brick as does Mould-B, due to easy insertion and removal of fasteners before and after brick formation. More so, the rate of production was higher (between 8-10minutes per brick) than in Mould-B. Mould-C was preferred and used for this research work.

3.12.4 Press forming of insulating bricks

The Batches for each bricks were introduced into the steel mould from the wrapped polythene bags. Insulating bricks were made by press forming method using the hydraulic press machine at the Laboratory of the Building Department, Ahmadu Bello University, Zaria. The bricks were produced at an average pressure range of between 1.7-3.4kg/cm³.

3.12.5 Casting of the batch in the mould

Batch per mould were already weighed, mixed and wrapped in polythene bags and allowed to age for two weeks. 625g of each batch were casted into the mould carefully and the plunger placed on the top Surface of charged mould before finally placed on the hydraulic press.

3.12.6 Release of the bricks from the mould

As soon as pressing is completed, forming is also completed by subsequent released of the formed bricks. This was achieved by pulling out the two fasteners from the two opposite ends of the mould.

3.12.7 Drying and firing of the bricks

The Bricks were dried in ambient air for fourteen days after which, oven-dried at 110°C for 24 hours at the Building Department Laboratory Ahmadu Bello University, Zaria. They were fired at 1280°C using ceramic test ring, Pyrometric cone equivalent (cone-9) and thermocouple as temperature measuring devices.

3.13 Production of Insulating Refractory Bricks

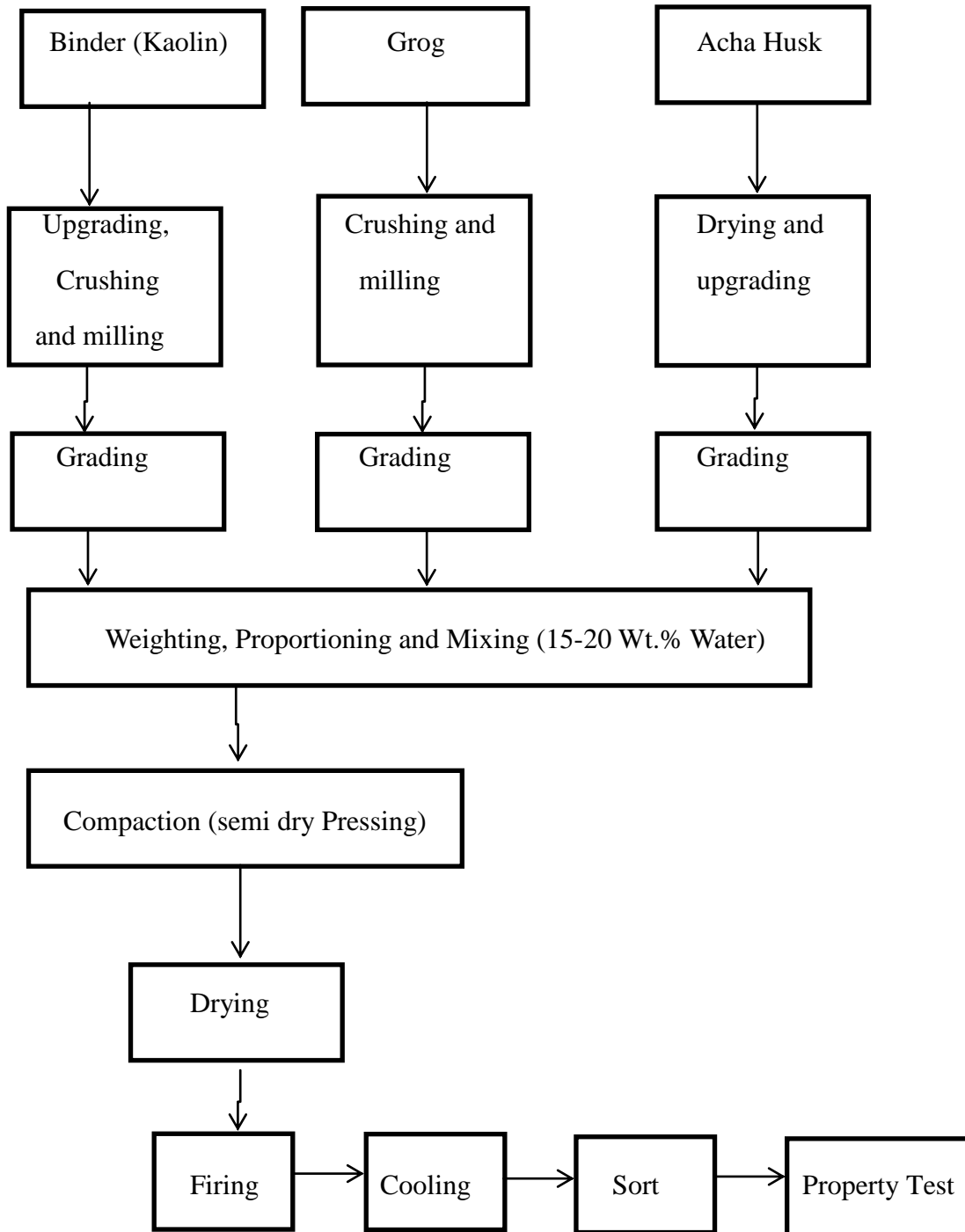


Figure 3.4: Flow Chart for the Production of Insulating Refractory Bricks.

3.14 Determination of Insulating Refractory Bricks Properties

Refractory products are tested of multi-component raw materials, coarse through ultra-fine particle sizing, contaminant possibilities, mixing efficiency, firing time and temperature and many more. Testing is necessary to ensure uniform and consistent production. In addition, tests are needed to compare “equivalent” products from different manufacturers to confirm that products meet or exceed user’s specification to determine the effects of service, and for research and development (R&D), among other purposes. The insulating refractory properties tested for were drying shrinkages, firing shrinkage, bulk density, apparent porosity, water absorption, cold crushing strength, spalling resistance and thermal conductivity.

3.14.1 Shrinkage test

The lengths of the as-cast bricks were measured length-wise, with Venier caliper. The measurement was repeated after drying to determine the drying shrinkage of each brick. For the fired shrinkage of the bricks, before and after firing dimensions were measured as in drying shrinkage. Similar procedure was used to determine the firing shrinkage of the test bricks fired to 1280°C (ASTM C179-14)

$$FS = \frac{DL - FL}{DL} \times 100 \dots\dots\dots(14)$$

Where: DL = Dry length (cm), FL = Fired length (cm)

3.14.2 Apparent porosity and bulk density

The boiling water method of determination of apparent porosity is applicable to burnt bricks only. Test bricks were dried at 110°C and weighed after cooling to room temperature. The test

specimen were placed in distilled water and boiled for two hours, and then allowed to cool to room temperature while still immersed in water. During boiling, the specimens were suspended in the water to avoid contact with the heated bottom or sides of the container (300ml). The specimens were cooled to room temperature and their weights(s) noted. Immediately after obtaining the suspended weight the test specimens were removed from water, blotted lightly with a moistened towel and weighed in air (W) (ASTM C20-00(2015)).

$$\text{Thus, apparent porosity} = \frac{W-D}{W-S} \times 100 \dots\dots\dots(15)$$

Where:

W= weight of soaked specimen suspended in air.

D= Weight of fired specimen

S= Weight of fired specimen suspended in water.

$$\text{Bulk Density} = \frac{D}{W-S} \text{ (g/cm}^3\text{)} \dots\dots\dots(16)$$

3.14.3 Cold crushing strength

25 samples of the same dimensions were placed on a flat bearing block surface of a Compressing Testing Machine, Salm Denison, Model T.I.B./MC in Department of Civil Engineering, Ahmadu Bello University, Zaria was used for the test. A uniform load was then applied through the bearing block in the compressing machine. The loads at which cracks appeared in the samples were recorded and the CCS computed as (ASTM C133-97 2015):

$$\text{CCS} = \frac{\text{Load Applied (Kg)}}{\text{Area of sample (cm}^2\text{)}} \dots\dots\dots (17)$$

3.14.4 Thermal shock resistance

Following the ASTM method and the method proposed by Abdullahi, (2009) was adopted for this test. Ten (10) pieces tested to be of optimum compositions were cut and fired at uniform rate to 1000°C in 3 hours and soaked at that temperature for 30 minutes. The specimens were removed with a pair of light tongs and placed on cold iron plate to cool for 10 minutes. They were replaced in the furnace for a further of 10 minutes. The test pieces were removed and replaced in the furnace in the same sequence in order to ensure the same period of heating and cooling. Toward the end of each cooling period the test pieces were examined for cracks and a slight force applied on a rig. They were then replaced in the furnace while still maintained at the test temperature for another period of 10 minutes. This circle of heating, cooling and testing was repeated for 30 cycles. The number of complete cycles of heating and cooling required to promote fracture in each specimen was noted.

3.14.5 Thermal conductivity

Thermal conductivity test was conducted in the Metallurgy Laboratory, Department of Metallurgical and Materials Engineering, Ahmadu Bello University Zaria. This test was based on IEEE standard 98-2002 with some slight modifications (Okereke, 2003). The quantity of heat which passes through a homogenous wall was determined using the formula:

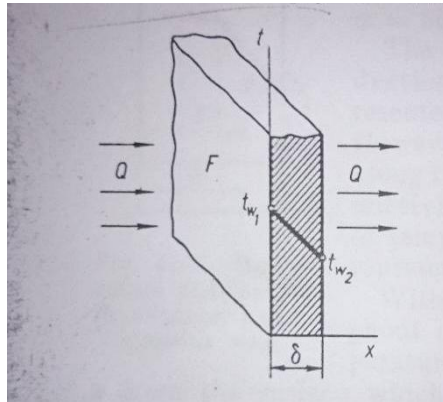


Figure: 3.5: Principle of Thermal Conductivity (Source: Okereke, 2003)

$$Q = qF = \lambda F \frac{tw_1 - tw_2}{\delta} \dots\dots\dots (18)$$

$$\lambda = \frac{Q\delta}{F(tw_1 - tw_2)} \text{ W/m.k} \dots\dots\dots (19)$$

(Shvets, 1980)

Where:

λ = Thermal Conductivity (W/m.°C)

F= Surface area (m²)

δ = Thickness (m)

tw_1 and tw_2 = boundaries surfaces of the wall at temperatures tw_1 and tw_2 .

Q= Coefficient of thermal conductivity (W/m.°C)

Where λ is the quantity of heat in joule or watt passed through from one face of the wall to the other (fig 3.1); Q- thermal coefficient of conductivity in W/m.°C; the cross-sectional area of the

bricks ($F=H \times D$)m²; δ = thickness of the bricks in meter; Z = the duration of the heat transferred in t_1 and t_2 = the temperatures at the two opposite faces in °C. From eqn. (18), it would be seen that if $F= 1\text{m}^2$, $t_1- t_2= 1^\circ\text{C}$, $\delta=1\text{m}$.

Then, $Q= \lambda$ (W/m.°C).....(20)

(Shvets, 1980)

Hence, by definition, the coefficient of thermal conductivity Q is that quantity of heat which passed through a unit cross sectional area of 1m^3 with a unit height and thickness of 1m in 1 hour, when there is a temperature difference of 1°C . The Q values of materials vary, depending on the following factors; the density, porosity, moisture content and of course the temperature. Materials with high density values have low porosity and therefore have higher thermal conductivity coefficient and vice-versa.

CHAPTER FOUR

RESULTS AND DISCUSSION

4.1 The Kaolin Characteristics

4.1.1 Sample description

The Kankara kaolin was sourced during the rainy season. The colour was pinkish with some brown and white stains. As it gets dried the colour changes to white and becomes whiter as it is being fired. Drying and firing of kaolin removes water and collapses the hydrated crystal structures within it. Carbonates and carbonaceous matter were also burnt. This process of firing induces a thermally activated, irreversible phase changes (Edomwonyi-otu *et al*, 2010).

Physically, the kaolin was lumpy (3-7cm) which easily crushed under slight pressure from the fingers giving a smooth powdery form.

4.1.2 Sinterability of kaolin sample

The drying and firing behaviours of the beneficiated Kaolin was determined at 110°C/24hrs and 1200°C at 8hrs respectively.

Table 4.1: Sinterability (Drying and Firing Behaviour) of Kaolin Sample

| Dried (110°C for 24hrs) | | | Fired (1200°C for 8hrs) | | |
|-------------------------|-----------------|------------------------------|-------------------------|-----------------|-----------------------------|
| Colour | Crack Formation | Average Linear Shrinkage (%) | Colour | Crack Formation | Average Linear Shrinkage(%) |
| Pale Pink | Crack formation | 6 | White | No crack | 7.4 |

The common ranges for Kaolin, 7-9% at 1200°C. Kankara kaolin had shrinkages within the range. The thermal stability of the kaolin sample is attributable to the presence of stabilizing materials which promote effective sintering of all clay particles together. Such materials are muscovite and feldspar (Ahmed, 1986).

The colour change noticed after firing Kankara kaolin, suggested the presence of trace coloured impurities especially iron oxides, carbonaceous matter and water of crystallization.

4.1.3 Atterberg plasticity index (API)

Results of the Atterberg plasticity tests conducted on the raw Kaolin sample showed that, moisture tolerance or liquid limit (LL) was 50% while its plastic limit (PL) was 32%, plasticity index therefore (PI) was 18%.The kaolin have good plasticity requirement needed for Iron and steel refractory. Plasticity of Kankara kaolin sample compared with some other Nigerian clay. Refer to Table 4.2 and Figure 4.1.

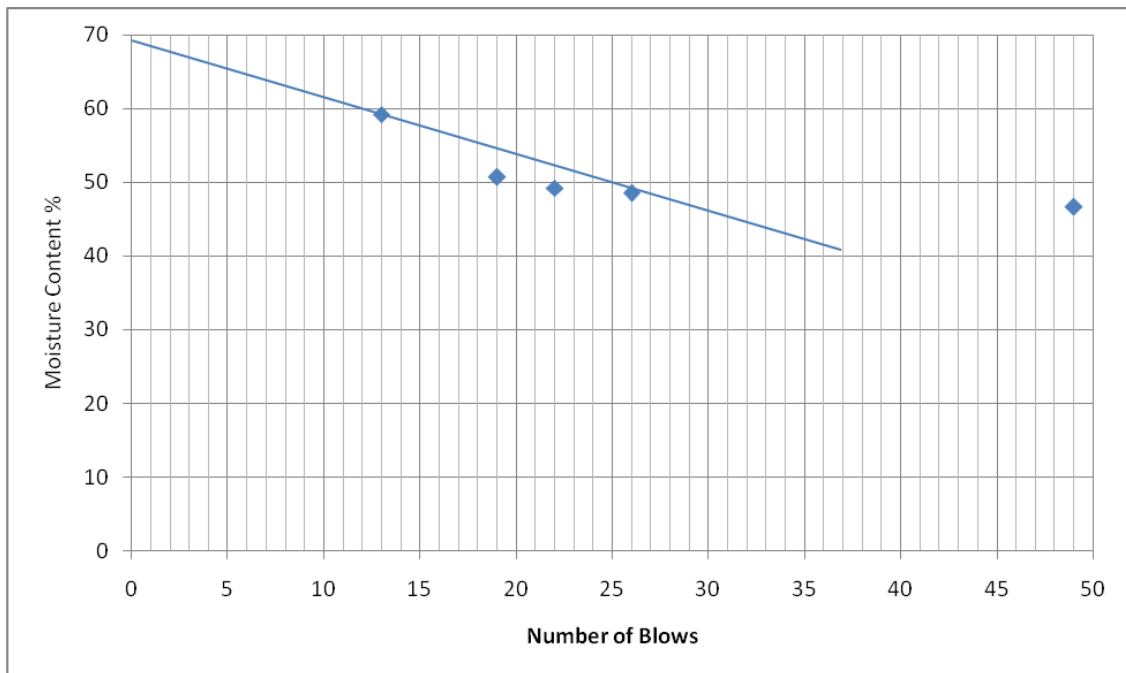


Figure 4.1: Liquid Limit Chart of Kankara Kaolin

Table 4.2: Kankara Kaolin Atterberg Plasticity Index Compared with Some other Nigerian Clays

| Clay | State | Liquid limit (LL) | Plastic limit (PL) | Atterberg Plasticity Index (API) |
|------------|--------------|-------------------|--------------------|----------------------------------|
| Kankara | Beneficiated | 50 | 32 | 18 |
| Enugu* | Beneficiated | 35 | 16 | 19 |
| Onibode** | Raw | 42 | 25 | 17 |
| Majohota** | Raw | 59.2 | 24.6 | 34.6 |
| Ahoko*** | | 35.45 | 21.91 | 13.54 |

Secondary Data Sources: (**Ahmed, 1986,*Sullayman, 2005 and ***Abdullahi, 2009)

4.1.4 Particle size distribution(PSD) of Kankara kaolin

Result of the sieve analysis of Kankara kaolin is presented in Figure 4.1. The sample had more than 71% of its particles finer than 75µm (200 mesh). This result shows that bulk of the kaolin sample (71%) falls within the required particle size for refractory materials.

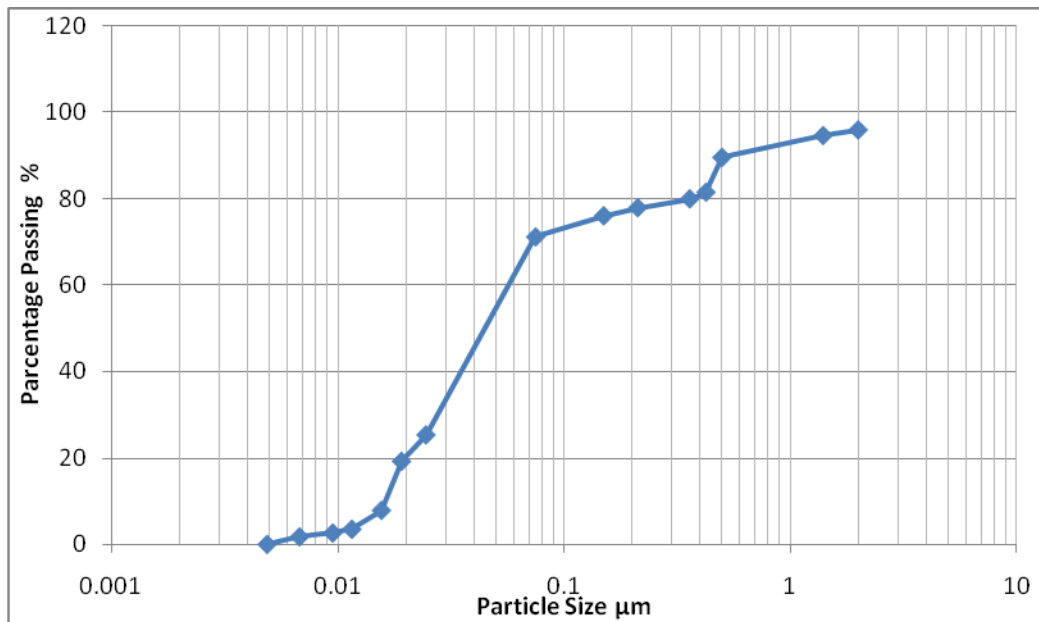


Figure 4.2: Particle Size Distribution Curve from Raw Kankara Kaolin Sample

4.2 The Chemical Composition of the Raw Materials

Results of the chemical analysis of the raw and beneficiated kaolin and AH are presented in Table

4.3

Table 4.3: XRF Results for the Kankara Kaolin Sample

| Oxide (%) | Un-beneficiated Kaolin | Beneficiated Kaolin |
|--------------------------------|------------------------|---------------------|
| SiO ₂ | 52.60 | 54.80 |
| Al ₂ O ₃ | 29.50 | 31.60 |
| Na ₂ O | 0.003 | 0.004 |
| K ₂ O | 0.80 | 0.88 |
| MgO | 0.001 | 0.002 |
| CaO | 0.28 | 0.409 |
| TiO ₂ | 0.13 | 0.27 |
| MnO | 0.02 | 0.02 |
| Fe ₂ O ₃ | 1.58 | 0.87 |
| L.O.I | 14.81 | 11.09 |
| BaO | 0.024 | 0.025 |
| ZnO | 0.029 | 0.010 |

4.2.1 Kankara kaolin:

From the results, principal oxides were detected and in quantities comparable with world class kaolin used for refractories (Ahmed, 1986). Average beneficiated and un-beneficiated kaolin have 53.70wt % SiO₂, 30.55wt % Al₂O₃ and 12.95wt % loss on ignition (LOI). These average values of the kaolin sample placed it within the stipulated requirement for fireclay refractory

materials (Abdullahi, 2009). The average values for 0.34wt % CaO, 0.20wt % TiO₂, and 1.22wt % Fe₂O₃ are within tolerable impurity range for the kaolin sample. Some of these oxides act as fluxing agents, while Fe₂O₃ could cause spalling, swelling and stains on the surfaces if in large quantities.

LOI measures the weight of water of hydration (combined water), carbonates and carbonaceous matter in kaolin sample. This was quite high – 12.95wt% on the average.

The disparity between raw and beneficiated kaolin is due to the fact that, Al₂O₃ and SiO₂ contents increases with fineness of the kaolin. After beneficiation, soluble salts and oxides are eliminated through wet sieving. Kankara kaolin, by its chemical composition compared favorably with good quality local and foreign kaolin, such as Majohota in Plateau, Onibode in Abeokuta (Ogun), Alkaleri in Bauchi and Ahoko in Kogi States all in Nigeria and the famous Cornish China Clay in Britain, Zettlitz kaolin in Czechoslovakia and Sergent China Clay in U.S.A. (Ahmed, 1986).

Table 4.4: Chemical Composition of Studied Kankara Kaolin Sample in Comparison with Previous Analyzed Kankara Kaolin (Major Oxides & LOI)

| Clay | ¹ Kankara | | ² Kankara | | ³ Kankara | | ⁴ Kankara | | ⁵ Kankara | | ⁶ Kankara | |
|--------------------------------|----------------------|-------------|----------------------|-------------|----------------------|-------------|----------------------|-------------|----------------------|-------------|----------------------|-------------|
| | Raw | Beneficiate | Raw | Beneficiate | Raw | Beneficiate | Raw | Beneficiate | Raw | Beneficiate | Raw | Beneficiate |
| SiO ₂ | 51.10 | 47.48 | 44.50 | 41.90 | 45.54 | 45.01 | 45.10 | 47.48 | 52.60 | 54.80 | 52.60 | 54.80 |
| Al ₂ O ₃ | 36.06 | 36.80 | 38.64 | 31.60 | 38.27 | 38.30 | 33.41 | 36.80 | 29.50 | 31.60 | 29.50 | 31.60 |
| Fe ₂ O ₃ | 0.72 | 0.56 | NIL | 14.94 | 0.72 | 0.70 | 0.56 | 0.56 | 1.58 | 0.87 | 1.58 | 0.87 |

| | | | | | | | | | |
|-------|-------|-------|-------|-------|-------|----|----|-------|-------|
| LOI | NA | 16.70 | NA | 14.04 | 14.28 | NA | NA | 14.18 | 11.09 |
| TOTAL | 87.88 | 99.84 | 88.44 | NA | NA | NA | NA | 98.49 | 98.36 |

1-Nurudeen, 2010, 2-Sullayman, 2005, 3-Edomwonyi-otu, 2013, 4-Ahmed, 1986, 5-Edomwonyi-out, 2009, 6-Sample results, 2015.

From Table 4.4, six different kaolin samples sourced from Kankara at different years, yet analysis gave close elemental compositions. The slight variation could be attributed to variations in kaolin quality with depth and locations at which it was mined at a time. More so, Kankara kaolin is of primary origin type, chemical reactions takes place in situ continuously to regenerate kaolin even at mined locations. These could lead to the slight variations noticed.

4.2.2 Acha husks

Five samples sourced from different locations were analyzed. The five samples were designated A, B, C, D and E. The contents of SiO₂ in the AH were 51.99wt%, 50.30wt%, 54.00wt% and 55.38wt% respectively. Al₂O₃ contents were 7.04wt%, 5.72wt%, 7.22wt%, 9.18wt% and 6.83wt% respectively. Other elements detected in significant quantities include, P₂O₅, Na₂O, K₂O, CaO and Fe₂O₃. Some are fluxes while some contaminants. Silica (amorphous) is believed to be highly porous and light weight. The properties of the AH could be influenced by temperature and duration, rate of heating, burning technique, crop variety and fertilizer used. Fourier transformation infra-red (FTIR) spectrometer, showed the presence of significant amount of hydrocarbon, cellulose and lignin content. This acts as activated carbon which is a complex porous structure (Kumar *et al*, 2012).

The high percentage of Fe₂O₃ particularly in samples B, C and D could be due to non-treatment of the husks. They were collected on bare floors which are believed to be contaminated with soil of high iron oxide. Sample A, was upgraded by reducing significantly the dust while the sample

used (E) in this work was collected directly after threshing by spreading wide PVC mats, to avoid contamination with soil

Table 4.5: XRF Results for Five Different Sources of Acha Husk from Kenya

| Oxides (%) | Acha Husk A (Siak) | Acha Husk B (Shano) | Acha Husk C (Jiek) | Acha Husk D (Gyeng) | Acha Husk E (Nganbyiok) |
|--------------------------------|--------------------|---------------------|--------------------|---------------------|-------------------------|
| SiO ₂ | 51.99 | 50.30 | 53.54 | 54.00 | 55.38 |
| Al ₂ O ₃ | 7.04 | 5.72 | 7.22 | 9.18 | 6.83 |
| SO ₃ | 1.93 | 1.20 | NA | 1.10 | NA |
| P ₂ O ₅ | 13.70 | 10.80 | 10.60 | 9.00 | 13.00 |
| Na ₂ O | 2.00 | 1.23 | 1.38 | 1.29 | 1.31 |
| K ₂ O | 12.01 | 10.20 | 11.50 | 10.30 | 12.80 |
| MgO | 0.84 | 0.62 | 0.64 | 0.73 | 0.54 |
| CaO | 4.71 | 3.80 | 3.65 | 3.52 | 5.84 |
| TiO ₂ | 0.719 | 1.88 | 1.97 | 1.72 | 0.55 |
| MnO | 0.075 | 0.42 | 0.48 | 0.46 | 0.656 |
| Fe ₂ O ₃ | 4.90 | 7.70 | 8.76 | 8.30 | 2.51 |
| BaO | NA | 0.032 | 0.34 | 0.30 | 0.14 |
| ZnO | 0.002 | 0.016 | 0.210 | 0.140 | 0.300 |
| CuO | 0.005 | 0.044 | 0.069 | 0.044 | 0.092 |

Table 4.6: Chemical Composition of Kenyi Acha Husks in Comparison with some Reference Samples of Acha Husks, Wheat Husk and Rice Husks.

| Constituents Parameter (%) | Kenyi Acha Husk | ¹ AchaHusk | ² RiceHusk | ³ RiceHusk | ⁴ WheatHusk |
|--------------------------------|-----------------|-----------------------|-----------------------|-----------------------|------------------------|
| SiO ₂ | 55.38 | 57.50 | 89.57 | 68.50 | 59.68 |
| Al ₂ O ₃ | 6.68 | NA | 1.32 | NA | NA |
| P ₂ O ₅ | 13.00 | 11.85 | 1.04 | 12.70 | 5.37 |
| Na ₂ O | 1.31 | NA | 0.77 | NA | NA |

| | | | | | |
|--------------------------------|-------|-------|------|------|-------|
| K ₂ O | 12.80 | 12.13 | 1.65 | 6.40 | 16.33 |
| CaO | 5.84 | 9.16 | 0.77 | 3.33 | 9.54 |
| TiO ₂ | 0.55 | 0.19 | 1.01 | 0.23 | 0.10 |
| Fe ₂ O ₃ | 2.51 | 1.43 | 1.43 | 2.42 | 0.84 |
| MnO | 0.66 | 0.66 | NA | 0.68 | 0.37 |
| MgO | 0.54 | 2.93 | 0.76 | 2.00 | 2.00 |

Secondary Data Source: 1, 3 and 4-Shamle, 2014, 2-Ghani, 2009.

From the Table 4.6, it is glaring that AH used for the study contained major constituents (SiO₂ and Al₂O₃) in comparable quantities needed as pore former in insulating refractories.

Table 4.7: Fourier Transform Infrared (FTIR) Spectrometry Analysis Results of Acha Husk Sample

| No. of Peaks | Functional Groups | Range(Frequency/cm ⁻¹) |
|--------------|-------------------------------------|------------------------------------|
| 1 = 419.53 | Alkali halides(R – I) | (200 – 500) |
| 2= 576.74 | Alkyl halides(R – Br) | (500 – 680) |
| 3 = 1037.74 | Alcohols(– C=C-CH ₂ OH-) | (1035 – 1050) |
| 4 = 1247.02 | Ethers (– Ar- OH) | (1200 – 1275) |
| 5 = 1429.3 | Alkanes (–Alkane – Branched) | (1450 – 1375) |

| | | |
|-------------|--|-----------------------------|
| 6 = 1638.58 | 1 ⁰ & 2 ⁰ Amines & Amides(N-H) | (1640 – 1550) (1640 – 1550) |
| 7 = 2272.22 | Aldehyde | (2800 – 2700) - |
| 8 = 2926.11 | Alkanes (– C – H) | (3000 – 2850) |
| 9 = 3382.29 | Alcohols (– OH) | (3300 – 3400) |

From Table 4.7, the characteristic infrared absorption bands of functional groups and a simplified correlation chart, the following organic matter are present in the AH used as pore former in the production of insulating refractory bricks, cellulose, lignin, etc., and other contents as Carbon, Hydrogen, Oxygen, Iodine, Bromine, Nitrogen. Other conventional husks used as Rice husk, a typical analyzed Rice husk showed the following compositions of organic matter 75 -90% such as cellulose, lignin, C = 35%; H = 4 – 5%; O = 31 – 37%; N = 0.23 -0.32%; S= 0.04 – 0.08%, (Kumar *et al*,2012).Refer to Figure A₂, Appendix B for FTIR Spectral results for AH.

4.3 Properties of Refractory Bricks

4.3.1 Physical appearance

On production of the insulating bricks, they appeared whitish with some black patches of aged AH. The whitish coloration improved as the bricks got dried at 110°C. The bricks were fired at 1280°C soaked for 1¹/₂ hours.

Set-1: Bricks (1-5) came out whitish with little or no flaking.

Set-2: Bricks 6 and 8 came out good while the rest flakes.

Set-3: Bricks 11 and 14 came out good while the rest flakes.

Set-4: Bricks 18 and 20 were good while the rest flakes.

Set-5: Bricks 23 and 25 were good, 22 warped the rest flakes.

Set-6: Bricks 28 and 30 were good, the rest flakes.

Set-7: Bricks 33 warped and 35 was good, the rest flakes.

Sets 8-10: Either warped or flakes badly.

The bricks not classified good, warped, cracked or flaked (i.e. weak and powdery). Evidently, they had not sintered well, while the good ones in the same kiln had sintered and appeared dense and strong. For those that were not good, either that the composition of AH was so much that, the binder could no longer hold the composites together, or the uneven heat distribution within the system affected their sinterability. Another reason could be the high ratio of the coarse grog deterred sinterability. As grog ratio 70:30, 66:34 and 55:45 had 3, 0 and 2 good bricks respectively while 60:40 and 50:50 ratios had 4 and 5 good bricks respectively. This is a pointer that good products could come from ratios 60:40 and 50:50.



Plate III: Physical Appearance

4.3.2 Shrinkage

Drying shrinkage increased with increased AH. This was because bricks with high AH content were expected to absorb more water, which subsequently shrinks more and they released water on drying and much more when fired. This trend was noted in the first few batches (sets) 1-4, thereafter, sets 5 and 6 average drying shrinkage decreased. Grog ratio 60:40 showed an increasing trend of drying shrinkage with increasing AH but interrupted at batch 4. These anomalies could be attributed to production and test methods errors. The maximum drying shrinkage was 1.37% with minimum (zero shrinkage). Averagely, there were low total shrinkage and they could be as a result of high percentage of grog materials which has attained thermal integrity when calcined. The result compares favorably with other insulating materials of 0.5%-

1.5% (ISTM-2014) and 0.059-7.15% depending on the wt% of combustible (Ahmed and Green, 2000).

Grog-wise, the trend in drying shrinkage is 50:50>55:45>60:40>66:34 while firing shrinkage 60:40>50:50>55:45>70:30>66:34. This shows that grog ratios 50:50 and 55:45 showed high shrinkage. The reason is not easily understood.

4.4 Drying Shrinkage, Bulk Density, Apparent Porosity, Water Absorption, Cold Crushing Strength, Thermal Shock Resistance and Thermal Conductivity.

4.4.1 Total drying shrinkage (g/cm³) results

From the results, the shrinkage increases with increase in AH content. The trend was regular in coarse: fine grog ratios, 50:50, 60:40, and intermittently in 70:30, 66:34 and 55:45. Generally, the drying shrinkage was within comparable range as seen in the Indian Standard Testing Methods (ISTM, 2014) which ranged from 0.5% - 1.5%. Even though the ratio of grog fraction and combustible material used for the ISTM materials was not known.

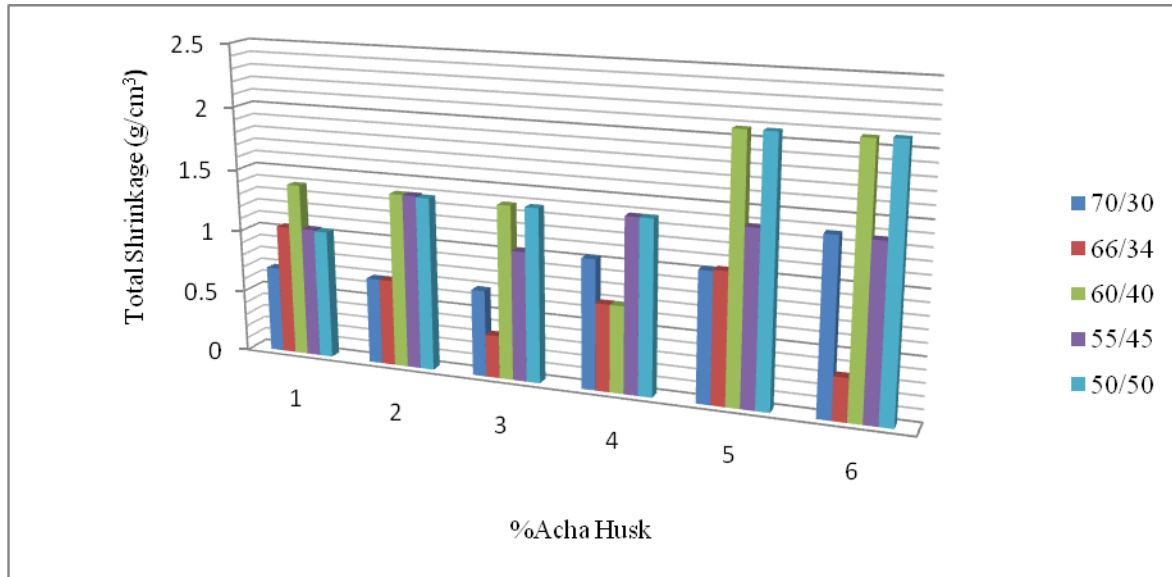


Figure 4.3: Total Drying Shrinkage Results

4.4.2 Apparent porosity (%) results

The expected trend in AH content increased with porosity is a proportional relationship. Higher the husk content the higher the porosity. The AH, when burnt-off creates pores. Coarse: fine grog ratios 60:40 and 50:50 showed increased in porosity with increased AH (1 – 6%). While grog ratios 70:30, 66:34 and 55:45, showed intermittent increased in trend. These values compared favourably with ISTM (2014) values which ranged from 45.0% - 70%. Although the compositions and materials not known. Insulating bricks developed by Ugheoke *et al*, (2009) ranged between 56.40% - 95.93%. The clay was not pre-fired.

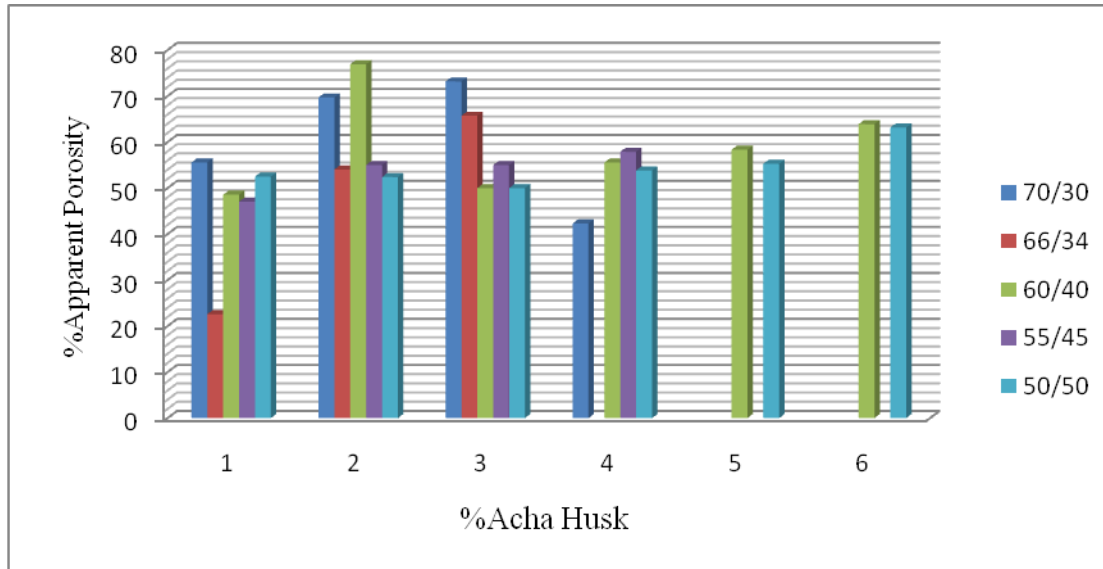


Figure 4.4: Apparent Porosity Results

4.4.3 Bulk density (g/cm^3) result

The expected trend of AH content with bulk density is an inverse relationship. That is, as AH additive increased, bulk density reduced. From the results, coarse/fine grog ratios, 55:45, 60:40 and 50:50 showed reduction in bulk densities with increase in AH additives (1-6%), although some anomaly were noted. This could be due to production or test errors. The result compared favorably with Skamol Moler (2014) insulating bricks whose average ranged bulk density is $0.45\text{g}/\text{cm}^3 - 0.95\text{g}/\text{cm}^3$ and ISTM (2014) ranged between $0.65\text{g}/\text{cm}^3 - 1.6\text{g}/\text{cm}^3$ although the composition were known.

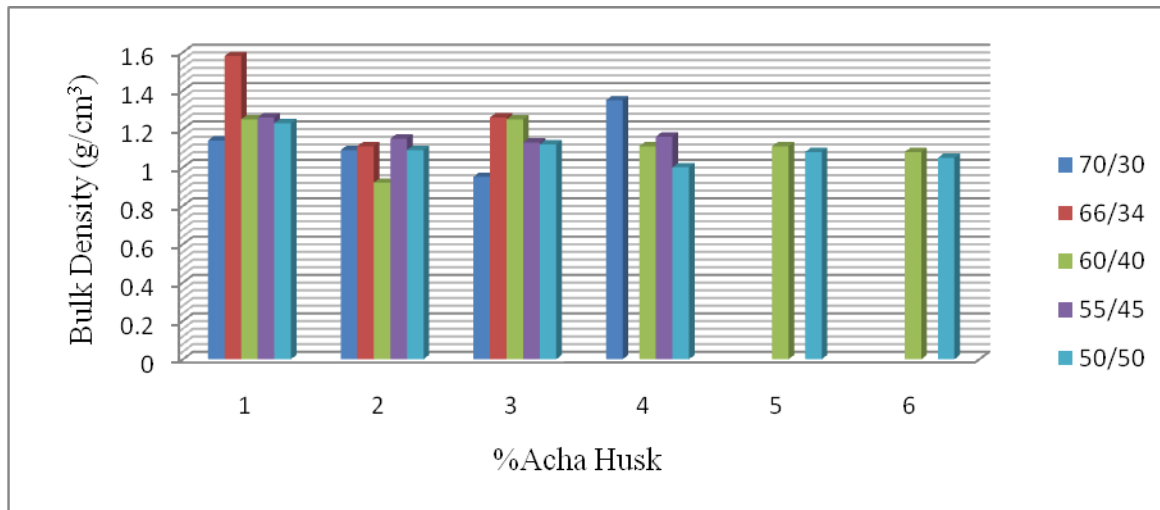


Figure 4.5: Bulk Density Results (g/cm^2)

4.4.4 Water absorption results

The effect of AH additive on water absorption is a proportional one. That is as AH additive increases, the pores produced after firing the bricks, absorbs water. As porosity increases, the absorption capacity of the bricks also increases. From the results, grog ratios, 70:30, 66:34, 60:40, 55:45 and 50:50 showed regularity in the trend except grog ratios, 60:40, AH(2)wt% had a very high value of 83.33%. The reason could be due to production or test error.

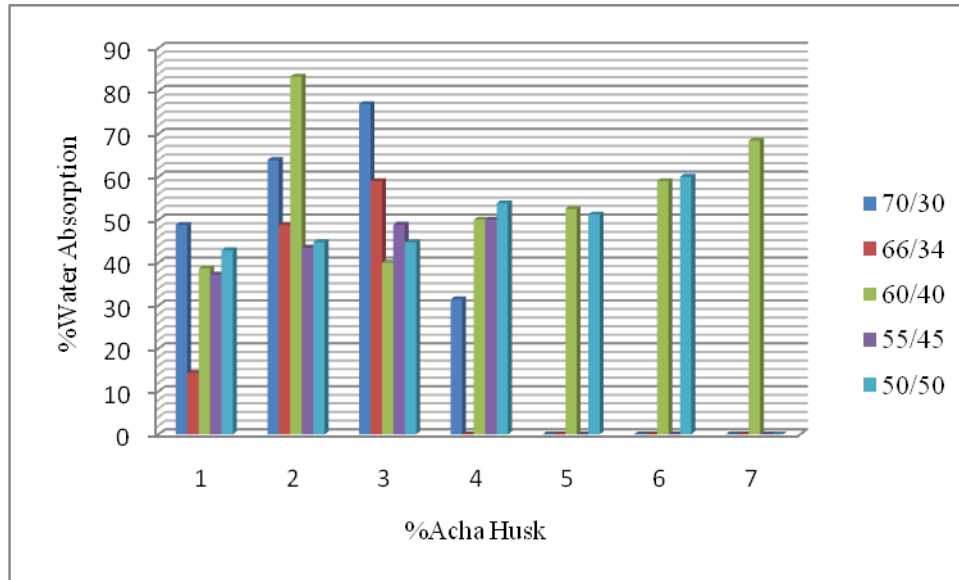


Figure 4.6: Water Absorption Results (%)

4.4.5 Cold crushing strength (MPa) result

From CCS results, each of the set of bricks, two parameters' effects were investigated. That is, the coarse: fine grog ratio and percentage of AH. The higher the coarse grog size, lower the CCS while increased in fine grog size lead to high CCS. From the chart Set-1, CCS increased in the order of grog ratio 70:30 < 66:34 < 60:40 < 55:45 < 50:50. This trend could be attributed to high porosity in coarse particles as a result of reduction in solid volume due to voids created by AH, leading to reduced CCS. While increased in fine grog size leads to reduced porosity and high compaction resulting to high CCS. This trend was same for sets 2-5, however, the CCS reduced from sets 1-5 generally as AH% increased. This could be due to increased in voids as AH was increased. The solid volume of the bricks is reduced, while porosity increased leading to reduced CCS of the bricks. The CCS results from the chart compared with Skamol Moler (2014) insulating bricks whose CCS ranged between 1.5-18 MPa. For ISTM (2014), it ranged between 17-60Kg/cm³.

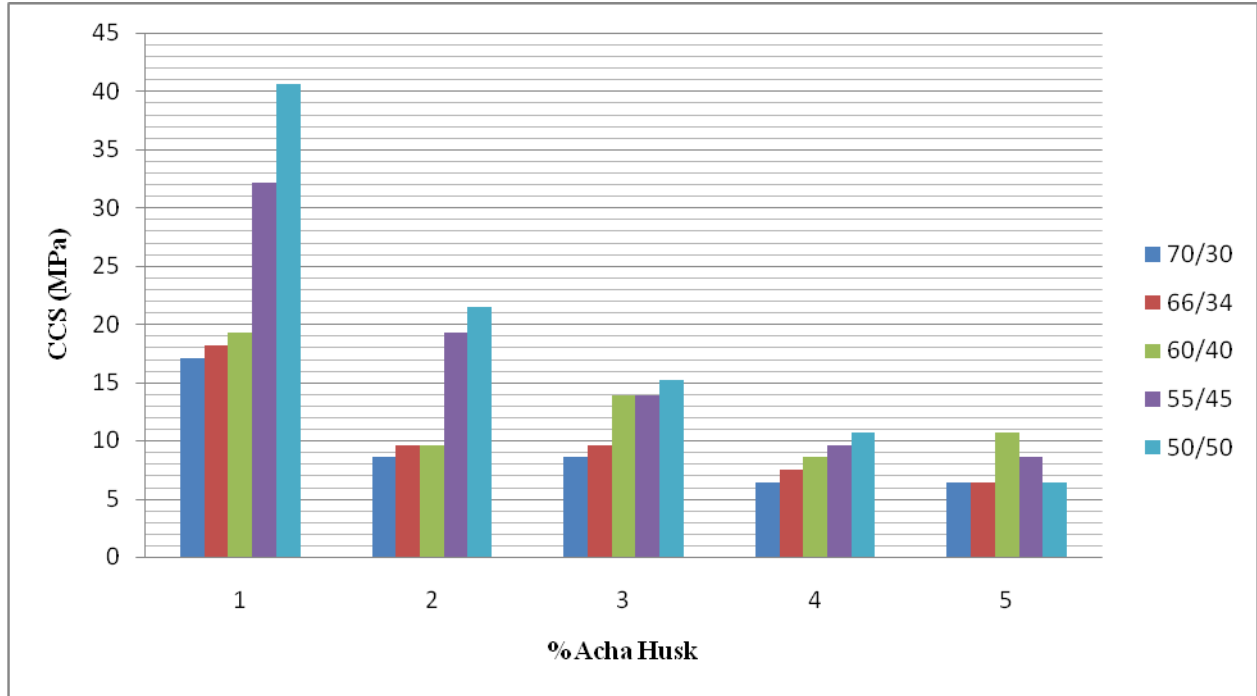


Figure 4.7: Cold Crushing Strength Results (MPa)

4.4.6 Thermal conductivity (W/m.°C) results at 500°C mean temperature

From the results obtained, thermal conductivity decreased with increase in AH additive from wt% of AH A-F (1-7%). However, some anomaly were observed in grog ratio, 60:40 4wt% of AH, had a value of 0.129 higher than 2wt% AH 0.005W/m.k. The results compared with insulating bricks using saw dust developed by Aramide, (2012), 0.005 – 0.134W/m.k, Manukaji, (2013), 0.134W/m.k, with 40% Sawdust and Callister, (1994), also 0.05 – 0.24W/m.k, ISTM 0.23 – 0.70W/m.k.

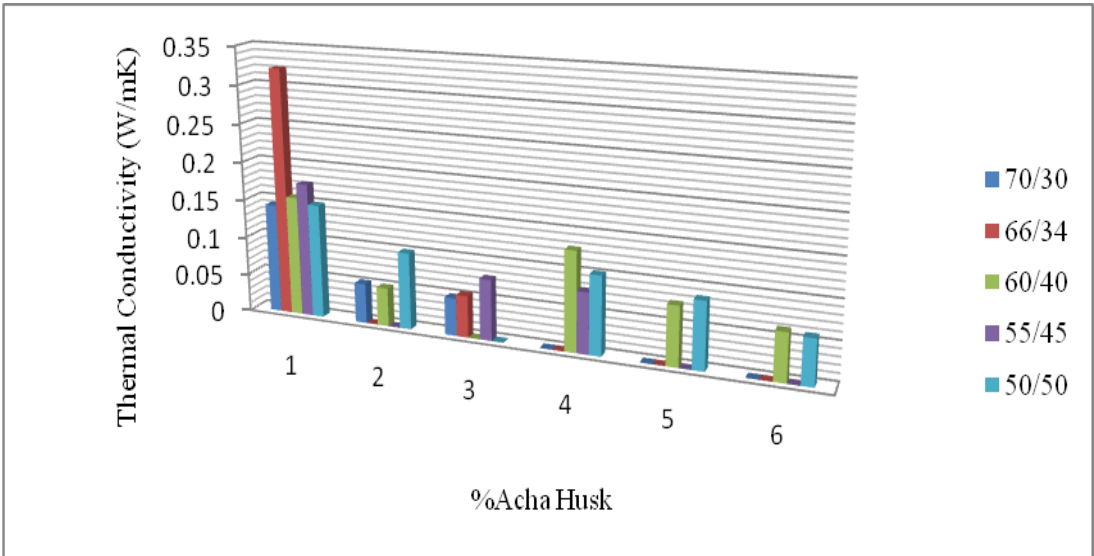


Figure 4.8: Thermal Conductivity Results

4.4.7 Thermal shock resistance results

From the results, on Table 4.8, the ten samples subjected to thirty cycles of heating and cooling showed no crack or warpage. Having passed the classification by Chesters (1973) that is refractories which go up to 30+ cycles are rated excellent thermal shock resistance. The excellent thermal shock resistance exhibited by Kankara kaolin sample could be attributed to its good insulating property due to uniformly distributed micro porosity. This made the bricks to absorb and release heat at lower rate, consequently, shielding it from the relative effect of high thermal shock experienced in dense bricks, by implication, the insulating bricks could have high life span.

Table 4.8: Thermal Shock Resistance

| Sample Batch | Fired Temperature °C | No. of Thermal Cycles | Thermal Shock Resistance Category | Bulk Density (g/cm³) |
|---------------------|-----------------------------|------------------------------|--|--|
| 3 | 1000°C (3hrs) | 30 | Very Good | 1.25 |
| 4 | 1000°C (3hrs) | 30 | Very Good | 1.26 |
| 5 | 1000°C (3hrs) | 30 | Very Good | 1.23 |
| 7 | 1000°C (3hrs) | 30 | Very Good | 1.11 |
| 8 | 1000°C (3hrs) | 30 | Very Good | 0.92 |
| 13 | 1000°C (3hrs) | 30 | Very Good | 1.25 |
| 15 | 1000°C (3hrs) | 30 | Very Good | 1.12 |
| 18 | 1000°C (3hrs) | 30 | Very Good | 1.11 |
| 23 | 1000°C (3hrs) | 30 | Very Good | 1.11 |
| 25 | 1000°C (3hrs) | 30 | Very Good | 1.08 |

4.5 Findings

The following are findings derived from the research study:

- i. API is 18%(intermediate plasticity range).
- ii. PSD showed that, 71% Kankara kaolin particle size passed through sieve 75µm.
- iii. XRF revealed that KK is suitable for high duty refractory bricks having 31.60%Al₂O₃.
- iv. AH(E) has added advantage of having 6.68% Al₂O₃. It enhances refractoriness of the bricks.

- v. FTIR showed AH to contain high percentage of organic matter which burnt off to create porosity in the bricks.
- vi. AH samples vary slightly in chemical contents due to the chemistry of the soil, and where or how the husk was procured.
- vii. As AH contents increased in the bricks, total shrinkage, porosity, water absorption and thermal shock resistance increased, but reduced with increased in fine grog ratio.
- viii. As AH contents increased in the bricks, bulk density, thermal conductivity and CCS of the bricks reduced. While these properties increased with increased in fine grog ratio.
- ix. Bricks codes 1, 2, 3, 4, 5, 6, 8, 10, 11, 12, 14, 18, 19 and 20 neither flaked nor warped relative to other bricks.
- x. Sample Numbers 6, 11, 12 and 14 gave optimum qualities.

CHAPTER FIVE

SUMMARY, CONCLUSIONS AND RECOMMENDATIONS

5.1 Summary

The study found out among other things that;

1. Kankara kaolin had a relatively low coarse and fine impurities with more than 71wt% of its constituent particles finer than 75 μ m (200mesh).
2. Kankara kaolin (KK) had an Atterberg Plasticity Index of 18wt%. The plasticity Index value by Casagrande Plasticity Chart placed the KK sample in the intermediate plasticity range.
3. The average linear drying and firing shrinkage of the beneficiated KK sample at 1280 $^{\circ}$ C was 6% and 7.4%. The samples burnt white with good thermal stability, due to their solid nature and were without cracks.
4. The chemical composition of KK revealed that they are suitable for high duty refractory bricks. This is because, the beneficiated sample showed the main elemental oxides to have 31.60wt% Al₂O₃, 55.80wt% SiO₂, which enhances the refractoriness of the material. Fe₂O₃ content was drastically reduced through beneficiation from 1.58wt% to 0.87wt%. Its presence devastates the quality and color of refractories.(spalling and stains on product)
5. The chemical contents of the AH(E) used compares favorably with conventional agro waste used,55.38wt% SiO₂, 6.84wt% Al₂O₃ and 5.84wt% CaO enhances the refractoriness of the bricks.

In most common agro wastes used as combustible materials for example, rice husk and wheat husk, they either do not contain or contain in small quantities Al₂O₃. Hence AH have an added advantage of having 6.68% of Al₂O₃.

FTIR result also showed AH to contain high percentage of organic matter which also is believed to be burnt off during firing of the brick at high temperature, thereby creating intended porosity.

From analysis, it was also discovered that, AH vary slightly in chemical contents due to the chemistry of the soil, climate, and where or how the husk was collected.

Sample E used for this study, was collected directly from the threshed mortar without being contaminated with soil, hence its Fe_2O_3 content was 2.51wt%, the treated husk (A) had 4.90wt%, and the untreated has an average of 8.25wt% Fe_2O_3 .

6. The rough kaolin bricks were fired at 1280°C which was sufficient for proper calcination for grog formulation. Andreason's Parameter was used to formulate the batches. Six different grain sizes of the crushed grog were used, with maximum 2.36mm and minimum 0.075mm. From these, five different grog ratios (coarse: fine) were also formulated into 70:30, 66:34, 60:40, 55:45 and 50:50. Ten sets of these grog ratios were used to formulate ten batches containing varied wt% of AH ranging between 1wt% to 10wt%. Hence, two parameters were studied. Each set of grog ratio had same quantities of AH up to ten. Variation within a set was due to grog ratios variations (intra-variation) and variation between batches was due to AH wt% variation (inter-variation).
7. Bond materials used were beneficiated Kankara kaolin of 10wt% of grog aggregates in each sample and Na_2SiO_3 , 5wt% of grog aggregates was also introduced in each sample to enhance bonding of the insulating bricks. This is because; the recommended 10wt% of kaolin could not give good binding of fire bricks.

Moisture introduced to enhance compaction and bonding range between 10wt% - 15wt% (17.50wt% range).

This is against 10wt% recommended, but for the presence of varied wt% of AH as blend, more moisture content was needed as AH content increased, owing to the high absorption of moisture by AH.

8. Three mild steel moulds (A, B and C) were made.

Mould-A; it was designed and fabricated. The mould is an assembly of a single component with a plunger. It has a problem of non-released of the brick even when lubricating oil was applied on the internal surface of the mould. This led to the design and fabrication of mould-B

Mould-C; this is an assembly of two components joined by four bolts (two bolts on each side) to enable coupling and decoupling during charging batch and discharging brick. The problems associated with it was that, it took longer time to screw and unscrew the bolts, each time batch charging and brick discharge was done. It took about 25minutes to 30minutes to produce one brick. More so, the formed bricks are being stressed and deformed in the process of bolting and unbolting. Consequently, another mould-C was designed and fabricated.

Mould-C; this is a two components assembly with two hinge-way device on both sides of the mould. The mould is firmly held by two fasteners easily inserted or removed into and out of the hinges during production. Easy operations of the fasteners do not stress nor deform the brick. More so, the rate of production was higher (8minutes – 10minutes per brick).

9. The produced insulating bricks appeared milky in colour with some black patches of AH, while some had dominantly black patches of AH with few milky colour. The higher the wt% of AH, greater the black patches and vice versa. As they were dried, they turned whiter, green strength of bricks increased and when fired at 1280°C, they also appeared whiter as the AH had been burnt-off thereby creating pores.

Bricks codes 1, 2, 3, 4, 5, 6, 8, 10, 11, 12, 14, 18, 19 and 20 neither flaked nor crumbled relative to other bricks. The rest either flaked or crumbled. The flaking and crumbling was attributed to inadequate firing temperature of between 1350°C – 1400°C. Poor heat distribution within the fired kiln and high wt% of AH-additives, with less binding blend.

10. The average drying shrinkage (DS) was expected to increase with increased in AH content since higher AH contents were expected to absorb more water which will subsequently be given off during ambient and oven (110°C) drying. However, the expected trend was not observed, except for grog ratio 60:40 which showed increase drying shrinkage interrupted at wt% AH-4. Grog ratio 55:45 showed intermittent increased in drying shrinkage from wt% AH 1-6. This non-linear changed could be due to inadequate drying and non-uniform drying of the green body. The maximum DS was 1.37% and some without observed shrinkage. While average firing shrinkage in grog ratios 70:30 and 50:50 showed an increasing trend of increasing trend with increased AH. Other ratios do not showed such trend. The total average shrinkage compared with (ISTM, 2014). The low total shrinkage value could also be attributed to the high percentage of grog materials which has attained high thermal integrity when calcined.
11. The results show a linear relationship between apparent porosity and the AH additive contents. As the AH contents is increased, the porosity is also increased, that is so because as

the AH contents is increased, the number of pores increased on firing and consequently, the higher the porosity, since this is directly dependent on number of pores present. The trend was pronounced in grog ratios 70:30; interrupted at batch (4) 42.31%, 66:34 show linear changed, 60:40 also showed a linear changed of increasing porosity with increased in wt% of AH except batch (2) which gave the highest porosity value of (76.92%). The effect of variation of grog ratios on apparent porosity was also observed. Compositions with Coarse: fine ratio of 70:30 had a higher average than 66:34, 60:40, 55:45 and 50:50. This could be attributed to the closed dense packing of subsequent ratios as the ratio of fine particles increased. Interruptions of the observed trend could be due to production errors or tests methods. Maximum and minimum average values of 76.92%-22.58% compared with 20%-80%. Aramide (2014). These are reasonable values for insulating bricks.

12. There is also a linear relationship between the Bulk density and AH in the blend. Bulk density decreased with increased in the combustible AH contents as more pores were produced on firing. Consequently, less solid volume and reduced density. This trend was pronounced in grog ratio 70:30, 66:34, 60:40 and 50:50 though some intermittent interruptions were noticed. The range 0.92g/cm^3 - 1.58g/cm^3 compared with standards and $0.8\text{-}0.9\text{g/cm}^3$ and $1.0\text{-}1.3\text{g/cm}^3$ (Ukrainian Scientific Research Institute of Refractories, 2014).
13. CCS increased as coarse grog size is reduced and reduced as AH contents increased.
14. Thermal shock resistance results showed that, the insulating bricks from Kankara kaolin went through 30 cycles at 1000°C . This implied that, the insulating effect confers low heat conductivity, low heat absorption and released and lower shock.

15. The linear relationship from the results was observed. As the amount of AH contents increased, more pores are produced when fired at 1280°C as the AH burnt off. Air which fills the pores of the refractory material acts as an insulator and hence heat conductivity is decreased. There was correlation between apparent porosity and thermal conductivity results in all grog ratios except some anomalies noticed in ratios 60:40, 55:45 and 50:50 batch-4. This could be due to either production or test errors. The minimum and maximum range obtained 0.049 W/m.k- 0.322 W/m.k compares with 0.02 W/m.k (Callister, 1994), 0.05- 0.24 W/m.k at (Aramide, 2012), 0.005- 0.134 W/m.k (Ugheo *et al*, 2014).
16. From these results, some necessary amount of insulating characteristics are developed by adjusting the grog ratios, number of solid particles and uniform pore spaces introduced by AH. This test method results are primary standard methods which are suitable for use in quality control, research development, establishing criteria for selection and use of refractory products in a variety of industrial applications.
17. From the results all the samples are insulating bricks that can withstand temperature up to 1000°C. Sample No.11 is the most porous, lowest bulk density and lowest thermal conductivity. Sample No.6 next most porous, lower density and lower conductivity. Sample No.12 is next with high porosity, relatively high density but low conductivity. They have optimum compositions for insulating bricks that can be used between 1000°C – 1200°C as backup in furnace linings, fore-hearts and so forth and the construction of kilns.

5.2 Conclusions

From the above analysis and discussions, the following conclusions were drawn:

1. The project was designed to assess the feasibility of producing insulating refractory bricks from Kankara kaolin and Acha (*Digitaria Exilis*) husks. In order to achieve this aim, objectives were set and attained.
2. The raw materials were sourced, beneficiated and characterized. The study established the baseline data for the chemical composition of AH, raw and beneficiated Kaolin along with insulating refractory brick's figures of merit for desired products.
3. The optimum mix of the raw materials was established as six percent Acha husks with coarse: fine grog ratios of 66:34, 55:45 and 50:50 of kaolin. The refractory brick's figures of merit such as Atterberg plasticity index of 18%, bulk density of 0.9 to 1.58g/cm³, ISTM of 1.0 to 2.1%, Thermal shock resistance and thermal shock conductivity of 0.005 to 0.32W/m.k were comparable with those obtained for this type of product worldwide.
4. Desirable temperature (1350°C – 1400°C) to fire the bricks could not be realized.
5. The study has provided an alternative utilization of this waste for insulating refractory bricks production.
6. The product can be used as backup in furnaces, in cement, metallurgical, glass, petroleum industries and kilns for ceramics industries at temperature 1000°C – 1200°C.

5.3 Recommendations

The following recommendations are suggested based on the result of this research.

1. There is the need to further investigate the potential properties of AH as a pore-former for insulating firebricks, with emphasis to its thermal integrity. The recommended

temperature range to study is 1350°C or 1450°C for improved chemical, physical, mechanical and thermal properties.

2. In most cases, it was not possible to establish consistent and conspicuous trends in most properties investigated. This could be attributed to poor batch mixing or uneven temperature distribution within the furnace system or property test error. It is therefore suggested that further investigation be conducted, particularly on grog ratio 60:40 and 50:50.
3. There are different Acha varieties whose husk could be explored as pore-former for insulating brick production.
4. The products could be used as back-up or bricks for kilns, furnace linings and so forth, for low temperature operations of between 1000°C-1200°C. Hence the need for such high temperature operating industries to establish refractory bricks manufacturing firms to exploit available local raw materials.
5. A Fast Test Firing Kiln that can go up to 1400°C – 1500°C is required for high temperature research work.

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APPENDIX A:

Batch Calculation Using Andreason's Parameter Equation:

I. The Average Sizes of Fractions (d) Used were Calculated as Follows:

$$\text{For } -2.36 + 1.18\text{mm, } d = \frac{2.36+1.18}{2} = 1.77\text{mm}$$

$$P1 = \left(\frac{1.77}{2.36}\right)^{0.7} \times 100 = 81.76$$

$$\text{For } -1.18 + 0.600\text{mm, } d = \frac{1.18+0.600}{2} = 0.89\text{mm}$$

$$P2 = \left(\frac{0.89}{2.36}\right)^{0.7} \times 100 = 50.53$$

$$\text{For } -0.600 + 0.300\text{mm, } d = \frac{0.600+0.300}{2} = 0.45\text{mm}$$

$$P3 = \left(\frac{0.45}{2.36}\right)^{0.7} \times 100 = 31.35$$

$$\text{For } -0.300 + 0.150\text{mm, } d = \frac{0.300+0.150}{2} = 0.225\text{mm}$$

$$P4 = \left(\frac{0.225}{2.36}\right)^{0.7} \times 100 = 19.30$$

$$\text{For } -0.150 + 0.075\text{mm, } d = \frac{0.150+0.075}{2} = 0.113\text{mm}$$

$$P5 = \left(\frac{0.113}{2.36}\right)^{0.7} \times 100 = 11.92$$

$$\text{For } -0.075, d = \frac{0.075}{2} = 0.0375$$

$$P6 = \left(\frac{0.0375}{2.36}\right)^{0.7} \times 100 = 5.51$$

II. The Percentage of Grog Fraction Adopted for the Batch Using the Above Values as Reference Point was Evolves as Follows:

$$P1 = \frac{81.76}{200.37} \times \frac{100}{1} = 40.80\% \text{ Coarse particle size fractions}$$

$$P2 = \frac{50.53}{200.37} \times \frac{100}{1} = 25.22\% \text{ Coarse particle size fractions}$$

$$P3 = \frac{31.35}{200.37} \times \frac{100}{1} = 15.65\% \text{ medium particle size fractions}$$

$$P4 = \frac{19.30}{200.37} \times \frac{100}{1} = 9.63\% \text{ Medium particle size fractions}$$

$$P5 = \frac{11.92}{200.37} \times \frac{100}{1} = 5.95\% \text{ Fine particle size fractions}$$

$$P6 = \frac{5.51}{200.37} \times \frac{100}{1} = 2.75\% \text{ Fine particle size fractions}$$

III. Total Amount of Grog Prepared = 56.06Kg. The Specific Grog Fraction Obtained Using the Percentage Value from Appendix A.II for the Calculation thus;

$$P1 = 40.80\% \text{ of } 56.06$$

$$= \frac{40.80}{100} \times 56.06$$

$$= 22.87\text{Kg.}$$

$$P2 = 25.22\% \text{ of } 56.06$$

$$= \frac{25.22}{100} \times 56.06$$

$$= 14.14\text{Kg.}$$

$$P3 = 15.65 \text{ of } 56.06$$

$$= \frac{15.65}{100} \times 56.06$$

$$= 8.77\text{Kg.}$$

$$P4 = 9.63 \text{ of } 56.06$$

$$= \frac{9.63}{100} \times 56.06$$

$$= 5.40\text{kg.}$$

$$P5 = 5.95 \text{ of } 56.06$$

$$= \frac{5.95}{100} \times 56.06$$

$$= 3.34\text{Kg.}$$

$$P6 = 2.75 \text{ of } 56.06$$

$$= \frac{2.75}{100} \times 56.06$$

$$= 1.54\text{Kg.}$$

Mixing the particle fraction to coarse and fine

$$P1 + P2 = \text{Coarse}$$

$$P3 + P4 + P5 + P6 = \text{Fine}$$

IV. Weight of Grog Fraction Required for the Laboratory Size Insulating Brick.

The mould dimensions measures 145mm × 75mm × 50mm

Thus, the volume of the brick = 14.5cm × 7.5cm × 5cm

$$= 543.75\text{cm}^3$$

Density for standard insulating Fireclay brick = 1.15g/cm³

$$\text{Density} = \frac{\text{mass (g)}}{\text{Volume (cm}^3\text{)}};$$

$$\therefore \text{Mass} = \text{Density} \times \text{Volume}$$

$$\text{Mass} = ?,$$

$$\text{Density} = 1.15\text{g/cm}^3,$$

$$\text{Volume} = 543.75\text{cm}^3$$

$$\text{Mass} = 1.15 \times 543.75 = 625.31\text{g}$$

$$\therefore 1000\text{g} = 1\text{kg}$$

$$\therefore 625.31 = \text{xKg}$$

$$\text{xKg} = \frac{625.31}{1000} = 0.62531\text{kg}$$

If one (1) laboratory size brick weight 0.62531kg.

$$\therefore 50 \text{ laboratory size bricks will weigh } 50 \times 0.62531\text{kg}$$

$$= 31.37\text{kg.}$$

V. Grog fraction weight (g) required per brick;

$$P1 = \frac{15.9}{100} \times 625.31$$

$$= 99.424$$

$$P2 = \frac{40.92}{100} \times 625.31$$

$$= 255.876$$

$$P3 = \frac{22.19}{100} \times 625.31$$

$$= 138.756$$

$$P4 = \frac{10.06}{100} \times 625.31$$

$$= 62.906$$

$$P5 = \frac{6.85}{100} \times 625.31$$

$$= 42.834$$

$$P6 = \frac{4.03}{100} \times 625.31$$

$$= 25.199$$

VI. Batch Composition Calculation for Each of the Five Coarse: Fine Ratios.

i. 70:30

Weigh % of coarse grog fraction

$$\frac{70}{100} \times 625.31$$

$$= 437.717$$

Weigh % fine grog fraction

$$\frac{30}{100} \times 625.31$$

$$= 187.59$$

ii. 66:34

Weigh % of coarse grog fraction

$$\frac{66}{100} \times 625.31$$

$$= 412.705.$$

Weigh % of fine grog fraction

$$\frac{34}{100} \times 625.31$$

$$= 212.604.$$

iii. 60:40

Weigh % of coarse grog fraction

$$\frac{60}{100} \times 625.31$$

$$= 375.186.$$

Weigh % of Fine grog fraction

$$\frac{40}{100} \times 625.31$$

$$= 250.124$$

iv. 55:45

Weigh % of coarse grog fraction

$$\frac{55}{100} \times 625.31$$

$$= 343.921$$

Weigh % of fine grog fraction

$$\frac{45}{100} \times 625.31$$

$$= 281.389.$$

v. 50:50

Weigh % of coarse grog fraction

$$\frac{50}{100} \times 625.31$$

$$= 312.655$$

Weigh % of fine grog fraction

$$\frac{50}{100} \times 625.31$$

$$= 312.655$$

VII. a. Determination of Density for Water Glass (Na_2SiO_3)

$$W_2 = \text{Bottle} + \text{Sample} = 74.1390\text{g}$$

$$W_1 = \text{Empty Bottle} = 21.3183\text{g}$$

$$\text{Weight of sample} = 52.8207\text{g}$$

$$\text{Density} = \frac{\text{mass}}{\text{volume}}$$

$$= \frac{52.8207}{50}$$

$$= 1.0564\text{g/cm}$$

VII b. Calculation for 5wt% Na_2SiO_3

$$\text{Mass of brick bodies} = 625.31\text{g}$$

$$10\% \text{ of the mass} = 62.53\text{g}$$

$$5\% \text{ of the mass} = 31.2655\text{g}$$

$$\text{Density} = 1.0564\text{g/cm}^3$$

$$\text{Mass} = 31.2655\text{g}$$

$$\text{Vol.} = ?$$

$$\text{Vol.} \times \text{Density} = \text{Mass}$$

$$\text{Vol.} = \frac{\text{Mass}}{\text{Density}}$$

$$= \frac{31.2655}{1.0564}$$

$$= 29.596\text{cm}^3 \text{ per batch.}$$

VIII. Determination and Adoption of Q Value (Coefficient of Thermal Conductivity of the Insulating Bricks).

Since the value was found in literature, the specific density and coefficient of thermal conductivity for other materials were given. So the specific density of the insulating bricks was determined and compared with the given specific density for correlation value.

Procedure:

1. Portions of 15 sampled insulating bricks were taken, crushed and thoroughly mixed and 10gm weighed for each of the 4-bottles.
2. 4-graduated bottles were washed and the following weighing were made;
 - $W_1 = \text{wt of bottle}$
 - $W_2 = \text{wt of bottle} + 10\text{gm of sample}$
 - $W_3 = \text{wt of bottle} + \text{sample} + \text{H}_2\text{O}$
 - $W_4 = \text{wt of bottle} + \text{water (full)}$

Table A8: Average Specific Gravity of Kankara Kaolin

| No of Bottle | W ₁ (g) | W ₂ (g) | W ₃ (g) | W ₄ (g) | W ₄ -W ₁ (g) | W ₃ -W ₂ (g) | W ₂ -W ₁ (g) | (W ₄ -W ₁) - (W ₃ -W ₂)=W (g) | Sg= $\frac{W_2-W_1}{W}$ |
|--------------|--------------------|--------------------|--------------------|--------------------|------------------------------------|------------------------------------|------------------------------------|---|-------------------------|
| 1 | 44.60 | 45.50 | 100.30 | 94.40 | 49.80 | 45.80 | 9.90 | 4.00 | 2.475 |
| 2 | 37.30 | 47.50 | 93.10 | 87.10 | 49.80 | 45.60 | 10.20 | 4.20 | 2.428 |
| 3 | 43.80 | 53.80 | 99.60 | 93.80 | 50.00 | 45.80 | 10.00 | 4.20 | 2.381 |
| 4 | 37.80 | 47.80 | 93.50 | 87.60 | 49.80 | 45.70 | 10.00 | 4.10 | 2.439 |

$$\text{Average Sg} = \frac{2.475 + 2.428 + 2.381 + 2.439}{4}$$

= 2.431g/cm³ ∴ the value correlated with 1.45W/m.°C

APPENDIX B

AHMADU BELLO UNIVERSITY, ZARIA

DEPT. OF CIVIL ENGINEERING

PLASTIC AND LIQUID LIMIT CHART

Site..... Job.....

Sample No.....Depth..... Date.....

Liquid Limit (LL).....plastic Limit (PL).....PI.....

Linear Shrinkage (LS).....Operator.....

Description of Soil: Kankara Kaolin.

PLASTIC AND LIQUID LIMITS

Table 1.B: Plastic Limit Instrumentation Table

| | | | | | | | | | |
|-----------------------------|-----------|--|-----------|--|-----------|--|-----------|--|-----------|
| Type of Test | LL | | LL | | LL | | LL | | LL |
| No of Blows | 49 | | 26 | | 22 | | 19 | | 13 |
| Container No. | N31 | | N50 | | N113 | | N104 | | N36 |
| Wt. of wet soil &cont..g | 22.2 | | 18.7 | | 19.0 | | 19.6 | | 25.4 |
| Wt. of dry soil and cint..g | 18.0 | | 15.4 | | 15.8 | | 16.0 | | 19.3 |
| Wt. of container. G | 9.0 | | 8.6 | | 9.3 | | 8.9 | | 9.0 |
| Wt. of dry soil (Wd)..g | 9.0 | | 6.8 | | 6.5 | | 7.1 | | 10.3 |
| Wt. of Moisture (Ww)..g | 4.2 | | 3.3 | | 3.2 | | 3.6 | | 6.1 |
| Moisture Content % | 46.67 | | 48.53 | | 49.23 | | 50.70 | | 59.22 |
| Type of Test | PL | | PL | | PL | | | | |
| No of Blows | | | | | | | | | |
| Container No. | N112 | | N41 | | N34 | | | | |
| Wt. of wet soil &cont..g | 9.4 | | 9.4 | | 9.3 | | | | |
| Wt. of dry soil and cint..g | 9.2 | | 9.2 | | 9.1 | | | | |
| Wt. of container. G | 8.5 | | 8.7 | | 8.4 | | | | |
| Wt. of dry soil (Wd)..g | 0.7 | | 0.5 | | 0.2 | | | | |
| Wt. of Moisture (Ww)..g | 0.2 | | 0.2 | | 0.2 | | | | |
| Moisture Content % | 28.57 | | 40.0 | | 28.57 | | | | |

Table 2.B: Hydrometer/ Particle Distribution Size Results

| SIEVE SIZES | MASS RETAINED D | % MASS RETAINED | CUM MASS RETAINED | % PASISNG |
|--------------------|------------------------|------------------------|--------------------------|------------------|
| 2 | 57.2 | 28.6 | 28.6 | 71.4 |
| 1.4 | 2.1 | 1.05 | 29.65 | 70.35 |
| 0.5 | 4.5 | 2.25 | 31.9 | 68.1 |
| 0.425 | 0 | 0 | 31.9 | 68.1 |
| 0.36 | 0 | 0 | 31.9 | 68.1 |
| 0.212 | 1.9 | 0.95 | 32.85 | 67.15 |
| 0.15 | 0 | 0 | 32.85 | 67.15 |
| 0.075 | 17.9 | 8.95 | 41.8 | 58.2 |
| PAN | | 0 | 41.8 | |

Table 3.B: Drying Shrinkage at 110°C and Firing Shrinkage at 1280° C of Insulating Firebricks Made From Kankara Kaolin Using Acha Husk Using equations 12 and 13

| Sample Code No | Original Length OL (cm) | Dry Length DL (cm) | Fired Length FL (cm) | Dry Shrinkage DS (%) | Fired Shrinkage FS (%) | Total Shrinkage FS (%) | Temperature (°C) |
|----------------|-------------------------|--------------------|----------------------|----------------------|------------------------|------------------------|------------------|
| 1 | 14.60 | 14.59 | 14.50 | 0.068 | 0.62 | 0.69 | 1,280 |
| 2 | 14.60 | 14.58 | 14.45 | 0.137 | 0.899 | 1.04 | |
| 3 | 14.60 | 14.58 | 14.40 | 0.136 | 1.25 | 1.39 | |
| 4 | 14.60 | 14.58 | 14.45 | 0.136 | 0.899 | 1.035 | |
| 5 | 14.60 | 14.50 | 14.45 | 0.684 | 0.346 | 1.03 | |
| 6 | 14.60 | 14.55 | 14.50 | 0.342 | 0.345 | 0.69 | 1,280 |
| 7 | 14.60 | 14.55 | 14.50 | 0.342 | 0.345 | 0.69 | |
| 8 | 14.60 | 14.48 | 14.40 | 0.136 | 1.25 | 1.39 | |
| 9 | 14.60 | 14.58 | 14.40 | 0.136 | 1.25 | 1.39 | |
| 10 | 14.60 | 14.50 | 14.40 | 0.684 | 0.694 | 1.38 | |
| 11 | 14.60 | 14.55 | 14.50 | 0.342 | 0.345 | 0.69 | 1,280 |
| 12 | 14.60 | 14.58 | 14.55 | 0.137 | 0.206 | 0.34 | |
| 13 | 14.60 | 14.50 | 14.40 | 0.685 | 0.649 | 1.38 | |
| 14 | 14.60 | 14.50 | 14.45 | 0.684 | 0.346 | 1.03 | |
| 15 | 14.60 | 14.50 | 14.40 | 0.685 | 0.694 | 1.38 | |
| 16 | 14.60 | 14.50 | 14.45 | 0.684 | 0.346 | 1.03 | 1,280 |
| 17 | 14.60 | 14.55 | 14.50 | 0.342 | 0.345 | 0.69 | |
| 18 | 14.60 | 14.55 | 14.50 | 0.342 | 0.345 | 0.69 | |
| 19 | 14.60 | 14.50 | 14.40 | 0.685 | 0.694 | 1.38 | |
| 20 | 14.60 | 14.50 | 14.40 | 0.685 | 0.694 | 1.38 | |
| 21 | 14.60 | 14.55 | 14.45 | 0.342 | 0.692 | 1.03 | 1,280 |
| 22 | 14.60 | 14.58 | 14.45 | 0.137 | 0.899 | 1.04 | |
| 23 | 14.60 | 14.50 | 14.30 | 0.685 | 1.399 | 2.08 | |
| 24 | 14.60 | 14.55 | 14.40 | 0.342 | 1.042 | 1.38 | |
| 25 | 14.60 | 14.50 | 14.30 | 0.685 | 1.399 | 2.08 | |
| 26 | 14.60 | 14.50 | 14.40 | 0.342 | 1.042 | 1.38 | 1,280 |
| 27 | 14.60 | 14.60 | 14.55 | 0.00 | 0.343 | 0.34 | |
| 28 | 14.60 | 14.50 | 14.30 | 0.685 | 1.39 | 2.08 | |
| 29 | 14.60 | 14.40 | 14.40 | 1.369 | 0.00 | 1.37 | |
| 30 | 14.60 | 14.58 | 14.30 | 0.137 | 1.96 | 2.09 | |

Table 4.B: Summary of Total Shrinkage (g/cm³) Results

| Variable Acha Husks (%) | | | | | | |
|---|----------|----------|----------|----------|----------|----------|
| Grog Fraction (Coarse/ Fine) | A | B | C | D | E | F |
| 70:30 | 0.69 | 0.69 | 0.69 | 1.03 | 1.03 | 1.38 |
| 66:34 | 1.04 | 0.69 | 0.34 | 0.69 | 1.04 | 0.34 |
| 60:40 | 1.39 | 1.39 | 1.38 | 0.69 | 2.08 | 2.08 |
| 55:45 | 1.04 | 1.39 | 1.03 | 1.38 | 1.38 | 1.37 |
| 50:50 | 1.03 | 1.38 | 1.38 | 1.38 | 2.08 | 2.09 |

Table 4.B: Average Bulk Densities, Apparent Porosities and Water Absorption of Some Fired Insulating Fireclay Bricks using Equations 15 and 16

| Batch Designation | | D (gm) | S (gm) | W (gm) | BD (g/cm ³) | Pa (%) | Water Absorption wt% $\frac{w - D}{D} \times 100$ |
|-------------------|--------|--------|--------|--------|-------------------------|--------|---|
| 70:30 | A1(1) | 410 | 250 | 610 | 1.14 | 55.56 | 48.78 |
| 66:34 | B1(2) | 490 | 250 | 560 | 1.58 | 22.58 | 14.29 |
| 60:40 | C1(3) | 440 | 260 | 610 | 1.25 | 48.57 | 38.64 |
| 55:45 | D1(4) | 430 | 250 | 590 | 1.26 | 47.06 | 37.21 |
| 50:50 | E1(5) | 490 | 300 | 700 | 1.23 | 52.50 | 42.86 |
| 70:30 | A2(6) | 360 | 260 | 590 | 1.09 | 69.69 | 63.89 |
| 66:34 | B2(7) | 410 | 240 | 610 | 1.11 | 54.05 | 48.78 |
| 60:40 | C2(8) | 360 | 270 | 660 | 0.92 | 76.92 | 83.33 |
| 55:45 | D2(9) | 460 | 260 | 660 | 1.15 | 50.00 | 43.49 |
| 50:50 | E2(10) | 460 | 260 | 680 | 1.09 | 52.38 | 47.83 |
| 70:30 | A3(11) | 390 | 280 | 690 | 0.95 | 73.17 | 76.92 |
| 66:34 | B3(12) | 390 | 270 | 620 | 1.26 | 65.71 | 58.97 |
| 60:40 | C3(13) | 400 | 240 | 560 | 1.25 | 50.00 | 40.00 |
| 55:45 | D3(14) | 450 | 270 | 570 | 1.13 | 55.00 | 48.89 |
| 50:50 | E3(15) | 380 | 210 | 550 | 1.12 | 50.00 | 44.74 |
| 70:30 | A4(16) | 350 | 200 | 460 | 1.35 | 42.31 | 31.43 |
| 66:34 | B4(17) | - | - | - | - | - | - |
| 60:40 | C4(18) | 400 | 240 | 600 | 1.11 | 55.56 | 50.00 |
| 55:45 | D4(19) | 440 | 280 | 660 | 1.16 | 57.89 | 50.00 |
| 50:50 | E4(20) | 390 | 210 | 600 | 1.00 | 53.84 | 53.85 |
| 70:30 | A5(21) | - | - | - | - | - | - |
| 66:34 | B5(22) | - | - | - | - | - | - |
| 60:40 | C5(23) | 400 | 250 | 610 | 1.11 | 58.33 | 52.50 |
| 55:45 | D5(24) | - | - | - | - | - | - |
| 50:50 | E5(25) | 410 | 240 | 620 | 1.08 | 55.26 | 51.22 |
| 70:30 | A6(26) | - | - | - | - | - | - |
| 66:34 | B6(27) | - | - | - | - | - | - |
| 60:40 | C6(28) | 390 | 260 | 620 | 1.08 | 63.89 | 58.97 |
| 55:45 | D6(29) | - | - | - | - | - | - |
| 50:50 | E6(30) | 400 | 260 | 640 | 1.05 | 63.16 | 60.00 |
| 70:30 | A9(41) | - | - | - | - | - | - |
| 66:34 | B9(42) | - | - | - | - | - | - |
| 60:40 | C9(43) | 380 | 280 | 640 | 1.06 | 72.22 | 68.42 |
| 55:45 | D9(44) | - | - | - | - | - | - |
| 50:50 | E9(45) | - | - | - | - | - | - |

Table 5.B: Summary of Bulk Density (g/cm³) Results

| Variable Acha Husks (%) | | | | | | |
|---|----------|----------|----------|----------|----------|----------|
| Grog Fraction (Coarse/ Fine) | A | B | C | D | E | F |
| 70:30 | 1.14 | 1.09 | 0.95 | 1.35 | - | - |
| 66:34 | 1.58 | 1.11 | 1.26 | - | - | - |
| 60:40 | 1.25 | 0.92 | 1.25 | 1.11 | 1.11 | 1.08 |
| 55:45 | 1.26 | 1.15 | 1.13 | 1.16 | - | - |
| 50:50 | 1.23 | 1.09 | 1.12 | 1.00 | 1.08 | 1.05 |

Table 6.B: Summary of Apparent Porosity (%) Results

| Variable Acha Husks (%) | | | | | | |
|---|----------|----------|----------|----------|----------|----------|
| Grog Fraction (Coarse/ Fine) | A | B | C | D | E | F |
| 70:30 | 55.56 | 69.69 | 73.17 | 42.31 | - | - |
| 66:34 | 22.58 | 54.05 | 65.71 | - | - | - |
| 60:40 | 48.57 | 76.92 | 50.00 | 55.56 | 58.33 | 63.89 |
| 55:45 | 47.06 | 50.00 | 55.00 | 57.89 | - | - |
| 50:50 | 52.50 | 52.38 | 50.00 | 53.84 | 55.26 | 63.16 |

Table 7.B: Summary of Water Absorption(%) Results

| Variable Acha Husks (%) | | | | | | |
|---|----------|----------|----------|----------|----------|----------|
| Grog Fraction (Coarse/ Fine) | A | B | C | D | E | F |
| 70:30 | 48.78 | 63.89 | 76.92 | 31.43 | - | - |
| 66:34 | 14.29 | 48.78 | 58.97 | - | - | - |
| 60:40 | 38.64 | 83.33 | 40.00 | 50.00 | 52.50 | 58.97 |
| 55:45 | 37.21 | 43.49 | 48.89 | 50.00 | - | - |
| 50:50 | 42.86 | 47.83 | 44.74 | 53.85 | 51.22 | 60.00 |

CALCULATION OF COLD CRUSHING STRENGTH

The Cold Crushing Strengths of the Insulating bricks were calculated using equation 17.

Table 8B: Cold Crushing Strengths of Fireclay Bricks Sample.

| Sample | Length (m) | Breath (m) | Area (M ²) | Force (KN) | CCs (MPa) |
|--------|------------|------------|---------------------------|------------|-----------|
| 1 | 0.085 | 0.055 | 4.675 X 10 ⁻⁰³ | 8 | 17.1 |
| 2 | 0.085 | 0.055 | 4.675 X 10 ⁻⁰³ | 8.5 | 18.2 |
| 3 | 0.085 | 0.055 | 4.675 X 10 ⁻⁰³ | 9 | 19.3 |
| 4 | 0.085 | 0.055 | 4.675 X 10 ⁻⁰³ | 15 | 32.1 |
| 5 | 0.085 | 0.055 | 4.675 X 10 ⁻⁰³ | 19 | 40.6 |
| 6 | 0.085 | 0.055 | 4.675 X 10 ⁻⁰³ | 4 | 8.6 |
| 7 | 0.085 | 0.055 | 4.675 X 10 ⁻⁰³ | 4.5 | 9.6 |
| 8 | 0.085 | 0.055 | 4.675 X 10 ⁻⁰³ | 4.5 | 9.6 |
| 9 | 0.085 | 0.055 | 4.675 X 10 ⁻⁰³ | 9 | 19.3 |
| 10 | 0.085 | 0.055 | 4.675 X 10 ⁻⁰³ | 10 | 21.4 |
| 11 | 0.085 | 0.055 | 4.675 X 10 ⁻⁰³ | 4 | 8.6 |
| 12 | 0.085 | 0.055 | 4.675 X 10 ⁻⁰³ | 4.5 | 9.6 |
| 13 | 0.085 | 0.055 | 4.675 X 10 ⁻⁰³ | 6.5 | 13.9 |
| 14 | 0.085 | 0.055 | 4.675 X 10 ⁻⁰³ | 6.5 | 13.9 |
| 15 | 0.085 | 0.055 | 4.675 X 10 ⁻⁰³ | 7.1 | 11.8 |
| 16 | 0.085 | 0.055 | 4.675 X 10 ⁻⁰³ | 3 | 6.4 |
| 17 | 0.085 | 0.055 | 4.675 X 10 ⁻⁰³ | 3.5 | 7.5 |
| 18 | 0.085 | 0.055 | 4.675 X 10 ⁻⁰³ | 4 | 8.6 |
| 19 | 0.085 | 0.055 | 4.675 X 10 ⁻⁰³ | 4.5 | 9.6 |
| 20 | 0.085 | 0.055 | 4.675 X 10 ⁻⁰³ | 5 | 10.7 |
| 21 | 0.085 | 0.055 | 4.675 X 10 ⁻⁰³ | 3 | 6.4 |
| 22 | 0.085 | 0.055 | 4.675 X 10 ⁻⁰³ | 3 | 6.4 |
| 23 | 0.085 | 0.055 | 4.675 X 10 ⁻⁰³ | 5 | 10.7 |
| 24 | 0.085 | 0.055 | 4.675 X 10 ⁻⁰³ | 4 | 8.6 |
| 25 | 0.085 | 0.055 | 4.675 X 10 ⁻⁰³ | 3 | 6.4 |

Table 9B: Summary of CCS (MPa) Results

| Variable Acha Husks (%) | | | | | |
|--|----------|----------|----------|----------|----------|
| Grog Fraction (Coarse/Fine) | A | B | C | D | E |
| 70:30 | 17.10 | 8.60 | 8.60 | 6.40 | 6.40 |
| 66:34 | 18.20 | 9.60 | 9.60 | 7.50 | 6.40 |
| 60:40 | 19.30 | 9.60 | 13.90 | 8.60 | 10.70 |
| 55:45 | 32.10 | 19.30 | 13.90 | 9.60 | 8.60 |

Table 10B: Calculation for Thermal Conductivity of Some Insulating Bricks Sample using equation 19

| Sample Code | t ₁ (°C) | t ₂ (°C) | (tw ₁ -tw ₂) (°C) | $\lambda = \frac{Q\delta}{F(T_1-T_2)}$ W/m°C | Sample Codes | Grog Ratio |
|-------------|------------------------|------------------------|---|---|--------------|------------|
| 1 | 500 | 365.30 | 134.70 | 0.143 | A1 (1) | 70:30 |
| 2 | 500 | 440.20 | 59.80 | 0.322 | B1 (2) | 66:34 |
| 3 | 500 | 376.60 | 123.40 | 0.156 | C1 (3) | 60:40 |
| 4 | 500 | 390.30 | 109.70 | 0.175 | D1 (4) | 55:45 |
| 5 | 500 | 371.0 | 129.00 | 0.149 | E1 (5) | 50:50 |
| 6 | 500 | 130.90 | 369.10 | 0.052 | A2 (6) | 70:30 |
| 8 | 500 | 135.70 | 364.30 | 0.0052 | C2 (8) | 60:40 |
| 10 | 500 | 308.40 | 191.60 | 0.100 | E2 (10) | 50:50 |
| 11 | 500 | 112.50 | 387.50 | 0.049 | A3 (11) | 70:30 |
| 12 | 500 | 139.90 | 360.10 | 0.053 | B3 (12) | 66:34 |
| 14 | 500 | 257.50 | 242.50 | 0.079 | D3 (14) | 55:45 |
| 15 | 500 | 360.10 | 130.90 | 0.147 | C4 (15) | 70:30 |
| 16 | 500 | 379.20 | 120.80 | 0.159 | D4 (16) | 60:30 |
| 18 | 500 | 350.80 | 149.20 | 0.129 | C4 (18) | 55:45 |
| 19 | 500 | 301.90 | 198.10 | 0.097 | D4 (19) | 50:50 |
| 20 | 500 | 311.30 | 188.70 | 0.102 | E4 (20) | 60:40 |
| 23 | 500 | 250.90 | 249.10 | 0.077 | C5 (23) | 50:50 |
| 25 | 500 | 277.70 | 222.30 | 0.087 | C6 (25) | 60:40 |
| 30 | 500 | 175.50 | 324.50 | 0.059 | E6 (30) | 50:50 |

Where:

$Q = 1.45 \text{ W/m.k}$ = Coefficient of Thermal Conductivity

$F = (\delta h) = 0.002944$ meter (Area of Bricks)

$\delta = 0.039$ (Thickness of the bricks)

$h = 0.0755 \text{ m}$. (Height of the bricks)

λ = Thermal Conductivity

tw_1 and tw_2 = boundaries surfaces of the wall at temperatures tw_1 and tw_2 .

tw_1 = initial temperature (source of heat)

tw_2 = final temperature (outer face of the material)

Table 11B: Summary of Thermal Conductivity (W/m.⁰C) Results at 500⁰C Mean Temperature

| Variable Acha Husks (%) | | | | | | |
|---------------------------------|-------|-------|-------|-------|-------|-------|
| Grog Fraction (Coarse/ Fine) | A | B | C | D | E | F |
| 70:30 | 0.143 | 0.052 | 0.049 | - | - | - |
| 66:34 | 0.322 | - | 0.054 | - | - | - |
| 60:40 | 0.156 | 0.05 | - | 0.129 | 0.077 | 0.062 |
| 55:45 | 0.175 | - | 0.079 | 0.079 | - | - |
| 50:50 | 0.149 | 0.100 | - | 0.102 | 0.087 | 0.059 |

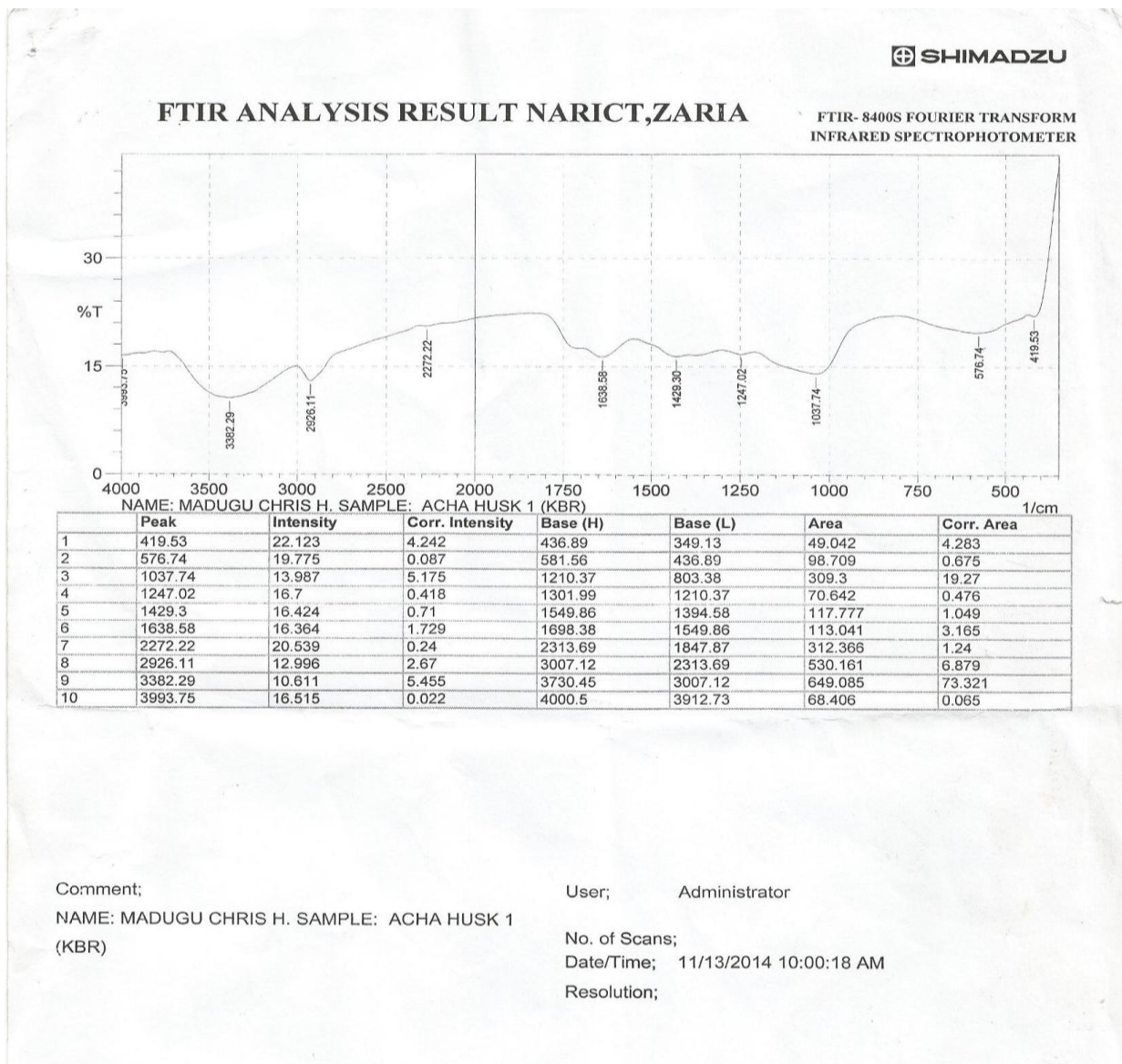
Table 12B: Designation of the Insulating Brick Batches

| Weight % of AH (g) | 70:30 (A) | 66:34 (B) | 60:40 (C) | 55:45 (D) | 50:50 (E) |
|-----------------------|-----------------|-----------------|-----------------|-----------------|-----------------|
| 1 | A ₁ | B ₁ | C ₁ | D ₁ | E ₁ |
| 2 | A ₂ | B ₂ | C ₂ | D ₂ | E ₂ |
| 3 | A ₃ | B ₃ | C ₃ | D ₃ | E ₃ |
| 4 | A ₄ | B ₄ | C ₄ | D ₄ | E ₄ |
| 5 | A ₅ | B ₅ | C ₅ | D ₅ | E ₅ |
| 6 | A ₆ | B ₆ | C ₆ | D ₆ | E ₆ |
| 7 | A ₇ | B ₇ | C ₇ | D ₇ | E ₇ |
| 8 | A ₈ | B ₈ | C ₈ | D ₈ | E ₈ |
| 9 | A ₉ | B ₉ | C ₉ | D ₉ | E ₉ |
| 10 | A ₁₀ | B ₁₀ | C ₁₀ | D ₁₀ | E ₁₀ |
| | | | | | |

Table 13B: Optimum Batch Formulated Results

| Code No | Grog Ratio | BATCH COMPOSITION | | | Pa (%) | BD (g/cm ³) | Thermal Conductivity (W/m.°C) | Remarks |
|---------|------------|-------------------|------------|--------|--------|-------------------------|-------------------------------|---------|
| | | Grog(g) | -Kaolin(g) | -AH(g) | | | | |
| 1. | 70:30 | 503.9 | 56.6 | 6.1 | 55.56 | 1.14 | 1.145 | Good |
| 2. | 66:34 | 504.9 | 56.6 | 6.1 | 22.58 | 1.58 | 0.322 | Good |
| 4. | 55:45 | 503.9 | 56.6 | 6.1 | 47.06 | 1.58 | 0.322 | Good |
| 5. | 50:50 | 503.9 | 56.6 | 6.1 | 52.50 | 1.23 | 0.149 | Good |
| 6. | 70:30 | 498.3 | 56.6 | 11.3 | 69.69 | 1.09 | 0.052 | Good |
| 10. | 50:50 | 498.3 | 56.6 | 11.3 | 52.38 | 1.09 | 0.100 | Good |
| 11. | 70:30 | 492.6 | 56.6 | 16.9 | 73.17 | 0.95 | 0.049 | V. Good |
| 12 | 66:34 | 492.6 | 56.6 | 16.9 | 65.71 | 1.26 | 0.053 | V. Good |
| 14 | 55:45 | 492.6 | 56.6 | 16.9 | 55.00 | 1.13 | 0.079 | V. Good |
| 19 | 55:45 | 486.9 | 56.6 | 22.7 | 57.89 | 1.16 | 0.097 | Good |
| 20 | 50:50 | 486.9 | 56.6 | 22.7 | 53.84 | 1.00 | 0.102 | Good |
| 30. | 50:50 | 475.6 | 56.6 | 33.9 | 63.16 | 1.05 | 0.059 | Poor |

APPENDIX C



FIGUER A3: Fourier Transform Infrared (FTIR) Spectrometry Analysis Result of Acha Husks (E)

Figure 1.C: FTIR Analysis of Acha