EVALUATION OF QUARTZ DEPOSIT IN ANGWAN DOKA, KOKONA NASARAWA STATE-NIGERIA FOR PRODUCTION OF DENSE REFRACTORY BRICKS

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A THESIS SUBMITTED TO THE SCHOOL OF POSTGRADUATE STUDIES, AHMADU BELLO UNIVERSITY ZARIA, NIGERIA, IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE AWARD OF MASTER OF SCIENCE DEGREE IN GLASS TECHNOLOGY

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ZARIA, NIGERIA

AUGUST, 2018

DECLARATION

I declare that the work in this dissertation entitled, "Evaluation of Quartz Deposit in AngwanDokaKokonaNasarawa State-Nigeria for Production of Dense Refractory Bricks" has been carried out by me in the Department of Glass and Silicate Technology. 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 or diploma at this or any other institution.

ZaiduYahuza ....
Name of Student Signature Date

CERTIFICATION

This dissertation entitled, “EVALUATION OF QUARTZ DEPOSIT IN ANGWAN DOKA KOKONA NASARAWA STATE-NIGERIA FOR PRODUCTION OF DENSE REFRACTORY BRICKS,” by ZAIDU YAHUZA meets the standard and regulation governing the award of the Master of Science degree in Glass and Silicate Technology ofAhmadu Bello University, Zaria Nigeria, and is approved for its contribution to knowledge and literary presentation.

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Chairman, Supervisory Committee Date
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DEDICATION

This dissertation is dedicated to the memory of my Beloved Aunty, **HajiyaHalima** (***Kande***).

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ABSTRACT

Samples of quartz were sourced from deposit site at AngwanDokaKokonaNasarawa State Nigeria. The samples were beneficiated and crushed to smaller particle sizes ranging from fine, medium to coarse. The samples were prepared by the semi-dry method, with moulding pressure of 300 kN to form brick. After drying at 110^oC, the bricks were burnt at 1450^oC. The refractory bricks turned brownish in colour with some spot-dot tint colour on their surfaces; the refractory bricks were investigated to determine their chemical and physical properties. X-ray fluorescence analysis result showed SiO_2 to be the major oxide at 96.25%. Other oxides present in the quartz sample as impurities were MgO-0.88%, Fe_2O_3-0.51%, Cr_2O_3-0.18%, CaO-0.02%, etc. Al_2O_3 and TiO_2 were not detected, loss on ignition (LOI) was 2.037% and the total sum of impurity contents of 1.713% falls within the tolerable impurities associated to silicates group of refractories. The refractory bricks have an average linear firing expansion value of 2.9% and it is in accordance with (ASTM C179-14 (2014). The apparent porosity value of the brick was 33.1% which is below recommended ASTM value of (17-25%). The bulk density was 1.7 g/cm^3; cold crushing strength was 202.56 kg/cm^2. Thermal shock resistance showed that 9 heat cycles were required at 900^oCbefore destruction. Therefore, it was categorized as having a good thermal shock resistance; this is due to the fact that the 9 heat cycles falls within recommended ranges of 7-12 cycles for silica dense refractory brick. The quartz from AngwanDokaKokonaNasarawa State formed a silica dense refractory brick which is among the medium heat duty group of silicate refractories. The produced refractory bricks can be used in applications such as construction of reheating furnace roofs, regenerator chambers, furnace doors etc. where moderate strength and heat treatments were required

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DEFINITION OF TERMS

Aggregates: Calculated materials crushed into particles of classified size fractions to be collected together from the fractions for the formulations of batch.

Aluminosilicates: It is an inorganic compound ceramic material whose major constituents are alumina and silica. Examples include fireclay, kaolin, ball clay, etc.

Beneficiation: This is the systematic process of refining or treatment of raw materials to optimize their performance for higher manufacturing process in order to achieve grade products.

Chemicomineralogical: This refers to the elemental oxide or chemical composition and the mineral phase present within the clay material.

Cold Crushing Strength: This measure the ability of bricks to withstand the structural load coming over them

Dense Refractory Brick: This is a refractory products made under high pressure by densification and compaction of refractory aggregates.

Fireclays: These are naturally occurring earthy materials having the chemical composition given by the formula $Al_2O_3.2SiO_2.H_2O$, whose alumina content is either above 25% or up to 45% of the weight, while the silica content is 50% to 80%.

Furnace: This is high temperature equipment used in the manufacturing or refining process where heat requirement is extreme. Examples of areas of application are the iron and steel, cement, glass and the petrochemical industries, etc.

Grog: This refers to pre-fired/calcined clay materials that are crushed into several particles sizes used for refractory production.

Porosity: This measure the percentage relationship between the volume of the open pore space and the total volume of a refractory.

Refractoriness: This measures the ability of bricks to withstand high temperatures over a long period of use without fusion or softening.

Thermal Shock Resistance: This measure the resistance of a refractory brick to sudden change in temperature as the result of alternate heating and cooling.

CHAPTER ONE

1.0 INTRODUCTION

1.1 Background of the Study

The term “REFRACTORY” means “HARD TO FUSE”. Refractories are therefore the class of materials which withstand high temperatures, resist the action of corrosive liquids and dust laden currents of hot gases that comes in contact with the material (Chesti, 1994). American Society for Testing and Materials (ASTM) defines refractories as inorganic materials, usually non-metallic that withstand high temperatures (ASTM, 1975). It was further asserted that any material withstanding temperatures above 538^oC (1000^oF) is considered as refractory. Such materials includes; aluminosilicates, silica, magnesites, chrome, chrome-magnesite, carbon and dolomite refractories. Others include zirconia, carbides, nitrides, silicides, borides and their compounds.

The criterion of ability to withstand exposure to environments above 538^oC (1000^oF) is the criterion of distinction separating refractories from other ceramics, fibres and coatings applicable only at lower temperatures. Thus, refractory materials are characterized by the ability to withstand not only the heat but also chemical and slag attack, abrasion impact, resist thermal shock and carry sustained structural stresses at high operating temperatures for as long as possible. Refractories belong to the class of ceramic materials which are employed for high temperature applications, usually above 1000^oC. Most refractories are made from naturally occurring high melting point oxides of SiO_2, Al_2O_3, MgO, Cr_2O_3, and ZrO. Refractories are used in almost every industry in which heat is employed such as in metallurgical, chemical, cement, glass and petrochemical industries. They are used for the construction and maintenance of furnaces, kilns, reactors and boilers (Jock *et al*, 2013). Refractory materials play a very significant role in high temperature environments, especially, in furnace linings and construction and other applications. They have wide applications in ceramics and glass industries, iron and steel plants, foundries, cement factories, petrochemical industries (Esezobor*et al.*, 2015).

Refractory materials have been developed to serve as heat shields in the construction and maintenance of lining of the interior walls of high temperature equipment like furnaces, flues and reactors thereby protecting the equipment shells (usually made of steel and or cast iron) from breakouts and premature failure during process operations. The iron and steel, ceramics and glass, sugar, cement, petroleum and petrochemical are some of the industries where high temperature operations are inevitable. In such industries, operating temperatures are as high as several hundreds to thousands of degrees (Nurudeen, 2010).

In Nigeria, numerous research works by Amuda*et al.*, (2005); Ameh and Obasi, 92009); Mark, (2010); Nurudeen, (2010); Musa, *et al.*, (2012); Jock, *et al.*, (2013) and Apeh, (2014) had been carried out on the characterization of indigenous refractory raw materials for possible production of quality refractory bricks for furnaces, kilns, ovens and lining to replace the high cost of imported bricks (Apeh*et al*., 2010). The results of these numerous researchers have indicated that high quality refractory bricks can be produced domestically.

It is regrettable, that a huge amount of money, about $229 million, is expended annually to import refractory materials into Nigeria (Apeh, 2014). Various researchers Hassan, (2005); Apeh*et al*., (2011) and Nurudeen, (2010) argued that, this huge spending could be avoided if the bricks were produced domestically in Nigeria.

The prime ingredient for silica refractories, sometimes termed acid refractories, is silica. These materials were well known for their high-temperature load-bearing capacity and are commonly used in the arched roofs of steel and glass-making furnaces; for these applications, temperatures as high as several hundreds may be attained. Under these conditions, some small portion of the brick will actually exist as a liquid. The presence of even small concentrations of alumina has an adverse influence on the performance of these refractories. These refractory materials are also resistant to slags that are rich in silica (called acid slags) and are often used as containment vessels for them. On the other hand, they are readily attacked by slags composed of a high proportion of CaO and or MgO (basic slags), and contact with these oxide materials should be avoided (Callister, 2007).

Silica brick is a refractory which is significant from the chemical point of view by its high content of SiO_2 (more than 95 %). As a mineral, it is composed of various modifications of SiO_2, particularly β-cristobalite and γ- tridymite (Brunk, 2000). Furthermore, it is composed of a low amount of unmodified β-quartz, calcium orthosilicates, and vitreous phase. The usage of refractory materials, usage of silica bricks as well, is influenced by technological development in the industries where these materials are applied. Their constantly increasing technical quality is an accompanying effect referred to as "*Hara-kiri* effect." Despite the unavoidable trend of continuous stagnation of the volumes required by industries, new refractory products are being developed, new technologies invented or new raw materials utilized. One of such products is silica material which is still used for its specific properties (Marinelli, 2008).

The use of silica material is quite specific, being implemented mainly in the following areas of application; in coke production, glass industry and metallurgy with completely different utilization. The biggest volumes are utilized in the construction of coke oven batteries, silica is still hardly replaceable in the crowns of glass melting furnaces and it is being used ever more frequently and also for the top parts of hot blast stoves in metallurgy. Even though the application of silica materials in the areas mentioned above is very effective there is still a potential for reaching an even higher level of quality. The success of this material based on SiO_2 is determined mainly by its outstanding performance at high temperatures. The melting point of SiO_2 is the lowest one in the category of technical oxides (1726^oC) which is why firing is the critical part of the manufacturing process (Brunk, 2000).

Despite this fact it is clear that the desired changes of parameters cannot be attained only by making changes in the firing regime. It is essential to attain maximum density at raw conditions and optimal dosage of mineralizers (maintaining thermodynamic properties) in order to have the optimal solution. Among other issues related to the use of silica bricks are adapting the material's behaviour to various operational conditions and optimizing its properties for the requirements of specific applications. Reaching better chemical (higher SiO_2 content and lower content of melting oxides), physical (lower apparent porosity and higher cold crushing strength) and thermodynamic properties (lower speed of creep) is mostly linked with higher thermal conductivity which is not always an advantage especially in glass industry application (Nevrivova et al., 2014).

AngwanDoka is a remote area in Kokona Metropolis. It is located at Agwada junction off Keffi-Akwanga road at kilometre 15 away from Nasarawa State University Keffi, see AppendixE for map and coordinate of AngwanDokaKokona, Nasarawa State. Presently the quartz from AngwanDokaKokona deposit in Nasarawa State has not been evaluated for possible use as a refractory material. The miners' minerals of interest are the precious minerals such as sapphire, tourmaline and aquamarine etc. They consider the quartz as just a vein that would lead them to these precious minerals; therefore to them it has less or no value. This research is however aimed at evaluating the quartz from this deposit for its refractory properties and applications. Thus, this study will strive to evaluate both the physical and chemical properties of quartz from AngwanDokaKokona deposit and to produce dense refractory bricks. The quartz used for this research was obtained primarily from AngwanDokaKokona, Nasarawa State.

1.2 Statement of the Problem

The development of the iron and steel industry via the rehabilitation of various inland rolling mills and the envisaged completion and commissioning of the Ajaokuta Steel Company Limited indicate that there will be a great increase in consumption of refractory materials. Ajaokuta Steel Company Limited is estimated to require 36,000 tonnes of refractory bricks worth over 120 million Naira just for furnace lining purposes (Musa *et al*., 2012).

In the past few years, there has been an increasing awareness on the scope, and the importance of refractory materials in the industrial development of Nigeria. Research records show that several workers have investigated the suitability of various locally sourced materials for the production of fire-clay refractories from various deposit (Ahmed, 1986; Garkida, 1998; Adekeye, 1999; Fasuba*et al.*, 2000, Sullayman, 2006; Abdullahi, 2009; Musa *et al.*, 2012; others include; Lawrence and Ayo, 2012; Obikwelu, 2012; Hassan and Aigbodion, 2014; Chinago*et al.*, 2015; Sani, 2016;). However, no records are available that investigate the properties of quartz from AngwanDoka, KokonaNasarawa State and its use in local production of dense refractory bricks despite the availability of the material. It is imperative, therefore, to attempt to produce silica bricks from this locally sourced raw material in order to curb costs that would result from foreign exchange dissipation due to the importation of the refractory bricks.

1.3 Aim of the Study

The aim of the study is to produce dense refractory bricks from quartz deposit, available at AngwanDokaKokonaNasarawa State Nigeria.

1.4 Objectives of the Study

The objectives of the study are to;

i. source and collect quartz sample from its place of deposition via mining at AngwanDoka, with the aid of mining tools and beneficiate the samples using physicochemical methods.
ii. analyze the upgraded sample with X-ray fluorescence (XRF) for determination of its oxide composition.
iii. produce proportionate aggregates, develop and mould a suitable body composition from the analyte sample, and then dry and fire the bricks to their fusion temperature for densification and sintering.
iv. determine the mechanical, physical and microstructural properties of the dense refractory.

1.5 Justification of the Study

Nigeria is endowed with vast reserves of solid minerals, including, but not limited to, precious metals, stones and industrial minerals. The country was a major exporter of tin, columbite and coal in the early 1970s; however, activities in the mining sector stoped considerably when crude oil production began to take the centre stage, and became a major source of foreign exchange for the country (Nigerian Mining Sector, 2012). With the current dwindling of oil prices, the need to diversify the revenue base of the country has become paramount.

Silica refractories are particularly useful in metallic and non-metallic industries, this is due to their rigidity at temperatures above the range of usefulness of ordinary fire-clay refractories and to the fact that they expand slightly when heated instead of shrinking as clay refractories do. Local production of silica refractory bricks is of vital importance for the sustenance of the Nigerian process industries which at present are in dilapidated condition. Successful completion of this research will lead to increase in local content for the production of refractories and sustenance of Nigerian process industries, thereby leading to diversifying and boosting the economy of Nigeria. So also, it will catalyse local investment in processes using quartz as raw material like; quartz clock, ceramics and glass, glaze, and enamel, production will also increase.

1.6 Significance of the Study

This research makes an attempt to utilize available raw materials for local production of dense silica refractory bricks for application in high temperature equipment (furnaces and kilns) in processing industries such as ceramic and glass, iron and steel etc. The success of this research is expected to further promote our local industries in the production and possible exportation of these bricks.

1.7 Scope of the Study

This research was delimited to the collection of quartz sample from AngwanDoka, KokonaNasarawa State Nigeria using appropriate tools, beneficiate and upgrades the sample; determine the sample oxides composition via (XRF) analysis. Mix proportionate aggregate, mould and press for compaction and finally produce dense refractory bricks from the sample and test the properties of the bricks for relevant refractory applications.

1.8 Limitation

During the cause of the research, the furnace that was available for use can only attain the maximum firing temperature of 1450℃. It is an electrical fired furnace therefore; it required uninterrupted power supply for maximum efficiency especially when longer soaking time is required. But unfortunately power instability is one of the major setbacks that affect some properties of the bricks.

Other properties test such as refractoriness under load, modulus of rupture and thermal conductivity were not achieved due to unavailability of necessary equipment to carry out the test.

CHAPTER TWO

2.0 LITERATURE REVIEW

2.1 Introduction

Refractories are materials of construction capable of withstanding high temperature and maintaining their physical properties in a furnace environment when in contact with corrosive slags, liquid metals and gases. They are widely used in most high-temperature manufacturing processes such as copper and aluminium smelting, glass and ceramic manufacture, cement and ore processing, petroleum refining and petrochemicals manufacture. However, iron and steel making account for almost two-thirds of all refractories used and it is in this industry where many refractory developments have occurred. The primary responsibility of refractories in the high temperature equipment like furnaces during operation is to minimize operation cost by controlling the consumption of energy required during production thereby profit is maximized (Muhammadu, 2013).

2.2 Refractory Material

According to Rahul (2011), materials that can withstand very high temperature without degrading or softening are known as refractory materials. Refractory materials include certain ceramics and super alloys and are used in heat insulation of furnaces. The main function of a refractory is to withstand and maintain high temperatures and resist the abrasive and corrosive action of molten material, slag and gases.

As mentioned earlier, the works of Amuda*et al*, (2005); Ameh and Obasi, (2009); Mark, (2010); Nurudeen, (2010); Musa *et al*, (2012); Jock *et al*, (2013); Apeh, (2014) and Odewale*et al*, *(*2015) had been carried out on the characterization of indigenous refractory raw materials for possible production of quality refractory bricks for furnaces, kilns and ovens lining to replace the high cost of imported bricks. The results of their work have shown the availability and the potentials of these local raw materials across the geopolitical zones of Nigeria for production of refractories, which needed to be properly used and a guide for exploration and application of these materials. These refractories are usually classified in terms of the ranges of temperature at which they are used, as either low refractory (below 1770°C); medium refractory (within the range of 1770°C -2000°C) and high refractory is above 2000°C (Nwannenn*et al*, 2014).

Similarly, in terms of their chemistry, (Ahmed, 1986; Gupta, 2008; Bhatia, 2012 and Rahul, 2011) agreed that, refractories can also be classified on the basis of chemical and mineralogical compositions, their methods of manufacture and their physical form. Therefore appropriate materials selection is required for the production of refractory materials of specified property.

2.3 Classification of Refractory Materials

Refractory oxides of metals, non-metals or their mixtures form the most widely used group of refractory materials as they include some of the most stable compounds known. Refractory materials are classified based on their chemical and mineralogical composition, their method of manufacturing and or based on their physical form

2.3.1 Classification base on chemicomineralogical composition

According to Chesti (1994) and Bhatia (2012) refractory can be classified based on the chemical and mineralogical composition of the material. The refractories are classified as acid, neutral and basic refractories depending on their chemical reaction with metallurgical slags, fluxes and furnace gases. Table 2.1 shows the classification of refractory base on chemicomineralogy.

Table 2.1 Chemicomineralogical Classification of Refractories

Acid Refractories	Basic Refractories	Neutral
Silica	Magnesite	Alumina
Aluminosilicates	Magnesite-Chrome	Carbon
Zirconia	Dolomite	Chrome
Zircon	Forsterite	Chrome-Magnesite

(Ahmed 1986) development of phosphate bonded fireclay refractory castable.

Acid refractories are those which are attacked by alkalis (basic slags). These are used in areas where slag and atmosphere are acidic. Neutral refractories are chemically stable to both acids and bases and are used in areas where slag and atmosphere are either acidic or basic. Basic refractories consist of the materials of basic chemical character. They are characterized by their withstanding power under basic environment at high temperature, but are easily attacked by acidic material. They are usually used in the basic environment of the metallurgical furnaces. They show improved performance at the slag zone of the furnaces (Damon, 2003).

Chemical characteristics of the furnace process usually determine the type of refractory required. Theoretically, acid refractories should not be used in contact with basic slags, gases and fumes whereas basic refractories can be best used in alkaline environment. Actually, for various reasons, these rules are often violated (Bhatia, 2011).

2.3.2 Classification based on method of production

According to (Chesti, 1994), the method by which refractories are produced is commonly used to classify them. There are four main methods by which refractories are manufactured. These are pressing, ramming, extrusion and castables. Pressing process is classified into dry pressing and semi-dry pressing processes. In the dry pressing process, the batch material usually contains about 20-50% of grog (pre-fired material) in the case of fire clay, mixed with relatively small amount of moisture, 5-8%. In semi-dry pressing, about 8-9% moisture is used in the batch. The batch is charged into steel moulds and compacted by means of mechanically or hydraulically applied pressure of about 200kg/cm^2 or more. These are dried and fired to desired temperature. Lower moisture contents in mixtures require high press and higher moisture containing mixes increase the slumping changes at the time of release from the mould Thus a compaction pressure from 200kgf/cm^2 to 800kgf/cm^2 is suggested.

2.3.3 Classification based on physical form

Refractories are classified according to their physical form. These are the shaped and unshaped refractories, either dense or porous. The shaped is commonly known as refractory bricks and the unshaped as "*monolithic*" refractories (Bhatia, 2011).

2.3.4 Classification based on refractoriness

Refractories are classified into following four types according to their refractoriness.

i. Low heat duty refractories – These refractories have refractoriness in the range of 1520^oC to 1630^oC and have pyrometric cone equivalent (PCE) value in the range of 19 to 28. Example of these refractories is silica bricks, (ASTM C416-97, 2013).

ii. Intermediate heat duty refractories – These refractories have refractoriness in the range of 1630^oC to 1670^oC and have pyrometric cone equivalent (PCE) value in the range of 28 to 30. Example of these refractories is fire clay bricks (Sani, 2016).

iii. High heat duty refractories – These refractories have refractoriness in the range of 1670^oC to 1730^oC and have pyrometric cone equivalent (PCE) value in the range of 30 to 33. Example of these refractories is chromite bricks (Chesti, 1994).

iv. Super heat duty refractories – These refractories have refractoriness greater than 1730^oC and have pyrometric cone equivalent (PCE) value greater than 33. Example of these refractories is magnesite bricks (Gupta, 2008).

Table 2.2 Refractoriness Classification of Refractories

Type of Refractory	PCE (Segar Cones)	Refractoriness/Fusion Point (^oC)
Low heat duty	19-28	1520-1630
Medium heat duty	28-30	1630-1670
High heat duty	30-33	1670-1730
Super duty	Greater than 33	Greater than 1730

(Gupta, 2008) elements of fuels, furnaces and refractories.

2.3.5 Shaped and unshaped refractories

Shaped Refractories are those which have fixed shaped when delivered to the user. These shaped category of refractories are been refer to as bricks. The shaped refractory may be further divided into two: standard shapes and special shapes. Standards shapes have dimension that are conformed to by most refractory manufacturers and are generally applicable to kilns and furnaces of the same type. Special shapes are specifically made for particular kilns and furnaces. This may not be applicable to another furnace or kiln of the same type. Shaped refractories are made from the different raw materials followed by pressing and firing at high temperature. Mostly shaped refractories are used to construct kilns and furnace. Unshaped refractories on the other hand are marketed as a mixture of refractory aggregates, binders, plasticizers etc. This ready to use mixture is mixed with water and the desired refractory lining is construed on site with it. The development of unshaped refractories gives the endless lining concept at the user industries. Various unshaped refractories include conventional castables, Low cement castable, ultra-low cement castable, gunning mixtures, ramming mixture and mortar (Rahul, 2011).

2.4 Dense and Porous Refractories

Refractories could be classified into two categories depending on the porosity present in it namely dense and porous refractories. Dense refractories contain porosity in the range 15-20%. These refractories were used in contact with hot liquid metal and gases. On the other hand porous refractories contain porosity as high as 80%. The porous refractories are light weight and possess very good thermal insulation properties. These refractories are used at high temperature or as back up lining of the furnaces. The high temperature insulation bricks are known as hot face insulation brick. The backup insulation bricks are known as cold face insulation brick (Charles, 2004).

2.5 Insulating Refractories

Insulating refractories are characterized by the presence of large amount of porosity in them. The pores are mostly closed pores. The presence of porosity decreases the thermal conductivity of these refractory drastically. The bulk densities of this type of refractories are usually low due to the presence of the porosity in them. The application temperature of these refractories depends on the constituents. For example, kyanite based insulating refractory is used as high temperature insulation. Whereas asbestos and fireclay based insulating refractories are usually used at low temperatures (Rahul, 2011). Glass-wool, slag wool, vermiculite are also used as insulating materials at low temperatures (Chesti, 1994).

2.6 Special Refractories

These include expensive materials which are used for the construction of crucibles and furnaces for experimental and other specials purposes where cost of refractory is no consideration. Refractory of this type include beryllia (BeO), thoria (ThO_2), zircon ($ZrSiO_4$), carborundum (SiC), sialons (Si-Al-O-N group of materials), and high purity refractory oxides like magnesia (MgO), silica (SiO_2), alumina (Al_2O_3). Others include alundum (mixture of fused alumina and clay), electrocast block of mullite, magnesia; and mixtures of chromite, bauxite and magnesia; and sillimanite ($Al_2O_3 \cdot SiO_2$); borides, carbides, nitrides and silicides, etc (Chesti, 1994).

2.7 Silica Refractories

Silica refractories are well adapted to high temperature service because of their high refractoriness, high, mechanical strength and rigidity at temperatures almost up to their melting points, as well as their ability to resist the action of dusts, fumes and acid slags. ASTM divides silica brick into Type A and Type B based on the brick’s flux factor. Flux factor is determined by adding the alumina content and twice the total alkali content. The Type A class includes silica brick with a flux factor of 0.50 or below; Type B includes all silica brick with a flux factor above 0.50. According to ASTM C416-97 (2013), both classes require that brick meet the following criteria: Al_2O_3 less than 1.50%; TiO_2 less than 0.20%; Fe_2O_3, less than 2.50%; CaO less than 40%; and average modulus of rupture strengths not less than 500 psi (3.45MPa).

2.7.1 Application of Silica Refractories

Silica refractories are particularly useful in industrial work. This is due to their rigidity at temperatures above the range of usefulness of ordinary fire-clay refractories, and to the fact that they expand slightly when heated instead of shrinking as clay refractories do. Silica refractory is the most abundant refractory used in the construction of a Coke Oven Battery (COB). Silica is the refractory of choice primarily because, at normal COB operating temperatures, silica refractories are subject to minimal creep. Also, since nearly all of the expansion of silica brick takes place below 650°C (ASTM, 1975), during normal operation of a COB, the moderate temperature fluctuations of the walls have no effect on the volume stability of the refractory comprising the wall. A COB design can have well over 400 different shapes used in its construction. These shapes are installed with a silica mortar. Lime-bonded silica refractories characterized by SiO_2 contents greater than 95% in terms of quantity represent the largest part in refractory materials used for coke oven construction. Their fields of application are the areas of oven sole, heating wall, oven top and the thermally higher stressed upper regenerator layer. Silica refractories have ideal thermo-mechanical properties and a high chemical resistance in those temperature ranges which are decisive for industrial coal carbonization (Brunk, 2000). Table 2.3 shows some region of COB and the types of refractories used in each region.

Table 2.3 Type of Refractories usually used in Different Zones of Coke Oven

Coke oven region	Refractories
Roof	Fireclay brick Insulating brick
Flue wall	Silica brick
Jamb (wall near oven door)	insulating brick, Fireclay brick
Curved wall	Silica brick
Regenerator	
Wall	Silica brick, Fireclay brick
Checker	Fireclay brick
Sole flue	Silica brick, Fireclay brick
Door	Precast brick
Ascension pipe	Precast brick
Chimney flue	Fireclay brick, Common brick

(Ispatguru, 2014) refractory for by-product coke oven batteries.

Other than COB, silica refractories are used mainly in glass melting furnaces, hot blast stoves, and electric arc furnace roofs and are useful for stiffening up walls, etc., clay base refractories would have otherwise soften and sag from the effect of heat and fumes arising from the glass.

The uses of silica refractories in copper reverberatory furnaces are similar to those in open-hearth steel furnaces, except that for melts of lead-free metallic copper, silica can be used throughout the furnace except at the slag line. For this work the brick should be as low in porosity as possible to avoid absorption of copper oxide, which slags away the bricks when the furnace is at its maximum temperature. Silica brick arches in copper furnace work wear much better if they are not insulated. In open-hearth steel furnace work, silica brick are used for the top, ends, and sometimes for the side walls of the furnace (Harbison-Walker Refractory Company, 2005).

2.7.2 Manufacturing of silica refractories

The raw material used in the manufacture of silica refractories consists essentially of quartz in finely crystalline form having the proper characteristics for conversion to the high temperature crystal modifications of silica. To assure the highest commercial quality in the refractory product, the mineral must be washed to remove natural impurities. After being formed, the brick must be fired at a temperature high enough to convert the quartz into forms of silica that are stable at high temperatures in the firing and cooling process, refractories must pass through several critical temperature ranges; consequently, it is necessary to maintain a carefully planned time-temperature schedule during the firing process, because proper schedule time-temperature factor results to the production of strong, well-bonded brick which attain their normal permanent expansion (Chesti 1994).

CHAPTER THREE

3.0 METHODOLOGY

3.1 Introduction

This chapter describes the materials and equipment, experimental procedures, and properties test that lead to realizing the aim of this research work. The procedures includes; mining of quartz sample from AngwanDokaKokona, beneficiation of the sample, chemical analysis of the sample, particles size distribution, mixing with binding material, pressing/moulding, drying and firing of the brick and finally, testing the product for refractory applications.

3.2 Materials

Materials that were used for this research include; quartz that was sourced via mining from the site at AngwanDokaKokona. Other materials include; distilled water, detergent powder, Hydrochloric acid (HCl), Molasses, Quicklime (CaO), and Mercury. Aside the quartz and water, the above aforementioned materials were sourced from chemical vendors.

3.3 Equipment

Some of the equipment and apparatus that were used during the cause of this research include the followings; shovel, digger, sample collection sack, analytical weighing balance, pipette, beaker, measuring cylinder, plastic container, jaw crusher model (Denver: BDA 15671) , cone crusher model (Denver: BMAE 20712), roll crusher MODEL (Deco B714:1A), ball milling machine, Tyler sieves, vibrating machine, Energy-Dispersive-X-ray fluorescence Spectrometer model (ED-XRFS MiniPAL 4), Scanning electron microscopy (SEM) model (Quarum with serial number Q150RES), Tecramicsdensometer, densometer weighing balance, 100ml beaker, one pair of crucible tongs pan mill mixer, hydraulic press machine model (Paul Weber D 7084), steel mould, electric oven, electric furnace model (Carbolite RHF 1600), electric hot plate model (Bio Tech serial No 06712), hammer etc.

3.4 Experimental Procedure

The experimental procedure include: collection of sample, beneficiation of sample, chemical analysis of sample, particle size distribution/batch formulation, mixing and bonding of sample, shaping, pressing and moulding the sample, drying and firing of the sample and finally testing the product for refractory applications.

3.4.1 Beneficiation of quartz

According to Abdullahi (2009), raw materials obtained directly from mines would usually require some purification treatment to separate the various impurities present. The most frequently used method is the sedimentation process which aims at separation of materials on a size basis, since no two materials are likely to have exactly the same size distribution. This method is often effective for separation of clay from sand and silt.

Sample of quartz obtained from AngwanDokaKokona, were both physically and chemically processed before use. The sample was washed thoroughly with distilled water, detergent and HCl and then finally rinsed with distilled water; all the clay, dust and other impurities present were washed away. The sample was crushed using two ways crushing; the Jaw crusher in National Metallurgical Development Centre Jos (NMDC) was the primary crusher used in reducing the size of the quartz. The Cone crusher in NMDC was the secondary crusher that further reduced the size of the quartz and finally ground to fine size in Roll crusher which was an adjustable crusher that has the ability to ground into the desired fineness. The fine, medium and coarse grains were screened using Tyler sieves on a vibrator machine. The required particle sizes were distributed based on percentage.

3.4.2 Sample Description

Quartz from AngwanDokaKokona is a hard crystal clear almost transparent to translucent mineral with hexagonal shape and pencil like tips at both ends, deformation occurs during mining when the mineral breaks, a conchoidal fracture with a glassy lustre smooth surface is revealed. Their sizes range between 1cm – 15cm and can weigh from 5g – 1kg. Quartz is generally rated number 7 in Mohrs scales of Hardness. The beneficiated sample was crushed to fine medium and coarse particle sizes, the resultant particles were crystal pure white with glowing reflection when exposed to sunlight.


Plate 3.1 Kokona Quartz before crushing

Plate 3.2 Kokona Quartz before crushing

Plate 3.3 Kokona Quartz after crushing

3.4.3 Chemical analysis

Chemical analysis test was carried out on the quartz sample in order to ascertain its elemental composition. Precisely, 20.00g of the sample was finely ground and sieved using 200 – 250μg mesh sieve; it was dried in an oven at 105°C for 1 hour and cooled. Thereafter, the sample was intimately mixed with a cellulose flakes binder in the ratio of 5.0g sample to 1.0g cellulose flakes binder and pelletized at a pressure of 15 tons / inch^2 in a pelletizing machine. The pelletized samples were stored in desiccators for and ready for analysis. Although, the researcher did not have access to operate the XRF machine but physically observed and noticed as the entire procedure is taken place during the experiment.

The XRF machine model (ED-XRFS MiniPAL 4) was switched on and allowed to warm up for 2 hrs. Appropriate programs for the various elements of interest were employed to analyse the samples. The result of the analysis was reported in percentage (%) for both the minor and major elements and non-detectable for those were absent.

3.4.4 Particle size distribution

There are three basic considerations in material proportioning; these are particle size distribution, maximum grains size and composite proportioning (Ahmed, 1986 and Chesti, 1994). Particle size distribution adopted by Muhammad (2009) was used. The method for batch formulation of the materials involves mixing the upgraded quartz sample with 2.0% CaO (quicklime) which acts as a binder and mineralizer in order to convert the free quarts to crystobalite and tridymite. Table 4.2 shows the range of particle size distribution which was adopted for this research work.

3.4.5 Mixing/bonding

The bonding solution was prepared by mixing the following additives: Bonding material-molasses of dextrin (about 1.0%), freshly burnt quicklime (2.0% of total mix) as a binder and mineralizer to hasten the inversion of quartz to cristobalite and tridymite, and water (about 7% of total mix). The lime was prepared by weighing out enough quicklime to furnish exactly 2 per cent of CaO to the weight of quartz. The weighing out was done on the basis of an analysis, thus, the lime contains only 90 per cent CaO, proportionately more was added. The lime was then placed in a small pan mixer; slaked and stirred for homogenisation; and then run through a screen of approximately 10 meshes per linear inch on its way to the wet pan.

3.4.6 Shaping, pressing and moulding

After the semi-dried well mixing (tempered mass), the semi dried bricks were well moulded; the brick moulds was made of steel and consist of a bottom plate on which lies a 3-4 brick form as shown on Plate 3.4. The moulded bricks were pressed with a pressure of (300kN) in a hydraulic pressing machine for proper compaction, shaping the brick into rectangular shape and to further minimize the moisture content. The moulded silica bricks were left at ambient condition to attain completely dried bricks, without cracking.

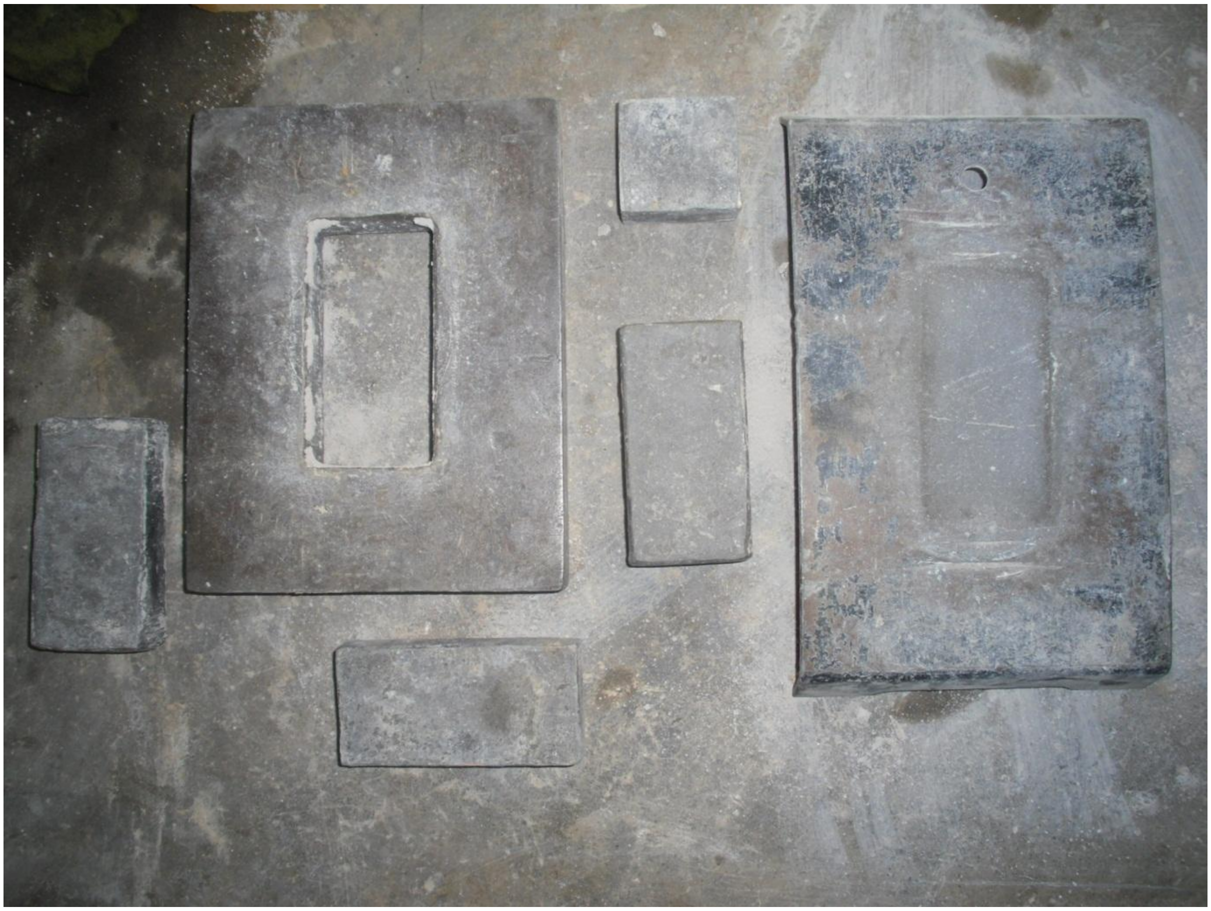

Plate 3.4 Steel mould(NMDC Jos)

3.4.7 Drying

The pressed bricks were allowed to dry for 72 hours under ambient temperature condition; the dried bricks were further exposed to heat in an oven to a temperature of 110^{o}C for 24 hours. This is to expel any moisture left in the bricks and to avoid cracks during firing.

3.4.8 Firing

The silica bricks were set into the furnace with approximately half inch space were left between the bricks to allow for expansion. They were placed with enough space wide by the width of the furnace and by the height of the furnace. This was done to prevent unnecessary cracking during brick expansion. The silica brick samples were fired at 1450^{o}C as recommended by Muhammad (2009) for 12 hours. The rate at which temperature increases was carefully controlled up to 900^{o}C, the temperature was varied at the rate of 5^{o}Cper minute. The temperature regulation is vital so as to avoid cracks formation in the product as a result of volume changes in the quartz due to conversions that might take place at those temperature ranges. The fired bricks products were tested and investigated for refractory applications.

3.5 Mechanical, Physical and Microstructural Properties

The inspecting and testing of refractoriness is vital in ensuring the suitability of a refractory product for an end use. Provision of suitable controls at various levels of production will guarantee minimum quality fluctuation and ensure conformity of the product to the specific consumer specifications. During the cause of this research, the following testing methods were used for testing the refractory product:

3.5.1 Linear Drying shrinkage

Linear shrinkage usually occurs from drying or firing of refractories and the values is expressed in linear. In reporting linear shrinkage, there are two bases on which the calculation is made. If L_{fd} is the as – formed dimension and $L_d$is the dimension of the dried unfired piece, percentage linear drying shrinkage on the as – formed basis is given according to ASTM C179-14 (2014) as:

$$\%LDS_f = \frac{L_{fd} - L_d}{L_{fd}} \times 100 \tag{3.1}$$

LDS_f = Linear Drying Shrinkage as formed
L_{fd} = Length as formed
L_d = Length when dried

On the other hand, the percentage linear drying shrinkage on the dried basis is also given according to ASTM C179-14 (2014) as:

$$\%LDS_d = \frac{L_f - L_d}{L_d} \times 100 \tag{3.2}$$

LDS_d = Linear Drying Shrinkage on the dried
L_f = Length as formed
L_d = Length of the dried

3.5.2 Firing Expansion/shrinkage

Firing linear expansion or shrinkage of refractory is based on the desired green length or unfired dimension. If FL represents the fired dimension, percentage linear firing expansion or shrinkage is given according to ASTM C179-14 (2014) as:

$$\%\mathrm{LFE} = \frac{\mathrm{FL} - \mathrm{GL}}{\mathrm{FL}} \times 100 \tag{3.3}$$

GL = Green length
FL = Fired length
%LFE = Percentage Linear Fired Shrinkage

3.5.3 Apparent porosity

Boiling point method is adopted for this test; the test brick were cut from a burnt refractory brick by a cut off wheel from within its core and cleaned from any dust or loose particles adhering to its surface. The specimen was allowed to dry in an oven at 110^{o}C to a constant weight (*D*) with an accuracy of 0.1g. The dried specimen was suspended in distilled water such that the specimen did not touch the base or sides of the container. It was boiled for two hours while still suspended in water and was allowed to cool to room temperature and the weight (S) was noted. The specimen were removed from water and excess water were wiped off from its surface by lightly blotting with a wet towel, and it's weighed in air was noted (*W*). The apparent porosity (Pa) was determined in accordance with ASTM C20–00 (2015):

$$\text{Pa} = \frac{\text{W} - \text{D}}{\text{W} - \text{S}} \times 100 \tag{3.4}$$

Pa = Apparent porosity
D = Dry Weight of Sample
W = Weight of Sample suspended in air including moisture in its open pores.
S = Weight of sample suspended in water.

3.5.4 Bulk density

The bulk density of refractory indicates the level of firing which the brick receive and thus its degree of densification. The sample of the silica brick refractory was cut off and grounded smooth to spherical shape and size of about 2cm in diameter, it was weigh on a laboratory balance and the value of this weight was designated as W_1. 1000g of clean mercury was pour into a 100ml beaker and was place on the balance pan centrally under the saddle of Tecramicsdensometer. The balance was adjusted until the gram scale read zero (0). The saddle was lowered into the mercury by hand-wheel and lock with the bridge locking screw. The micrometer was adjusted for the pointer to touch the surface of the mercury, and the balance was tare back to zero. The adjustment of the saddle, micrometer and resetting the balance to zero was repeated until the pointer touches the surface of the mercury and the balance gram scale reads zero. The saddle was raised by means of the hand-wheel and the brick sample was placed on the surface of the mercury using tongs. The saddle was lowered to immersed the brick sample into the mercury and the bridge was locked with locking screw, the micrometer was then adjusted so as to touches the mercury surface. The balance was read with + or – 0.01g and the value obtain was represented by W_2. Finally, the volume of the brick sample was calculated by this formula:

$$V=\frac{W2}{d} \tag{3.5}$$

Where d = density of mercury.

Bulk Density is determined according to ASTM C179-14 (2014) was used to calculate the bulk density:

$$BD = \frac{W_1 d}{W_2} \tag{3.6}$$

W_1 = Dry Weight
W_2 = Weight Deep in Mercury (Hg)
d = Density of Mercury
BD = Bulk Density

3.5.5 Cold crushing strength (CCS)

This is the ability of a refractory material to withstand the structural load coming over it. Dense and fine grained refractories generally possess good crushing strength whereas porous and coarse grained refractories have poor crushing strength. The fired bricks were cut in cubic form with different dimensions and were tested for crushing strength, using hydraulic machine. The cut samples were placed on a flat block surface. A uniform load was applied axially by turning the hand wheel at a uniform rate until fracture occurs. The loads at which cracks appeared in the samples were recorded and the crushing strength was then calculated using the relationship below in accordance with ASTM C133-97 (2015):

$$\text{Cold Crushing Strength} = \frac{\text{Load applied (Kg)}}{\text{Area (cm}^2\text{)}} \tag{3.7}$$

3.5.6 Thermal shock resistance

Thermal shock resistance, also known as spalling resistance is a measure of the resistance of a refractory to sudden changes in temperature. Refractories were subjected to alternate cycles of heating and cooling and must not spall, crack or crumble to pieces. The fired dense brick sample of size 2.3cmx2.26cmx2.26cm was used. The thermal shock was carried out using the test rig in accordance with ASTM-C16-03 (2012). The test rig consists of a sample holding Table which is designed so that the heat transfer due to conduction is reduced to its minimum value. Stainless steel sheathed K-type insulated junction thermocouples of 0.5 mm in diameter and 150 mm long were used to measure the surface and the centre temperatures. These thermocouples can measure temperatures ranging from 0 to 1100^{o}C. The sample was placed in the test rig and maintained at about 900°C and soaked for 15 minutes, after which, it was air cooled and observed for any cracks. If no cracks were observed, it was returned to the furnace and the same process was repeated until the sample cracked. The number of cycles were noted and recorded

3.5.7 Microstructural Analysis

The scanning electron microscopy analysis was carried out on the sample of the brick to determine its morphology. The experiment was carryout using SEM (model Quarum with Serial number Q150RES) in the Department of Chemical Engineering Ahmadu Bello University, Zaria. The sample being insulating in nature does not conduct electrons therefore it was grounded with gold (to conduct electricity which will improve the resolution of the image) and was placed on the aluminium holder stub using a double sticky carbon tape and was allowed to dry on oven at 60^{o}C for 4 hours. The valves of the two nitrogen gas tanks were opened and the “Vent” button located at the display panel of the microscope table was pressed, after a clicked sound it takes about 1 minute to fill up the chamber with Nitrogen gas and lever on the bottom of the door was gently pulled up to open the chamber door.

The sample mounting stage was brought down by pressing the keyboard of the microscope control table, the sample holder stubs were placed into the mounting holes. With the aid of long screwdriver, the set screws for the mounting holes were well tight. Precautionary measures were taken in ensuring nothing gets into the door before it was closed, EVAC button was pressed to start the rotary pump and after 2 minutes the green light was displayed. After about 30-45 minutes the vacuum reaches proper level and the filament light of the SEM and the monitor were all turned ON. Dialled positions status were in order, the acceleration voltage was adjust to 10 kV. The filament was turned ON and the red light indicates that the filament has burned out and the high voltage was turned ON as well. Precaution is being taken to ensure that the filament emission, beam alignment, aperture alignment, and astigmatism were all in order. The lowest magnification (10x) was used to choose the TV scan mode and the track ball was used to find or locate the sample. The coarse focus switch was turned ON and it was used to change the working distance to 14 mm, the sample stage was slowly brought up by means on command key from the key board and the sample position (z-position) where the image focuses was located the record was noted and the coarse focus was turned OFF while the medium light was turned ON.

Slow scan mode was activated, spot size was switch to medium and the magnification was further increased 500x respectively. Variable button was pressed to open up a small variable window on the screen, the size of the screen was adjusted and it’s been overlaid in the region of interest. The image focus was within the small screen, the outer and inner focus ring was used to fine tone the focus. The scanning speed was adjusted to S1 to allow electronic imaging acquisition; the spirit icon was double clicked on the computer, file menu was clicked and folder name and mapping option were selected to normal resolution of 1024. The image acquire icon was selected to record the image and software takes the control over the SEM and monitor was freeze, no parameter was changed during the image recording and the image was finally saved on the image folder.

CHAPTER FOUR

4.0 RESULT

4.1 Chemical Composition of the Quartz

Table 4.1 represents the XRF analysis result of the quartz sample after beneficiation.

Table 4.1: XRF Analysis of the Quartz Samples

Composition	Parameters (in %) ND=None detectable
SiO_2	96.25
Al_2O_3	ND
Fe_2O_3	0.51
K_2O	ND
CaO	0.02
TiO_2	ND
V_2O_5	0.007
Cr_2O_3	0.18
MnO	0.02
CuO	0.009
As_2O_3	0.003
P_2O_5	ND
NiO	0.005
ZnO	0.009
PdO	0.03
Au	0.01
PbO	0.02
SO_3	ND
MgO	0.88
Ag_2O	0.01
L.O.I	2.037

Table 4.2

Particle size distribution in percentage	**Size**
6%	0.15 mm
65%	0.177 mm
25%	0.42 mm
2%	0.599 mm
2%	0.71 mm

4.2 Linear Drying Shrinkage

Data obtained fromlinear drying shrinkage(LDS) were the length of the bricks as formed (L_{fd}) and length of the bricks as when dried (L_d).

Length as formed =10cm and Length when dried=9.9

4.3 Calculation of Linear Drying Shrinkage

Using Equation (3.1) where L is in cm, the result for percentage linear drying shrinkage was calculated and is given below:

For Sample 1:

Length as formed =10cm

Length when dried=9.9

$$\%\text{LDS} = \frac{10 - 9.9}{10} \times 100 = 1\%$$

For Sample 2:

Length as formed =10cm

Length when dried=9.9

$$\%\text{LDS} = \frac{10 - 9.9}{10} \times 100 = 1\%$$

For Sample 2:

Length as formed =10cm

Length when dried=9.9

$$\%\text{LDS} = \frac{10 - 9.9}{10} \times 100 = 1\%$$

$$\text{Average} = \frac{1+1+1}{3} = 1\%$$

Table 4.3 Linear Drying Shrinkage of the Brick Samples

Test Bar No.	Initial Length (cm)	Final Length (cm)	Drying Shrinkage (%)
1	10	9.9	1
2	10	9.9	1
3	10	9.9	1
Average			1

4.4 Data Obtained from Linear Firing Expansion

Data obtained fromlinear firing expansion (LFE) were the green length of the bricks as (GL) and fired length of the bricks as (FL).

4.5 Calculation of Linear Firing Expansion

Using Equation (3.3) where L is in cm, the percentage linear firing expansion was calculated and is given below:

For Sample 1:

Green Length = 9.9

Fired Length = 10.2

$$\%\mathrm{LFE} = \frac{10.2 - 9.9}{10.2} \times 100 = 2.9\%$$

For Sample 2:

Green Length = 9.9

Fired Length = 10.2

$$\%\mathrm{LFE} = \frac{10.2 - 9.9}{10.2} \times 100 = 2.9\%$$

For Sample 3:

Green Length = 9.9

Fired Length = 10.2

$$\%\mathrm{LFE} = \frac{10.2 - 9.9}{10.2} \times 100 = 2.9\%$$

$$\text{Average} = \frac{2.9 + 2.9 + 2.9}{3} = 2.9\%$$

Table 4.4 Firing Expansion of the Brick Samples

Test Bar No.	Initial Length (cm)	Final Length (cm)	Firing Expansion (%)
1	9.9	10.2	2.9
2	9.9	10.2	2.9
3	9.9	10.2	2.9
Average			2.9

4.6 Produce Dense Refractory Brick

Plate 4.1 Surface Appearance of the Refractory Brick after Firing at 1450°C

4.7 Data Obtained from Apparent Porosity

4.8 Calculation of Apparent Porosity

The apparent porosity was calculated using Equation (3.4) is given below:

For Sample 1:

Dried weight D (g) = 8.66,

Wet weight W (g) = 10.57

Suspended weight S (g) = 5.12

$$\%\mathrm{Pa} = \frac{10.57 - 8.66}{10.57 - 5.12} X100 = 35.05\%$$

For Sample 2:

Dried weight D (g) = 10.72

Wet weight W (g) = 12.85

Suspended weight S (g) = 6.01

$$\%\mathrm{Pa} = \frac{12.85 - 10.72}{12.85 - 6.01} X100 = 31.14\%$$

$$\mathrm{Average} = \frac{35.05 + 31.14}{2} = 33.1\%$$

Table 4.5 Apparent Porosity of Brick Samples

Specimen No.	Weight of Specimen in gram (g)			Apparent Porosity (%)
	Dried (D)	Wet (W)	Suspended (S)	
1	8.66	10.57	5.12	35.05
2	10.72	12.85	6.01	31.14
Average				33.1

4.9 Data Obtained from Bulk Density

4.10 Calculation of Bulk Density

The bulk density was calculated using Equation (3.6) and is given below:

For Sample 1:

Dried weight W1 (g) = 7.91

Weight deep in mercury Hg W2 (g) = 70.25

Density of mercury (g/cm^3) at 25°C = 13.5337

$$\text{B.D} = \frac{7.91 \times 13.5337}{70.25} = 1.52$$

For Sample 2:

Dried weight W1 (g) = 8.59

Weight deep in mercury Hg W2 (g) = 64.85

Density of mercury (g/cm^3) at 26°C = 13.5312

$$\text{B.D} = \frac{8.59 \times 13.5312}{64.85} = 1.79$$

For Sample 3:

Dried weight W1 (g) = 8.59

Weight deep in mercury Hg W2 (g) = 64.85

Density of mercury (g/cm^3) at 26°C = 13.5312

$$\text{B.D} = \frac{8.59 \times 13.5312}{64.85} = 1.79$$

$$\text{Average B.D} = \frac{1.52 + 1.79 + 1.79}{3} = 1.70$$

Table 4.6 Bulk Density of Brick Samples

Specimen No.	Dry Weight (W_1) (g)	Weight Hg (W_2) (g)	Bulk Density (g/cm^3)
1	7.91	70.25	1.52
2	8.59	64.85	1.79
3	8.59	64.85	1.79
Average			1.70

4.11 Data Obtained from Cold Crushing Strength

4.12 Calculation of Cold Crushing Strength (C.C.S)

The cold crushing strength was calculated using Equation (3.7) is given below:

For Sample 1:

Length L (cm) = 2.4

Breath B (cm) = 2.5

Area A (cm^2) = 6.0

Force F (KN) = 12.4

Mass M (kg) = 1240

$$\text{C.C.S} = \frac{1240}{6} = 206.66.\text{kg/cm}^2$$

For Sample 2:

Length L (cm) = 2.5

Breath B (cm) = 2.6

Area A (cm^2) = 6.5

Force F (KN) = 12.9

Mass M (kg) = 1290

$$\text{C.C.S} = \frac{1290}{6.5} = 198.46\text{kg/cm}^2$$

$$\text{Average C.C.S} = \frac{206.67 + 198.46}{2}. = 202.56\text{kg/cm}^2$$

Table 4.7 Cold Crushing Strength of Brick Samples

Specimen	Length (cm)	Breadth (cm)	Area (cm^2)	Force (KN)	Mass (kg)	C.C.S (kg/cm^2)
1	2.4	2.5	6.0	12.4	1240	206.66
2	2.5	2.6	6.5	12.9	1290	198.46
Average						202.56

4.13 Thermal Shock Resistance

The result for thermal shock resistance showed that the number of heat cycles at900^oC before destruction was 9.

4.14 Microstructural Analysis

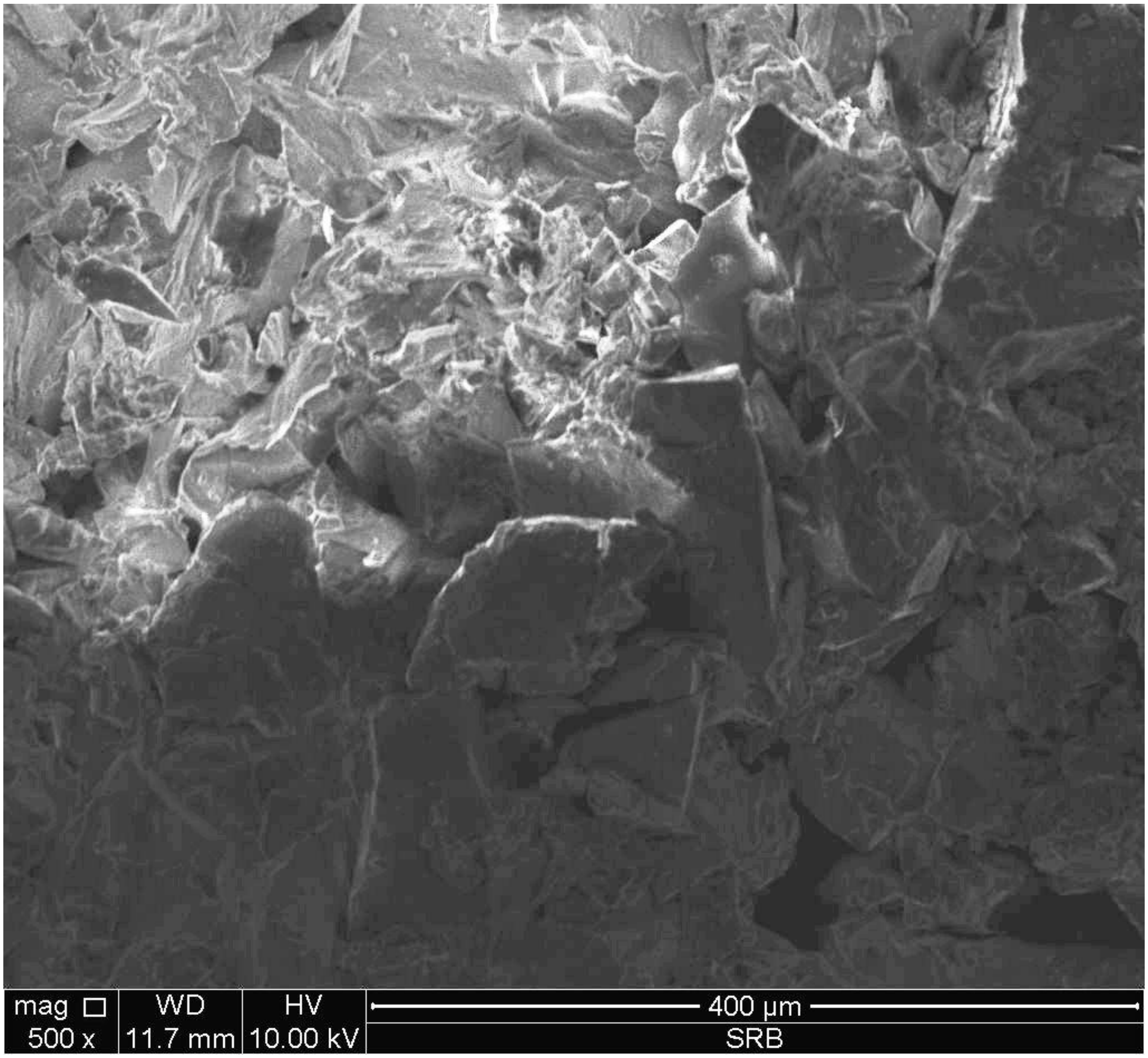

Plate 4.2 SEM micrograph of dense refractory brick after firing

CHAPTER FIVE

5.0 DISCUSSION

5.1 Chemical Composition

The result obtained from XRF analysis of the beneficiated quartz sample from AngwanDokaKokona in Table 4.1 indicated that the principal oxides present to be silica (SiO_2). The XRF analysis showed that the silica (SiO_2) content was 96.25% (Table 4.1). This value for silica content in the quartz sample makes it suitable for production of dense refractory brick. The total sum of impurities values summed together was 1.703% and it fall within the tolerable range for impurities associated to silica dense refractory which should not exit 4% ASTM (1982). The high percentage for SiO_2 (96.25%) coupled with the relatively low impurity contents of (1.703%) make the quartz suitable for the production of super-heat duty silica refractory material. This is in accordance with earlier works by (Chesti, 1994).

5.2 Linear Drying Shrinkage

The average linear drying shrinkage of quartz sample was 1.0%. This is because the moisture content within the brick was drastically reduced during pressing and the drying was carried out with less than 0.2% moisture in the product. This value of 1.0% falls within the recommended drying shrinkage values of silica ASTM C179-14 (2014). The value is satisfactory, because much higher shrinkage value may result in warping and cracking of the brick during drying and that may hinder the maximum efficiency of the final product.

5.3 Linear Expansion of the Quartz Samples

The fired brick product showed a fired linear expansion value of 2.9%, this value falls within the range at which silica brick should expand to change in volume as reported by (Chesti, 1994). The linear change in volume due to drying is of little significance, since the value change with moisture content during casting.

5.4 The Surface Appearance of the Brick

The product did not develop any crack during and after firing. The silica bricks produced had a brownish colour with dot-spot all over the brick as observed in Plate 4.1. The dense silica brick contains the crystalline SiO_2 modifications cristobalite, tridymite and some residual quartz. During the firing process, the lime reacts with the finest quartzite components to form wollastonite ($CaO.SiO_2$). The matrix also contains very small quantities of calcium ferrite, hematite, magnetite, calcium olivine and calcium ferrous silicate, [$CaFe(SiO_3)_2$], which are formed from impurities. These crystalline phases are the reason for the discoloration and spot formation on the fired bricks.

The discoloration of brown reflex appearing in the dense refractory bricks, might be due to the combination of iron oxide (Fe_2O_3) with calcium oxide (CaO) forming calcium ferrite [$CaFe(SiO_3)_2$] and wollastonite($CaO.SiO_2$) respectively as reported by Chesti, (1994) andIspatguru, (2014).

5.5 Apparent Porosity

Apparent porosity values were determined using hydrostatic gravity test and high pressure mercury porosimetry. The pore structure of silica brick is open and it can be presumed that the average pore size is one of the important parameters for assessing the durability of the material (Nevrivova, 2014). Compressive strength is also important for the use of silica bricks in the construction of thermal aggregates. The average apparent porosity value of the bricks was 33.1% for the quartz sample. Hence the porosity of the brick does not fall within the acceptable level as recommended for silica bricks value of 17-25% (ASTM C20-00, 2015). The very high porosity value of the refractory bricks could be due to insufficient firing temperature of the furnace which could not go beyond 1450°C to allow proper sintering and densification of the brick. Thus firing the bricks to higher temperatures above 1450°C would allow for proper sintering and densification to take place. This could enhance achieved recommended range value of 17-25% porosity in silica dense refractory bricks. This is in accordance with earlier works by Chesti (1994); Ispatguru (2008) and ASTM C20-00 (2015).

5.6 Bulk Density

The average bulk density value of the bricks was 1.7g/cm^3 for the quartz sample, hence the bulk density of the brick fall within the acceptable range of 1.7-1.9g/cm^3 as recommended by (ASTM, 1982 and ASTM C20-00, 2015). The relatively average value of bulk density of the dense refractory brick could be due to the fact that the compaction pressure of 300 kN is sufficient to compress the brick during pressing process of the semi dried brick to maximum compaction pressure. This also indicate that the different particle size distribution of the materials yielded positive results in terms of bulk density and so also the plasticity of the bonding material (molasses) was adequately strong.

5.7 Cold Crushing Strength (CCS)

The average cold crushing strength value obtained was 202.56 kg/cm^2 for the brick. The CCS of the produced refractory bricks was not in conformity with ASTM standard for cold crushing strength value of 250 kg/cm^2 minimum for silica brick (ASTM, 1982). The decrease in cold crushing strength may be due to increase in porosity and to loosening of the structure which was caused byvolume changes within the brick as a result of incomplete conversion of tridymite and cristobalite due to temperature fluctuations during firing. The practical significance of this value is that the brick will be strong enough to withstand handling, transportation and abrasion in services. Thus, a higher firing temperature above 1450^{o}C is required to reduce the percentage on porosity and to improve on CCS.

5.8 Thermal Shock Resistance

The use of the heat-cycle method as a means of evaluating the spalling resistance of refractories is satisfactory for quality control purposes ASTM-C16-03 (2012). The result for thermal shock resistance showed that the bricks were heated for 30 minutes at 900°C in an electric furnace then was suddenly exposed to room temperature. After cooling, the specimens were re-fired in the furnace and left there for 15 minutes. The cycle of cooling and heating was repeated until the specimen broke apart.

The result of this test showed that the number of heat cycles at 900°C before destruction was 9. The bricks with high content of silica were more subject to volume changes due to the quartz conversion into its various forms. The volume changes which accompany their conversion lead to a loosening of the structure and to a lower resistance against thermal shock.

5.9 Microstructural Analysis

The SEM analysis showed that the fired silica brick contains the crystalline SiO_2 modifications cristobalite, tridymite and some residual quartz as observed on plate 4.2. During the firing process, the lime reacts with the finest quartzite components to form wollastonite ($CaO.SiO_2$). The matrix also contains very small quantities of calcium ferrous silicate, [$CaFe(SiO_3)2$], which are formed from impurities. The transformed coarse grain generally consists of cristobalite, a proportion of residual quartz corresponding to the degree of transformation and very little tridymite, whereas the fine grained matrix is enriched with tridymite, glass and wollastonite. Silica bricks with identical chemical composition can have differing mineralogical compositions and this can cause quite different behaviour during use. Therefore, it is not always sufficient to evaluate silica bricks solely by their chemical compositions; hence it is essential to also consider the degree of transformation (residual quartz content) and the thermal expansion behaviour of the bricks.

4.10 Findings

i. The chemical composition of the quartz from AngwanDokaKokonaNasarawa State showed that SiO_2 has the maximum value of about 96.25%. This implies that the material can be used in the production of dense refractory bricks. It also has a low level of impurities for other oxide contents summed together as 1.703%, and LOI value of 2.037.

ii. The brownish colour with dot spot tint on the produced dense refractory brick after firing at a temperature of 1450°C might be due to the presence of transitional oxides in trace amount.

iii. The dense refractory bricks showed no cracks as-formed, drying and after firing at 1450°C, but expand slightly due to the linear expansion nature of silica during various conversions at high temperatures.

iv. The dense refractory brick had an average apparent porosity value of 33.1% and an average bulk density of 1.7 g/cm^3. The relatively higher percentage in porosity could be due to inadequate proportional ratio of particle size distribution or insufficient firing temperature of (1450^oC).

v. The average cold crushing strengths value of 202.56 kg/cm^2 of the bricks did not meet standard values of 250 kg/cm^2 for silica bricks as recommended by (ASTM 1982). This means that for a high quality bricks to be produced from this quartz, the brick need to be fired to a higher temperature above 1450°C.

vi. The average thermal shock resistance value of 9 cycles at 900°C falls within the acceptable range for silica bricks.

vii. The value of 1.7 g/cm^3 for bulk density falls within the acceptable range of 1.7 - 1.9 g/cm^3 for bulk density. This shows that the application of 300 kN for compaction during the production of the brick was grossly adequate.

CHAPTER SIX

6.0 CONCLUSIONS AND RECOMMENDATIONS

This chapter entails the conclusions drawn from the aim and objectives of the research work and general recommendations were made by the researcher.

6.1 Conclusions

The present research was centred on the evaluation of quartz deposit in AngwanDokaKokonaNasarawa State for production of dense refractory bricks. From the evaluation carried out, the mineral quartz is readily available at aforementioned mining site. The samples were randomly collected after digging from different points at the mining site, beneficiated to improve on the purity and ready for XRF analysis. The result of XRF analysis have shown that the quartz meet up with the requirement in terms of SiO_2 content, the result also revealed that the SiO_2 was not only present in the quartz but it was the major oxide with maximum content of 96.25%. The undesirable Fe_2O_3 was 0.51%; Al_2O_3 and TiO_2 were not present in the quartz while the total sum of other oxides as impurity was 1.703%. This proves that the quartz is suitable for the production of dense refractory brick which is among the silica group of refractories. The beneficiated quartz blended well with the binding and mineralizing material (quicklime and molasses) and was moulded, shaped and pressed in hydraulic pressing device.

After complete drying and firing there was no visible crack on the bricks, which indicated the product to be silica brick because linear expansion has taken place instead of shrinking as clay base refractory product does. The relative increase in porosity (33.1%) and low value of CCS (202.56 kg/cm^2) drastically reduced the quality of the product. Therefore the resultant product of quartz from AngwanDokaKokonaNasarawa State can only be used in application where moderate strength and heat treatment operation is required such as lining of roofs, side walls, flue walls and sole flue in kiln, COB and arc furnace.

6.2 Recommendations

The results obtained has produced some important information on the characterization and suitability of quartz from AngwanDokaKokona as refractory materials and thus, the following recommendations are proposed for further investigation on the characterization of quartz from the location mentioned above.

i. The quartz should further be investigated to ascertain its refractoriness under load (R.U.L) and modulus of rupture (M.O.R).

ii. The quartz should further be investigated by firing the brick made from the quartz using different proportion of particle size distribution, so as to attain a higher bulk density of a dense brick and a relatively low percentage of porosity.

iii. The dense refractory brick should be subjected to application where moderate strength and heat treatment operation is required such as lining of roofs, side walls, flue walls and sole flue in kiln, COB and arc furnace.

iv. The quartz from AngwanDokaKokonaNasarawa State meet the ASTM C416-97 standard for classification of silica in terms of purity, therefore I highly recommend the quartz for production of high quality refractory bricks, optical glasses, ceramic glazing.

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APPENDICES

Appendix I Hydraulic Pressing machine

Appendix II   Laboratory electric furnace

Appendix III Roll crusher

Appendix IV Air-float separator

Appendix V Rapid magnetic separator

Appendix VI Satellite map of AngwanDokaKokona