# LOCAL RAW MATERIAL EXPLORATION FOR THE PRODUCTION OF REFRACTORY POTS FOR MELTING GLASS.

**BY**

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A PROJECT SUBMITTED TO THE DEPARTMENT OF INDUSTRIAL DESIGN, FACULTY OF ENVIRONMENTAL DESIGN,AHMADU BELLO UNIVERSITY, ZARIA. IN PARTIAL FULFILMENT FOR THE AWARD OF MASTER OF ARTS DEGREE (M.A. INDUSTRIAL DESIGN)

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## DECLARATION

I declare that the material presented in this thesis report is an original work. It has not been submitted in any other form, to any institution for the award of a degree, except here, to Ahmadu Bello University for the award of the Degree of Master's of Arts in Industrial Design (Ceramics). Works of other authors are duly referenced

CERTIFICATION

This Thesis entitled "Local Raw Material Exploration for the Production of Refractory Pots for Melting Glass" by Garkida, Adele Dzikwi, meets the regulations governing the award of the Degree of Master of Arts in Industrial Design (Ceramics), of Ahmadu Bello University, Zaria. It has this day been approved for its contribution to knowledge and literary presentation.

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# DEDICATION

This work is dedicated to all members of my family.

"To seeking knowledge there is no end......"

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## ABSTRACT

The melting of glass depends on the generation of high temperatures and suitable containers for the molten glass. Attempt is made to develop these containers for molten glass from local refractory clay materials comprising of Kaolin from Kankara Katsina State whose deposit has been found to be of economic value and ball clay from Bomo-village - Zaria, Kaduna State. Five different composite bodies were prepared from the local clay materials, and have been used to cast refractory pots which were fired and tested for the following properties; (i) Refractoriness (ii) Thermal Shock resistance and (iii) Resistance to chemical attack by molten glass.

All pots made were able to withstand high temperatures without crumbling although some showed higher crack formation. The refractory pots were able to withstand the chemical action of the molten glass of lead crystal and soda-lime silica glass batches. The composite body of 60% grog and 40% Kaolin was found to be the most suitable for the production of pots that would be used for melting glass, having showed the best results of the tests that were carried out, thus it is being recommended for the production of refractory pots for melting glass.

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## DEFINITION OF SPECIAL TERMS

**Abrasion:** Wearing away of the refractory by the rubbing action of the material in contact with it.

**Annealing:** A process of cooling of ceramics/glass for stress relaxing

**Ball clay:** Clay that has been dispersed by weathering agencies and deposited somewhere else.

**Bauxite:** Hydrated alumina, containing iron and silica as impurities

**Bogeys:** Trolleys

**Composite:** Body composition containing different materials or parts.

**Corrosion:** Act of weathering or wearing away of material caused by chemical reaction.

**Crucible:** Pot in which metal or glass is melted.

**Eutectic:** A composition having the lowest melting point in a series of two or more components

**Extant:** Still in existence.

**Furnace:** A thermal plant or thermal installation

**Flues:** Channels of hot waste gases.

**Grog:** Fired clay particles

**Kiln:** A furnace made of refractory clay materials for firing ceramic products.

**Levigation:** A method of refining clay by water flotation the heavier particles settling out.

Lead crystal glass: A cut glass ware used for table wares and characterised by high brilliance. It is composed of silicon oxide, alkali and high proportion of lead oxide.

Plasticity: The quality of clay that allows it to be manipulated and still maintain its shape without cracking or sagging.

Porosity: Condition that allows liquid to pass through

Pneumatic ramming: Worked or driven through by compressed air.

Pyrometer: Meter for measuring very high temperatures

Refractory: Hard to fuse.

Refractoriness: A term used as an index of the heat resulting properties of refractories.

Soaking: Wetting the composition bodies with water.

Soaking: Maintaining the firing of the refractories at the highest temperature reached for some hours to achieve a product of uniform properties.

Spatula: A tool with a wide, flat, flexible blade used for mixing or spreading various substance.

Soda-lime silica glass: The most common commercially produced and widely used family of glass, composed chiefly of silicon oxide, sodium oxide and calcium oxide.

Tongs: One of various kinds of hinged tool for taking and holding something.

# CHAPTER ONE

## 1.0 INTRODUCTION

Trial and error methods have for centuries been used in the making of glass. Over time, certain limits of silica-alumina refractories have come to be adopted for the production of containers for molten glass.

Scholes (1974) reports that the melting of glass is carried out at temperatures ranging from 1300-1600°C, and refractories make possible the process of glass melting, since they supply containers for the glass and walls for the retention of heat.

Chesti (1986) refers to refractories technologically as the materials employed for the construction of furnaces, flues, crucibles, pots etc. used in high temperature operations. According to Scholes (1974) the glass furnace demands of its refractories that they shall be able to withstand temperatures of 1620°C or even higher in some cases. While the interiors of the walls must be able to endure for long periods temperatures of this level, the walls themselves must be able to retain without too great loss, the heat of the furnace, and must be able to sustain the weight of its own bricks at high temperatures without appreciable settling or sagging.

The properties of a good refractory material would include:

i) High refractoriness - resistance to fusion and softening at the working temperature.

ii) High resistance to thermal fatigue - capacity to withstand sudden changes in temperature.

iii High resistance to abrasion - ability to resist the rubbing action of the material coming in contact with it.

iv. High resistance to corrosion - inertness to the chemical action of metal, slag or gases when it comes in contact with it.

v. High refractoriness underload - resistance to crack, crumble or deform, under the prevailing conditions of pressure and temperature.

vi. High thermal conductivity - ability to promote heat transfer as in recuperative type of furnaces.

vii. Low thermal conductivity: ability to prevent heat losses from the furnace.

viii. Low coefficients of thermal expansion and contraction - ability to undergo the least structural change, on heating and cooling and resist disintegration.

No material is available which possesses all the above mentioned properties. Refractories with a given set of properties are chosen for a particular purpose to meet the specific service conditions and other special requirements. The generation of high melting temperatures demands that the furnace refractories must be of high quality if it were to last long for the production of glasses free from defects.

## 1.1 STATEMENT OF THE PROBLEM

The problem of this study is the development of suitable containers for molten glass, from local clay materials.

## 1.2 OBJECTIVES OF THE STUDY

The objectives of this study are:

i. To locate sources of suitable refractory clays

ii. To analyse the refractory clays in order to determine their chemical composition, thus their suitability.

iii. To produce different composite bodies that would withstand the melting of glass batches.

iv. To test the refractory bodies for the following properties

a. Resistance to chemical attack

b. Thermal shock resistivity

c. Refractoriness

v. To identify suitable composition for making refractory pots for melting glass.

## 1.3 LIMITATIONS

Inadequate facilities in the Department of Industrial Design for processing and treatment of raw materials constitute a limitation, likewise the lack of sufficient literature on glass technology and refractories in Nigeria.

## 1.4 DELIMITATIONS

The use of materials for the production of refractory pots for melting glass is delimited to Kaolin from Kankara, Katsina

State and Ball clay from Bomo-village, Zaria, Kaduna State. The scope of this study is based on easy accessibility of the materials.

## 1.5 BASIC ASSUMPTIONS

i. It is assumed that suitable raw materials would be found locally.

ii. It is assumed that all the available equipment are in working condition.

iii. Results of analysis of clay minerals taken at different positions would not differ.

## 1.6 SIGNIFICANCE OF THE STUDY

The production of glass depends on the generation of high temperatures and suitable containers for the molten glass. As such, it is necessary that there is a continuous search for improved processes and materials for containers for molten glass.

This study would determine the suitability of locally produced contact refractories for molten glass. The result obtained would determine the level of import substitution that is possible for a Nigerian based manufacturer. Presently, 100% of refractories are imported resulting in high production cost and a discouragement of growth of small scale glass factories.

## 1.7 BACKGROUND OF THE STUDY

Glass-making raw materials exists as rocks and grains, which must be melted before glass can be produced. The melting of glass is carried out at temperatures ranging from 1300-1600$^{o}$C. Scholes (1974) emphasises that the heat must be intense, because it facilitates the reactions between the ingredients of the batch and dissolves the silica. It also reduces the viscosity of the molten glass, allowing the bubbles or gas to escape.

It is therefore very necessary to have suitable containers that can withstand the melting temperatures of glass. Research on materials and processes in the manufacture of these containers, have used clays and high alumina base materials. Doyle (1979) asserts that clays occur in widely geographically scattered areas throughout the world. Likewise, the Geological survey of Nigeria (1992) reports the occurrence of refractory clay deposits at Bauchi, Edo, Kwara, Katsina and Sokoto States, yet contact refractories for melting glass is not developed.

Fwatmwol (1992) designed and constructed a double chamber furnace. The contact refractories used for the construction were supplied by West African Glass Industry (WAGI) Port Harcourt, who also imported them. He recommended an intensive research to be carried out in the area of refractories. To the authors knowledge, every glass company currently operating in the country imports its refractories.

## 1.8 HISTORICAL DEVELOPMENT OF GLASS MAKING POTS

Historically information on the making of furnace, crucibles and other tools of the glass makers trade has been handed down verbally as recorded by Singer et al (1958) Remains of glass work which belongs to premedieval times, have revealed no extensive evidence of furnace patterns. Descriptions of early medieval furnaces for the first and second firing and for annealing are however given. There is a contemporary drawing of an eleventh century furnaces, which were often in three storeys; fuel chamber, pot chamber and annealing oven. The remains of the furnace seem to conform to this pattern and these glass works are thought to have been founded by Egyptians. There is much archaeological evidence in Surrey and Sussex and elsewhere for the later medieval furnaces up to C1600.

As regards crucibles our evidence again begins mainly in medieval times, Petrie found at El-Amarna (Dynasty XVIII) fragments of clay crucibles about two or three inches deep and of the same diameter. Much longer crucibles must have been used elsewhere and is suggested by the size of ancient glass vessels and of extant masses of crude glass, one of which would require a crucible of 5000cc capacity according to singer et al (1958).

Gunther (1955) asserts that these crucibles had to be carefully prepared to withstand not only the intense heat of the fire, but the solvent action of the molten glass.

Open pots are the oldest form of containers for molten glass and the modern ones differ mainly in size from the crucibles of the ancients. Open pots are made from clay by hand moulding or by slip casting in plaster moulds. They are cylindrical in form or slightly tapering round or oval in cross-section. They range in height up to three feet and in diameter to five feet

CHAPTER TWO

# 2.0 SYNTHESIS OF RELATED LITERATURE

## 2.1 INTRODUCTION.

Refractories make possible the process of glass melting since they supply containers for the glass and walls for the retention of heat. The melting of glass is carried out at temperatures ranging from 1300-1600°C and it is expected that the refractories shall be able to withstand temperatures of 1620°C or even higher according to Scholes (1974). Alper (1970) identifies the most common refractories as comprising of oxides of aluminium, silicon, magnesium, calcium, chromium. These oxides can be used individually, but they are usually employed in the form of naturally occurring minerals in which they are associated. The natural ores are in many cases objectionable from the standpoints of refractory performance. Chesters (1973) reports that contact refractories for melting glass have been developed in other parts of the world and they have been grouped based on their alumina contents as shown in Table 2.1:

Table 2.1 Refractory Types By Alumina Content

| Group | Refractory Type | Percent Alumina |
|---|---|---|
| 1. | Fire clay | 25-45 |
| 2. | Sillimanite & other alumina-silica type mineral | 45-65 |
| 3 | Mullite | 65-75 |
| 4 | Bauxite based | 75-90 |
| 5 | Corundum | 90-100% |

These refractories are referred to as fireclay and high alumina refractories generally. They form the `Alumino-Silicate' group of refractories comprising mainly of two basic oxides $Al_2O_3$ and $SiO_2$; associated with impurity oxides of Ti $Fe_2O_3$, MgO CaO, $K_2O$ etc to the tune of about 2- 5% when put together. Appendix 1, figure 1, represents the equilibrium of various minerals of the $Al_2O_3$: $SiO_2$ system.

## 2.2 DEVELOPMENT AND USE OF REFRACTORIES

The alumina-silica phase diagram in appendix 1 figure 1 has wide application in refractories technology. Alper (1970) contributes that the diagram explains the thermochemical characteristic of many refractories whose compositions are substantially all alumina and/or silica. Clays are seldom pure and generally contain excess silica over that of the Kaolinite molecule, but many of them serve useful purposes as refractories in a wide range of industrial furnaces. These and other compositions that are substantially all alumina and silica can be looked upon as members of the system and much of their pyrochemical behavior can be attributed to the relationship expressed in the phase diagram. the system is characterised by its two end members, alumina and silica and an intermediate compound mullite having the formula $3Al_2O_3$. $2SiO_2$. There are two eutectics in the system, one between cristobalite and mullite, which melts at 1545°C and the other between mullite and corundum, which melts at 1850°C. Mullite takes a limited amount of these also into solid solution. compositions between mullite and cristobalite will develop liquid at 1545°C, as this eutectic contains only 5% alumina, it is evident that alumina is a powerful flux for silica.

A large number of aluminosilicate minerals including clays, whose compositions contain less than 72% alumina when calcined, fall between mullite and silica on this diagram. In this category several types of fireclays and kaolins containing up to 45% alumina when fired and minerals such as pyrophyllite, kyanite/ andalusite and sillimanite. Each of the last three contain 63% alumina. These refractories have varying uses owing to their impurities, but they certainly have useful applications.

The phase diagram shows that the melting points of compositions between 5-72% alumina increase with their alumina contents, however all compositions in this range will be liquid, in some degree, at temperatures above 1595°C. Any of these compositions that lie between mullite and corundum may therefore be used with assurance up to temperatures well above 1840°C.

Clays for refractories related to this system are generally used as mined. They therefore bring with them constituents other than alumina and silica which are indigenous to their natural environment. Titanium, iron oxide and the alkalies are the principal impurities. The principal effect of the alkalies is to lower the fusion point, even in quite pure raw materials such as kaolins. It is considered that the alkalies are a major problem in attaining high temperature stability and good load bearing ability in alumina-silica refractories. Equally severe, is the effect of alkalies on clay refractories when they are used in glass melting furnaces.

The iron oxide present in clay refractories should not be ignored, though present in relatively small percentages around 1.5-2% and may be as high as 3-4% in plastic fireclays that are less pure. Under oxidising condition, the iron oxide may not be seriously detrimental to the refractory, as liquid will not form below 1400°C (Appendix 2. figure 2). However, under reducing conditions, liquid will develop below 1150°C (Appendix 3 figure 3).

Most refractory clays are considered to have the approximate composition of the mineral Kaolinite ($Al_2O_3 . 2SiO_2 . 2H_2O$). When this mineral is fired, it first loses its water and further firing results in the formation of mullite and free silica as follows:

$$3(Al_2O_3 + 2SiO_2) = Al_2O_3 . 2SiO_2 + 4SiO_2$$

Glass-making and metallurgical processes require alumina-silica refractories that are more refractory and volume stable than clays without fortification by adding alumina.

## 2.3 RAW MATERIALS

While it seems fortunate that we are able to find in nature, in great quantities, materials which will ensure practically the refractory requirements, it is also somewhat disappointing that, in the scheme of things, there are only a few such materials and these have very definite limitations. These materials are almost entirely composed of fewer than a dozen elements combined as oxides.

The choice of materials is further narrowed to the truly refractory combinations of these oxides with the added provision that they should not colour the glass. Until recently glass makers were compelled to rely on silica, alumina and their compounds. Since the mid 1940's a third, Zirconia $ZrO_2$ which is now added to many refractories in common use, has brought about a marked improvement in service characteristics.

The oxides above in common with other rock formers, are readily soluble in glass and do indeed form definite additions to glass batches. Zirconia is also some-what soluble and in addition undergoes a destructive volume change due to phase transformation in the temperature range encountered in service. Chromic oxide, which would be an excellent ingredient for glass refractories because it dissolves very slowly in molten glass is generally excluded from consideration because it is a potent colouring oxide. Platinum is excluded for its cost, except for special purposes such as manufacture of optical glass. It becomes apparent that the problem of refractories, for glass containers especially, is a serious one. The selection of any material for a practical retaining wall for molten glass is at best a compromise between the ideal and the practical.

### 2.3.1 Clays

The type of plastic fireclay which enters very largely into the preparation of glass house refractories is chosen for several characteristics. Fundamentally it must be sufficiently refractory and must not soften completely below 1650°C. Another requirement is sufficient plasticity, hence it can be moulded into shapes when mixed with enough grog to permit ready drying and to prevent undue shrinkage. The clay must also contain enough fusible material so that vitrification will begin at a sufficiently low temperature to produce the required mechanical strength.

This process of vitrification is the formation of glass with a very high softening temperature. This permanent glassy bond between the refractory particles of a clay body is responsible for the utility of the body. It also limits its refractoriness as noted by Scholes,(1974).

Kingery (1980) describes some of the common types of refractory clays as follows:

i. Kaolin or china Clay; residual sedimentary, white burning, of poor to fairly good plasticity and dry strength.

ii. Fireclay; not white burning, but free from fluxes such as iron, alkaline earths, alkalies and excess silica.

iii. Ball clay; sedimentary, white burning clay of fine particle size, excellent plasticity and dry strength.

iv. Flint Clay, Non plastic, hard, refractory clay, having a low firing shrinkage.

#### 2.3.1.1. Kaolin ($Al_2O_3.2SiO_2.2H_2O$)

Kaolin or china clay is defined as a white powdery mineral which consists principally of micro-crystalline compound, approximating to the mineral Kaolinite ($AL_2O_3$. $2SiO_2$. $2H_2O$) It has a specific gravity of 2.6 and fusion point of about 1985$^oC$. kaolin is distinguished from other clays primarily by its whiteness, softness, ease of dispersion in water and other liquids and free from impurities, particularly iron compounds and its negligible content of titanium and alkaline earths. Pure Kaolin is analysed as 46% silica, 40% alumina, and 14% water. according to Johnstone and Johnstone(1961).

Generally clay minerals are formed from the decomposition of igneous rocks such as granite. Granite consists of Mica, quartz, and feldspar in approximately equal proportions. Ryan (1978) asserts that Feldspar is the least stable when exposed to the action of water and air. The process of Kaolinisation is from the decomposition or Kaolinisation of feldspar in the presence of air and water over long period of time. The potash and part of the silica in feldspar would have dissolved, the residue combining with water to give the clay mineral Kaolinite.

$$\underbrace{K_2O,\ Al_2O_3.\ 6SiO_2}_{\text{feldspar}} + 2H_2O = \underbrace{Al_2O_3\ 2SiO_2.\ 2H_2O}_{\text{kaolin}} + 4SiO_2$$

Kaolin is extremely refractory. Owing to their very simple mineralogy and the absence of colloidal inclusions of carbonaceous matter, residual China Clays are generally of low plasticity as explained by Sutherland (1987) There are known deposits of Kaolin or China Clay in various parts of the world and in Nigeria, there are deposits in Katsina, Edo, Kwara, Bauchi and Sokoto States. Nehikhare (1985) puts the reserves of the Kaolin deposit in Kankara (Katsina State) at 3,400.000 tonnes. The analysis of Kankara Kaolin is given by Umar (1988) and Hendsman (1979) in table 2.2;

Table 2.2 Analysis of Kankara Kaolin

| Compound | Formula | Percent |
|---|---|---|
| Silica | $SiO_2$ | 44.55% |
| Alumina | $Al_2O_3$ | 31.45% |
| Iron Oxide | $FeO$ | 1.07% |
| Magnesia | $MgO$ | 0.87% |
| Lime | $CaO$ | 0.28% |
| Sodium Oxide | $Na_2O$ | 0.17% |
| Potassium Oxide | $K_2O$ | 0.03% |
| Excess water | $H_2O+$ | 12.03% |
| Chemical water | $H_2O$ | 9.06% |
| Titanium Oxide | $TiO_2$ | 0.41% |
| Manganese | $MnO$ | 0.01% |
| Sulphur | $SO_4$ | 0.05% |

Kankara Kaolin which burns white has been found to have the following qualities by Gukas (1985) as shown in Table 2.3;

Table 2.3 Some other Properties of Kankara Kaolin

| Property | Measured value |
|---|---|
| Plasticity | Low |
| Dry Shrinkage | 6% |
| Bisque Shrinkage | 2% |
| Glaze Shrinkage | 6.5% |
| Total Shrinkage | 14.5% |
| Adsorption at Bisque | 13.5% |
| Adsorption at 1300oC | 12.5% |

Kaolin is a refractory material which on heating as pure Kaolinite, looses its water as steam at 450°C-500°C The dehydration product, metakaolin, has no binding power like that of the clay and hence the body so formed is weakened.

### 2.3.1.2 Fire Clay ($Al_2O_3$. $2SiO_2$. $2H_2O$).

Fireclays are naturally occurring earthy minerals. This was probably the first refractory material used by man. It occurs in most areas of the world and being plastic, when mixed with water, was found singularly useful for the production of crucibles for metal and glass manufacturers. Chesti (1986) agrees that the alumina content of fireclay generally ranges from 25-45% and they fuse at 1580-1750$^{o}$C The presence of alkalies, sand, gravel, calcium carbonate, iron oxide, calcium silicate, magnesium silicate, iron silicate, magnesium carbonate, iron sulphide etc. in fireclays seriously lower their fusion temperature and therefore fireclay should be free from them as much as possible. Alkalies and basic oxides are the most harmful elements because they form double silicates of lower refractoriness.

2.3.1.3 Ball Clay ($Al_2O_3$, $2SiO_2$, $2H_2O$).

Ball clays result from direct levigation and transportation of primary kaolin of pneumatolytic origin and impure sedimentary clays derived from the slow weathering of feldspars from a granite parent rock Cardew (1960) mentions. Ball clays are generally highly plastic and have particles ranging in size from 0.2 microns to 2.0 microns and they include, as do most angilic rocks, some colloidal material probably montmorillonite. The effect of montmorillonite in clays is to increase dramatically their green strength, plasticity and drying shrinkage. Many ball clays consist predominantly of disordered kaolinite together with some micaceous minerals and quartz, but it is important to note that the proportions of these minerals vary widely from one ball clay to another according to Sutherland (1987).

The following data on ball clay deposited at Bomo-Zaria, Kaduna State is given by Aluwong (1988).

| | |
|---|---|
| Plasticity | Medium |
| Shrinkage | 11% |
| Fusibility | Vitrifiable |

Using the Instrumental Neutron Activation Analysis (NAA) 26 elements were reportedly determined in Bomo ball clay. by Oladipo (1989), Akomolafe (1991),

Table 2.4 Results of NAA Reported for Bomo Ball Clay

| Element (ppm) | Conc. (ppm) | Element | Conc. (ppm) | Element | Conc |
|---|---|---|---|---|---|
| Na | 3499 | Sb | 0.03 | Cr | 188 |
| M | 5059 | Cs | 0.75 | Mn | 336 |
| Al | 7.49% | Ba | 75.4 | Fe | 0.91 |
| K | 1.50% | La | 92.0 | Co | 4.29 |
| Ca | 3932 | Ce | 170 | Rb | 6.07 |
| Sc | 15.5 | Sm | 15.1 | Lu | 9.07 |
| Ti | 3516 | Eu | 1.64 | Ta | 5.33 |
| V | 51.4 | Dy | 5.76 | Pa(Th) | 21.2 |
| U | 10.3 | Hf | 9.07 | | |

#### 2.3.1.4 Flint Clay

Flint clays are non-plastic fireclays. These flint clays may be used as refractory grog. They are used in fire bricks and certain special shapes not in contact with the glass. They seldom enter the composition of blocks or pots, Scholes (1974) presents..

### 2.3.2 High Alumina Containing Raw Materials.

These comprise of bauxite, bauxitic clays, silimanite, kyanite, and andalusite and even chemically produced alumina. Chemical compositions of some high alumina materials are given in Table 2.5.

Table 2.5. Composition of Some High Alumina Materials

| Composition | Type of clay (%) | | | |
|---|---|---|---|---|
| | Kyanite | Sillimanite | Bauxite | Diaspore |
| $Al_2O_3$ | 58-62 | 61 | 58-62 | 64 |
| $SiO_2$ | 34-37 | 36.5 | 1.2-2.6 | 25 |
| $Fe_2O_3$ | 0.3-0.8 | 1.1 | 1.4 | 0.75 |
| $TiO_2$ | 0.88-1.0 | 0.75 | 3.4 | 1.2 |
| CaO | - | 0.6 | - | - |
| Alkalies | - | - | - | - |
| L.O.I | 2.3-3.3 | - | 25-30 | 9 |
| Fusion Temp ($^{O}c$) | >1800 | >1800 | >1800 | >1800 |

#### 2.3.2.1. Sillimanite ($Al_2O_3$, $SiO_2$)

This mineral is in the form of a rock and may be used as mined or blended with clays to obtain intermediate compositions. (Gilchrist, 1977).

#### 2.3.2.2 Kyanite And Andalusite

These are identical to sillimanite chemically, but have different crystal structure. (Chesti, (1986).

#### 2.3.2.3 Bauxite ($Al_2O_3H_2O$ or $Al_2O_3.3H_2O$).

Bauxite is a hydrated alumina containing iron and silica as impurities. (Chesti, (1986).

### 2.4 POT MAKING

An ordinary batch for glass pots consists of about 40% raw plastic fireclay with 60% grog ground to pass a screen of 12 wires per mesh according to Scholes (1974). Grog is a term used for fired clay particles. A good refractory clay is hard fired in kilns and crushed to desired particle size. It is also made by crushing previously fired bricks and scrap bricks. Grog is added to the clay mainly as an anti-shrinkage element in the form of angular particles of various sizes to achieve better interlocking of grains. Rounded particles are avoided for their decreased bonding capacity.

There are several advantages associated with the addition of grog to clay. Some of these include; less shrinkage on heating, increased strength of the fired refractory, decreased apparent porosity and increased specific gravity.

There is also greater resistance to sudden changes in temperature and a requirement of less water to get a workable plasticity and lesser time required for drying the raw refractories; hence an increased rate of production. The quantity of grog added, ranges from 20-90% in the clay mixture and this depends on the properties of the raw clays and the desired properties of the finished products. High percentages of grog necessitate the use of highly plastic clays for the development of proper bonds.

Quality of high grog products can be improved by the use of:

a) Fine dispersion and thorough tempering of a highly plastic bond clay to ensure its proper distribution and effective binding by as thin layers of clay as possible.

b) Calcined materials of high grain density for improved strength and soundness of the product.

c) properly graded raw materials in the mixture to ensure highest packing density and reduction in the use of plastic clay for binding purposes. A proper sequence of charging various raw materials in the mixture ensures proper blending and uniform properties in the product.

### 2.4.1 Mixing

Finely crushed refractory clay is mixed with an appropriate quantity of the prepared grog and if necessary, with the bond clay or calcined bauxite. Mixing is carried out either in dry state or by wet method. In the former case different ingredients are taken in the requisite proportions, either on weight or on volume basis, and mixed. In wet mixing the ingredients are mixed with water in a pug. or pan-mill, before transferring to pug-mill, the mixture may be soaked in water for about 30-36 hours or more, so that the water is absorbed by the clay and distributed uniformly throughout the mass. After thorough mixing, the mixture is ready for moulding into desired shapes.

The mixture may be allowed to mature for a number of days in a cool cellar before moulding to increase it plasticity.

### 2.4.2 Moulding

Moulding is carried out either by hand, pneumatic ramming or by mechanical pressing. About 7-9% water is used in hand moulding mixtures and 4-7% in case of machine moulding mixtures. Lower moisture content in mixtures requires high moulding pressures. Lower moisture content in mixtures increases the slumping chances at the time of release from mould. Hand moulded refractories may not be of the appropriate size and may be less dense than those produced by machine or pneumatic ramming using 10 to 12% and 7.5 to 9.5% moisture respectively in the moulding mixtures. Machine moulding gives a high rate of production.

### 2.4.3 Drying

This is carried out either on large drying floors where the refractories are laid out in open array, or in tunnel kilns where they are stacked on bogeys and passed through a tunnel against a stream of hot air. This is the faster and more compact process but cannot be readily adopted where sizes and shapes are not fairly constant.

### 2.4.4. Firing

This is the final stage and it is carried out in batch or continuous type kilns. The kilns may be down draught, up draught or horizontal draught type. Tunnel kilns are used for mass production and chamber kilns are generally preferred for larger and complicated shapes.

Firing of refractories may be divided into five stages depending upon the temperature of the furnace and the changes that take place in the dried refractories. The first stage is known as steaming and it is the expulsion of moisture which has been left in the material after drying takes place. Most of the moisture is expelled until a temperature of about 120$^{o}$C is reached, however, the expulsion of chemically combined water continues as the temperature of the furnace increases. This stage is also known as smoking and lasts for about 20 hours.

Second stage is said to begin after a temperature of about 350$^{o}$C has been reached and extends up to 900$^{o}$C. It lasts for about 20 hours and results in the decomposition of clay. Any molecular water present is driven out, and the combustible and volatile matter is also expelled. The breakdown of the clay impurities takes place according to the following reactions (Nurton)

Table 2.6 Reaction temperatures of Clay Impurities.

| Reaction | Temperature $^oC$ |
|---|---|
| $S+O_2 = SO_2$ | 250-920 |
| $C + O_2 = CO_2$ | 350 |
| $FeS_2 + O_2 + FeS + SO_2$ | 450 |
| $MgCO_3 = MgO = CO_2$ | 400-900 |
| $4FeS + CO_2 = 2Fe_2O_3 + 4SO_2$ | 500-800 |
| $Fe_2(SO_4)_3 = Fe_2O_3 + 3SO$ | 560-775 |
| $CaCO_3 = CaO + CO_2$ | 600-1050 |
| $4FeCO_3 + O_2 = 2Fe_2O_3 + 4CO_2$ | 800 |
| $CaSO_4 + CaO + SO_3$ | 1250-1300 |

The third stage is the \`Oxidation' or \`Full fire Stage' it lasts for about 12 to 36 hours and extends up to a temperature of 1350$^o$C permitting various minerals transformations.

At a temperature of about 950$^o$C alumina begins to combine with silica to form mullite ($3Al_2O_3.2SiO_2$) and this is accompanied by the evolution of heat, being an exothermic reaction. Crystallization of mullite is completed between 1350 to 1380$^o$C. The clay shrinks and gets hardened and no shrinkage of the refractories takes place on subsequent heating or when in use.

The fourth stage, called the \`Incipient fusion' extends up to 1500$^o$C and is accompanied by the incipient fusion of the impurities and filling up of the interstices between the solid particles by the fused mass. As the pores are filled, bonding of the clay and grog particles takes place, which subsequently results in the production of compact and hard mass. Firing is finished at a temperature higher than that at which the refractories are to be used, the work is soaked at the highest temperature for some hours to achieve a product of uniform properties.

The fifth stage is known as annealing or cooling' stage. This involves a very slow cooling of the fired refractories in the Kiln itself. In batch type kilns the fire is put off and the damper closed, and heat is lost mostly by radiation. This cooling takes about the same time as in heating the goods to the maturing temperature. In continuous kilns, cooling takes place as the fired refractories move towards the discharge end. Low rates of firing are maintained for low grog refractories as they undergo higher shrinkages. High grog refractories on the other hand involve low shrinkage and therefore allow higher rates of firing. High alumina refractories involve high firing temperature of the order of 1500$^{o}$C - 1680$^{o}$C depending upon the percentage of alumina present therein. Firing temperature can be reduced by use of certain additives which on firing form liquid phases necessary to develop binding of refractory grains. However such refractories have reduced refractoriness underload.

## 2.5 THERMAL CHANGES IN REFRACTORIES

Refractories consist of aggregate and matrix. The purpose of firing of refractories is to adjust the forming of matrix to stabilize the sintering and assure simultaneously the quality of refractories.

Clay breaks down on heating to temperatures above 500$^{o}$C, as water is being expelled from the crystal, an amorphous material sometimes called metakaolin is formed, according to Chesters (1973).

Jay (1939) in order to build up standards, X-rayed a number of clay test pieces, ranging in type from the siliceous to the aluminus after firing in the temperature range 900°C-1400°C. His conclusions which do not appear to have been upset by any recent work are as follows:

1. Quartz in siliceous clays beginning to convert to cristobalite at about 1100°C and is not usually observable in the x-ray pattern after the 1300°C firing

ii. Gamma alumina is present at temperatures between 900°C and 1000°C, it is not observed at 1100°C or over.

iii. Cristobalite is first observed at 1100°C, as shown below. It is formed mostly from the free silica from the dissociated clay rather than from quartz.

$$3(Al_2O_3.2SiO_2.2H2O) = 3Al_2O_3.2SiO_2 + 4SiO_2 + 6H_2O$$

(Clay) (Mullite) (Cristobalite)

At higher temperatures (1300°C-1400°C), cristobalite tends to disappear with the appearance of a diffused reflection, suggesting glassy material.

iv. At 1100°C mullite is first observed, there does not appear to be any marked increase in the amount with increasing firing temperature. There is however distinct crystal development after the 1400°C firing as indicated by the sharpness of the line pattern, especially for the high order reflections.

In a Combined X-ray studies with electron micrographs of the products formed on heating kaolin by Comeforo et al (1948), it was shown that the non crystalline compound formed. When the water is expelled, it retains the hexagonal form of the original kaolin. At a higher temperature however, it collapses to yield crystal nuclei of mullite, which grow in the normal way. By thermal analysis, Y - alumina was observed between 960°C - 1000°C and if a suitable flux ($B_2O_3$) is present, mullite could be formed after long heating periods (200hrs) at temperatures as low as 900°C. Chesters (1973) explains that the mullite so formed is not by direct combination of amorphous alumina, but in the presence of mineralizer, by a reaction between Y-alumina and amorphous silica.

## 2.6 PROPERTIES

The properties of refractories vary with their composition and manufacturing processes. The adjustment of the dependent variables is done to produce refractories to suit specific applications (Chesters (1973).

i) Colour: This varies from light buff to reddish buff depending upon the iron content of the raw materials. The more the iron content the darker the colour.

ii) Slag Attack: Fire clay refractories are slightly acidic in nature. Hence they are not resistant to the action of basic slags, alkalies, molten fluxes etc. The finer the grain texture of the refractories, the better their resistance to the action of otherwise corrosive media.

iii) Chemical Activity: Fireclay refractories combine with salts such as chlorides, sulphates and carbonates or bases like lime magnesia, iron oxide ash, forming fusible aluminates, silicates and alumino - silicates.

iv) Porosity: This varies for fireclay refractories from 8 to 24% depending upon the grain fineness and the temperature of firing. if the temperature of firing is high, the incipient fusion of the impurities, is highly accelerated, producing compact, hard and less porous refractories. Amount and fineness of grog used also influences the porosity of these refractories.

v) Thermal Fatigue: Fireclay refractories have a low coefficient of thermal expansion and hence are resistant to thermal fatigue (spalling). Coarse textured and high grog containing refractories have a greater resistance to thermal fatigue than those produced from fine textured and low grog mixtures.

vi) Refractoriness: Softening point of fireclay refractories varies widely depending upon the type of clay, their alumina content and the type of grog used and correspond to PCE values of orton 31 to 34.

vii) Refractoriness Under Load (RUL). This depends on the chemical composition and physical structure of the material. Presence of impurities like iron and alkalies have a marked effect on their R.U.L. Fireclay refractories do not fall below 1350$^{o}$C under a load of 2 kgf/cm$^2$.

viii) Cold crushing strength: This is about 200 - 400kg/cm$^2$ depending on the composition, texture and firing temperature of the refractories. Cold crushing strength of a well fired refractory is higher than that of a soft fired one.

# CHAPTER THREE

## 3.0. PROCEDURE FOR COLLECTION AND TREATMENT OF DATA:

### 3.1 POPULATION:

Population of the study comprises of Kaolin or China clay and Ball clay. The choice of these materials is guided by their known physical, chemical and thermodynamics properties as obtained from available literature.

### 3.2 FIELD WORK

Location of samples was guided by information obtained from available literature, professionals in related fields and residents in the areas. Kaolin was procured in 50kg bags from Kankara, Katsina State and Ball clay was also procured in 50kg bags from Bomo-Zaria, Kaduna State.

### 3.3 SAMPLING

Representative samples of the whole material was obtained by coning and quartering methods of sampling. (Akomolafe (1991). Each sample was made into a heap divided into four parts and two opposite sides brought together while the remaining two were put away. This was done until the required size or weight of sample was obtained for further processes.

### 3.4 CHEMICAL ANALYSIS

Kaolin and Ball Clay from the same deposits as for the ones in this study have already been analysed and the results are stated in the previous chapter (Chapter two.) The analysis were carried out by Hendsman (1979) and Oladipo (1989) respectively. Hendsman's result put the value of alumina at

31.45%, hence it could be used to make contact refractories for melting glass.

### 3.5 TREATMENT AND PROCESSING OF RAW MATERIALS

The identified materials were processed as shown in table 3.1

Table 3.1 Treatment and Processing of Raw Materials.

| Kaolin | Ball Clay | Grog |
|---|---|---|
| Levigated | Levigated | Levigated |
| Dried | Dried | Fired |
| Crushed | Crushed | Crushed |
| Ground | Ground | Ground |
| Seived | Seived | Seived |

#### 3.5.1 **Kankara Kaolin.**

This was obtained in the form of a fine grain material with some lumps. The Kaolin was soaked for 3 weeks and levigated to reduce the excess free silica, so that the Kaolinite content would improve and also improve the clays plasticity. The levigated kaolin was poured into plaster moulds to dry. This was later sun dried, crushed, ground, and sieved in a 44 mesh.

#### 3.5.2. **Bomo-ball Clay.**

This was obtained in form of fine grain material with some lumps. The Ball clay was soaked for 3 weeks and levigated to remove all the free silica to obtain very fine clay particles to enhance its plasticity, since it would be used as a binder. The levigated clay was poured into plaster moulds to dry, later sun dried, crushed, ground and sieved in a 44 mesh.

### 3.5.3 **Grog.**

Some of the dried levigated kaolin was fired to 1280$^{o}$C to form grog. The grog was crushed, ground and sieved in a 22 mesh.

## 3.6 POT MAKING:

### 3.6.1 **Compositions:**

The following compositions were made as shown on the table below:

Table 3.2: Composition of Experimental Samples

| Sample | 1 | 2 | 3 | 4 | 5 |
|---|---|---|---|---|---|
| Kaolin | 50 | 40 | 30 | 20 | 10 |
| Grog | 50 | 60 | 70 | 80 | 90 |

Ball clay was added proportionately as thus; samples 1 and 2, 20% of total weight of each. Samples 3 and 4,22% and sample 5, 25% of the weight of sample, 0.6ml of water per gramme of composite body was added to make slips. The amount of grog added is taken from 50% and upward because the refractory pots when made are desired to have less shrinkage on firing, increased strength, decreased apparent porosity and increased specific gravity, and greater resistance to sudden changes in temperature since grog is normally added to improve these properties, the higher the grog content the better and so less than 50% of grog is not desired for this study.

### 3.6.2 **Mixing:**

The compositions as shown on Table 3.2 were mixed, soaked in water for 8 weeks, to improve the plasticity of the samples. The compositions were mixed thoroughly by stirring to facilitate the even distribution of fine to coarse particles in the whole mass.

3.6.3 **Moulding:**

Slipcasting method was used in the making of the pots. Two moulds were made from Plaster of Paris for this purpose. Five pots of each of the samples were casted.

3.6.3.1 Mould Making:

Two clay models were made. 4.5 litres of water was poured into a plastic container and 3kg of plaster was added to it and quickly stirred. This was used to cover the first mould. The plaster was allowed to set and the model was removed, the mould was washed and dried. The same procedure was repeated for the second model. but 7.5 litres of water and 5kg of plaster.

3.6.3.2 Slipcasting:

The liquid clay which was of thick syrupy consistency was poured into the hollow absorbent plaster moulds and within few minutes after pouring, a filter of firm clay appeared on the inner surface of the mould. When the desired thickness had developed, the mould was appended and the center portion which was still liquid poured out. The remaining clay coating continued to harden and eventually shrank away from the mould and was taken out. The process was repeated until all the prepared slip got exhausted.

3.6.4 **Drying**

The pots were dried to remove the moisture from them thereby increasing their strength and making them safe for subsequent handling. Drying rate was kept at a minimum to avoid higher shrinkage and crack formation. Drying was carried out under shade.

3.6.5 **Firing:**

The pots were fired in an electric kiln in stages under an oxidising atmosphere as follows;

STAGE I

This stage lasted for 5 hours and the firing was done slowly from 0-350$^{O}$C to expel the moisture.

STAGE 2

This stage also lasted for another 6 hours and the temperature was raised slowly from 350 - 900$^{O}$C. This would result in the decomposition of the clay, any molecular water present would be driven out and the combustible volatile matter expelled.

STAGE 3

This was full fired stage and it lasted for 8 hours from 900 - 1350$^{O}$C, this stage permits various minerals transformations.

STAGE 4

This stage was extended from 1350 - 1400$^{O}$C and it lasted for 2 hours, to bring about the incipient fusion of the impurities filling up of the interstices between the solid particles by fused mass. Bonding of the clay and grog takes place..

STAGE 5

This was the annealing stage, the kiln was put off and the fired pots were allowed to cool gradually in the kiln, mostly by radiation for 18 hours, to prevent the formation of cracks due to rapid cooling.

### 3.7 TESTING OF THE REFRACTORY POTS

The most important requirements of the refractory pots are:

i. Rigidity and maintenance of size, shape, strength at operating temperature which will be high.

ii. Ability to withstand thermal shock, such as is met in heating and cooling of furnaces or in fluctuations which occur during charging and during normal operation.

iii. Resistance to chemical attack by gas, slag or glass is likely to be encountered. For these requirements, the following tests were carried out on the refractory pots made.

#### 3.7.1 **Determination Of Refractoriness:**

This test was carried out to determine the softening temperature of the refractories to help in the selection to suit specific working temperature. One test piece from each was taken, crushed, ground and sieved using a 44 mesh. A 10g sample was drawn from this ground material using the standard method of sampling and was mixed with the desired quantity of water and glue for each sample. The mixture was then moulded into test cones and sintered to a temperature of 900°C so that the cones could be handled easily. Keeping in view the anticipated fusion temperature of the test cones, the cones were placed in a gas furnace and fired. A pyrometer was used to check the temperature at which each test cone would begin to bend and when it bent completely. The observation of the test cones was made through the peep-hole of the furnace. After cooling the cones were removed and examined.

### 3.7.2 Determination Of Resistance To Thermal Shock:

One test refractory pot from each of the composition made was taken, placed in a cold furnace and gradually heated until it attained the test temperature of 100°C in 3 hours. This temperature was maintained for 30 minutes after which the test pieces were removed with a prewarmed pair of tongs and cooled for 10 minutes on a brick floor in a position that was free from draughts. The test pieces were examined to note the presence of cracks. The test pieces were again recharged in the furnace and maintained at the temperature of the test for 10 minutes and were then cooled again for 10 minutes as above. These cycles of heating and cooling were repeated to note the appearance of cracks in the test pieces.

### 3.7.3 Determination of Resistance to Chemical Attack:

Two common glass compositions were prepared and used to carry out this test.

#### 3.7.3.1 Glass Batch Preparation:

Glass compositions for this study were based on lead crystal composition of; $SiO_2$ -54%, PbO - 35%, and $Na_2O$ - 11%. The soda-lime silica composition was; $SiO_2$ - 65%, $Na_2O$ - 25%, CaO - 7%, $Al_2O_3$ - 3%.

These batches were chosen because they are used in producing a wide variety of glass wares and their melting temperature can be reduced sufficiently while the glass still maintains its properties.

3.7.3.2 Melting of glass Batches:

The lead crystal glass and sodalime silica glass batches were made and melted in the refractory pots, in a gas kiln, at a temperature range of 1300 - 1350$^{o}$C, after which they were cooled gradually to ambient temperature, in the kiln.

3.7.3.3 Action of glass melt on Refractory pots:

The glass melts having cooled in the pots, were sectioned, the action of the glass melts on the refractory pots were examined.

## 3.8 IDENTIFICATION OF SUITABLE COMPOSITION FOR THE PRODUCTION OF REFRACTORY POTS FOR MELTING GLASS:

The results of the various compositions from formation to testing of the refractory pots were closely examined to identify which composition most suits the production of refractory pots.

### 3.8.1 **Chemical analysis:**

Chemical analysis on the most suitable refractory body composition was carried out using X-ray fluorescence method to determine its elemental composition and also the alumina-silica ratio by Neutron Activation Analysis.

## CHAPTER FOUR

### 4.0 ANALYSIS OF DATA:

#### 4.1 CHEMICAL ANALYSIS OF RAW MATERIALS:

The literature results of the analysis of Kankara Kaolin and Bomo-Ball clay are given below (Hendsman, (1979), Umar, (1988))

Table 4.1 Chemical Analysis of Kankara Kaolin

| Compound | formula | Results |
|---|---|---|
| Silica | $SiO_2$ | 44.55% |
| Alumina | $Al_2O_3$ | 31.45% |
| Iron oxide | FeO | 1.07 |
| Sodium oxide | $Na_2O$ | 0.17% |
| Potassium oxide | $K_2O$ | 0.03% |

These five oxides stated above have values that are within range for the production of refractory pots.

#### 4.2 MOULDING:

Since Kaolin is not plastic, Ball clay was added cautiously to avoid lowering the refractoriness. A range of 5 to 25% was added while pots were being casted. If the pots cracked, after excess slip was poured out of the moulds, the composition was then considered to be unsuitable. Calculated addition of the ball clay was done until the slip became castable for each composition, as thus: Samples 1 and 2 having 20% ball clay, Samples 3 and 4 having 22% ball clay and Sample 5 having 25% ball clay.

#### 4.3 DRYING:

Drying was done under shade. Ambient high humidity slowed the drying process and as a result non of the pots showed any crack formation.

## 4.4 FIRING

Eighty percent of each of samples 3, 4 and 5 survived the firing without any crack, while the 20% of samples 3 and 4 showed minor cracks, the 20% of sample 5 showed severe cracks.

Table 4.2 Percent of Crack on Experimental Samples

| Sample | % Crack |
|---|---|
| 1 | Nil |
| 2 | Nil |
| 3 | 20 |
| 4 | 20 |
| 5 | 20 |

## 4.5 DETERMINATION OF REFRACTORINESS:

The test cones were fired in a kiln, the maximum temperature obtained was 1350°C which was maintained for 2 hours. the test cones did not show any sign of bending. This is to say that the softening temperatures of these refractories are beyond 1350°C.

## 4.6 DETERMINATION OF RESISTANCE TO THERMAL SHOCK:

The samples went through 15 cycles successfully without any appearance of a crack, after which the experiment was stopped. the samples having survived 15 cycles should have good thermal shock resistance.

4.7 DETERMINATION OF RESISTANCE TO CHEMICAL ATTACK BY MOLTEN GLASS: The following plates show results of the action of the molten glass on the sample refractory pots.

ate 1. Sectioned pot made from sample 1 containing lead ystal glass melt. The molten glass does not show any attack the pot. Although there is a slight propagation of a crack, e molten glass only slightly sipped through it.

ate II: the above photograph shows a sectioned pot of sample
containing molten soda-lime silica glass. The molten glass
.ows no reaction on the pot.

ate III: This is a sectioned pot of sample 2 containing lead
ystal glass, which also has shown no effect on the refractory
:.

Plate IV: Sectioned pot of sample 2 containing soda-lime silica glass showing no reaction on the pot.

ıte V:  Sectioned pot of sample 3 containing lead crystal ıss. Although there is no reaction of the molten glass on the :, the pot shows some cracks formed during the firing of the .ss batch.

ate VI: Sectioned pot of sample 3 containing soda-lime silica
ass. The molten glass shows no reaction on the pot, but the
t shows further crack formations.

VII: Sectioned pot of sample 4 containing lead
l glass. The glass shows no reaction on the pot but the
s a large crack that allowed some glass melt to flow out.

te VIII: Sectioned pot of sample 4 containing soda-lime
.ca glass. The molten glass did not have any effect on the

ate IX: Sectioned pot of sample 5 containing lead crystal
ass: The molten glass has shown no reaction on the refractory
:, except that the wall of the pot has crack big enough to
: some molten glass flow out.

ıte X: Sectioned pot of sample 5 containing soda-lime silica ıss: The molten soda lime silica glass shows no chemical .ction on the refractory pot: The pot also has some cracks ·pagated.

4.8 IDENTICATION OF SUITABLE COMPOSITION FOR THE PRODUCTION OF REFRACTORY POT FOR MELTING GLASS:

Considering the results of the refractory pots made from drying, firing to melting of glass batches in the pots, it was observed that sample 2 which contained 40% Kaolin and 60% grog was the most suitable for the production of refractory pots to be used for melting glass.

4.9. CHEMICAL ANALYSIS:

4.9.1 **X-ray Flourecence**

The result of quantitative analysis of the sample body by the XRF technique carried out at the Centre for Energy Research and training ABU Zaria is given below.

Table 4.3 X-ray Fluorescence Analysis Result for Sample Body

| Element | Concentration |
|---|---|
| Al | 42.85+4.88 %W |
| Si | 60.75+4.68 %W |
| P | < 1829ppm |
| Cl | - |
| Ar | - |
| K | 33+ 0.18 %W |
| Ca | 3064.82 ppm |
| Ti | 4520.31ppm |
| V | 49.90ppm |

** Note: Analysis Method was by Direct Comparison of count rates of sample and standard.

4.9.2 **Neutron Activation Analysis Technique (NAA)**

At the Centre for Energy Research and Training, ABU.Zaria, the sample was analysed to determine the ratio of $Al_2O_3$ to $SiO_2$ via the NAA method. The results are presented as follows:

$Al_2O_3/SiO_2$ 30.47/64.33 %W

## CHAPTER FIVE

### 5.0 FINDINGS, CONCLUSION AND RECOMMENDATIONS:

### 5.1 FINDINGS:

The following findings have been made:

i. It was possible to produce refractory pots from castable slip that can withstand very high temperatures.

ii. All the refractory pots made from all the samples were able to withstand high temperatures without crumbling, except that some samples showed higher crack formations, which could be attributed to the amount of binder used and firing conditions.

iii It was found that to maintain a sequence in the amount of binder used in the composite bodies, it became necessary to reproduce pots of samples 5 containing 24% ball clay as binder against the already made containing 25% ball clay which was made and could not obtain the same firing conditions due to breakdown of equipment. Since the basis for comparison could not have been created, the pots earlier made of sample 5 containing 25% ball clay were considered as reported in chapters 3 and 4.

iv. The refractory pots made were able to withstand the chemical action of the molten glass of the lead-crystal glass and soda-lime silica glass batches.

v. Sample 2 with the composition of 60% grog and 40% kaolin was found to be the most suitable for the production of refractory pots that would be used for melting glass.

vi. Levigation of the clays improved their refractoriness.

vii. There was no effect on the varying thickness of the pots by the action of the molten glass since the pots were of slightly different thickness.

viii. The range of grain size of grog used in the study was smaller than those seen in the imported pots and they showed better interlocking after the firing.

ix. The alumina-silica content of the sample analysed shows that the refractory pots made fall within the group of fireclay refractories which have an alumina content ranging from 25-45%.

## 5.2 CONCLUSION

Local raw materials from Kankara and Bomo were found to be suitable for the production of refractory pots for melting glass, having been tested to have good resistance to chemical attack, high thermal shock resistivity and high refractoriness.

The composition of 60% grog and 40% Kaolin was found to be most suitable and its chemical analysis, which puts the alumina-silica ratio at 30.47 - 64.33 %W, places it within the group of fire clay refractories.

## 5.3 RECOMMENDATION:

Based on the operational performance of the refractory pots, the following recommendations are made;

i. The composite body made up of 60% grog and 40% kaolin is recommended for the production of refractory pots for melting glass.

ii. Composite bodies consisting of 70% grog and above could be used for making contact refractory bricks for glass melting tanks.

iii. The refractory pots are also recommended for studio glass practice, as it would enable the melting of different glass batches at the same time, especially when dealing with different colours of glass.

iv. Other refractory materials in Nigeria could also be explored, they could have better properties than the Kankara kaolin and Bomo Ball Clay used in this study.

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APPENDIX I

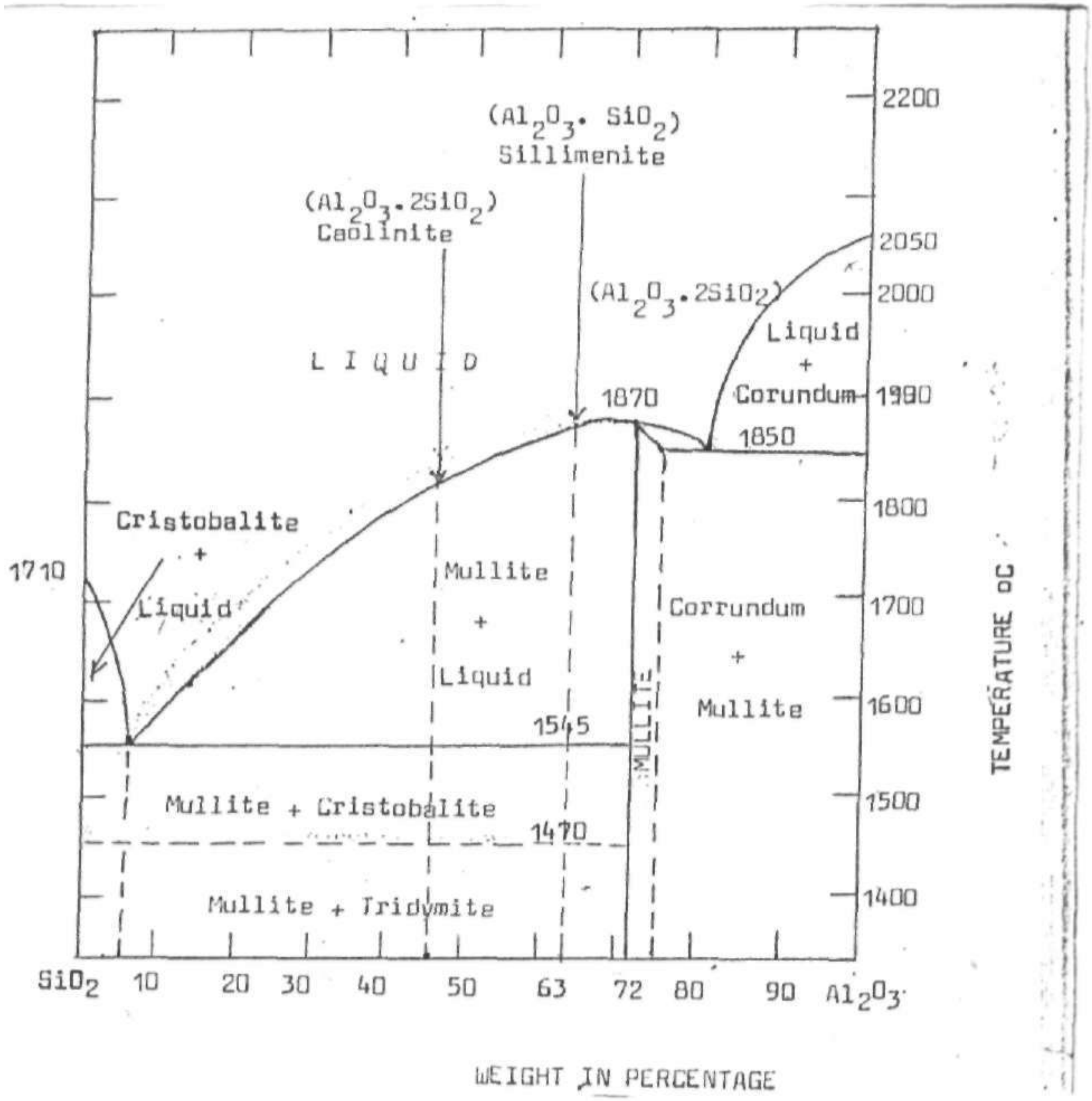


Figure 1: Silica-Alumina Phase Diagram

Adapted and Modified from Chesters (1973) Originally from Bowen and Greig Based on more recent data of Muan

## APPENDIX II

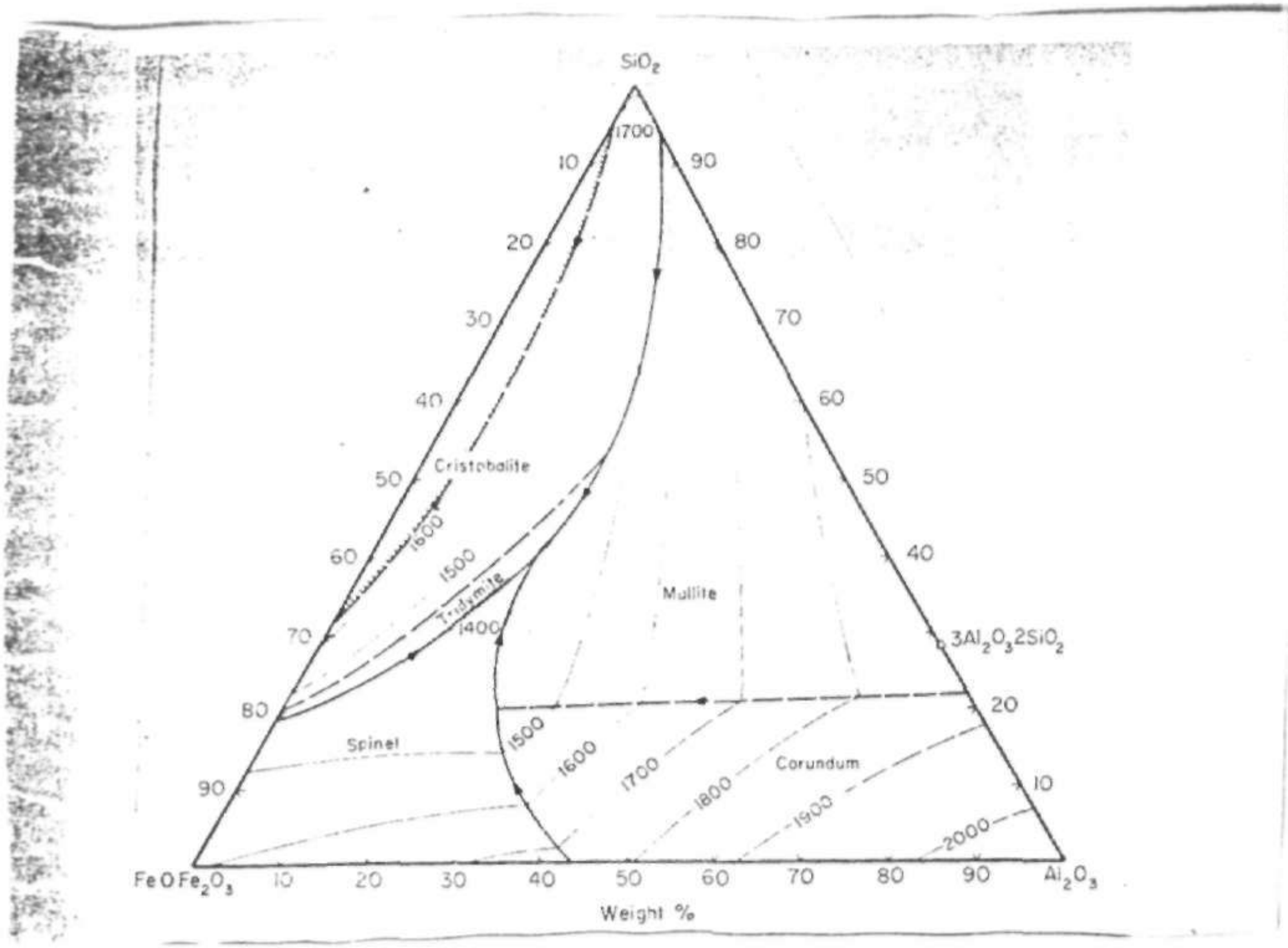


Fig: 2 Phase relations at liquidus temperatures in the system iron oxide-$Al_2O_3$-$SiO_2$ in air. Based mainly on Muan, (1957). Reprinted from Muan and Osborn (1965).

## APPENDIX III

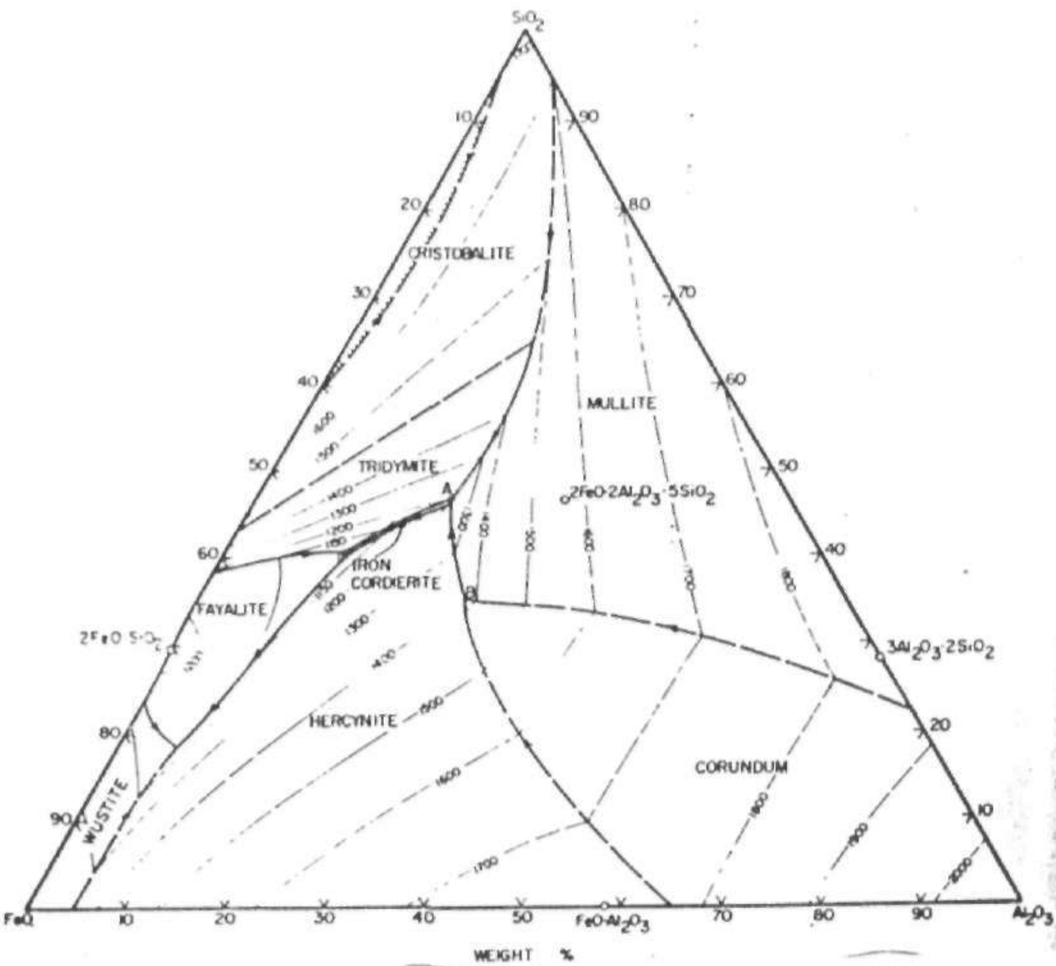


Fig. 3 The system $FeO-Al_2O_3-SiO_2$. (Bowen and Schairer, 1932)
Reprinted from Muan and Osborn (1965)