DETERMINATION OF THE SUITABILITY OF GROUNDNUT HUSK ASH AS A SOURCE OF FLUXING AGENT FOR GLASS MAKING

By

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DECLARATION

I declare that the work in this thesis entitled "DETERMINATION OF THE SUITABILITY OF GROUNDNUT HUSK ASH AS A SOURCE OF FLUXING AGENT FOR GLASS MAKING" has been carried out by me in the Department of Industrial Design. The information derived from literature has been duly acknowledged in the text and a list of references provided. No part of this thesis was previously presented for another degree or diploma at this or any other Institution.

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CERTIFICATION

This dissertation entitled "DETERMINATION OF THE SUITABILITY OF GROUNDNUT HUSK ASH AS A SOURCE OF FLUXING AGENT FOR GLASS MAKING" by Kazashim Seth AYUBA, meets the regulations governing the award of Master of Science in Glass Technology, 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 the Almighty God, the One who never fails, the Beginning and the Ending of all knowledge and wisdom.

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ABSTRACT

Agricultural waste like Groundnut Husk Ash(GHA) generated can inevitably affect the environment by the emission of methane and leachate when rotten. The evaluation of the possible utilization of agricultural waste is important in not only protecting the environment but also in minimizing the cost of disposal, conservation of land for other purposes and reduction in the use of other raw materials. Groundnut husk Ash as a source of fluxing agent for Glass making was experimentally investigated using the ashes from groundnut husk sourced from Gumel, Kujama and Narayi in Kaduna State. The ash obtained from 100g of GH fromGumel, Kujama and Narayi was 2.00g, 2.20g and 1.80g respectively. The solubility test on each of the samples of GHA indicated that the ashes were fairly soluble in water. The XRF analysis carried out on the ash samples showed thatK_2O as obtained on the samples from Gumel, Kujama and Narayi were 46.2%, 32.7% and 33.94% and the CaO were 34.5%, 26.8% and 30.9% respectively. The batches formulated corresponding to Soda Lime Silicate glass container composition (SiO_2 70%K_2O 15%, Na_2O 2%, CaO 12%, MgO 1%, Fe_2O_3 0.03%) and test melted at about 1300^0C formed glass.10% solution of $HCl_{(aq)}$ was used for acid resistant test and 10% NaOH was used for Alkali resistant test. The test revealed the glass were resistant to both media and the densities of glass as obtained with the GHA samples from Gumel, Kujama and Narayi stood at 2.45gcm^{-3},2.47$gcm^{-3,}$, 2.48gcm^{-3} respectively when measured. SEM analysis of the glass samples showed the external morphology while the XRD analysis showed a broad peak zone but absence of sharp Bragg's peak that signified the amorphous nature of the glass melts. From the result of analysis carried out on GHA and the test carried out on the formed glass, it has shown that GHA is a suitable source of fluxing agent for glass making.

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CHAPTER ONE

1.0 INTRODUCTION

In recent years, agricultural, industrial, and economic growth has been on a rise which is contributing to an improved quality and well-being of citizens.Due to the increase in the world's population and most of it moving to urban cities, there is increased demand for food, and this has resulted in the production of large amounts of agricultural wastes (Sabiti, 2011). However, wastes and by-products are created by the production systems which inevitably affect the environment. Some agricultural wastes emit methane and leachate when rotten, while some open burning by farmers to clear the lands, generates CO_2 and other local pollutants (UNEP, 2009).These wastes can be generated at any point in the production cycle, whether during obtaining the raw materials, transformation and production, or when the end user disposes the products which are no longer needed.

The enormous amount of waste generated is still far from being used in its totality, making technological alternatives imperative to reduce its possible environmental impact such as leaching of potentially toxic substances into soils and groundwater, reductions in plant establishment and growth due primarily to adverse chemical characteristics of the waste. Other effects are, changes in elemental composition of vegetation growing on the waste and an increased mobility as well as accumulation of potentially toxic elements throughout food chain.

According to Garkida (2002),waste prevention has economic benefits and reduces pollution. Generation of new products from recycled waste is thus an irreplaceable alternative.

The evaluation of the possible uses of agricultural waste is important not only from a scientific point of view but as a resource that can be extracted and used in the production of new materials. In addition to helping protect the environment, there are many reasons to increase the amount of waste being utilized. These include disposal costs are minimized, less area is reserved for disposal, thus enabling other uses of the land. There may be financial returns from the sale of the by-product or at least an offset of the processing and disposal costs. The use of such waste offers a series of advantages such as a reduction in the use of other raw materials, contributing to an economy of natural resources.A major advantage of agricultural residues is that they are widely distributed in the country; in parts of thecountry that cannot support abundant growth of trees, agricultural produce are cultivated (Ogunwusi, 2014). The high cost associated with raw materials extraction, as well as the damage that the extraction causes to the environment are important reasons to motivate the use of industrial / agricultural process waste.

Potassium salts were commonly used as flux in the melting of glass in the fifteenth century at which time the glass industry was well established in Bohemia. The Bohemian factories were generally located in the forests where wood fuel was cheap and a crude potassium carbonate was obtained by lixiviation of the wood ashes. But as the industry grew, potash was replaced to some extent by soda ash from Spain(Finn, 1938). Soda ash has been commonly used as a fluxing material in glass making.

Groundnut is an important cash crop produced in large quantity in Nigeria. The productions of ground nut reached its peak in Nigeria during the period of 1969 to 1971when approximately 1.7 million tonnes of the pods were produced from an area of 1.8million hectares (Elinwa and

Awari, 2001).According to the global food security index Nigeria is rated the 80th and among the top countries that produce protein food crops and the third in the production of groundnut after India and China (Baraka, 2014).

Groundnuthusk or hull is the shell of groundnut (peanut)left after the removal of the seed from the pod.The husk is used in most crop farms as residuewhere they decay and add nitrogen to the soil Okorie *et al.* (2013).Majority ofgroundnut husk are either burnt or allow to degrade naturally and advised that anumber of these agricultural residues can beutilized after a proper appraisal of theirchemical and physical properties(Faugdar& Diwaka, 1993).

The shell or husk is considered to be waste product with little or no use, after the groundnut is extracted, disposed off to prevent pollution. Currently, husk solids are used as fuel for fire. After combustion, about 5% ash by weight of husk is produced and since the ash does not have sufficient nutrients to be used as fertilizer, hence alternative means of disposing the ash is requiredAdole (2011). The utilization of groundnut husk will promote waste management at little cost, reduce pollution and increase the economic base of farmers when the waste is sold thereby encouraging more production,Ketukah (2006).

Groundnut husk as an agricultural waste its conversion to value added products makes it a secondary resource material. The analysis of groundnut husk ash (GHA) elemental composition revealed that it contains 15.46% K_2O (Adole, 2011).

The study of groundnut husk ash (GHA) may not be new; its application is seen in other areas such groundnut husk ash as a partial replacement of cement in mortar Ketukah (2006), effect of groundnut husk ash blended cement on chemical resistance of concrete Adole (2011), an investigation into the possible use of bagasse in glass making Tagwoi (2009).

1.1 Statement of the Problem

For many purposes, potassium oxide is employed to give a glass desirable working qualities. The potassium oxide glasses are more resistant to weathering than the sodium oxide glass. The high potassium and low silica content of this glass influences its chemical stability (De Bardi *et al*, 2015) which confirms that potash glass has more quality in terms of durability than soda glass. But due to cheap alternative which sodium carbonate provides and economic factors (cost of production), the use of potassium oxide as a flux has been abandoned which in turn discouraged the production of potash glass. The use of groundnut husk ash as a cheaper alternative source of potassium oxide will be explored in this study to tackle this problem.

1.2 Aim of the Study

The aim of this study is to utilize groundnut husk ash (GHA) as a source of fluxing agent for glass making.

1.3 Objectives of the Study

The objectives of this study are to;

i. Source the groundnut husks from the locations
ii. analyzing the chemical composition of the groundnut husk ash (GHA)

iii. formulate glass batch

iv. tests melt the formulated batches.

v. test for the physical, mechanical and chemical properties of the formed glass.

1.4 Research Questions

i. What effect will the chemical composition of GHA have on the glass to be produced?

ii. How can groundnut husk ash be used to flux a glass?

iii. How can the groundnut husk ash significantly lower the melting temperature of the glass?

iv. How can the formed glass be made to meet the required standards?

1.5 Basic Assumption

i. That the groundnut husk ash can be used as a flux in glass making.

ii. The chemical composition of the ash will have an effect on the glass to be produced.

iii. That the groundnut husk ash will lower the temperature for glass melting to occur.

1.6 Justification of the Study

The glass industry is the largest user of soda ash and almost all the bulk tonnage of glass produced in the world today uses soda ash as a source of its alkali-metal oxide (Doyle, 1994). The need for the promotion of indigenous technology and the consequent demands for diverse locally available raw materials at csompetitive prices make it necessary to introduce new kinds of raw materials into the manufacturing process Mulchlyonov et al., (1986).Theneed to promote the use of agricultural residues is being influenced by increasing agricultural production

andproductivity globally as man continually sought to improve the quality of life by transforming nature to providemore food, better living conditions and long life (Hall, Guo, Dore & Chow, 2009). An avenue can also be provided for complete utilization of raw materials. For example, oil which is extracted from the nuts for cooking and other applications, the husk which is used as fuel and the ash that results can be used in glass making. This study also proposes the reduction in environmental waste and also, the reduction in cost of procuring and processing raw materials.

1.7 Significance of the Study

The groundnut husk which is a residue after the nuts are removed can be completely utilized for production. Therefore, it will promote the complete utilization of residues for production.

Most waste is hurriedly disposed without proper consideration whether it contains resources that can be exploited or not. This study will serve as a means of exploiting the resources contained within waste that would have been lost.

It will reduce the negative environmental impact associated with landfill, thereby minimizing manufacturing cost and preserve non-renewable natural resources.

It will promote and expand the frontiers of knowledge, as more research can be done on this study and other related works. The study will promote the generation of wealth from agricultural wastes.

1.8 Scope of the Study

This research will utilize ground nut husk gotten from Narayi, Kujama and Gumel in Kaduna State, while the quartz as a source of silica will be obtainedfrom an already sourced, beneficiated and analysed sample from Kaduna State.

CHAPTER TWO

2.0 LITERATURE REVIEW

2.1 Glass and its Raw Materials

Glass is a unique material which combines the properties of both solids and liquids but are also distinctively different from each other and thus can be defined as a "supercooled liquid".

Glasses are usually formed by melting crystalline materials at a very high temperature and then cooling them 'rapidly' before the atoms get a chance to settle into a nice, well-ordered arrangement. It has been observed that with enough heat, 100% of the earth crust could be made into glass (Glenn, 2007).

Shelby (2005), a glass can be defined as an amorphous solid completely lacking in long range, periodic atomic arrangement with a time dependant ability to transform to a crystalline phase. Any material, inorganic, organic, or metallic formed by any technique, which exhibits glass transformation behavior is a glass.

Glass has three compositions: the formers, fluxes and stabilizers.Silica is the glass former. Silicon dioxide, also known as silica is a chemical compound that is a dioxide of silicon with the chemical formulaSiO_2. Silica is most commonly found in nature as quartz. Fluxes help the formers to melt at lower temperatures, some of which are soda, potash, lithium carbonate. Lime, litharge, alumina are used as stabilizers to keep the finished glass from dissolving or crumbling.

Some additives might be needed to add to the glass properties some of which are lead, boron, iron, copper.

2.1.1 Quartz

Quartz is the second most abundant mineral in the earth's crust, after feldsparRudnick (2003) and Barrie (2012). It is made up of a continuous framework of SiO_4silicon–oxygentetrahedra, with each oxygen being shared between two tetrahedra, giving an overall formula SiO_2. It is present in a wide range of variety, from colorless and transparent quartz (clear quartz) to colored (amethyst, citrine, rose) quartz. Natural quartz is a key raw material to produce fused silica, which is an important material for several high technology applications (Murilo, Santos, Egont, Jacinta & Carlos, 2015).Such applications includeceramic production, the manufacturing of crucibles used to produce solar grade silicon (Minami et al., 2011, Kodama et al., 2010 and Yamahara *etal.*, 2000) and in glass making; the production of transparent bulbs for UV light-induced water purification (Macaluso et al., 2009 and Barkhudarov et al., 2008). It is also used as abrasives and in the construction industry. High quartz sand used in glass making contains small amounts of impurities such as iron oxide, calcium oxide, aluminum oxide and magnesium oxide. It is the major glass former.

2.1.2 Silica sand

Silica is the main component in most commercial glasses and silica sand is the main raw material for this constituent (Simmingskold, 1997). This fine white sand is needed because it is very pure and does not contain other unwanted materials. It is a major glass former used in commercial

glasses. Silica sand is one of the abundant natural minerals which occur in several forms. The form of most significance to the glass industry is quartz.

The main sources of the silica sand needed to make clear glass are in Australia and South East Asia in countries like Indonesia, Malaysia and Vietnam. Silica sand deposits found in countries such as China, India and Egypt. The deposits in Britain and Ireland are mostlyused in the glass factories in the U.K. (Bryson, 2015). Nigeria has extensive deposits of good quality silica sands. Many of which are associated with the coastal plain of sedimentary areas in the southern part of the country, although deposits also occur in some inland areas. The major silica sand deposits in the country are located in Ughelli, Delta State, Igbokoda, Ondo State, Baure, Katsina State, Badagry, Lagos State, and along the sandy shoreline of the Atlantic, some inland deposits are also reported at Shebu, Plateau State and Ilaro, Ogun State. (Foraminifera, 2013)

Most glass quality sand is at least 99% $SiO_2$and the iron content (Fe_2O_3) must be less than 0.03%.Though float glass, colored container glass and fibre glass can tolerate higher iron content.Chemical analysis of glass quality sand generally involves quantitative spectrographic analysis (QSA) combined with atomic absorption spectroscopy (AAS).

2.1.3 Limestone ($CaCO_3$)

Limestone is a sedimentary rock composed primarily of calcium carbonate ($CaCO_3$) in the form of the mineral calcite. It most commonly forms in clear, warm, shallow marine waters. It is usually an organic sedimentary rock that forms from the accumulation of shell, coral, algal and fecal debris. It can also be a chemical sedimentary rock formed by the precipitation of calcium

carbonate from lake or ocean water (Geoscience news, 2016). It is either a high calcium ($CaCO_3$) or dolomitic [$CaMg(CO_3)_2$] product. Technically, dolomitic limestones contain less than the theoretical 46% MgO. Most of the glass industries use these materials in their carbonate form, although there are still some limited oxides, known as lime, quicklime, or dolomitic lime.

Limestone is by definition a rock that contains at least 50% calcium carbonate in the form of calcite by weight. All limestones contain at least a few percent other materials. These can be small particles of quartz, feldspar, clay minerals, pyrite, siderite and other minerals. The calcium carbonate content of limestone gives it a property that is often used in rock identification - it effervesces in contact with a cold solution of 5% hydrochloric acid.In glassmaking,Lime is an essential component of the glass as it reduces its solubility in water (Dieter, 2012)which acts as a stabilizer combining with the glass former and the flux.

When the carbonate form is used in glass batches, the CO_2 is driven off during melting. The CaO and MgO act as fluxes to hasten the melting of silica. The substitution of MgO for some of the CaO in soda lime silica glasses increases meltability, reduces the tendency for divitrification, and extends the working range

2.1.4 Soda ash (Na_2CO_3)

Sodium carbonate (also known as washing soda, soda ash and soda crystals), Na_2CO_3, is a sodiumsalt of carbonic acid (soluble in water). It most commonly occurs as a crystallineheptahydrate, which readily effloresces to form a white powder, the monohydrate. Pure sodium carbonate is a white, odorless powder that absorbs moisture from the air, has an alkaline taste, and forms a strongly alkaline water solution. The most common used quality is dense granular soda ash which should contain min. 99% Na_2CO_3 (min. 58%

Na_2O)(Simmingskold, 1997). Sodium carbonate is domestically well known for its everyday uses as a water softener. It can be extracted from the ashes of many plants growing in sodium-rich soils. It is synthetically produced in large quantities from salt (sodium chloride) and limestone by a method known as the Solvay process.

The manufacture of glass is one of the most important uses of sodium carbonate. Sodium carbonate acts as a flux for silica, lowering the melting point of the mixture to something achievable without special materials. This "soda glass" is mildly water soluble, so some calcium carbonate is added to the pre-melt mixture to make the glass produced insoluble. This type of glass is known as soda lime glass: "soda" for the sodium carbonate and "lime" for the calcium carbonate. Soda lime glass has been the most common form of glass for centuries.

2.1.5 Potassium Carbonate (K_2CO_3)

Main raw material for K_2O in glass. A white crystalline powder, very hygroscopic. Tends to harden even in closed containers(Simmingskold, 1997). Another source of K_2O is Potassium carbonate, hydrated ($K_2CO_3.15H_2O$): hydrated potash; pearl ash. It is white crystalline granular material and Less hygroscopic than the calcined salt.

2.1.6Finning agents

They are used to minimize bubbles, blisters and seeds in glass melts. These agents include sulfates, arsenic, antimony, fluorides, phosphates, and chlorides. Finning is a very complex process that is dependent on viscosity, composition, raw materials and redox conditions in the batch and melter.

2.1.7Colorants and decolorisers

These include selenium, cobalt, iron, manganese oxides, rare earth compounds, such as nickel, copper, cadmium. The most common decolorizing system involves the use of selenium and cobalt to mask the blue and yellow (green) color imparted to glass by iron impurities in the raw materials. The selenium pink complements the iron yellow, and the cobalt (blue) produces a neutral gray to the eye.

2.2 Types of Glass

2.2.1 Soda lime glass

This is one of the most common types of glass which is used for windowpanes and glass containers for beverages and food.

The raw materials for soda glass, which are sodium carbonate, lime, dolomite, silicon dioxide, aluminum oxide with small quantities of fining agents like sodium sulphate and sodium chloride, are heated in a glass furnace at 1675 °C temperatures. Color is added to the glass by using iron oxide, which provides green and brown color to glasses. However the chemical composition can be varied according to its uses.

2.2.2 Fused silica (vitreous) glass

A type of glass that is composed of purely silica (SiO_2). It has a very low thermal expansion and resists high temperatures which make it suitable in high temperature applications.

2.2.3 Alumino silicate glass

It is a type of glass which its composition consist of a relatively high amount of alumina (15-17%). It is extensively used for fibre glass, glass-reinforced plastics and halogen bulb glass.

2.2.4 Borosilicate (Pyrex glass)

Pyrex glasses were first introduced by Corning Incorporated in 1915. These types of glasses have high heat resistance due to the presence of boron oxide. Since boron oxide expands very little on heating, the glass does not crack on strong heating. Because of the presence of boron oxide, these types of glasses are also known as borosilicate glass (Werner, 1994). They are widely used in manufacturing laboratory equipment.Borosilicate glass is chemically resistant, has a low thermal expansion coefficient and can be used at relatively high temperatures. It is available in many forms and sizes such as rod, tube, plate and as machined or hot formed components (Goodfellow, 2013).

2.2.5 Lead crystal glass

In lead crystal glass, lead is added in the place of the calcium content of a typical potash glass. Generally, lead oxide is added to the raw materials of glass to make this type of glass. This glass contains 18–40 weight% lead (II) oxide (PbO). Due to the presence of lead content in the glass, it gets a high refractive index and maximum brilliance. Due to these characteristics, it sparkles and is used for many art objects and for making expensive glassware.

2.2.6 Potassium oxide glass

Potassium oxide glass contains potassium oxide and silica as major ingredients. They are more resistant to weathering than the sodium oxide glasses. Used in the manufacture of tubing for the electric lamp industry, illuminating glass ware and fine tableware (Finn, 1938).

2.3 Properties of Glass

Most glass is a mixture of silica obtained from beds of fine sand or from pulverized sandstone; an alkali to lower the melting point, usually a form of soda or, for finer glass, potash; lime as a

stabilizer; and cullet (waste glass) to assist in melting the mixture. The properties of glass are varied by adding other substances, commonly in the form of oxides, e.g., lead, for brilliance and weight; boron, for thermal and electrical resistance; barium, to increase the refractive index, as in optical glass; cerium, to absorb infrared rays; metallic oxides, to impart color; and manganese, for decolorizing (Glass, 2013).

2.3.1 Physical properties

Many physical properties of glass are "additive"in character: an additive property is one which depends not on only upon the oxide which a glass contains but also upon the relative amounts of each oxides present in the glass. The physical properties includes density and surface tension. Mendelev, *et.al.* (*2006*).

2.3.2 Optical properties

In the field of optics, the components utilized in imaging systems are mostly manufactured from glass. A faithful geometric and colour image of an object is generated by using the property of refraction (Alexander, 1998)

Glass is in widespread use largely due to the production of glass compositions that are transparent to visible wavelengths of light. In contrast, polycrystalline materials do not in general transmit visible light. The individual crystallites may be transparent, but their facets (grain boundaries) reflect or scatter light resulting in diffuse reflection. Glass does not contain the internal subdivisions associated with grain boundaries in polycrystals and hence does not scatter light in the same manner as a polycrystalline material. The surface of a glass is often smooth since during glass formation the molecules of the supercooled liquid are not forced to dispose in rigid crystal geometries and can follow surface tension, which imposes a microscopically smooth

surface. These properties, which give glass its clearness, can be retained even if glass is partially light-absorbing that is coloured(Glass, 2009).

These prominent optical properties are refractive index, dispersion and transmission. These properties are mainly determined by the chemical composition and to lesser degree, by the melting process and the subsequent thermal treatment. These procedures fix the molecular structure of glass which is responsible for the interaction with the electromagnetic waves (Alexander, 1998).Glass has the ability to refract, reflect, and transmit light following geometrical optics, without scattering it. It is used in the manufacture of lenses and windows. Common glass has a refraction index around 1.5. According to Fresnel equations, the reflectivity of a sheet of glass is about 4% per surface (at normal incidence in air), and the transmissivity of one element (two surfaces) is about 90%. Glass also finds application in optoelectronics—e.g., for light-transmitting optical fibers.

2.3.3 Thermal properties

The most important thermal property of glass is its rate of thermal expansion; the lower the rate, the greater the ability to withstand high temperature and sudden changes in temperature i.e the thermal shock of heat resistance. Thomas *et. al*(2005). Glass characteristically shows low thermal conductivity and high emissivity. Fused silica glass and quartz glass thermal expansion closely matches that of a metal. Thus, some glasses are used as seal to some particular metals. And when a seal is made at a high temperature where the glass softens, both glass and metal will shrink at the same rate and not develop high stress at the bonding surface. (Larger, *et. al; 1982*).

2.3.4 Chemical properties

Glass has an ability to relatively resist attack by chemicals. Due to its high corrosionresistance, concentrated phosphoric acids, concentrated alkalis can be safely kept in glass. Generally, the high silica, heat resisting glass shows the highest resistance to acids, whereas, some sodalime glasses show the highest resistance to alkalis (Werner, 1994).

2.3.5 Electrical properties

Glasses can be classified as electrical insulating materials. The electrical behaviour of glass is determined principally by four properties: insulation resistance, dielectric constant, dilectric loss and dilectric strength.

2.3.6 Mechanical properties

Glass behaves as a perfectly elastic material when it's under load because it has the ability to return to its original shape after deformation due to applied load or stress. The failure of glass always results from tensile stress. Glass can be strengthened by pre-stressing so that all surfaces are in compression, since failure mostly originates on the surface.

2.4 Batch Formulation

Glass batch calculation or glass batching is used to determine the correct mix of raw materials (batch) for a glass melt. Without chemical knowledge of the reactions which take place when the glass batch melts, it is impossible to calculate the amount of glass resulting from a certain batch (Shilvey, 1981). The weight of the raw materials used expresses glass composition. The chemical analysis of the raw material has little or no meaning unless the corresponding batch composition is also known Lyle (1981).

The raw materials mixture for glass melting is termed "batch". The batch must be measured properly to achieve a given, desired glass formulation. This batch calculation is based on the common linear regression equation:

$N_B = (B^T . B^T)^{-1} . B^T . N_G$(Glass Batch calculation, 2010).

With N_B and N_G being the molarities 1-column matrices of the batch and glass components respectively, and B being the batching matrix. The symbol "^{T}" stands for the matrix transpose operation, "$^{-1}$" indicates matrix inversion, and the sign "·" means the scalar product. From the molarities matrices N, percentages by weight (wt %) can easily be derived using the appropriate molar masses Khaimovich *et al;* (2005).

2.5 Fluxes and their Types

The introduction of alkali oxide to vitreous silica produces a weakening of the structure through the formation of non-bridging oxygen, therefore, lower elastic modulus. In contrast, the additions of Al_2O_3 or B_2O_3 to alkali silicate glasses have the effect of raising the modulus of elasticity, since the number of non-bridging oxygen is diminished. In general, one will anticipate that the strength of glass increases with increasing bonding strength of the glass structure. Therefore, glass strength increases with increasing B_2O_3 and CaO.

2.5.1 Lithia(Li_2O)

Lithia is a very effective flux, especially when used in conjunction with potash and soda feldspars. It is a valuable constituent in certain glasses having low thermal expansion because it permits the total alkali content to be kept at a minimum. Glasses containing lithia are much more

fluid in the molten state than those containing proportionate amount of sodium or potassium.Therefore, much smaller amounts are required to produce a glass of the necessary viscosity for working without sacrificing the desired physical and chemical properties.

2.5.2 Lime(CaO)

Lime offers stability, hardness, viscosity, strength, and facilitates melting and refining. Lime decreases the viscosity at high temperature, but increases the rate of "setting" in the working range. It greatly reduces the crushing strength when present in quantities > 12.9%. Lime will provide the highest tensile strength when it is properly mixed with soda and silica.

2.5. Boric Oxide (B_2O_3)

In general, those glasses containing the greatest amount of boric oxide show a minimum expansion, a property which is of great importance in obtaining thermal durability. In addition to its beneficial effects in melting, the presence of small amounts of borax in ordinary soda-lime-silica glass (resulting from use of borax in the batch) imparts greater brilliance, strength, durability and thermal shock resistance. It also decreases the tendency for glass to devitrify or crystallize. Boric oxide in the glass composition has been found to increase both the impact and tensile strength of glass containers. It also decreases the coefficient of expansion and increases rate of heat transfer and strength, all of which play important roles in thermal endurance.

2.5.4 Zinc oxide (ZnO)

Reduces the coefficient of thermal expansion, thus making possible the production of glass products of high resistance to thermal shock. It imparts high brilliance of luster and high stability against deformation under stress (i.e. higher elasticity). As a replacement flux for the

more soluble alkali constituents, it provides a viscosity curve of lower slope. Specific heat is decreased and conductivity increased by the substitution of zinc oxide for BaO and PbO.

2.6 Ash and Ashing Process

Ash is the products of firing, incineration or combustion. It can also be referred to as the solid remains of fire. Examples of ashes include: Ash, the compounds that remain after a sample is burned; commonly reported as a percentage.Bottom ash, products of coal combustion and cigar ash, the ash produced when a cigar is smoked, Fly ash, product of coal combustion., Incinerator bottom ash, a form of ash produced in incinerators, Vibhuti, the ash of cow dung and several other substances,Wood ash, products of wood combustion, Cremation, Ashes or remainsleft fromdried bone fragments. In practice, the type of ash varies considerably according to the burning technique (Opoku and Garkida, 2012).

2.6.1 Dry Ashing

Dry ashing procedures use a high temperature muffle furnace capable of maintaining temperatures of between 500^{o}C and 600^{o}C. Water and other volatile materials are vaporized and organic substances are burned in the presence of the oxygen in air to CO_2, H_2O and N_2. Most minerals are converted to oxides, sulfates, phosphates, chlorides or silicates. Although most minerals have fairly low volatility at these high temperatures, some are volatile and may be partially lost, *e.g.,* iron, lead and mercury. If an analysis is being carried out to determine the concentration of one of these substances then it is advisable to use an alternative ashing method that uses lower temperatures.

2.6.2 Wet Ashing

Wet ashing is primarily used in the preparation of samples for subsequent analysis of specific minerals. It breaks down and removes the organic matrix surrounding the minerals so that they are left in an aqueous solution. A dried ground food sample is usually weighed into a flask containing strong acids and oxidizing agents (*e.g.*, nitric, perchloric and/or sulfuric acids) and then heated. Heating is continued until the organic matter is completely digested, leaving only the mineral oxides in solution. The temperature and time used depends on the type of acids and oxidizing agents used. Typically, a digestion takes from 10 minutes to a few hours at temperatures of about 350^{o}C. The resulting solution can then be analyzed for specific minerals.

2.6.3 Low Temperature Plasma Ashing

A sample is placed into a glass chamber which is evacuated using a vacuum pump. A small amount of oxygen is pumped into the chamber and broken down to nascent oxygen by application of an electromagnetic radio frequency field. The organic matter in the sample is rapidly oxidized by the nascent oxygen and the moisture is evaporated because of the elevated temperatures. The relatively cool temperatures (< 150^{o}C) used in low-temperature plasma ashing cause less loss of volatile minerals than other methods.

2.7 Groundnut

Groundnut or peanut, a common name for an annual warm-season plant of the legume family, and for its seeds. The peanut belongs to the subfamily Papilionoideae, family Fabaceae (formerly Leguminosae) and is classified as *Arachis hypogaea.* (William, 2009).

Peanuts originated in South America, probably in Brazil, and have been cultivated since ancient times by Native Americans. Plants grow about 75 cm (about 30 in) tall and can spread 1.2 m (4 ft). Some types develop a bunchy erect growth; others, called runners, spread over the ground.

The peanut is unusual in that, after the flower is fertilized, the elongated receptacle, called the peg, turns downward from the base of the flower stalk to bury the ovary tip in the soil, where the fruit or pod develops.

Many types of peanuts exist, and both large- and small-seeded kinds are grown extensively in the world. Large-seeded varieties are used for roasting and confections and small-seeded types are used for peanut butter and oil. After peanuts are harvested, the plants are used as forage for livestock. The traditional use as fodder and grain feeds for cattle and hogs have declined.

Groundnuts require sunshine, a warm growing season of 120 to 140 days, and moderate rainfall. They do best on well-drained sandy soils; dark soils tend to stain the shells. Traditionally, production has been in the southern United States and in the warm-season areas of South America, Africa, and Asia.

Groundnuts are nutritious and high in energy. The seeds contain 40 to 50 percent oil and 20 to 30 percent protein, and they are an excellent source of B vitamins. Groundnuts are made into peanut butter, and can be sold as roasted peanuts. Peanut oil is also popular as a high-quality salad and cooking oil and is commonly used in margarine.

The leading peanut-producing countries, in order of production, are China, India, and the United States. In the United States peanuts are grown primarily for food; in other countries they are used principally for edible oil. Production in the United States averages 1.9 million metric tons

annually, about one-tenth of the world crop, with nearly half the United States crop grown in Georgia.

Groundnut is currently grown on over 22.2 million hectares worldwide with a total production of over 35 million tonnes. India and China are the world's largest producers of groundnuts, accounting for over 41% and over 18% of world production respectively. Millions of small-holder farmers in Sub-Saharan Africa grow groundnut as a food and cash crop, accounting for over 9 million hectares of cultivated farmland. Nigeria, Sudan and Senegal are Sub-Saharan Africa's leading producers, and the crop also does well in southern Mali and adjacent regions of Cote D'Ivoire, Burkina Faso and Ghana (IDP, 2014).

Even as India and China are the world's largest producers, they account for only a small part of international trade because most of their production is consumed domestically as peanut oil. Exports from India and China are equivalent to less than 4% of world trade. 90% of India's production is processed into peanut oil. Only a nominal amount of hand-picked select-grade peanuts are exported. The major exporters are the United States, Argentina, Sudan, Senegal, and Brazil. These five countries account for 71% of total world exports, with the United States being the major exporter. The major peanut importers are the European Union, Canada and Japan. These three areas account for 78% of the world's import.

2.8Waste Management

Waste can be defined as any garbage or refuse; sludge from a wastewater treatment plant, water supply treatment plant, or air pollution control facility; and other discarded material, including solid, liquid, semi-solid, or gaseous materials resulting from industrial, commercial, mining, and agricultural operations, and from community activities (Environmental Protection Agency,

2012). Waste is always a danger to the society if not properly managed. Its effects ranges from environmentalinclude air pollution, which includes odour, smoke, noise, dust, etc. Waste pollution – pollution from disposal sitevia flooding because of blocked drains and land degradation (Adewole 2009).

2.8.1 Agricultural Waste Management

The by-products of agricultural activities are usually referred to as "agricultural waste" because they are not the primary products. These wastes chiefly take the form of crop residues (residual stalks, straw, leaves, roots, husks, shells etcetera) and animal waste (manure). According to Sabiti *et al*(2005), agricultural wastes are widely available, renewable and virtually free, hence they can be an important resource. Thoughmany of the agricultural wastes are still largely underutilized, and left to rot or openly burned in the field, especially in developing countriesSabiti (2012).All these wastes are known to contain high nutrient levels of Nitrogen, Potassium, Phosphorus that would improve soil fertility and increase crop yields such as vegetables, maize that fetch high prices and hence enhance food security.This alternate method of utilization by farmers for agricultural production has also reduced the rate of accumulation, with subsequent reduction on environmental pollution thus improving environmental healthand will lead to diminished fears and preconceived notions of nuisance problems that decrease land values and environmental degradation Westerman (2005). Another way of managing waste is recycling.

2.8.2 Recycling

Recycling involves the collection, processing, and reuse of materials that would otherwise be thrown away. Materials ranging from precious metals to broken glass, from old newspapers to

plastic spoons, can be recycled. The recycling process reclaims the original material and uses it in new products. Roy (2009).

In general, using recycled materials to make new products costs less and requires less energy than using new materials. Recycling can also reduce pollution, either by reducing the demand for high-pollution alternatives or by minimizing the amount of pollution produced during the manufacturing process. Recycling decreases the amount of land needed for trash dumps by reducing the volume of discarded waste.

The recycling process involves three stages. In the first stage, the old products are collected and processed, where they are sorted, cleaned and made ready for recycling or manufacturing new products. The second stage involves the manufacturing of new products from the raw material obtained by the processing of the old products. Finally, the process ends with the purchasing of recycled goods by the consumers. The more people step forward to buy recycled products, the better the success of the process can be ensured. Buying recycled products will only increase when every individual will develop an awareness of the difference that can be made by utilizing their old household objects as raw materials to produce new goods and help the environment by recycling. (Paramita, 2011). Reasons for recycling includes;Resource conservation, energy conservation, pollution reduction, Land conservation, economic savings, saves environmental conditions and reduces pollution

2.9 Analytical Techniques

2.9.1 Atomic Absorption Spectroscopy (AAS)

Is a spectroanalytical procedure for the quantitative determination of chemical elements employing the absorption of optical radiation (light) by free atoms in the gaseous state.In analytical chemistry the technique is used for determining the concentration of a particular element (the analyte) in a sample to be analyzed. AAS can be used to determine over 70 different elements in solution or directly in solid samples. In order to analyze a sample for its atomic constituents, it has to be atomized. The atomizers most commonly used nowadays are flames and electrothermal (graphite tube) atomizers.

The technique makes use of absorption spectrometry to assess the concentration of an analyte in a sample. It requires standards with known analyte content to establish the relation between the measured absorbance and the analyte concentration and relies therefore on the Beer-Lambert Law. In short, the electrons of the atoms in the atomizer can be promoted to higher orbitals (excited state) for a short period of time (nanoseconds) by absorbing a defined quantity of energy (radiation of a given wavelength). This amount of energy, i.e., wavelength, is specific to a particular electron transition in a particular element. In general, each wavelength corresponds to only one element, and the width of an absorption line is only of the order of a few picometers (pm), which gives the technique its elemental selectivity. The radiation flux without a sample and with a sample in the atomizer is measured using a detector, and the ratio between the two values (the absorbance) is converted to analyte concentration or mass using the Beer-Lambert Law.

2.9.2 X-Ray Fluorescence (XRF)

It is a phenomenom that is widely used for element and chemical analysis. It involves the use of high energy X-rays or gama rays to bombard a material which emits a characteristic florescence or X-ray typical of the material. It is widely used in fields of archeology, forensic science and geochemistry for researches. Also for investigating building, metals, glass and ceramic materials.

Ionization occurs when these materials are exposed to X-rays or gamma rays. Ionization consists of the ejection of one or more electrons from the atom, and may occur if the atom is exposed to radiation with an energy greater than its ionization potential. These rays can be energetic enough to dislodge highly bonded electrons from the inner orbitals of the atom which renders the electronic structure of the atom unstable, making the electrons in higher orbitals "fall" into the lower orbital to fill the space left behind. During this falling, energy is released in the form of a photon, the energy of which is equal to the energy difference of the two orbitals involved. Thus, the material emits radiation, which has energy characteristic of the atoms present. This method is used as a non-destructive analytical technique, and as a process control tool in many extractive and processing industries.

2.9.3 Energy-Dispersive X-ray Spectroscopy (EDS, EDX, or XEDS)

It is an analytical technique used for the elemental analysis or chemical characterization of a sample. It relies on the investigation of an interaction of some source of X-ray excitation and a sample. Its characterization capabilities are due in large part to the fundamental principle that each element has a unique atomic structure allowing unique set of peaks on its X-ray spectrum. To stimulate the emission of characteristic X-rays from a specimen, a high-energy beam of charged particles such as electrons or protons, or a beam of X-rays, is focused into the sample

being studied. At rest, an atom within the sample contains ground state (or unexcited) electrons in discrete energy levels or electron shells bound to the nucleus. The incident beam may excite an electron in an inner shell, ejecting it from the shell while creating an electron hole where the electron was. An electron from an outer, higher-energy shell then fills the hole, and the difference in energy between the higher-energy shell and the lower energy shell may be released in the form of an X-ray. The number and energy of the X-rays emitted from a specimen can be measured by an energy-dispersive spectrometer. As the energy of the X-rays are characteristic of the difference in energy between the two shells, and of the atomic structure of the element from which they were emitted, this allows the elemental composition of the specimen to be measured. Four primary components of the EDS setup are

1. The excitation source (electron beam or x-ray beam)
2. The X-ray detector
3. The pulse processor
4. The analyzer.

2.9.4 X-ray Crystallography (Diffraction)

Itis a method used for determining the atomic and molecular structure of a crystal, in which the crystalline atoms cause a beam of X-rays to diffract into many specific directions. By measuring the angles and intensities of these diffracted beams, a crystallographer can produce a three-dimensional picture of the density of electrons within the crystal. From this electron density, the mean positions of the atoms in the crystal can be determined, as well as their chemical bonds, their disorder and various other information. X-ray crystallography is still the chief method for

characterizing the atomic structure of new materials and in discerning materials that appear similar by other experiments.

XRD is traditionally regarded as being of limited valuein determining mineral proportions (i.e. as a tool for quantitativemineralogical analysis) due to factors such as theinherent variability of some crystal structures, sample preparation,grain size effects, preferred orientation, and theabsorption of X-rays by other components in the mixture.For this reason XRD studies are often regarded as semiquantitative Vassilev (2002).The use of XRD as a quantitative tool has been considerably enhanced in recent years following development of purpose-specific computer processing systems based on the principles of full-profile XRD analysis developed by Rietveld Colin (2006).

In an X-ray diffraction measurement, a crystal is mounted on a goniometer and gradually rotated while being bombarded with X-rays, producing a diffraction pattern of regularly spaced spots known as reflections. The two-dimensional images taken at different rotations are converted into a three-dimensional model of the density of electrons within the crystal using the mathematical method of Fourier transforms, combined with chemical data known for the sample. Poor resolution (fuzziness) or even errors may result if the crystals are too small, or not uniform enough in their internal makeup.

2.10 Standard Tests

2.10.1 Glass density test

Density, a fundamental property of glass, it varies with changes in composition and thermal history. Density determined either by direct measurement or comparison.

Density Comparator (DC) is a laboratory device that provides fast, reliable measurement of glass density for routine control of glass composition. The Density Comparator determines the density of a glass sample by the sink-float method as described in ASTM Standard Method C729.

With the Density Comparator, the specimen of unknown density will be compared with a reference standard of known density. The specimen and the standard will be placed in a test tube containing a liquid of known density. The tube will then be immersed in a variable-temperature bath. Initially, the standard and specimen will both float in the solution. As the temperature rises, both the standard and specimen will sink or settle in the solution. As the standard and specimen reach a reference line on the test tube, the temperature of the liquid in the test tube will be measured. The temperature is then referenced to a table of Density Difference and the density of the specimen will be calculated.

2.10.2 Thermal shock resistance test

Thermal shock occurs when a thermal gradient causes different parts of an object to expand by different amounts. This differential expansion can be understood in terms of stress or of strain, equivalently. At some point, this stress can exceed the strength of the material, causing a crack to form. If nothing stops this crack from propagating through the material, it will cause the object's structure to fail.During a thermal shock, a transient temperature gradient occurs, inducing thermal stresses. The shock intensity is related to the level of temperature difference between the initial temperature and that of the environment Malou *et al.* (2013).

Experimental methods used for controlling thermal shock damage are numerous Hamidouche *et al.* (2003). The thermal shock resistance of a material is determined by the difference between the peak temperature of the material which was heated, rapidly cooled, and then fractured and that of the cooling media. Stresses are generated by temperature differences between the interior and surface of a test piece, which occur during rapid cooling. When this stresses exceed the strength of the material, fracturing occurs. These temperature differences are determined by the thermal conductivity of the material, as well as the coefficient of heat transfer between the material and the cooling media. The stresses generated are determined by multiplying young`s modulus, the coefficient of thermal expansion, and the temperature differences between the interior and surface of the material.

2.10.3 Resistance to Chemical Attack Test

The solubility of glass in contact with food, beverages, or pharmaceutical products is an important consideration for the safe packaging and storage of such materials. Autoclave conditions are specified since sterilization is often employed for the packaging of the product. It also represents one of the most extreme conditions, particularly of temperature, that containers will ordinarily experience. ASTM C255-85 (2009) gives the standard test methods for resistance of glass containers to chemical attack.

2.10.4 Water absorption test

Water absorption is used to determine the amount of water absorbed under specified conditions. Factors affecting water absorption include: type of plastic, additives used, temperature and length

of exposure. The data sheds light on the performance of the materials in water or humid environments.For the water absorption test, the procedure according to ASTM D570, the specimens are dried in an oven for a specified time and temperature and then placed in a desiccator to cool. Immediately upon cooling the specimens are weighed. The material is then emerged in water at agreed upon conditions, often 23°C for 24 hours or until equilibrium. Specimens are removed, patted dry with a lint free cloth, and weighed.

Water absorption is expressed as increase in weight percent.

Percent Water Absorption = [(Wet weight - Dry weight)/ Dry weight] x 100.

CHAPTER THREE

3.0 METHODOLOGY

The research methodology that was adopted for this study is experimental. The procedures followed were in stages. Sourcing of raw materials, beneficiation of the raw materials, chemical analysis of the raw materials, batch composition and formulation, melting and property tests.

3.1 Sampling

Groundnut husks that were collected from three different sites around southern parts of Kaduna namely: Gumel, Kujama and Narayi and denoted as sample A, Sample B and Sample C

respectively. The quartz used as source of silica was an already analysed one sourcedfrom Kaduna containing 98.69% SiO_2 .



Plate 3.1a Groundnut husk from Gumel



Plate 3.1b Groundnut husk from Kujama



Plate 3.1c Groundnut husk from Narayi

3.2 Pretreatment of Groundnut Husk

The sourced groundnut husk was gathered and sorted so as to remove spoilt husks and possibly the nuts that might still be in the husks, then washed to remove soil attached to the husk, and dried. The dried husk was pounded in a wooden mortar into a fine powder.

3.3 Ashing

The powdered husk was heaped in an enclosure where it was not be easily blown away by air due to its light weight and then it was set on fire in the open air to carbonize it. The carbonized husk was placed in a muffle furnaceat a temperature of 600^0C for 6 hours.The ash was then collected and sieved through a 0.09mm sieve (Alexander, 2009).



Plate 3.3a Groundnut husk ash from Gumel



Plate 3.3b Groundnut husk ash from Kujama



Plate 3.3c Groundnut husk from Narayi

3.4 Chemical Analysis of the Ash.

X-ray florescence (XRF) was used to determine the chemical element and elemental oxides of the ash. This analysis was carried out at the Nigerian Survey Agency; National Geosciences Research Laboratory (NGRL), Kaduna.

3.5 Loss on Ignition (LOI)

1g of each sample was weighed and poured into a crucible, which was then placed in an electric furnace and heated for an hour at 200^0C. The samples were subsequently cooled and weighed again. A constant weight was obtained after the process has been repeated. The difference in weight was calculated which represented loss on ignition of the samples.

Calculation

Weight of empty crucible (W_1) g = 71.9967g

W_1 + Sample A (W_2) g = 72.9967g

W_2 after ignition (W_3) g = 72.8950

Loss in weight (W_2-W_3) g = 0.1017

% LW $= 100 \times 0.1017 = 10.17\%$

The same procedure was followed for the other samples.

3.6 Solubility Test

5g of the sample was introduced into a test tube; distilled water was added and shaken until it was observed to have dissolved. It was then allowed to stand. It was observed that the ash sample dissolved but with some sediments seen. The same method was used in treating the other samples. (IS 13855:1993)

3.7 Batch Formulation

Table 3.7 Basis of calculation for glass container composition

Soda-lime	Composition Wt%
SiO_2	70-74
$Na_2O + K_2O$	13.0-16.0
CaO + MgO	10.0-13.0
BaO	0-0.5
Al_2O_3	1.5-2.5

Source: Glass bottle for carbonated soft drinks, voluntary product standard Ps 73- 89, U.S Department of commerce, National Institute of Standards and Technology. 1989

From table 3.7,the composition of soda lime silica was prepared which also served as basis for calculation of the batches (SiO_2 70%, K_2O 15%, Na_2O 2%, CaO 12%, MgO 1%, Fe_2O_3 0.03%).

Three batches were formulated from each of the three samples of the ash and it comprises of different composition based on the percentages of the oxides present in the ash samples. The sum total of individual oxide in each case was taken as the oxide composition of the batch. Silica sand was used as the major glass former while the ash was used to provide other supplementary oxides required in each batch since it has the required amount of the oxides needed. The approximation method was used in calculating the batch based on the percentage yield of the major oxides introduced by the various raw materials.

3.8 Test Melting

After the formulation and mixing, the batch was then placed in a crucible and fired to a temperature of about $1{,}300^0$C for 9 hours. The crucible was removed from the furnace after gradual lowering of the temperature to room temperature.

3.9 Property Tests

Property test of the formed glass was done to check if it conformed to the required standard needed for a glass.

3.9.1 Chemical Resistant Test

According to ASTM Method C-650, the glass samples were tested to determine their resistance to chemical attack. The resistance of the glasses to acidic and alkaline substance were tested with 10% solutions of $HCl_{(aq)}$ and $NaOH_{(aq)}$respectively for 24 hours at ambient temperatures. The samples were observed for color, weight and texture changes. This test was carried out at the department of Chemical Engineering, Ahmadu Bello University, Zaria.

3.9.2 Water absorption test

According to ASTM C 373, the test samples were oven dried at 150^0C followed by cooling in a desiccator where the dry masses (D) were determined to the nearest 0.01g. The samples were placed in a pan of distilled water and boiled for 5 hours and then allowed to soak for additional 24 hours. The samples were lightly blotted with a moisten cotton to remove excess water from the surface and the saturated mass (M) to the nearest 0.01g were determined.This test was carried out at the department of Chemical Engineering, Ahmadu Bello University, Zaria.

Calculations

Water Absorption $A = [\frac{M-D}{D}] \times 100$

Where

D= Dry mass of the sample

M= Saturated mass of the sample

3.9.3 Glass Density Measurement

According to ASTM C729, glass density was determined by direct measurement. Density is an important property of glass which varies with composition and thermal history SWGMAT (2004). The glass samples were weighed to determine their masses. 50ml of water was measured in a graduated cylindrical flask and the samples were gently immersed into the cylinder to measure their volumes. The weight of the sample in the fluid is equal to the volume of fluid displaced as described by Varshneya (1994). Their densities were determined using this relation

$$\text{Density} = \frac{Mass}{Volume}$$

This test was carried out at the Department of Building, Ahmadu Bello University, Zaria.

3.9.4 Scanning electron microscopy (SEM)

Scanning electron microscopy shows high resolution images of surfaces. The Microstructural morphology of selected spots on each of the glass samples were analyzed. This analysis was done with (SEM, Model:PhenomProx Instrument) at the Department of Chemical Engineering Ahmadu Bello University, Zaria.

3.9.5 X-ray Diffraction Analysis (XRD)

XRD is used to determine the atomic and molecular structure of a crystal by measuring the angles and intensities of diffracted beams of the rays. The glass samples were analyzed for crystalline phases with a Pan-analytical diffractotmeter (Model: Empyrean). This analysis was carried out at the Nigerian Survey Agency; National Geosciences Research Laboratory (NGRL), Kaduna.

CHAPTER FOUR

4.0 RESULTS AND DISCUSSON

4.1 Groundnut Husk Ashing

From the result of the groundnut husk ashing, the ash obtained from 100g of groundnut husk is 2.0g on the average (Table 4.1a). This implies that only 2% of ash will be obtained in every 100.00g of groundnut husks in other words, more groundnut husk will be needed to yield plenty ash.

Table 4.1a Weight of ash obtained by burning groundnut husk in a furnace

Samples	Wt of groundnut husk (g)	Wt of ash obtained (g)	% weight of ash
A	100.00	2.00	2.00
B	100.00	2.20	2.20
C	100.00	1.80	1.80

The percentage weight loss on the samples after ignition stands at 10.12% on the average (Table 4.1b). This shows that the volatile substance such as moisture and impurities constitute about 10% of the ash.

Table 4.1b Loss on ignition

Samples	%
A	10.17
B	10.08
C	10.12

From the solubility test (Table 4.1c); all the groundnut husk ash samples were fairly soluble which shows the absence of insoluble materials present in the ash as well as confirming that the beneficiation of the ash was properly done.

Table 4.1c Solubility test

Samples	Solubility
A	Fairly Soluble
B	Fairly Soluble
C	Fairly soluble

4.2 Chemical Analysis

From the result of XRF analysis, the percentage concentration of the elements present in the ash samples are in Table 4.2a – 4.2c

Table 4.2a Percentage composition of elements in sample A

Elements	%Composition

Si	12.20
K	19.06
Na	0.02
Ca	24.52
Mg	1.16
Fe	1.84

Table 4.2b Percentage composition of elements in sample B

Elements	%Composition
Si	27.54
K	13.48
Na	0.04
Ca	19.28
Mg	1.05
Fe	1.22

Table 4.2c Percentage composition of elements in sample C

Elements	%Composition
Si	7.45
K	14.08
Na	0.75

Ca	22.07
Mg	2.81
Fe	1.5

The percentage composition of the elemental oxides present in the ash samples are presented in Table 4.2d – 4.2f

Table 4.2d Percentage composition of elemental oxides in sample A

Elemental Oxides	%Composition
SiO_2	26.17
K_2O	46.20
Na_2O	0.09
CaO	34.5
MgO	2.65
Fe_2O_3	5.30

Table 4.2e Percentage composition of elemental oxidein sample B

Elemental Oxides	%Composition
SiO_2	27.69
K_2O	32.70
Na_2O	0.06

CaO	26.8
MgO	1.82
Fe_2O_3	3.79

Table 4.2f Percentage composition of elemental oxides in sample C

Elemental Oxides	%Composition
SiO_2	16.16
K_2O	33.94
Na_2O	2.06
CaO	30.9
MgO	3.80
Fe_2O_3	4.72

These results shows that ash from groundnut husks sourced from Gumel, Kujama and Narayi contains K_2O with 46.2% (19.06% K), 32.7% (13.48% K), 33.94% (14.08% K) respectively, making them good source of K_2O as a flux. Also, the CaO content of the ash is relatively high with 34.5% (24.52% Ca) from Gumel, 26.8% (19.28% Ca) from Kujama and 30.9% (22.07% Ca) from Narayi. Showing the Calcium content from the ash is sufficient enough to be a good source of CaO. The content of Fe_2O_3as observed in the three samples can be as a result of environmental factors or contaminations during the ashing process.

4.3 Batch Formulation and Composition

Batch formulation defines the amount in which various raw materials are used to produce a glass of the preferred composition. Different compositions of the GHA samples and other raw material were calculated by percentage weight to formulate a batch. The glass batch composition that was adopted was that of a typical glass container batch. From elemental oxide result, the high presence of potassium and calcium content informed the decision to use the sample as source of both the alkali (K_2O) and the stabilizer (CaO). It can also be seen that the oxides present in the ash samples differ with composition due to location difference and as such used in the formulation of glass batch as shown in Table 4.3.1 - 4.3.6. The glass batch formulated may be regarded as a three component system (K_2O- SiO_2-CaO).

Table 4.3a Summary of oxides introduced by 100g of glass containing GHA sample A to form batch 1

Wt/100g	SiO_2	K_2O	Na_2O	CaO	MgO	Fe_2O_3
Quartz	61.50	-	-	-	-	-
Sample A	8.50	15.00	0.03	11.20	0.86	1.72

Table 4.3b Weights of raw materials that form batch 1

Raw material	Ash	Quartz
Wt/100g	32.47	62.30

Table 4.3c Summary of oxides introduced by 100g of glass containing GHA sample B to form batch 2

Wt/100g	SiO_2	K_2O	Na_2O	CaO	MgO	Fe_2O_3

Quartz	57.30	-	-	-	-	-
Sample B	12.70	15.00	0.031	2.29	0.83	1.74

Table 4.3d Weights of raw materials that form batch 2

Raw material	Ash	Quartz
Wt/100g	45.87	58.06

Table 4.3e Summary of oxides introduced by 100g of glass containing GHA sample C to form batch 3

Wt/100g	SiO_2	K_2O	Na_2O	CaO	MgO	Fe_2O_3
Quartz	62.86	-	-	-	-	-
Sample C	7.14	15.00	0.91	13.65	1.86	2.09

Table 4.3f Weights of raw materials that form batch 3

Raw material	Ash	Quartz
Wt/100g	44.19	63.69

4.4 Test Melting

To determine the suitability of groundnut husk ash (GHA) as the main flux in the glass composition, a test melting was carried out. The raw materials were accurately weighed based on the information gotten from the chemical analysis of the samples and were thoroughly mixed for homogeneity in the glass. It was then transferred into crucibles for melting in a kerosene kiln at the Department of Industrial Design, Ceramics Section where the temperature reading from the pyrometer reached about $1{,}300^{0}$C. After the gradual lowering of temperature, the fired crucibles with the formed glass in it were removed.



Plate 4.4a Glass melt from sample A

Sample A as shown in Plate VII showed a homogeneous melt with little air pockets. However, the glass was colored which attested to the fact that sufficient temperature was attained which allowed proper reaction of the glass forming oxides during firing. Also the presence of impurities from iron and possibly other oxides and the reactions from the crucible may have coloured the glass.



Plate 4.4b Glass melt from Sample B

The melt, Sample B as shown in Plate VIII showed the presence of scum on the surface which usually results from rapid melting of the alkali. When the alkali melts too rapidly, it prevents the proper sequence of glass forming reactions to occur which results to foaming. This scum prevents heat from penetrating into the glass which hinders proper refining and homogenizing. There was however, the presence of gaseous inclusions (bubbles or air space) which its occurrence could be related to the improper fining of glass. It may also signify an incomplete melt. Colour was observed which could be attributed to the presence of impurities from iron and possibly other oxides. It also indicated that sufficient temperature was attained.



Plate 4.4c Glass melt from Sample C

Sample C as shown in Plate IX showed presence of plenty of gaseous inclusions (bubbles or air space) which may be related to improper fining of glass. Also, there was the presence of scum on the surface which may be the result of rapid melting of the alkali. Impurities from iron and possibly other oxides may also, account for the colour seen on the melt. It also indicates that sufficient temperature has been reached for melting of the glass batch.

4.5 Chemical and Physical Tests

Table 4.5aAcid Resistance Test (10% HCl)

Sample	Initial weight (g)	Final weight (g)	Difference	Changes
A	0.3352	0.3346	0.0006	No color change
B	0.6086	0.6000	0.0086	No color change
C	0.7412	0.7378	0.0034	No color change

The result of the acid test from Table 4.5.1 showed that the samples didn't react with the 10% solution of $HCl_{(aq)}$ because there was no color change and the weight difference were very negligible 0.0006, 0.0086, and 0.0034 for sample A, B and C respectively.

Table 4.5b Alkali Resistance Test (10%NaOH)

Sample	Initial weight (g)	Final weight (g)	Difference	Changes
A	0.2870	0.2861	0.0009	No color change
B	0.3128	0.3128	0.0000	No color change
C	0.3991	0.3966	0.0025	No color change

For the alkali test, the result from Table 4.5.2 shows that the samples didn't react with the 10% solution of $NaOH_{(aq)}$ because there was no color change and the weight difference were very negligible also 0.0009, 0.0000, and 0.0025 for sample A, B and C respectively.

Table 4.5c Water Absorption Test

Sample	Dry mass (g)	Saturated mass (g)	Absorption percentage (%)
A	0.4107	0.4123	0.38
B	0.6503	0.6526	0.35
C	0.6050	0.6094	0.72

The water absorption test showed that sample A, B and C absorption percentage was 0.38%, 0.35% and 0.72% respectively. Although, the expected result was 0% water absorption, the measurement was found to be otherwise. This might be as a result of improper fining and melting of the samples and also the observed air pockets (bubbles) in the samples. But these values were in the acceptable range.

Table 4.5d Glass Density

Sample	Density (gcm^{-3})
A	2.49
B	2.47
C	2.48

The values of glass density provided by Glenn (2004) and Glass (2009) for different types of glasses showed that vitreous silica had a density of $2.20gcm^{-3}$, soda lime silica (ordinary bottles and window glasses) had $2.49gcm^{-3}$ - $2.5gcm^{-3}$, sodium borosilicate had $2.23gcm^{-3}$, lead glass had $3.1gcm^{-3}$, aluminosilicate glass $2.64gcm^{-3}$and optical glass with $2.51gcm^{-3}$. The result obtained from Table 4.4d above shows that the density of sample A was in accordance to the density of soda lime silica glass. For sample B and C, their densities were close to the range of soda lime silicate glass and could also be inferred as falling under the same density.

4.6 Scanning Electron Microscopy (SEM)

The glass samples A, B and C were enlarged to $500\times536\mu m$ in magnification and were scanned using back scattered electron. The surfaces of the glass samples reveal rough surfaces, pores and fibers of different sizes.Fig. 4.6a to 4.6c showed the surface texture and morphology. The surface texture looked rough with irregular shapes on it. Fig 4.6d to 4.2f showed the pore histogram of the glass samples.

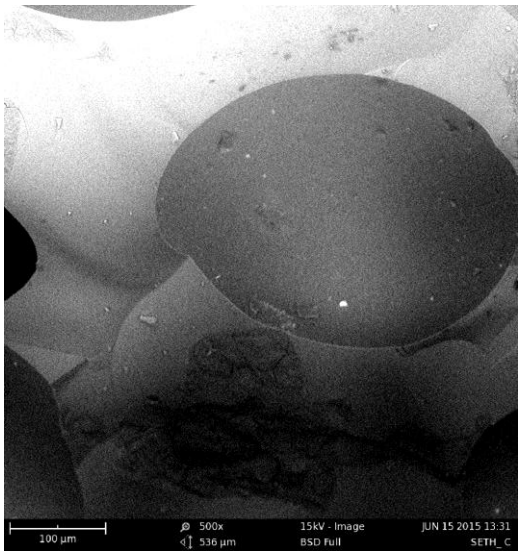


Fig. 4.6a External morphology of glass sample A

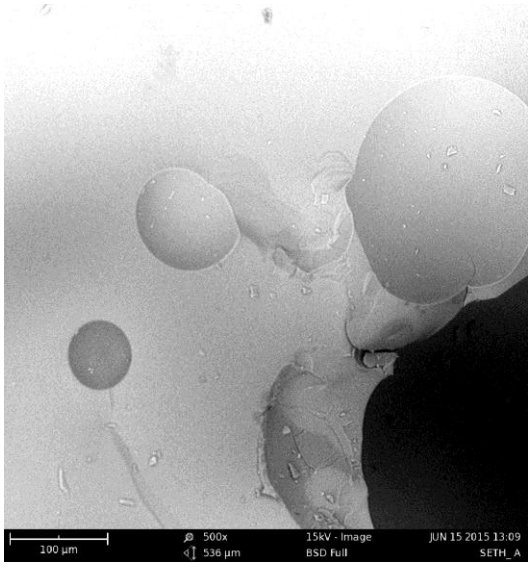


Fig. 4.6bExternal morphology of glass sample B

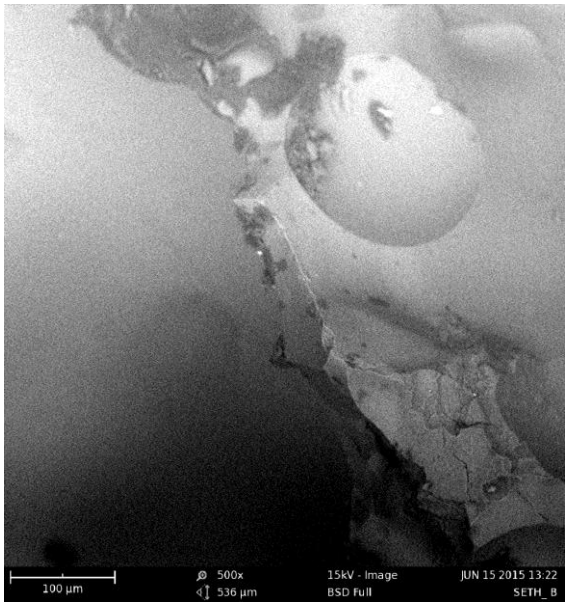


Fig 4.6cExternal morphology of glass sample C

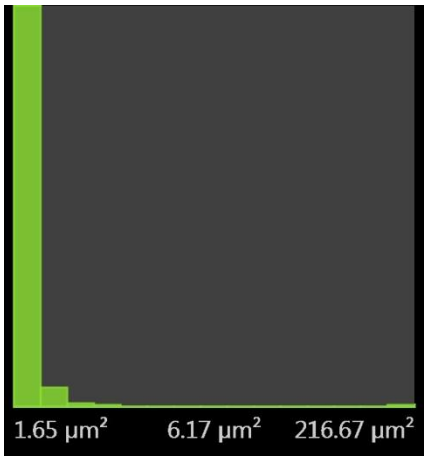


Fig. 4.6d Pore histogram of sample A

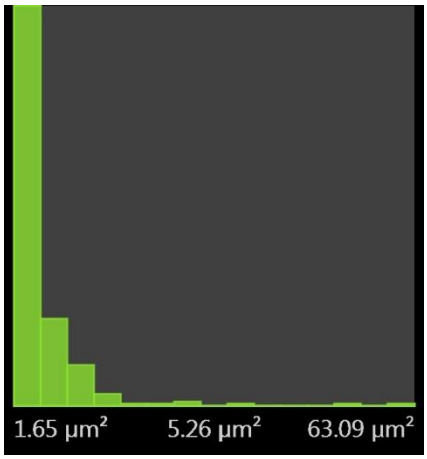


Fig. 4.6e Pore histogram of sample B

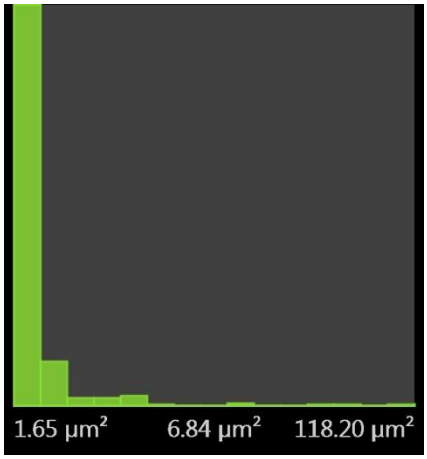


Fig. 4.6f Pore histogram of sample C

From the SEM analysis, the external morphology of the glass samples shows rough surfaces which might be as result of incomplete finning and homogeneity. The pores exist in the glass due to improper fining of the melt. Homogenization did not occur because no stirring was done. The highest pore size stands at 1.65μm .

4.7 X-ray Difrraction (XRD)

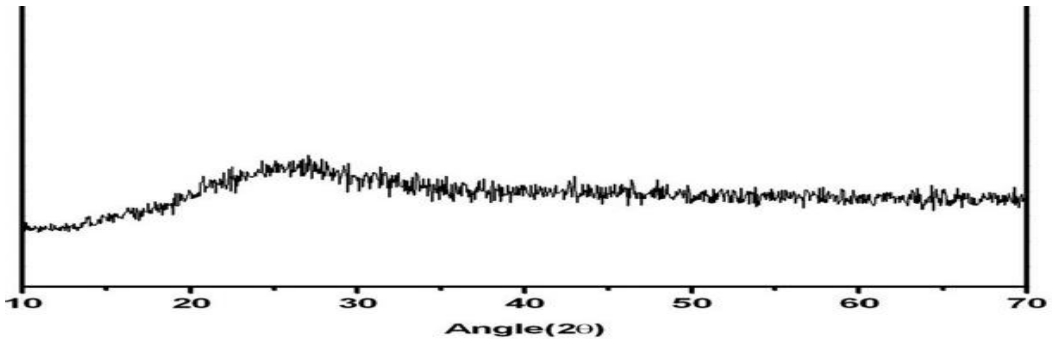


Fig 4.7a shows result of XRD pattern of sample A

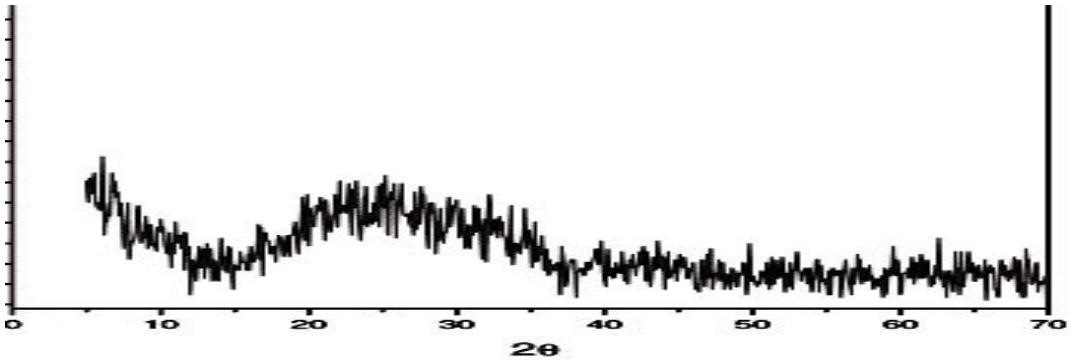


Fig 4.7b shows result of XRD pattern of sample B

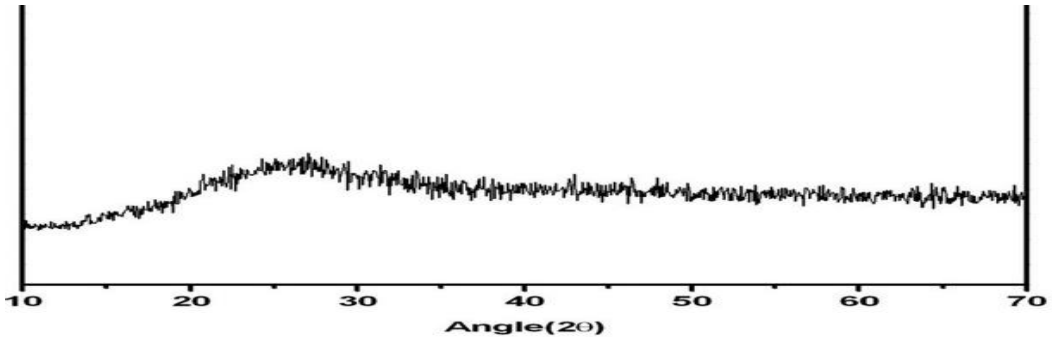


Fig 4.7c shows result of XRD pattern of sample C

Fig 4.7a-4.7c showed the X-ray diffraction patterns of the formed glass samples. There was no sharp Bragg's peaks, but a broad peak zone near $2\theta=22^0$C which signifies the amorphous nature of the melts.

4.8 Findings

1. It was revealed from chemical analysis that groundnut husk ash (GHA) had a high percentage of K_2O and CaO which could be a good source in supplying these oxides in glass batches.
2. The glass density test showed that the glasses formed falls within the same value of soda lime silicate glass.
3. The GHA was able to lower the melting temperature, which enabled the glass raw materials to melt at $1{,}300^0$C.
4. From the result of chemical resistance test, the glass could be suitable for both acidic and basic applications.
5. From the result of groundnut husk ashing, toobtain high yield of groundnut husk ash, high amount of groundnut husk was needed. Which in turn contribute in combating environmental pollution caused by improper disposal of groundnut husk.

CHAPTER FIVE

CONCLUSIONS AND RECOMMENDATONS

5.1 Summary

The study was able to establish that itis possible for GHA to be used in producing potash glass at a temperature of $1{,}300^0$C. The formed glasses were resistant to both acidic and basic conditions and also had a density of soda lime silicate glass (2.5 - 2.6gcm^{-3}). The presence of scum and bubbles in the formed glass was as a result of incomplete fining, homogenizing and the ability of the alkali to rapidly fuse. The use of GHA as a glass raw material especially in Nigeria with regards to agricultural waste will go a long way in tackling environmental challenges posed by these wastes and promoting a safe environment and also, encouraging potash glass production in the country.

5.2 Conclusions

The result of the research showed that the characterization of GHA had high percentage of K_2O and CaO. From the result obtained and the tests carried out, the glasses formed could be termed as potassium glass with density of 2.47 gcm^{-3}-2.49 gcm^{-3} (same with soda lime silicate glass). Its water absorption rate stood at 0.38%, 0.35% and 0.72% for sample A, B and C respectively. The formed glasses can function in both acidic and basic applications as the chemical resistant test showed that both substances do not attack them. The result from the XRD showed broad peak zone near $2\theta=22^0$C which indicated the amorphous nature of the melts.

This research was carried out within the limits of available materials, equipment and experimental errors; however the study has proved successful in showing the potentials of GHA as a source of fluxing agent for glass making and more research could be carried out on the subject matter.

It was also shown that the unutilized abundant and growing biomass of GHA that was an environmental problem in terms of pollution and disposal could be used to produce potash glass.

5.3 Recommendations

1. More research should be embarked upon on other agricultural waste to determine their suitability for glass production.
2. GHA and Soda ash should be used as mixed alkali in various ratios in glass making and investigate the resultant effects.
3. Facilities for various analyses such as XRF and XRD should be made available or procured for easy access and use during similar research.

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