# ASSESMENT OF THE SUITABILITY OF SAW DUST ASH AS FLUXING RAW MATERIAL IN SODA LIME SILICA GLASS PRODUCTION

**BY**

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**AUGUST, 2015**

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**BY**

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**A THESIS SUBMITTED TO THE POSTGRADUATE SCHOOL, AHMADU BELLO UNIVERSITY, ZARIA NIGERIA**
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**DEPARTMENT OF INDUSTRIAL DESIGN,**
**AHMADU BELLO UNIVERSITY,**
**ZARIA NIGERIA**

**AUGUST, 2015**

## DECLARATION

I declare that the work in the thesis entitled **'Assessment of the Suitability of Saw Dust Ash as Fluxing Raw Material in Soda Lime Silica Glass Production'** has been performed by me in the Department of Industrial Design under the supervision of Dr. A.D. Garkida and Dr. E.A. Ali. The information derived from the literature has been duly acknowledged in the text and a list of references provided. No part of of this thesis was previously presented for another degree or diploma at any university.

**Abdulmumini Jibril** _____ _____

**Signature** **Date**

## CERTIFICATION

This Thesis entitled 'ASSESSMENT OF THE SUITABILITY OF SAW DUST ASH AS FLUXING RAW MATERIAL IN SODA LIME SILICA GLASS PRODUCTION' by Abdulmumini Jibril meets the regulations governing the award of Master of Science of Ahmadu Bello University, Zaria, and is approved for its contribution to knowledge and literary presentation.

| | | |
|---|---|---|
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## ABSTRACT

This research was conducted to study the suitabilty of saw dust ash in glass preparations. Saw dust from Isoberlinea doka (doka), Terminalia superba (Afara), Anogeissus leiocarpus (marke) and the mix saw dust (without taking note of the wood type) were used. They were collected from local market Sabon Gari, Zaria. The saw dusts were ashed using muffle furnace at $550^0$C and analysed using affluent absorption spectrophotometer and percentage oxides were calculated. The following results were obtained: Terminalia superba (Afara) 7.85% $SiO_2$, 1.89% $Al_2O_3$, 18.00% $K_2O$, 24.80% CaO, 5.40% $Na_2O$, 5.07% MgO, 1.97% $Fe_2O_3$. Anogeissus leiocarpus (marke) 7.31% $SiO_2$, 2.27% $Al_2O_3$, 22.80% $K_2O$, 19.05% CaO, 9.17% $Na_2O$, 9.04% MgO, 1.37% $Fe_2O_3$. Isoberlinea doka (doka) 7.10% $SiO_2$, 6.42% $Al_2O_3$, 21.50% $K_2O$, 8.42% CaO, 7.55% $Na_2O$, 24.21% MgO, 1.92% $Fe_2O_3$. Mixed saw dust 7.27% $SiO_2$, 7.56% $Al_2O_3$, 29.90% $K_2O$, 24.47% CaO, 5.40% $Na_2O$, 15.61% MgO, 4.57% $Fe_2O_3$. Four glass batches containing these ashes were prepared and melted in refractory crucibles at $1300^0$C for five hours where two batches 1 and 4 melted and formed glasses while 2 and 3 sintered. Some properties of the two glasses obtained was determined, the density was determined by measurement and by calculation. The result from measurement was found to be 2.46gdm$^{-3}$ while by calculations, the result was 2.42gdm$^{-3}$. The variation in densities might be as a result of foreign inclutions from refractory crucibles or the introduction of excess $K_2O$ into the glass batches. Chemical durability test of the glasses was found to be in compliance with standard soda lime silica glass. The Xray diffraction of the melt showed the formation of amorphous phase in sample 1 and 4, which indicated the formation of glass while that of 2 and 3 indicated the formation of crystalline phase which confirmed the formation of sintered bodies. The electron dispersive spectroscopy from scanning electron microscopy (SEM) of glasses from batch 1 and 4 showed the consistency of major elements forming the glass compositions, Si, Na and O, indicating the homogeinity of the melts and accuracy of the temperature range. While the external morphology of the glasses showed rough surfaces as a result of not subjecting the glasses into mechanical stirring or any of the forming processes, and also presence of pores but not large enough to cause any form of deformity to the glasses. Based on the results obtained, saw dust ash can best be used as sources stabilizers in glass making due to high percentage of CaO and $K_2O$.

## TABLE OF CONTENTS


Cover page - - - - - - - - - i

Title page - - - - - - - - - - ii

Dedication - - - - - - - - - - iii

Certification - - - - - - - - - - iv

Acknowledgement -- - - - - - - - - v

Abstract - - - - - - - - - - vi

Table of Content - - - - - - - - - vii

List of Tables - - - - - - - - - xii

List of Figures - - - - - - - - - xiii

**CHAPTER ONE**

1.0 Introduction - - - - - - - - - 1

1.1 Background of the Study - - - - - - - - - 1

1.2 Glass - - - - - - - - - - 2

1.3 Glass making raw materials- - - - - - - - - 4

1.3.1 Glass forming oxides - - - - - - - - 4

1.3.2 Flux - - - - - - - - - 5

1.3.3 Property modifiers - - - - - - - - 5

1.3.4 Oxidizing agents - - - - - - - - 5

1.3.5 Reducing agents - - - - - - - - 6

1.3.6 Fining agents- - - - - - - - - - 6

1.3.7 Colouring agents - - - - - - - - 7

1.4 Glass making raw materials sources - - - - - - 7

1.4.1 Mined or Quarried material - - - - - - - 7

1.4.2 Synthetic raw materials - - - - - - - 8

1.4.3 By product materials - - - - - - - - 8

1.5 The Ash - - - - - - - - - 8

1.5.1 Uses os Ash - - - - - - - - - 9

1.6 Statement of the Problems - - - - - - - 10

1.7 Aim and Objectives of the Study - - - - - - 10

1.8 Research Questions - - - - - - - - 11

1.9 Significance of the Study - - - - - - - 11

1.10 Scope of the Study - - - - - - - - 12

1.11 Justification of the Study - - - - - - - 12

**CHAPTER TWO**

2.0 Literature Review - - - - - - - 13

2.1 Historical Background - - - - - - - 13

2.2 Saw Dust - - - - - - - - - 14

2.3 Sources of Saw Dust - - - - - - - - 14

2.4 Uses of Saw dust - - - - - - - - - 15

2.4.1 Application in Agriculture - - - - - - - - 16

2.4.2 Saw dust as Source of Energy - - - - - - 16

2.4.3 Application in the production of composite bricks - - - - 18

2.4.4 Apllication in the synthesis of chemicals - - - - - - 19

2.4.5 Application in environmental protection - - - - - 19
2.5 Effects of Saw dust to the Environment - - - - - 20
2.5.1 Effect to Land - - - - - - - - - 20
2.5.2 Effect to water body - - - - - - - - - 20
2.5.3 Effect to farm animals - - - - - - - - - 21
2.6 White Afara (Terminalia superba) - - - - - - 21
2.7 Marke (Anogeissus leiocarpus) - - - - - - 23
2.8 Doka (Isoberlinea doka) - - - - - - - 23
2.9 Chemical composition of saw dust - - - - - 24
2.10 Factors affecting chemical composition of saw dust - - - - 24
2.11 Application of saw dust in glass preparation - - - - 25
2.12 Types of glass - - - - - - - - - 25
2.12.1 Soda lime silica glass - - - - - - - - - 25
2.12.2 Vitreous silica glass - - - - - - - - - 26
2.12.3 Boro silicate glass - - - - - - - - 27
2.13 Physical properties of soda lime silica glass - - - - - 27
2.14 Chemical properties of soda lime silica glass - - - - 28
2.15 Raw material anlysis - - - - - - - - - 29
2.16 Sample preparation - - - - - - - - - 29
2.17 Atomic absorption spectrophotometry - - - - - 30
2.18 X ray flourescence - - - - - - - - - 30
2.19 Scanning electron microscopy - - - - - 31

**CHAPTER THREE**


| | | |
|---|---|---|
| 3.0 | Methodology - - - - - - - - - | 32 |
| 3.1 | Appararus and materials - - - - - - - - | 32 |
| 3.2 | Method - - - - - - - - - | 32 |
| 3.2.1 | Raw material sourcing - - - - - - - - | 32 |
| 3.2.2 | Sample Preparation - - - - - - - - - | 33 |
| 3.2.3 | Digestion of saw dust ash - - - - - - - | 33 |
| 3.2.4 | Chemical analysis of ash - - - - - - - | 34 |
| 3.3 | Batch formulation - - - - - - - - | 35 |
| 3.4 | Test melting - - - - - - - - - | 45 |
| 3. 5 | Property testing - - - - - - - - | 45 |
| 3.5.1 | Density test - - - - - - - - - | 45 |
| 3.5.2 | Chemical durability test - - - - - - - | 47 |
| 3.5.3 | X ray diffraction - - - - - - - - | 48 |
| 3.5.4 | Scanning elecro microscope - - - - - - - | 49 |


**CHAPTER FOUR**


| | | |
|---|---|---|
| 4.0 | Result and discussion - - - - - - - - | 51 |
| 4.1 | Result of saw dust ashing - - - - - - - | 51 |
| 4.2 | Result of chemical analysis - - - - - - - | 51 |
| 4.3 | Batch formulation - - - - - - - | 53 |
| 4.4 | Result of test melting - - - - - - - - | 56 |

4.5 Result of density test -- - - - - - - - 57

4.6 Result of chemical durability test - - - - - - 58

4.7 Ressult of X ray diffraction analysis - - - - - - 59

4.8 Result of scanning electron microscopy - - - - - 61

4.8.1 Morphology - - - - - - - - - 61

4.8.2 Energy dispersive spectroscopy (EDS) - - - - - 68

4.9 Findings - - - - - - - - - - - 80

**CHAPTER FIVE**

5.0 Conclusion and recommendations - - - - - - 82

s5.1 Conclusion - - - - - - - - - - 82

5.2 Recommendations - - - - - - - - 82

REFERENCES - - - - - - - - - - 83

## LIST OF TABLES


| | | |
|---|---|---|
| Table 3.1: | Soda lime silica glass composition - - - - | 36 |
| Table 3.2: | Contributions of oxides by 100g of raw materials - - | 38 |
| Table 3.3a: | Summary of oxides introduced by 100g of glass from batch 1 - | 41 |
| Table 3.3b: | Weight of raw materials forming batch 1 - - - - | 41 |
| Table 3.4a: | Summary of oxides introduced by 100g of glass from batch 2- - | 42 |
| Table 3.4b: | Weight of raw materials forming batch 2 - - - - | 42 |
| Table 3.5a: | Summary of oxides introduced by 100g of glass from batch 3 - | 43 |
| Table 3.5b: | Weight of raw materials forming batch 3 - - - - | 43 |
| Table 3.6a: | Summary of oxides introduced by 100g of glass from batch 4 - | 44 |
| Table 3.6b: | Weight of raw materials forming batch 4 - - - - | 44 |
| Table 4.1: | Weight of ashes obtained - - - - | 51 |
| Table 4.2: | Percentage concentrations of elements in the ash samples - - | 52 |
| Table 4.3: | Percentage concentrations of oxides in the ash samples - - - | 52 |

## LIST OF FIGURES


| | | |
|---|---|---|
| Figure 3.1: | Spectograph of glass sample 1 showing the seven spots examined- - - - - - - - | 49 |
| Figure 3.2 | Spectograph of glass sample 4 showing the seven spots examined - - - - - - - - | 50 |
| Figure 4.1: | Weight of raw materials to form batch 1 - - - - - - | 54 |
| Figure 4.2: | Weight of raw materials to form batch 2 - - - - - | 55 |
| Figure 4.3: | Weight of raw materials to form batch 3 - - - - - | 55 |
| Figure 4.4: | Weight of raw materials to form batch 4 - - - - - | 56 |
| Figure 4.5a: | XRD spectrum of glass sample 1 - - - - - - | 59 |
| Figure 4.5b: | XRD spectrum of sintered sample 2 - - - - - | 60 |
| Figure 4.5c: | XRD spectrum of sintered sample 3 - - - - - - | 60 |
| Figure 4.5d: | XRD spectrum of glass sample 4 - - - - - - | 60 |
| Figure 4.6a: | External morphology of glass sample 1, x420 magnification - - - - - - - - | 62 |
| Figure 4.6b: | External morphology of glass sample 1, x1000 magnification - - - - - - - - | 62 |
| Figure 4.7a: | External morphology of glass sample 4, x420 magnification - - - - - - - - | 63 |
| Figure 4.7b: | External morphology of glass sample 4, x1000 magnification - - - - - - - - | 63 |
| Figure 4.8a: | Fiber histogram of glass sample 1 - - - - - - | 64 |
| Figure 4.8b: | Fiber histogram of glass sample 4 - - - - - - | 64 |
| Figure 4.9a: | Pore histogram of glass sample 1 - - - - - - | 65 |
| Figure 4.9b: | Pore histogram of glass samples 4 - - - - - - | 65 |
| Figure 4.10a: | Fiber metric of glass sample 1 - - - - - - | 66 |

Figure 4.10b: Fiber metric of glass sample 4 - - - - - 66

Figure 4.11a: Spectrum of elements in spot 1 of glass sample 1 - - - 68

Figure 4.11b: Histogram of elements in spot 1 glass sample 1 - - - 68

Figure 4.12a: Spectrum of elements in spot 2 of glass sample 1 - - - 69

Figure 4.12b: Histogram of elements in spot 2 glass sample 1 - - - 69

Figure 4.13a: Spectrum of elements in spot 3 of glass sample 1 - - - 70

Figure 4.13b: Histogram of elements in spot 3 glass sample 1 - - - 70

Figure 4.14a: Spectrum of elements in spot 4 of glass sample 1 - - - 71

Figure 4.14b: Histogram of elements in spot 4 glass sample 1 - - - 71

Figure 4.15a: Spectrum of elements in spot 5 of glass sample 1 - - - 72

Figure 4.15b: Histogram of elements in spot 5 glass sample 1 - - - 72

Figure 4.16a: Spectrum of elements in spot 6 of glass sample 1 - - - 73

Figure 4.16b: Histogram of elements in spot 6 glass sample 1 - - - 73

Figure 4.17a: Spectrum of elements in spot 7 of glass sample 1 - - - 74

Figure 4.17b: Histogram of elements in spot 7 glass sample 1 - - - 74

Figure 4.18a: Spectrum of elements in spot 1 of glass sample 4 - - - 75

Figure 4.18b: Histogram of elements in spot 1 glass sample 4 - - - 75

Figure 4.19a: Spectrum of elements in spot 2 of glass sample 4 - - - 76

Figure 4.19b: Histogram of elements in spot 2 glass sample 4 - - - 76

Figure 4.20a: Spectrum of elements in spot 3 of glass sample 4 - - - 77

Figure 4.20b: Histogram of elements in spot 3 glass sample 4 - - - 77

Figure 4.21a: Spectrum of elements in spot 4 of glass sample 4 - - - 78

Figure 4.21b: Histogram of elements in spot 4 glass sample 4 - - - 78

Figure 4.22a: Spectrum of elements in spot 6 of glass sample 4 - - - 79

Figure 4.22b: Histogram of elements in spot 6 glass sample 4 - - - 79

# CHAPTER ONE

# INTRODUCTION

## 1.1 Background of the Study

The unavailability of raw materials for industrial production of any product including glass can cause some difficulties, or problems to mass production or research within that particular area. This problem is among observations raised at the seminar organized by Raw Materials Research and Development Council on the Prospects and Challenges of Sourcing Raw Materials for the Chemicals and Pharmaceutical Industries in Lagos, Nigeria in the year 2001. It was highlighted that over 90% of raw materials used in chemical and pharmaceutical industries are imported and the cost of importation of such chemical raw materials is enormous. Some locally available raw material such as kaolin, carbon black etc, are of very poor quality and do not meet the specifications of the industries. The report also stated that there is lack of intermediate industries to produce the required raw material. Because of these and similar problems affecting glass production, there is need to have easier and cheaper means of obtaining these required oxides in glass preparation at the local, national or universal level. So, ash is something that can be obtained any where worldwide.

Ash utilization in glass production will immensely reduce environmental pollution and at the same time provide extra income to some individuals that will be involve in sourcing or marketing of the ash or materials that when burnt will provide the required ash at the required quality and quantity. The practice of recycling solid waste into something useful is an ancient one. Metal implements were melted down and recast in prehistoric times. Today,

according to Huang (2008), recyclable materials are recovered from municipals refuse by a number of methods, including shredding, magnetic separation of metals, air classification that separates light and heavy fractions, screening, and washing. Another method of recovery is the wet pulping process: Incoming refuse is mixed with water and ground into slurry in the wet pulper, which resembles a large kitchen disposal unit. Large pieces of metal and other non pulpable materials are pulled out by a magnetic device before the slurry from the pulper is loaded into a centrifuge called a liquid cyclone. Here the heavier non combustibles, such as glass, metals, and ceramics, are separated out and sent on to a glass- and metal-recovery system; other, lighter materials go to a paper-fiber-recovery system. The final residue is either incinerated or is used as landfill.

Waste materials residues of incineration generated from combustion of coal from thermal power station such as coal ash and fly ash, as well as bottom ash from grate of incinerators, sewage sludge ash, sludge from pulp paper waste, slag from metallurgical process and sugar cane bagasse are common waste residues that are useful in glass ceramic production. (Isa and Abdulmumini, 2015).

## 1.2 Glass

Glass is defined as an amorphous solid completely lacking in long range, periodic atomic structure, and exhibiting a region of glass transformation behavior. Any material whether it is inorganic, organic, or metallic, formed by any technique, which exhibits glass transformation behavior is a glass. (Shelby, 2005).

He adopted this definition due to the fact that there are numbers of discoveries in the field of glass science and technology that earlier definitions had to be looked in and therefore, had to be reviewed. Some of the advancements according to him are that silica is not the required component of a glass as other inorganic materials which do not contain silica can be used to produce glass, example $P_2O_5$ and GeO are glass network forming oxides after cooling from melt. Also cooling from the melt is not requirement for glass formation. Glass can be produce by other methods like sol gel process of solutions, vapour deposition and neutron irradiation of crystalline materials. Varshneya (2005) stated that $As_2O_5$, $Sb_2O_3$ and $V_2O_5$ are able to produce glass by vapour phase condensation.

Apart from the above mentioned advancements, Shelby (2005), also described that glasses inorganic or non metallic nature of glasses can not be used to define glass because, currently there are organic glasses and metallic glasses. Varshneya (1999) cited some examples of glass formation in metallic systems which include, Transition metals metalloid (T-M) system, example $Pb_{80}Si_{20}$, $Ni_{80}P_{20}$ and $FeNi_{40}P_{14}$. Early and late transition metals (TE, TL); examples are $Nb_{60}Ni_{40}$, $W_{45}Fe_{55}$, $Zr_{76}Fe_{24}$, $Co_{33}Gd_{67}$ and $Ni_{30}Gd_{70}$. The alkaline earth (AE) with rare earth (RE) and simple metals (S), examples $Ca_{67}Mg_{33}$, $Mg_{70}Zn_{30}$, $Ca_{67}Pb_{35}$ and $Al_{30}La_{70}$. The transition metals actinides (T-AC) system, example $U_{70}Cr_{30}$. Shelby (2005) therefore, stated two common characteristics of all glasses, firstly, no glass has a long range, periodic atomic arrangement and secondly, time dependent glass transformation behavior. Therefore, any material inorganic, organic, metallic or non metallic formed by any technique which exhibits glass transformation behavior is glass.

## 1.3 Glass Making Raw Materials

Before any type of glass is produced, good selection of raw materials is put into consideration. The raw materials should be abundant, cheap and free from impurities. Physical nature such as grain size is also an important factor that plays some roles in success of obtaining the type of glass intended to be produced. Glass making oxides are categorized into various groups, Samuel (1974) classified glass raw materials into Glass forming oxides, flux, oxidizing agents, reducing agents, fining agents and colouring agents. Shelby (2005), categorized glass making oxides into Glass Former, Flux, Property Modifier, Colourant, and Fining Agent.

### 1.3.1 Glass Forming Oxides

The primary glass formers in commercial oxide glasses are silica (SiO,), boric oxide ($B_2O_3$), and phosphoric oxide ($P_2O_5$), which all readily form single component glasses. A large number of other compounds may act as glass formers under certain circumstances, including GeO, $As_2O_3$, $Sb_2O_3$, TeO,, $A1_2O_3$, $GaO_3$, and VO. With the exception of GeO, these oxides do not readily form glasses by themselves unless very rapidly quenched or vapor deposited, but can serve as glass formers when mixed with other oxides. The elements S**,** Se, and Te act as glass formers in chalcogenide glasses. Although halide glasses can be made in many systems, with many different compounds acting as glass formers, the two most common halide glassformers are BeF, and ZrF (Shelby, 2005).

### 1.3.2 Flux

This is a group of oxides which when impact into glass; modify the glass structure resulting in the decrease in the temperature of fusion. Shelby (2005), mentioned that the most common fluxes are the alkali oxides, especially Na,O (soda) and PbO. Most commercial glasses contain soda, including those used for containers and window glasses. Potassium oxide is also used extensively in commercial glasses, while lithium oxide is used in a number of commercial glass-ceramics. Addition of large amounts of alkali oxides results in serious degradation in many properties.

### 1.3.3 Property Modifier

Addition of fluxes to silica lead to decrease in cost of glass formation, the addition of large amounts of alkali oxides results in serious degradation in many properties. The degradation in properties is usually countered by addition of property modifiers, which include the alkaline earth and transition metal oxides, and, most importantly, aluminum oxide. They also improve many of the properties of the resulting glasses. The properties are thus modified, or adjusted, by careful control of the amount and concentration of these oxides to obtain precisely the desired results (Shelby, 2005).

### 1.3.4 Oxidizing Agents

These are oxides that liberate oxygen during glass melting by decomposing organic matter present in the batch, change ferrous to ferric state (FeO to $Fe_2O_3$) by this, it also prevent discolouration of glass in the manufacture of colourless glass. Oxidizing agents hinder chemical reduction to obtain specific colour in the manufacture of coloured glass. It

also help in fining by evolving oxygen bubbles. Examples are $KNO_3$, $NaNO_3$, $Ba_2O_2$ and MnO (Samuel, 1974)

### 1.3.5 Reducing Agents

These are oxides that extract oxygen from some constituents of the batch. They reduced ferric to ferrous ($Fe_2O_3$ to FeO) in specific case of ultra violet light transmitting glasses. These oxides are also introduced to promote incorporation of some batch forming oxides into a glass by reducing sodium sulphate to sodium sulphite ($Na_2SO_4$ to $Na_2SO_3$) which reacts quicker with $SiO_2$. Example is carbon as coal or coke. And it hinders oxidizing atmosphere in the development of certain coloured glass. Examples of reducing agents are coal, anthracite, $K_2NO_2$ and tartaric acid,

### 1.3.6 Fining Agents

Fining agents are added to glass forming batches to promote the removal of bubbles from the melt. Fining agents include the arsenic and antimony oxides, potassium and sodium nitrates, NaCl, fluorides such as CaF,, NaF, and Na,AlF,, and a number of sulfates. These materials are usually present in very small quantities usually less than 1 wt%, and are usually treated as if they have only minor effects on the properties of the final glasses. Their presence, however, is essential in many commercial glasses, which would be prohibitively expensive to produce without the aid of fining agents in reducing the content of unwanted bubbles in the final product. (Shelby, 2005)

### 1.3.7 Colouring Agents

These are substances that impacts colour to glass, and in most cases according to Shelby (2005), are oxides of either the 3d transition metals or the 4f rare earths. For example $Cr_2O_3$ introduces green colour, $CrO_3$ gives orange – red colour, while $V_2O_3$ impact yellow to glass. Iron oxides, which are common impurities in the sands used to produce commercial silicate glasses, act as unintentional colorants in many products.

## 1.4 Glass Making Raw Materials Sources

Isa (2009) described that all glass making raw materials are being sourced from Mined or quarried raw material, Synthetic raw materials or by product materials.

### 1.4.1 Mined or Quarried Raw Material

These are mined materials and beneficiated in some ways after extraction from their various ores to meet the requirements for glass production and market demands. Such raw materials are sand, quarts, limestone, dolomite and feldspar (Isa, 2009)

Sand which is near the surface is explored by open cast method in which the upper top soil is removed and then the sand dug up. Generally, machines are used to dig up minerals by this method. While feldspar, limestone and dolomite are explored by bulk extraction in which explosives devices are used to break it into smaller fragments for easy handling and transportation from one place to another. (RMRDC, 2009)

### 1.4.2 Synthetic Raw Materials

These are manufactured chemical substances which include the soda ash, boric acid, aluminium hydrate, potash, borax etc. Fwotmwol and Garkida (2005) stated that these oxides are supplied in Nigeria by vendors from importation or by locally based chemical processing plants. Example of such plant is the soda ash processing plant in Borno state.

### 1.4.3 By Product Materials

These are materials produced as a result of the manufacture or production of something else, often useful or commercially valuable. These include the cullet from within the factory as a result of defects along the production line and from external sources. Also, blast furnace slag and phosphate slag are chemical by products.

## 1.5 The Ash

Ash as defined by Redmond (2009), as simply a solid product of combustion. If combustion is complete, the ash is wholy inorganic. The ash from wood or similar plant material generally consists principally of sodium carbonate and potassium carbonate. Ash from animal bones also contains oxides similar to that from plants and some others not obtainable from plants. Alemaka (2002) found out that bones and bone ashes contain calcium phosphate (v) in addition to calcium carbonate, fat and organic matter containing nitrogen.

### 1.5.1 Uses of Ash

Ash can serve as both waste and item of economic importance depending on individual need, trade or culture. It is waste to a person that run a restaurant and to those that use woods as source of domestic energy in cooking, room warming or trades like blacksmithing. It is at the same time a good source of manure to gardeners and crop farmers. Ewule, (2002) stated that wood ash is used by farmers as a source of manure because of its high content of potash and potassium. In some parts of Nigeria, wood ash is also applied as insect repellants to farm crops due to its toxicity, and as traditional source of easy and quick method of fruit ripening. Because of its high alkaline content, Serafimova, Mladenov and Pelovski, (2011) recommended the use of wood ash for soil acid neutralization.

Ash is also a good source of locally obtainable glazes to ceramics as stated by Ojie and Egede (2002). “In a majority of cases, wood ash contains considerable soluble alkalis such as potash and soda in addition to silica and alumina. Combination of these alkali and the glass formers gives very good glazes. Ash does not only serve as raw material in production but also serve as food additives in some areas of Northern Nigeria”.

Serafimova *et al* (2011) stated that in most cases, ash from combustion of plants waste does not contain heavy metals and other toxic elements in concentration that could lead to environmental contamination. Also, such type of ashes could be used as a secondary raw material contributing to the reduction of the quantity of generated wastes and achieving greater sustainability. Analyses confirmed that the basic components of wood ash are

$CaCO_3$, $SiO_2$ and $K_2Ca(CO_3)_2$. Therefore, this determined the nature of alkaline extracts in wood ash is attractive material for use in glass production.

## 1.6 Statement of the Problem

The problem of this research is to find a way of recycling waste saw dust ash to curb environmental pollution. The general way of waste disposal in Nigeria is dumping in an open land, land filling and in few cases by incineration. Isa and Abdulmumini (2015) pointed out that these methods have adverse effects such as emission of green house gases and making environments so irritating to its inhabitant. Therefore, recycling and utilization of wastes is an important aspect of environmental sanitation and minimizes the exploitation of virgin raw materials for any form of production.

## 1.7 Aim and Objectives of the Study

The aim of this research was to use saw dust ash as cheaper source of flux in the production of soda lime silica glass. This aim was achieved through the following objectives which were to;

1. Source for saw dust from saw mills.
2. Burn the saw dust obtained from the saw mill into ash.
3. Carry out chemical analysis of the saw dust ash to determine its chemical composition.
4. Prepare a glass batch based on soda lime silica glass with the ash as an additive.
5. Test melt the glass at temperature range of $1250^oC$ to $1300^oC$

6. Carry out some properties tests on the glass prepared; chemical durability test, density test, x ray diffraction (XRD) and scanning electron microscopy (SEM).

## 1.8 Research Questions

1. What are the sources of saw dust?
2. What is the best method of obtaining ash from saw dust?
3. What is the chemical composition of saw dust ash?
4. Can soda lime silica glass be prepared from saw dust ash?
5. What is the temperature range for melting soda lime silica glass containing saw dust ash?
6. What are the properties of glass containing saw dust ash?

## 1.9 Significance of the Study

Considering the unavailability and cost of importation of glass making ingredients like soda ash, borax etc, there is need to find cheaper ways obtaining soda ash, borax etc. or its supplements locally to discourage importation of glass making raw materials.

The utilization of saw dust ash in glass production especially soda lime silica glass, which is produced in mass will make saw dust to be utilized in large quantity thereby converting waste to wealth and reduce environmental pollutions caused by saw mills.

The research will contribute not only to the field of glass technology but also to areas like agriculture in synthesizing organic fertilizer from plant wastes and ash. The

success of the research will also help in increasing earning of saw mill owners as saw dust will no more be discarded as waste. Different individuals may be engaged in collection and sale of saw dust if it becomes an industrial raw material.

## 1.10 Scope of the Research

The research was delimited to the use of saw dust ash as fluxing raw material in the preparation of soda lime silica glass. The saw dust was obtained from saw mills in Sabon Gari market, because of its ready availability and effects to the environment, particularly the saw mills and their neighborhood.

## 1.11 Justification of the Research

Saw dust is selected instead of wood ash for environmental reason, as anything that has to do with cutting down wood from trees; goes hand in hand with deforestation, which is a major cause of desert encroachment, soil erosion and global warming. NEEDS (2005), stated that Nigeria used to have some 92000 hectares of forest but only half of that forest remained today. It further described that apart from deforestation and erosion, desertification in some areas is another environmental problem facing Nigeria.

# CHAPTER TWO

# LITERATURE REVIEW

## 2.1 Historical Background

Use of ash from plants and plant products in glass making is as old as glass itself. Brown (1967) pointed out that glass was discovered by accident about 10000 years ago when the Phoenician sailors who used blocks of natrium (natural soda) to support their cooking pots over fires and the fish bones became the lime source on a sandy beach. Though, there is no evidence to prove that this actually happened, the fish bones can only be part of the glass ingredient if it was burnt and become ash.

In about 1000 BC, Iberian (present day Portugal/Spain) glass workers collected special plants which grow in the salt marshes, dried them in the sun and then burnt them. The ashes contained a proportion of carbonates of soda, and a smaller proportion of lime. This ash has been known as *barilla*. And french ancient glass houses used ashes from fern plant and made pale amber or greenish glass called *verre de fourgere.*

Using plant ash as glass making ingredient was also practiced in Germany when glass houses were established in forest areas and *waldglas* (forest glass), a bluish green glass with many impurities, bubbles and striation were made from potash derived from the ashes of beach wood.

Golozubov et al (1992), explored the possibilities of obtaining some forms of glass, in the $SiO_2$-CaO-$Al_2O_3$-$FeO_3$-$Na_2O$-$P_2O_5$ system, from thermal plants ash, while Alemaka (2002), prepared non silicate hydro flouric resistant glass from bone ash.

## 2.2 Saw Dust

Saw dust or wood dust is a byproduct of cutting, grinding, drilling or otherwise pulverizing wood with a saw or other tools; it is composed of fine particles of wood. It is also the byproduct of certain animals, birds and insects which live in wood, such as the woodpecker and carpenter ant. It can present a hazard in manufacturing industries, especially in terms of its flammability.

Saw dust plays an important role in foundry operations. It is used in making insulatory bricks for glass melting furnaces and blast furnaces for metallurgical process. Acoording to Adegoke and Ijagbemi (2006), when saw dust is embedded into the clay to make the bricks, it fills the interstices between the clay particles and burns off at high temperature with pores and vacuum being created. Heat flow is retarded and insulating ability is enhanced.

## 2.3 Sources of Saw Dust

Saw dust is a waste product from timber processing saw mills. It was noted by Fajobi and Adewumi (2006), that the practice in most timber mills in Nigeria is to burn saw dust . The resulting saw dust ash like other ashes and incineration wastes need to be disposed off. The burning may contribute to air pollution in the neighbourhood of the saw

mill and beyond, so finding a beneficial use of saw dust and its ash such as its use as glazes in ceramics and glass making will reduce the incidence of such pollution.

The wood from which saw dust is obtained are of various types. Example, the Terminalia superba (white afara), Terminalia ivorensis (black afara), Isoberlinea doka (doka), Isoberlinea tomentosa, Anogeissus leiocarpus (marke) etc.

## 2.4 Uses of Saw Dust

A major use of saw dust is for particle board; coarse saw dust may be used for wood pulp. It has also a variety of other practical uses, including serving as mulch, or as fuel in saw dust stove. Until the advent of refrigerator, it was often used in ice houses to keep ice frozen during the summer in cold temperate region. Saw dust is also used in the manufacture of charcoal briquette. The claim for invention of the first commercial charcoal briquette goes to Henry Ford who created them from the wood scraps and saw dust produced by his automobile factory.

Saw dust is also used for bio remediation, soil amendment; additive such as saw dust, peat, waste cotton, manure, fertilizer etc. are added to increase micro organism activities. A soil amendment is any material added to soil to improve its physical properties, such as water retention, permeability, water infiltration, drainage, aeration and structure. Adedokun and Ataga (2007) conducted a research on the growth of cowpea cultivated on soil polluted with crude oil (COIL), automobile gasoline oil (AGO), and spent engine oil (SEO) with saw

dust as an additive for bio remediation and found it very effective from seed germination, growth and yield.

Small sized sawmill residues such as saw dust in many part of the world are simply dumped or burned, but it has many useful applications in so many ways of human life ranging from domestic use, in agriculture, industries etc.

### 2.4.1 Applications in Agriculture

Saw dust has wide applications in agiculture such as:

**i. Compost Making:**

Humus, the product of decayed wood, gives soil nutrition and structure, which is important to plant health. If soil is gloppy, it does not provide a plant's roots with the air they need for proper absorption of nutrients. No all woods are toxic after they have been adequately decomposed, as most of the toxins in these woods will break down in the presence of decomposers.

Also, if lots of sawdust is added, trace amounts of various elements are added, including some heavy metals. as described by Gene (2012). If too much saw dust is added, it will increase the concentration of these heavy metals above safe levels. The forest does the same thing as it adds leaves and falling branches much more slowly and not so concentrated above safe level.

### ii. As Poultry Bedding and Nesting:

Bedding is used to cover the floor of chicken cages and to provide nest materials in the nesting boxes. Two or three inches of saw dust are spread to cover the cage floor insulates the chicken from cold, absorbs water from their dropping and soaks up any spilled drinking water. Saw dust is an interesting poultry bedding material because it is usually inexpensive and its absorbency is enough to keep the cage and manure dry. Because of its light weight, chickens can easily scratch through saw dust looking for insects or bits of food and getting some exercise. However, because so dust is so light and fine, it can cause some respiratory problems when the chicken scratching sends its air born. It also becomes a digestive problem, especially for baby chicks, if accidentally swallowed. Saw dust bedding is preferable in well ventilated cages and with adult chickens only. It also provides good nesting materials because of its light and soft nature, chickens easily peck at it, picking it up and dropping it to make the perfect nest, ideal for the hen to hide and lay an egg.

It is also suitable in cattle and horse ranch as stalls and horse stables because it can be easily disposed as part of manure, absorbency and do not block spaces between slots. It disadvantage is that if pointed sawing are used, it can cause injury to cows' teats and lead to some infections (Gene, 2012)

### iii. In Soil Remediation:

For efficient bioremediation in case of places affected by pollution, such as areas where oil exploration takes place soil amendment or additives, such as sawdust, peat, waste cotton, manure, fertilizers etc. are added to increase micro-organisms activities. A soil amendment is any material added to a soil to improve its physical properties, such as water

retention, permeability, water infiltration, drainage, aeration and structure (Davis and Wilson, 2005).

### 2.4.2 Saw Dust as Source of Energy

Saw dust are used as source of domestic fuel, as there are stoves that utilize saw dust as fuel are in many households. A research conducted by Forest Product Laboratory in 2000 shows that apart from using saw dust as domestic fuel, saw mills in the U.S use saw dust as fuel by constructing fire box to burn the saw dusts which provide energy. It is also used in making saw dust briquette of different sizes, which is also another accepted safe source of domestic fuel.

### 2.4.3 Application in the Production of Composite Bricks

Composite containing saw dust and Portland cement in a ratio of 2:1 was prepared by Zziwa et al (2006), and found to be of practical application in building because of its low density and weight. Its disadvantage is that it is only suitable for interior wall paneling, because of its low strength and is affected by damp conditions.

Gene (2012) also made mention of ceiling boards and tiles made from saw dust and cement presented by Nigeria at the World Exposition EXPO 2000. Apart of its application as composite brick, ceiling board and tiles, according to Forest Product Laboratory, it is also compacted into wall to serve as sound and heat insulation in buildings and as absorbent in composition flooring.

### 2.4.4 Application in the Synthesis of Chemicals

Forest product laboratory described saw dust as source of many synthetic chemicals. Methyl or wood alcohol can be produced from it not in large commercial quantity due to the minuteness of the particles that make it difficult for destructive distillation to take place. Ethyl or grain alcohol can be produced from saw dust in commercial quantity provided there is very large daily supply of at least 250 tons of saw dust. Oxalic acid can also be produced from saw dust with a large yield. Apart from those chemicals, Forest Product Laboratory further described the usage of saw dust in paper pulp, but the paper from it is of poor quality.

### 2.4.5 Application in Environmental Protection

By mixing with some materials like manure, Gene (2012) stated that saw dust can serve as structural material to adjust water content to an optimal value. He also described that a mixture of saw dust chips and compost can serve as bio filter to treat odorous air. A well managed bio filter according to him can reduce odour emission by 85%, reduce hydrogen sulfide by 90% and ammonia by 60%. Shulga et al (2007) stated that there is some practices of using saw dust as mulch and materials for tackling soil erosion in countries of Eastern Europe Baltic.

## 2.5 Effects of Saw Dust on the Environment

The saw mills that processed the lumber from the forests if carelessly dump their saw dust into the water drainage, lake, stream, and river; it will cause clogging of the small water ways. It contaminate environment, when mixed with water causes unpleasant smell.

Continues accumulation of saw dust in rivers and lakes will make the fish and other animals that lived in the area began to ingest the debris, causing a drop in the animal populations, and the water became contaminated with bacteria and floating debris. The bacteria that contaminated the water, found a way to creep into the supply of drinking water. The bacterial contamination may cause an epidemic such as typhoid fever to spread through the population.

### 2.5.1 Effect to Land

Saw dust like other semi solid or solid matters that are created by human or animal activities, and which are disposed because they are hazardous or useless are known as solid waste. Most of the solid wastes are not biodegradable, which means they do not get broken down through inorganic or organic processes. Thus, when they accumulate they pose a health threat to people, plus, decaying wastes also attract household pests and result in an environment becoming unhealthy, dirty, and unsightly places to reside in. Moreover, it also causes damage to terrestrial organisms, while also reducing the uses of the land for other, more useful purposes. Land or soil pollution can affect wildlife, plants, and humans in a number of ways, such as: causing problems in the respiratory system and on the skin.

### 2.5.2 Effect to Water Body

Saw dust and other unwanted pieces of woods if left on the floor of the forest are being washed into the lakes and streams by winds and rain, causing small waterways to become clogged. The sawmill that processed the lumber from the forests began to dump their sawdust into the lakes, streams, and rivers as well, causing even more clogging of the

small waterways. According to Putatunda (2011), the fish and other animals that lived in the area began to ingest this debris, causing a drop in the animal populations. She also highlighted that such waste give some bacteria a home that contaminated the water in the lakes and streams which subsequently found its way to creep into the supply of drinking water. This bacterial contamination was responsible for causing an epidemic of Typhoid Fever or other chronic disease to spread through the population.

### 2.5.3 Effects to Farm Animals

Lots of saw dust in animals nest and bedding add trace amount of various elements as described by Gene (2012), putting too much saw dust, heavy metals can increase above safe level.

## 2.6 White Afara (*Terminalia superba*)

Kimpouni (2009) described Terminalia superba as popularly known as Limba, white afara, shinglewood, white mukonja, Congo walnut. In france and french speaking africa, it is known as Limba, fraké, noyer du Mayombe.

According to Orwa and Mutua (2009) This tree is widespread in West and Central Africa, from Angola, Benin, Cameroon, Central African Republic, Congo, Cote d'Ivoire, Democratic Republic of Congo, Equatorial Guinea, Gabon, Ghana, Guinea, Liberia, Nigeria, Sierra Leone, Togo. Kimpouni (2009) added that it has been planted in many tropical countries outside the natural distribution area as a promising timber plantation

species, e.g. in Uganda, Tanzania, Zimbabwe, Madagascar, Indonesia, Malaysia, the Philippines, the Solomon Islands, Fiji, Australia, Brazil and Argentina.

White afara is used as solid wood and also as veneer and plywood, and is especially popular for blond furniture, school and shop fittings, radio and television cabinets, parquetry and joinery. It has been used in construction of bridges ship building, interior trim, vehicle bodies and rail lines. It is also suitable for paper making, although the paper is of moderate quality. The wood is also used as firewood and for charcoal production. A yellow dye is present in the bark; it is used traditionally to dye fibres for matting and basketry. The bark is also used for dyeing textiles blackish. Bark decoctions and macerations are used in traditional medicine to treat wounds, sores, haemorrhoids, diarrhoea, dysentery, malaria, vomiting, gingivitis, bronchitis, aphthae, swellings and ovarian troubles, and as an expectorant and anodyne. The leaves serve as diuretic and roots as laxative. In Côte d'Ivoire Terminalia superba is occasionally used as a shade tree in cocoa and coffee plantations and in DR Congo it is used as shade tree in coffee, cocoa and banana plantations (Kimpouni, 2009).

Another specie of this tree is the Black Afara (*Terminalia ivorensis*). According to Hawthorn (1998), it is a native of Cameroon; Côte d'Ivoire; Ghana; Guinea; Liberia; Nigeria; Sierra Leone. It is widely used as timber, firewood and provides good shade and is planted with coffee, banana and cocoa.

## 2.7 Marke (A*nogeissus leiocarpus*)

Evi, (2014) described Anogeissus leiocarpus as a tall evergreen tree native to savannas of Tropical Africa. It is widely used according to Sore, H; Hilou, A; Sombie, P.E.D; Compaore, M; Meda, R; Millogo, J. and Nacoulma, O.G (2012), to dye textiles and leather as well as in the traditional treatment of many diseases. The decoction and maceration of the stem bark are used against anorexia, constipation, malaria, jaundice, itching, wounds, eczema, psoriasis, carbuncles, boils and various forms of ulcers. The woods are also used as timber in tropical Africa.

## 2.8 Doka (Isoberlinea *doka*)

Abdulkadir *et al* (2011) described Isoberlinia doka *as* a tree native to northern Nigeria and known as *doka in* Hausa. Burkill (1995) in Abdulkadir *et al* (2011) stated that it grows to about 20 m high, with spreading crown and thrives in both temperate and tropical areas. is distributed in west and central Africa from Guinea in the west to Sudan and Uganda in the east (Benin, Burkina Faso, Burundi, Cameroon, Central African Rep., Chad, Ghana, Guinea, Ivory Coast, Mali, Niger, Nigeria, Sudan, Togo, Uganda and Zaire); it is not known to occur south of the equator. Isoberlinea doka is mainly used for the timber. In Togo the wood is reduced to ash for soap-making.

Isoberlinia tomentosa is specie of this tree. It is described by Louppe (2012) as a "small to medium-sized tree up to 18 m tall, occurring from Guinea and Mali eastward to Sudan in wooded savanna together with Isoberlinia doka, and southward to Tanzania, Malawi and Zambia in Brachystegia woodland. Its wood resembles that of Isoberlinia doka and used for similar purposes, e.g. for posts, planks, joinery and furniture. In Burkina Faso

the leaves are eaten as vegetable and in various sauces, whereas the bark is used in the treatment of sores. The bark is used in medications against stomach problems and is used in Nigeria in the preparation of arrow poison".

## 2.9 Chemical Composition of Saw Dust Ash

Okunna (1983), pointed out that analysis of ashes including other vegetable ashes like grass and straw, show that they contain oxide like, potash,lime, magnesia, and glass formers such as silica. There are also small amount of coloring matter such as the oxide of iron. According to Ewule (2002), ash is used in dyeing due to the following chemical composition in wood ash: 10-15% Alumina; 30-70% Silica; Up to 15% potash and 30% lime. Ojie and Egede (2002) also found out that, in a majority of cases, wood ash contains considerable soluble alkalis such as potash and soda in addition to silica and some alumina.

## 2.10 Factors Affecting Chemical Composition of Saw Dust Ash

The factors responsible for the variable nature of wood ash according to Okunna (1983) are the soil type, the time of the year the wood was cut, wood cut in the rainy season or water logged areas have less concentration of oxides such as lime, potash and that of sodium. And the wood type, hard word may differ from soft ones in term of chemical composition. Different parts of a plant store up certain group of mineral. For example grass grain according to Okunna (1983), obtain much of their brittle strength from silica. He also pointed out that, ash is more likely to be more fluxing if the plant is harder and woodier.

## 2.11 Application of Saw Dust in Glass Preparation

Saw dust has been used in making insulating bricks for furnaces used in glass melting. The saw dusts are incorporated into mixture of raw materials to form the bricks, when fired it leave space within the bricks with entrapped air. Because air is poor conductor of heat, it gives such bricks an insulating property.

## 2.12 Types of Glass

For a particular type of glass to be produced, good selection of raw materials most be made. Such materials as described by Shand (1958) should be weighed accurately in correct proportions to yield glass of desired composition. Slight variation in the amount of particular material can lead to the formation of different type of glass; therefore, glasses are classified based on the raw materials used to yield that glass. Major silicate glass types include soda lime silica glass, borosilicate glass and vitreous silica glass

### 2.12.1 Soda Lime Silica Glass

This is the earliest glass made and still the commonest. It is believed to account for over 90% of all glasses in present day use. It is used for the production of bottles, jars, window sheets and electric lamp (bulb) etc. Soda lime silica glass has this composition: $SiO_2$ 73.0, $Al_2O_3$ 0.5,CaO 8.0, MgO 3.0, $Na_2O$ 15.5 (Samuel, 1974).

Shand (1958) stated that Soda ($Na_2O$), and sometimes a small quantity of potash ($K_2O$), are added as fluxing agents, while lime (CaO), with small quantity of alumina, ($Al_2O_3$), and magnesia (MgO) are added to improve the chemical durability of the glass. The

proportion of these oxides may differ slightly depending upon the particular uses for which the glass is intended. Soda lime silica glass according to Shelby (2005) is used in flat glasses used for windows, mirrors, and automotive applications, container glasses, or glasses used for incandescent lamps.Some soda lime glass composition are:

**i. Electric Lamp Bulbs:** Samuel (1974) gave the following as American bulb and tubing glass composition, 72.5% $SiO_2$, 1.6% $Al_2O_3$, 4.9% CaO, 3.5% MgO, 17.5% $Na_2O$, 0.5% $K_2O$. Shand (1958), provided the following as soda lime electric lamp bulbs glass composition 73.6% $SiO_2$, 1.0% $Al_2O_3$, 5.2% CaO, 3.6% MgO, 16.0% $Na_2O$, 0.6% $K_2O$. While Schole (1988), gave the following as lamp bulbs glass composition, 71.5% $SiO_2$, 2.0% $Al_2O_3$, 6.6% CaO, 2.8% MgO, 15.5% $Na_2O$, 1.0.% $K_2O$.

**ii. Window Glass:** Schole (1988), provided the following as composition of glass containers, 72.2% $SiO_2$, 1.9% $Al_2O_3$, 9.6% CaO, 1.5% MgO, 14.6% $Na_2O$, Samuel (1974), presented this composition as British cheap table ware 72.0% $SiO_2$, 4.3% ZnO, 10.8% $K_2O$, 6.3% $Na_2O$,

### 2.12.2 Vitreous Silica Glass (Pure Fused Quartz)

Vitreous silica is made up of 100% silica. This is an extremely hard glass with a very high melting temperature. It has a low coefficient of thermal expansion and thus a high thermal shock resistance. It has a high chemical durability, Thus it is used in the manufacture of prisms and windows for optical instruments especially where a high degree of transmission in the ultraviolet and near infrared region is required. They are not normally

found in the homes. This is high temperature material; it can be used up to 1000 degrees centigrade. Vycor is 97% pure silica or sand. This type of glass is also used for optical fibers, ultraviolet and infrared transmitting optics.

### 2.12.3 Borosilicate Glass

This type of glass contains about 75-81% silica, 10-13.5% boric oxide, 4.5-6.0% alkali oxide and 2-5% alumina. It is a glass with a lower coefficient of thermal expansion, but with a lower resistance to thermal shock when compared to vitreous silica glass. It has a lower melting temperature when compared to fused silica. It also has a high chemical durability. These properties explain its versatile application as laboratory glass wares, oven wares, table wares and industrial glass wares. Borosilicate glasses are crystal-white in cross-section. Relatively low thermal expansion helps to make this glassware resist cracking from heat during experiments.

## 2.13 Physical Properties of Soda Glass

i. **Tensile Strength:** Normally the tensile strength of soda glass ranges between 280 and 560 kg per sq cm (4,000 and 8,000 lb per sq in), but exceed 7,000 kg per sq cm (100,000 lb per sq in) if the glass is specially treated.

ii. **Density** Density of glass ranges from 2.16gcm$^{-3}$ to 2.89gcm$^{-3}$ Schole (1958). Shand (1958), provided the densities of some glasses as: fused silica glass 2.20 gcm$^{-3}$, soda lime silica, window sheet 2.46 gcm$^{-3}$ to 2.49 gcm$^{-3}$, soda lime electric lamp bulbs 2.47 gcm$^{-3}$, borosilicate glass 2.23 gcm$^{-3}$.

## 2.14 Chemical Properties of Glass

Shand (1958) pointed out that "chemical durability of silicate glasses results from the inert nature of silica itself. Silica is practically insoluble in water except at high temperature. Acids have little effect on silica, with the exception of hydrofluoric acid and at high temperature phosphoric acid. The solubility of silica increases with the alkalinity of the solution, and with highly alkaline solutions at elevated temperatures the rate of attack may become pronounced. The alkali oxides $Na_2O$ and $K_2O$ commonly used as flux, do not enter the silica network but remains in holes in the structure where they retain some degree of mobility and whence they can be extracted by various reagents. They can however, be held more securely in their places by the addition of the other modifying oxides, such as CaO, MgO, $Al_2O_3$, $B_2O_3$, that increase the durability of the glass".

Van Thoor (1971) pointed out that such extraction (chemical action) of the mobile oxides and even glass forming oxides occur in two stages:

i. Reaction of involving diffusion of ions in the glass network out into the attacking materials and counter attacking of the materials into the glass. example of such reaction is as indicated by the equation,

$$Si\text{-}O^- Na^+ + H^+ \longrightarrow Si - OH + Na^+$$

(Glass) (Attacking soln) (Glass) (Solution)

This type of reaction occurs in early stages of attack by water, acids and their vapours.

ii. reaction in which in which silicate network is broken, example

$$Si\text{-}O\text{-}Si + OH^- \longrightarrow Si\text{-}O^- HO\text{-}Si$$

(Glass) (Attacking soln) (Glass)

This type of attack occurs in the advanced stages of attack by water, water vapour and alkali solutions.

## 2.15 Raw Materials Analysis

For any material to be used as glass making raw material, it most obtain certain quality requirement, such requirement is certified on if the raw material is subjected to any standard analysis that give detail information of its quantitative and qualitative chemical constituents. Raw material analysis is carried out in stages as follows:

## 2.16 Samples Preparation

Solid samples are digested before being analysed. Digestion simply means the burning of organic substances in the sample. It is of two forms, dry digestion (Ashing) and wet digestion.

i. *Dry digestion or Ashing* is carried out according to Christian (2004) on animals and plant samples. Their tissues are decomposed at temperature of $400^0$c – $700^0$c in a muffle furnace. Porcelain crucibles can be used and heating should be gradually done to full temperature to prevent rapid combustion and foaming.

ii. *Wet digestion* is carried out with a mixture of nitric acid and sulphuric acid in a ratio of 1:4. Nitric acid destroys the bulk of organic matter, but does not get enough to destroy the last traces. It is boiled off during digestion process until sulphuric acid remains and dense, while $SO_3$ fumes are evolved and began to refluxing the flask. At this point, the solution gets very hot, and sulphuric acid acts on the remaining organic material.

Digestion can also be done with mixture of nitric acid, perchloric acid and sulphuric acid in a ratio of 3:1:1 (Christian, 2004).

Organic samples has to undergo the two processes if need to be analyzed by Atomic absorption spectroscopy or Flame photometer, but if it is to be analyzed by x ray florescence, it only have to pass through ashing depending on what is to be analyzed.

## 2.17 Atomic Absorption Spectroscopy

In atomic absorption spectroscopy the sample is heated to a high temperature and thereby decomposed into atoms and ions that absorb or emit visible or ultraviolet electro magnetic radiation (e.m.r) at energies characteristic of the elements involved. The yellowing of a flame by the addition of salt, for example, occurs because the sodium in salt emits strongly in the yellow portion of the visible light e.m.r spectrum. These methods are especially useful for low concentrations of metallic elements in both qualitative and quantitative analysis. (Motz, 2007).

## 2.18 X ray Flourescence

X-ray fluorescence (XRF) spectrometer as described by Wirth and Barth (2013) "is an x-ray instrument used for routine, relatively non-destructive chemical analyses of rocks, minerals, sediments and fluids. It works on wavelength-dispersive spectroscopic principles that are similar to an electron microprobe (EPMA). However, an XRF cannot generally make analyses at the small spot sizes typical of EPMA work (2-5 microns), so it is typically used for bulk analyses of larger fractions of geological materials. The relative ease and low

cost of sample preparation, and the stability and ease of use of x-ray spectrometers make this one of the most widely used methods for analysis of major and trace elements in rocks, minerals, and sediment". It has application in the field of soil surveys, mining (e.g., measuring the grade of ore), cement production, ceramic and glass manufacturing, metallurgy (e.g., quality control), environmental studies (e.g., analyses of particulate matter on air filters) etc. (Watson and Suchy, 2007).

## 2.19 The Scanning Electron Microscope (SEM)

As described by Swapp (2013), scanning electron microscope uses a focused beam of high-energy electrons to generate a variety of signals at the surface of the solid specimens. The signals that are derived from electron-sample interactions reveal information about the sample including external morphology (texture), chemical composition, and crystalline structure and orientation of materials making up the sample. In most applications, data are collected over a selected area of the surface of the sample, and a 2-dimensional image is generated that displays spatial variations in these properties. Areas ranging from approximately 1 cm to 5 microns in width can be imaged in a scanning mode using conventional SEM techniques (magnification ranging from 20X to approximately 30,000X, spatial resolution of 50 to 100 nm). The SEM is also capable of performing analyses of selected point locations on the sample; this approach is especially useful in qualitatively or semi-quantitatively determining chemical compositions using energy dispersive spectroscopy (EDS).

# CHAPTER THREE

# METHODOLOGY

This research looked at the possibility of producing soda lime silica glass from saw dust ash. Purified sand, sodium carbonate and calcium carbonate were used to provide balance oxides to form the required batching proportion.

## 3.1 Apparatus and Materials

Atomic absorption spectrophotometer, furnace, crucible, pestle and mortar, XRD, SEM, saw dust ash, soda ash (sodium carbonate), lime (calcium carbonate), potassium carbonate, alluminium hydroxide and magnesium carbonate.

## 3.2 Methods

This involves the raw materials sampling, ashing, analysis of ash, preparation of raw materials, test melting and properties testing.

### 3.2.1 Raw Material Sourcing

The saw dust used for this research was obtained from Sabon Gari, Zaria Saw Mills. Four different types of saw dust were collected.

i. Terminalia superba (White Afara)

ii. Anogeissus leiocarpus (Light red marke, chew stick tree)

iii. Isoberlinia doka (Red doka)

iv. Mixed saw dust , this is the saw dust from trees without taking a record of the type of tree.

v. Purified sand produced by BDH limited, England with product number 33094.

vi. Sodium carbonate produced by Hopkin and Williams limited, England with product number 777000G. It has an assay of 99.5% $Na_2CO_3$.

vii. Calcium carbonate produced by Burgoyne and Burbide and company, United kingdom. It has an assay of 98.0% $CaCO_3$.

viii. Aluminium hydrate produced by Burgoyne and Burbide and company, United Kingdom. It has an assay of 98% $Al_2O_3.3H_2O$.

ix. Magnesium carbonate produced by BDH limited, England with product number10198. It has an assay of 99.9% $MgCO_3$.

### 3.2.2 Samples Preparation

The saw dust collected was washed to remove unwanted materials such as clay. It was then sun dried and its ash was obtained by putting the saw dust samples in a crucibles and heated in the muffle furnace at a temperature of $550^0$C for one hour.

Obtaining ash from by muffling dry ashing is achieved by putting the sample in the muffle furnace at a temperature of $400^0$c - $700^0$c. The organic matter will burn off leaving an inorganic residue (Christian, 2004).

### 3.2.3 Digestion of Saw Dust Ash

Before embarking on chemical analysis of the samples, it was digested by taking 1g of each ash samples into a beaker, 15ml concentrated HCl and 5ml concentrated $HNO_3$ was added and kept over a hot plate. After some times, 20ml dilute $H_2SO_4$ was added. It was

heated to dryness. It was allowed to cool and about 100ml water and few drops of dilute $H_2SO_4$ was added until a clear solution was obtained. 30ml 40N HF was added and boiled for one minute and was filtered at once using 40 whatman filter paper. The residue was washed with hot water 3 times.

### 3.2.4 Chemical Analysis of Ash

The amounts of Si, Al, Ca, Mg and Fe in the ash samples were analyzed by atomic absorption spectrophotometry (A.A.S) in the Multi User Laboratory of Department of Chemistry, Ahmadu Bello University, Zaria. While the amount of K and Na were determined by flame photometry at the Soil Science Depatrment, Ahmadu Bello University, Zaria. The concentrations of elements and oxides in each sample were calculated using the following relationships.

#### i. Concentration of Elements in the Ash Samples

The percentage elements in the samples were calculated by converting the concentration in ppm (mg $dm^{-3}$) to $mgKg^{-1}$ using the relationship:

$$conc.of\ elememt\ in\ mgKg^{-1} = \frac{concentration\ in\ ppm \times volume\ of\ digested\ sample}{weight\ of\ sample\ taken}$$

$$percentage\ elememt = \frac{concentration\ in mg Kg^{-1} \times volume\ of\ digested\ sample}{10^6} \times 100$$

The percentage concentrations of elements in each sample are presented on table 1.

**ii. Percentage Concentration of Oxides in the Ash Samples**

The percentage concentrations of oxides in the ash samples were obtained using the relationship:

$$percentage\ oxide\ = \frac{percentage\ element \times molar\ mass\ of\ oxide}{atomic\ weight\ of\ element}$$

The results obtained are presented on Tables 4.2 and 4.3.

## 3.3 Batch Formulation

The composition of soda lime silica glass prepared is represented in Table 3.1. This is the composition given by Samuel (1974) as the composition of American bulb and tubing glass. This forms the basis for calculation of the batches.

**Table 3.1 Soda Lime Silica Glass Composition**

| Oxide | Weight Percentage |
|---|---|
| **$SiO_2$** | 72.5 |
| **$NaO_2$** | 17.5 |
| **CaO** | 4.9 |
| **$K_2O$** | 0.5 |
| **$Al_2O_3$** | 1.6 |
| **MgO** | 3.5 |

Samuel (1974)

Four glass batches were prepared from the saw dusts ashes obtained. Different composition of the batches were obtained from the percentage oxides in the four ash samples coded 1 to 4. Sand was used as the major glass former while the ash, sodium carbonate, calcium carbonate, potassium carbonate, aluminum hydroxide and magnesium carbonate were used to provide other oxides required in each batch.

i. Sand is the source of $SiO_2$ and it contained 98.80% $SiO_2$.
ii. Sodium carbonate is the supplementary source of $Na_2O$ and is obtained as the decomposition product of $Na_2CO_3$.

$$Na_2CO_3 \longrightarrow Na_2O+CO_2 \qquad \text{eqn 1.}$$

106g $Na_2CO_3$ supply 62g $Na_2O$

100g $Na_2CO_3$ will supply 62/106x100 = 58.50g

iii. $CaCO_3$ is the supplementary source of CaO and is obtained as the decomposition product of $CaCO_3$ according to the following equation:

$$CaCO_3 \longrightarrow CaO+CO_2 \qquad \text{eqn 2.}$$

100.1 supply 56.1 CaO

100g $CaCO_3$ will supply 56.00g CaO

iv. $K_2CO_3$ is the supplementary source of $K_2O$ and is obtained as the decomposition product of $K_2CO_3$ according to the following equation:

$$K_2CO_3 \longrightarrow K_2O+ CO_2 \qquad \text{eqn 3.}$$

138.2g $K_2CO_3$ supply 94.2g $K_2O$

100g $K_2CO_3$ will supply 68.16g $K_2O$

v. $Al_2O_3.3H_2O$ is the supplementary source of $Al_2O_3$ and is obtained as the decomposition product of $Al_2O_3.3H_2O$ according to the following equation:

$$Al_2O_3.3H_2O \longrightarrow Al_2O_3+3H_2O \qquad \text{eqn 4.}$$

$Al_2O_3.3H_2O$ 137.8 supply 101.8g $Al_2O_3$

100g $Al_2O_3.3H_2O$ will supply 73.90g $Al_2O_3$

vi. $MgCO_3$ is the supplementary source of MgO and is obtained as the decomposition product of $MgCO_3$ according to the following equation:

$MgCO_3 \longrightarrow MgO+CO_2$ eqn 5.

84.3g $MgCO_3$ supply 40.3g MgO

100g $MgCO_3$ will supply 47.81g MgO

**Batch 1**

The oxides in this batch was based on the result of the chemical analysis of Afara saw dust and oxides introduced by other raw materials. The weight of oxides in 100g of raw materials is tabulated in table 3.2:

**Table 3.2 Contribution of Oxides by 100g of Raw Materials**

| | **$SiO_2$** | **$Na_2O$** | **CaO** | **$K_2O$** | **$Al_2O_3$** | **MgO** |
|---|---|---|---|---|---|---|
| **Afara ash** | 7.85 | 5.40 | 24.80 | 18.00 | 1.89 | 5.07 |
| **Sand** | 98.80 | _ | _ | _ | _ | _ |
| **$Na_2CO_3$** | _ | 58.50 | _ | _ | _ | _ |
| **$CaCO_3$** | _ | _ | 56.00 | _ | _ | _ |
| **$K_2CO_3$** | _ | _ | _ | 68.16 | _ | _ |
| **$Al_2O_3.3H_2O$** | _ | _ | _ | _ | 73.90 | _ |
| **$MgCO_3$** | _ | _ | _ | _ | _ | 47.81 |

Calculations using approximation method based on the 100g of glass of composition $72.5SiO_2.17.5Na_2O.4.9CaO.0.5K_2O.1.6Al_2O_3.3.5MgO$

**Assumption 1**

Afara ash will supply all $Na_2O$ (17.5g) required in the glass batch

100g Ash introduce 5.40g $Na_2O$

17.5g $Na_2O$ will be supplied by 100/5.40x17.5= 324.10g ash

Glass batch requires 4.9% CaO

Amount of CaO to be introduced by 324.10g ash which contains 24.80% CaO = 24.80/100x324.10 = 80.38

This introduces 80.38-4.9 = 75.48g in excess of CaO required to form the batch, therefore it proved assumption 1 wrong.

**Assumption 2:**

Afara ash will supply all CaO (4.9g) required in the glass batch

100g Ash introduce 24.80g CaO

4.9g CaO will be supplied by 4.9/24.8x100 = 19.76g afara ash

Amount of $SiO_2$ introduced by 19.76g ash which contains 7.85g $SiO_2$= 7.85/100x19.76 = 1.55g

Balance to be introduced by sand = 72.5-1.55 = 70.95g $SiO_2$

Amount of sand to introduce 70.95g $SiO_2$ (sand contains 98.80g $SiO_2$)

= 98.80/100x70.95 = 70g

Amount of $Na_2O$ to be introduced by 19.76g afara ash which contains 1.89% $Na_2O$ = 1.89/100x19.76 = 0.37g

Balance to be introduced by $Na_2CO_3$ = 17.5-0.37= 17.13g

100g $Na_2CO_3$ introduce 58.50g $Na_2O$

Amount of $Na_2CO_3$ to introduce 17.13g $Na_2O$ = 100/58.50x17.13= 29.30g

Amount of $Al_2O_3$ introduced by 19.76g ash which contains 1.89g $Al_2O_3$ = 1.89/100x19.76 = 0.37%

Balance to be introduced by $Al_2O_3.3H_2O$ = 1.6-0.37 = 1.23%

Amount of $Al_2O_3.3H_2O$ to introduce the balance of 1.23% (100g $Al_2O_3.3H_2O$ contains 65.34g $Al_2O_3$) = 100/65.34x1.23= 1.9g

Amount of $K_2O$ introduced by 19.76g of Afara ash which contains 18.00% $K_2O$ = 18.00/100 x 19.76 = 3.60% this introduces an excess of 3.10% $K_2O$.

Amount of MgO introduced by 19.76g ash which contains 5.07% MgO= 5.07/100x19.76 = 1.00%

Balance to be introduced by $MgCO_3$ =3.5-1= 2.5%

Amount of $MgCO_3$ 2.5% MgO (100g $MgCO_3$ contains 47.81g MgO)

= 100/47.81x2.5 = 5.23g

**Table 3.3a: Summary of Oxides Introduced by 100g of Glass containing Saw Dust Ash of White Afara (*Terminalia superba)***

| Wght/100g of glass | Ash | Sand | $Na_2CO_3$ | $CaCO_3$ | $K_2CO_3$ | $Al_2O_3.3H_2O$ | $MgCO_3$ |
|---|---|---|---|---|---|---|---|
| **$SiO_2$** | 1.55 | 70.95 | _ | _ | _ | _ | _ |
| **$Na_2O$** | 0.37 | _ | 17.13 | _ | _ | _ | _ |
| **CaO** | 4.90 | _ | | _ | _ | _ | _ |
| **$Al_2O_3$** | 0.37 | _ | _ | _ | _ | 1.23 | _ |
| **MgO** | 1.00 | _ | _ | _ | _ | _ | 2.50 |

**Table 3.3b: Weight of the Raw Materials to Form Batch 1**

| Raw materials | Weight/100 g |
|---|---|
| **Ash** | 19.76 |
| **Sand** | 70.00 |
| **$Na_2CO_3$** | 29.30 |
| **$Al_2O_3.3H_2O$** | 1.90 |
| **$MgCO_3$** | 5.23 |

Same procedure was repeated for batch 2, 3 and 4 taking into consideration the possibility of adopting any assumption to form the batch, until more reasonable result is obtained, so it is taken as most acceptable for batch formation.

**Batch 2**

The oxides in this batch was based on the 7.31% $SiO_2$; 2.27% $Al_2O_3$; 22.80% $K_2O$; 19.05% CaO; 9.17% $Na_2O$; 9.04%MgO obtained from the ash of light Marke Saw dust, and oxides contribution by other raw materials as indicated in table 3.4b.

**Table 3.4a: Summary of Oxides Introduced by 100g of Glass Containing Saw Dust Ash of Marke (*Anogeissus leiocarpus)***

| **Wt/100g of glass** | **Ash** | **Sand** | **$Na_2CO_3$** | **$CaCO_3$** | **$K_2CO_3$** | **$Al_2O_3.3H_2O$** | **$MgCO_3$** |
|---|---|---|---|---|---|---|---|
| **$SiO_2$** | 2.00 | 70.50 | _ | _ | _ | _ | _ |
| **$Na_2O$** | 2.36 | _ | 15.14 | _ | _ | _ | _ |
| **Cao** | 4.90 | _ | _ | _ | _ | _ | _ |
| **$K_2O$** | 5.86 | _ | _ | _ | _ | _ | _ |
| **$Al_2O_3$** | 0.58 | _ | _ | _ | _ | 1.02 | _ |
| **MgO** | 2.33 | _ | _ | _ | _ | _ | 1.17 |

**Table 3.4b: Weight of the Raw Materials to Form Batch 2**

| **Raw materials** | **Weight/100 g** |
|---|---|
| **Ash** | 25.72 |
| **Sand** | 71.40 |
| **$Na_2CO_3$** | 25.80 |
| **$Al_2O_3.3H_2O$** | 2.33 |
| **$MgCO_3$** | 2.45 |

**Batch 3**

The oxides in this batch was based on the 7.10% $SiO_2$; 6.42% $Al_2O_3$; 21.50% $K_2O$; 8.42% CaO; 7.55% $Na_2O$; 24.21%MgO obtained from the ash of red Doka Saw dust and oxides contribution by other raw materials as indicated in table 3.5b.

**Table 3.5a: Summary of Oxides Introduced by 100g of Glass Containing Saw Dust Ash of Doka (*Isoberlinea doka)***

| Wt/100g of glass | Ash | Sand | $Na_2CO_3$ | $CaCO_3$ | $K_2CO_3$ | $Al_2O_3.3H_2O$ | $MgCO_3$ |
|---|---|---|---|---|---|---|---|
| **$SiO_2$** | 1.03 | 71.47 | _ | _ | _ | _ | _ |
| **$Na_2O$** | 1.10 | _ | 16.40 | _ | _ | _ | _ |
| **Cao** | 1.22 | _ | _ | 3.68 | _ | _ | _ |
| **$K_2O$** | 2.62 | _ | _ | _ | _ | _ | _ |
| **$Al_2O_3$** | 0.90 | _ | _ | _ | _ | 0.70 | _ |
| **MgO** | 3.50 | _ | _ | _ | _ | _ | _ |

**Table 3.5b: Weight of the Raw Materials to Form Batch 3**

| Raw materials | Weight/100 g |
|---|---|
| **Ash** | 14.50 |
| **Sand** | 72.30 |
| **$Na_2CO_3$** | 28.00 |
| **$CaCO_3$** | 6.60 |
| **$Al_2O_3.3H_2O$** | 1.07 |
| **$MgCO_3$** | 0.00 |

**Batch 4**

The oxides in this batch was based on the the 7.27% $SiO_2$; 7.56% $Al_2O_3$; 29.90% $K_2O$; 24.47% CaO; 5.40% $Na_2O$; 15.61%MgO obtained from the ash of mixed Saw dust and oxides contribution by supplementary raw materials as indicated in table 3.6b.

**Table 3.6a: Summary of Oxides Introduced by 100g of Glass Containing Saw Dust Ash of Mixed Saw Dust**

| **Wt/100g of glass** | **ash** | **Sand** | **$Na_2CO_3$** | **$CaCO_3$** | **$K_2CO_3$** | **$Al_2O_3.3H_2O$** | **$MgCO_3$** |
|---|---|---|---|---|---|---|---|
| **$SiO_2$** | 1.34 | 71.16 | _ | _ | _ | _ | _ |
| **$Na_2O$** | 1.10 | _ | 16.50 | _ | _ | _ | _ |
| **Cao** | 4.50 | _ | _ | _ | _ | _ | _ |
| **$K_2O$** | 5.00 | _ | _ | _ | _ | _ | _ |
| **$Al_2O_3$** | 1.40 | _ | _ | _ | _ | 0.10 | _ |
| **MgO** | 2.90 | _ | _ | _ | _ | _ | 0.60 |

**Table 3.6b: Weight of the Raw Materials to Form Batch 4**

| **Raw materials** | **Weight/100 g** |
|---|---|
| **Ash** | 18.40 |
| **Sand** | 72.02 |
| **$Na_2CO_3$** | 28.21 |
| **$Al_2O_3.3H_2O$** | 0.15 |
| **$MgCO_3$** | 1.25 |

## 3.4 Test Melting

The four batches formulated were melted in an open refractory crucible in an electric furnace at a temperature of $1300^0$C for 5 hours. It was conducted at the National Research Institute for Chemical Technology, Zaria. Soda lime silicate glass according to Varshneya (1994) is expected to melt at temperature of $1400^0$C – $1500^0$C. When the required temperature is reached, the furnace will be off and will be allowed to cool to room temperature while the samples are still in.

## 3.5 Properties Test

The properties tested were the density, by measurement and by calculation; the chemical durability test by weight loss method. Also X-ray diffraction (XRD), as well as scanning electron microscopy (SEM) of the glass prepared was tested:

### 3.5.1 Density Test

Density of glass is measured by any of the following methods:

1. *Determination by Archimedes principle:* Varshneya (1994), provided that density of glass is measured most commonly by weighing a suitable selected piece of glass in air and dividing this weight by the buoyancy, which is reduction in weight when the sample is suspended in water. According to Archimedes, buoyancy equal to the volume of displaced water. Therefore, density $\rho = W_a/(W_a-W_b)$ , where $W_a$ = weight of specimen in air and $W_b$ = weight of specimen in water.

2. *Sink Float Method:* the unknown glass is gently dropped into a tube containing a slightly denser solution of organic liquid such as methylene iodide. The temperature around the tube is gradually changed until the previously floating specimen begins to sink. Calibrations of the liquid density against temperature yield the density of the unknown.

3. *By using pycnometer:* Samuel described that this method is carried out by crushing the glass sample to a definite size and sieved to remove dust. It is then dried and small quantity is weighed. A pycnometer or density bottle is first weighed and then the glass is put into it and is re weighed. The density is the calculated using the relationship:

$$\rho = \frac{w}{w+(w1-w2)}$$

Where w = weight of the glass

$w_1$ = weight of density bottle with water

$w_2$ = weight of weight of the density bottle with the glass and water.

4. *By calculation:* Samuel further stated that density of glass can be determined by calculation using the formula:

$$\frac{100}{p} = \frac{p1}{di} + \frac{p2}{d2} = \frac{p3}{d3} \dots\dots\dots\dots..$$

Where $p_1$, $p_2$, $p_3$ ----- are the percentages of the oxides present

$d_1$,$d_2$,$d_3$,-------- are the corresponding density factors of the oxides.

The densities of the glass samples were measured at room temperature using Archimedes principle (Displacement method) with water as an immersion fluid at the chemistry laboratory of Nuhu Bamalli Polytechnic, Zaria. The density of each glass was also calculated using the relationship,

$$\rho = \frac{m}{v}$$

Where m is the weight of glass before immersing in fluids and v is the volume of water displaced which is equal to the volume of glass sample immersed in it. The weight of the sample in the fluid is equal to the volume of fluid displaced (Varshneya, 1994).

For further verification, density was also calculated using the relationship:

$$\frac{100}{\rho} = \frac{p1}{d1} + \frac{p2}{d2} + \frac{p3}{d3} \quad \text{--------}$$

Where, ρ is the density, p percentage of oxides introduced into glass and d is the density factors of the oxides.

### 3.5.2 Chemical Durability

Chemical durability of glass can be measured by one of the following methods:

1. ***Durability test:*** Varshneya (1994), pointed out that chemical durability of glass can be measured by immersing the glass sample in a given quantity of fluid contained in a covered non reacting polymer beaker at a controlled temperature in shaker bath. The quantity of material leached is obtained by either measuring the weight change, flame photometry or atomic absorption analysis of the leachate. Conducting this experiment with pulverized samples or at elevated temperature take shorter time. Samuel (1974)

also considered this method as effective, but emphasized determining of alkali liberated, that is applying flame photometry or atomic absorption spectroscopy.

2. ***Powder test:*** By this method, Samuel (1974) described that 5g of powdered glass 40 to 50 mesh size is heated with 100cm$^3$ water, in platinum basin at 80$^0$c for an hour. It is then filtered and residue washed free from alkali, the filtrate is then titrated against $H_2SO_4$ solution and calculation of $H_2SO_4$ value is made for 100g of glass.

The test was carried out using the method described by Samuel (1974) as weight loss test at the chemistry laboratory of Nuhu Bamalli Polytechnic, Zaria. 5g of crushed glasses were measured and transferred into two conical flasks and labeled A and B respectively for glass samples 1 and 4. 25cm$^3$ of concentrated HCl was added to each glass samples and allowed to stay for an hour on a shaker bath. It was then filtered. The residue was allowed to dry and reweighed.

### 3.5.3 X ray Diffraction Analysis

X ray diffraction was conducted at the Steel Raw Materials Exploration Agency, Kaduna. It was used to determine the minerals formation in the glass samples. It was conducted in Shimadzu 6000XRD model.

### 3.5.4 Scanning Electron Microscopy

This was conducted at the Department Chemical Engineering, Ahmadu Bello University Zaria, using Pro X SEM model. Morphology of the glass samples obtained was scanned and energy dispersive spectroscopy of selected spots on each glass samples was analyzed.

i. Morphology to view the external features of the glasses prepared, it was carried out on a SEM machine.

ii. Energy-Dispersive X ray Spectroscopy (EDS) of the glasses was taken using SEM machine. Seven spots on each glass was scanned as indicated in spectrograph in figure 3.1 and 3.2

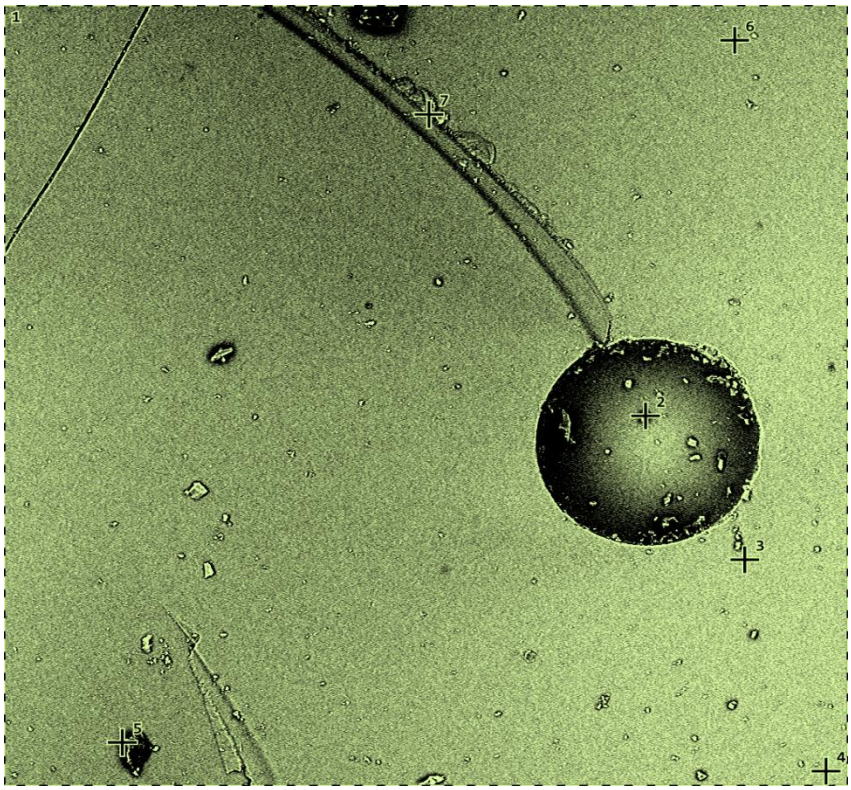



**Figure 3.1: Spectograph of Glass Sample 1 Showing the Seven Spots**

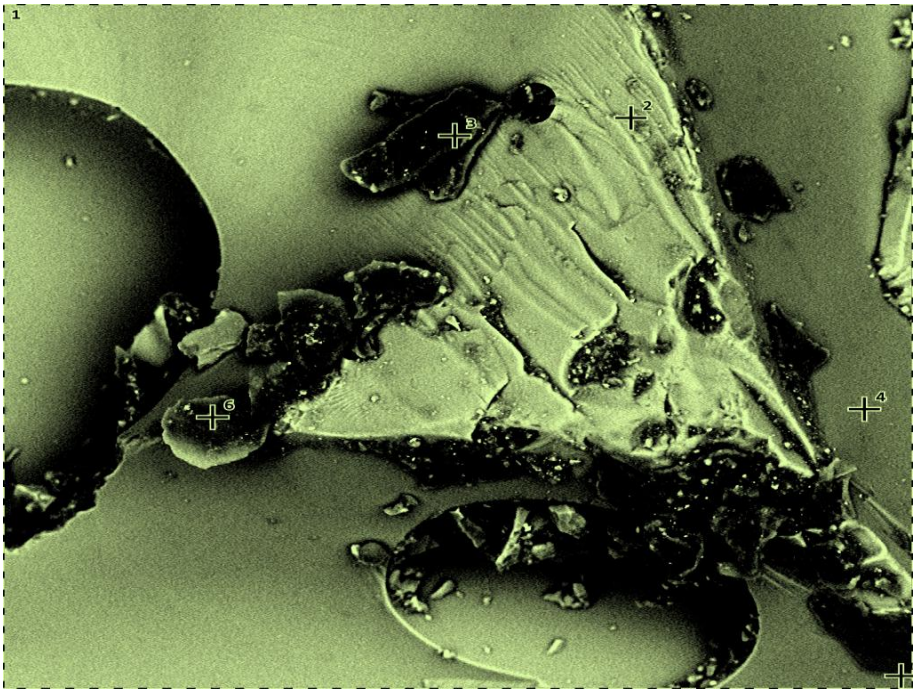



**Figure 3.2: Spectograph of Glass Sample 4 Showing the Seven Spots**

# CHAPTER FOUR

# RESULTS AND DISCUSSION

## 4.1 Result of Saw Dust Ashing

Table 4.1 gives the weight of ash obtained by burning 50g of saw dust in muffle furnace.

**Table 4.1: Weight of Ash Obtained by Burning Saw Dust in a Muffle Furnace**

| | Wt of saw dust(g) | wt of ash obtained (g) | % wt of ash |
|---|---|---|---|
| **1.Terminalia superba (Afara)** | 50.00 | 1.27 | 2.54 |
| **2.Anogeissus leiocarpus (Marke)** | 50.00 | 1.88 | 3.76 |
| **3.Isoberlinea doka (Doka)** | 50.00 | 1.11 | 2.22 |
| **4.Mixed** | 50.00 | 0.96 | 1.92 |

The result of saw dust ashing shows effectiveness of using muffle furnace because there is temperature control and total destruction of organic matter is achieved. This is proved by the difference in weight of ashes obtained to the weight of saw dust taken before ashing.

## 4.2 Result of Chemical Analysis

Elemental and oxides concentrations of the ashes are respectively presented in tables 4.2 and 4.3

**Table 4.2: Percentage Elements in the Ash Samples**

| | Si | Al | K | Ca | Na | Mg | Fe |
|---|---|---|---|---|---|---|---|
| **1. Afara (*Terminalia superba*)** | 3.67 | 0.05 | 7.46 | 17.72 | 0.20 | 3.06 | 0.69 |
| **2. Marke (*Anogeissus leiocarpus*)** | 3.42 | 0.06 | 9.46 | 13.62 | 0.34 | 5.45 | 0.48 |
| **3. Doka (*Isoberlinea doka*)** | 3.31 | 0.17 | 8.92 | 6.02 | 0.28 | 14.6 | 0.67 |
| **4.Mixed** | 3.40 | 0.02 | 12.41 | 17.47 | 0.20 | 9.41 | 1.60 |

**Table 4.3: Percentage Oxides in Ash Samples**

| | $SiO_2$ | $Al_2O_3$ | $K_2O$ | CaO | $Na_2O$ | MgO | $Fe_2O_3$ |
|---|---|---|---|---|---|---|---|
| **1. Afara (*Terminalia superba*)** | 7.85 | 1.89 | 18.00 | 24.80 | 5.40 | 5.07 | 1.97 |
| **2. Marke (*Anogeissus leiocarpus*)** | 7.31 | 2.27 | 22.80 | 19.05 | 9.17 | 9.04 | 1.37 |
| **3. Doka (*Isoberlinea doka*)** | 7.10 | 6.42 | 21.50 | 8.42 | 7.55 | 24.21 | 1.92 |
| **4.Mixed** | 7.27 | 7.56 | 29.90 | 24.47 | 5.40 | 15.61 | 4.57 |

The results of chemical analysis show that ash from Marke *(Anogeissus leiocarpus),* Doka *(Isoberlinia doka)* and mixed saw dust contains more $K_2O$, 22.80% (9.42% K), 21.50% (8.92% K) and 29.90% (12.41% K) respectively as shown in tables 4.2 and 4.3, with very minimal amount of $Fe_2O_3$. This is in accordance with the composition obtained by Tsotumu and Erich (1994) on wood ash having composition 24.80% Ca, 14.60% Si, 5.72% Al, 3.80 Mg, and 29.90% K. Ash from Afara saw dust contains 24.80% CaO (17.72% Ca) as the highest oxide obtained, this is in accordance with the composition of wood ash obtained by Mahindra *et al* (1993); 31.35% Ca, 10.25% K, 7.57% Mg, 0.09% Fe, 0.03% Al, 0.06% Na and 0.13% Si. Pit man also obtained Ca as highest element present in the two

ashes he obtained as comparative studies of hard and soft wood which is as indicated: Hard wood contain 18.00% K, 21.40% Ca, 3.30% Mg, 0.50% Fe while soft wood contains 12.00% K, 22.80% Ca, 3.80% Mg and 0.70% Fe.

If the results obtained are compared with the compositions above, it can be stated that Ca, Na and K are within permissible range while the high content of Mg in both Doka and mixed saw dust 14.60% and 9.41% respectively, can be as a result of environmental factors, or contaminations along the analysis line. While the high content of Fe in mixed saw dust might be as a result of contaminations in the saw mill, because it was collected from a bump of saw dust from different types of woods without considering its purity, it can also be contaminated in the laboratory environment.

### 4.3 Batch Formulation

The batches for this research was formulated by approximation method, although with modern technological advancements it is not usually used, but it become necessary because other methods cannot be applied to glass prepared from the ashes used in this work as its molecular weight cannot be calculated since it may contains elements outside the scope of this research, and it is an essential requirement for adopting Matrix or Gravimetric factor methods as contain in the two formulae below:

i. $$Gf = \frac{\text{Molar weight of raw material}}{\text{Molar weight of oxides}}$$

$G_f$ = Gravimetry factor

ii. $N_B = (B^T.B)^{-1}.B^T.N_G$

$N_B$ = Molarity of batch components,

$N_G$ = Molarity of glass components

B = Batching matrix

This method can still lead to formation of glass as stated by Samuel (1974), glass batch is obtainable in most cases by trial and error method, but its disadvantage is that there is always difficulty in getting exact quantities of oxides to be introduced in small quantities, such as colouring agents. This is what made it impossible to get exact percentage of $K_2O$ required in all the four batches formulated.

The saw dust ashes made an important contribution to the glass batches formulated. It formed approximately 20% of batch 1, 26% of batch 2, 15% of batch 3 and 18% of batch 4. The contribution of the ashes as contained in 100g of batch materials is shown in figures 4.1, 4.2, 4.3 and 4.4.

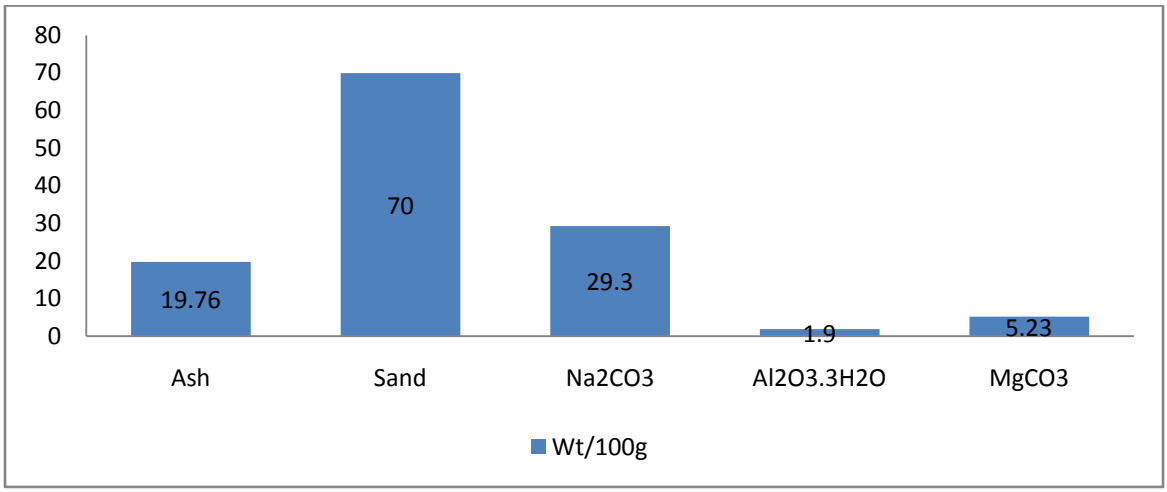


**Figure 4.1: Bar chart Showing Percentage Weight of the Raw Materials to Form Batch 1.**

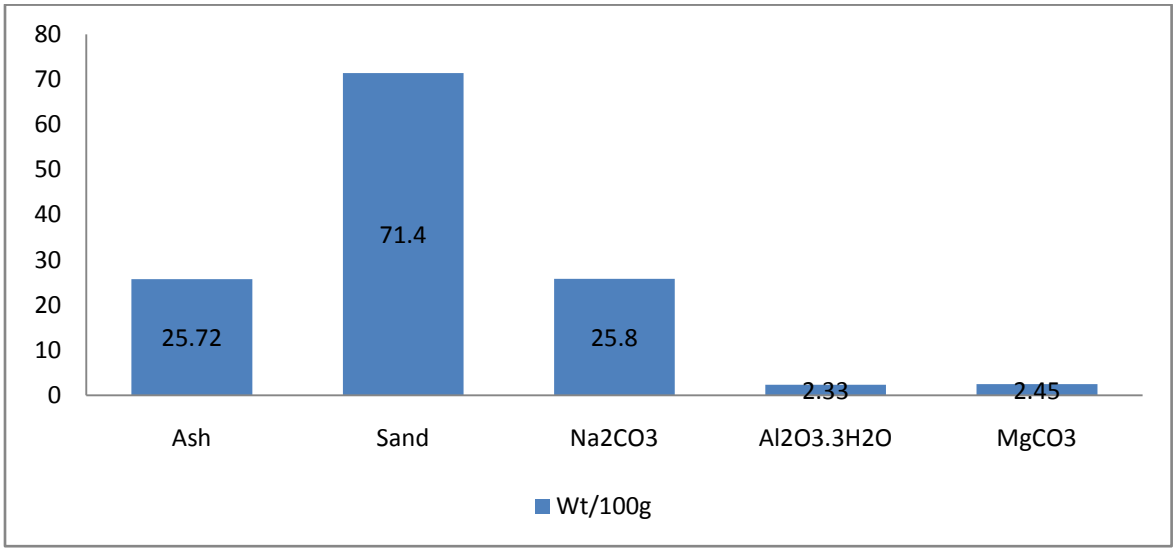



**Figure 4.2: Bar chart Showing Percentage Weight of the Raw Materials to Form Batch 2.**

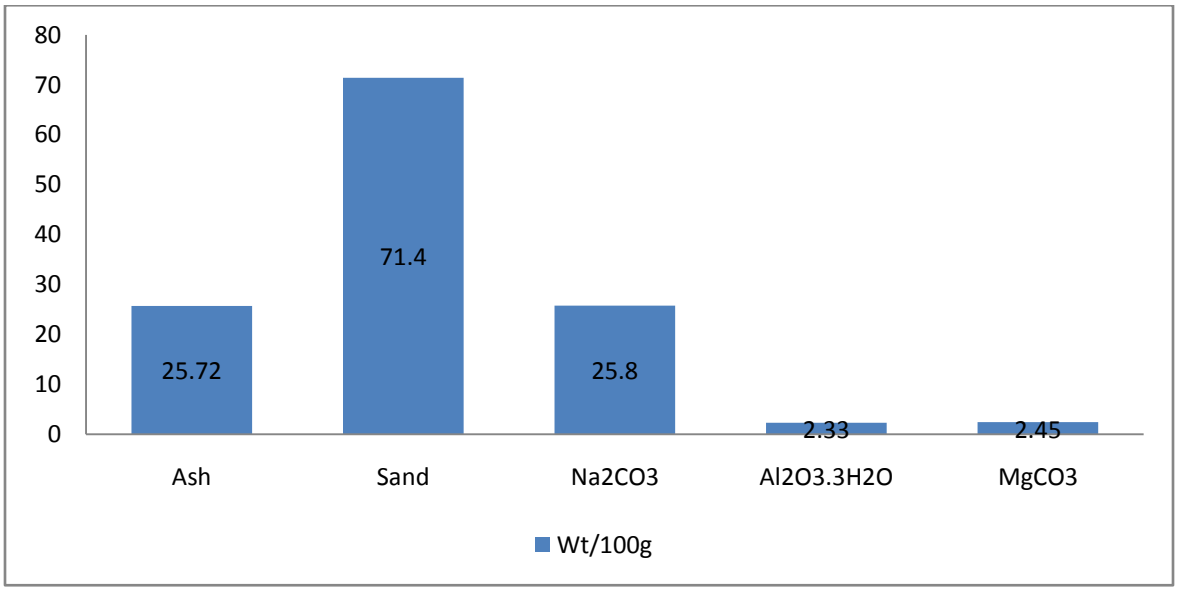



**Figure 4.3: Bar chart Showing Percentage Weight of the Raw Materials to Form Batch 3.**

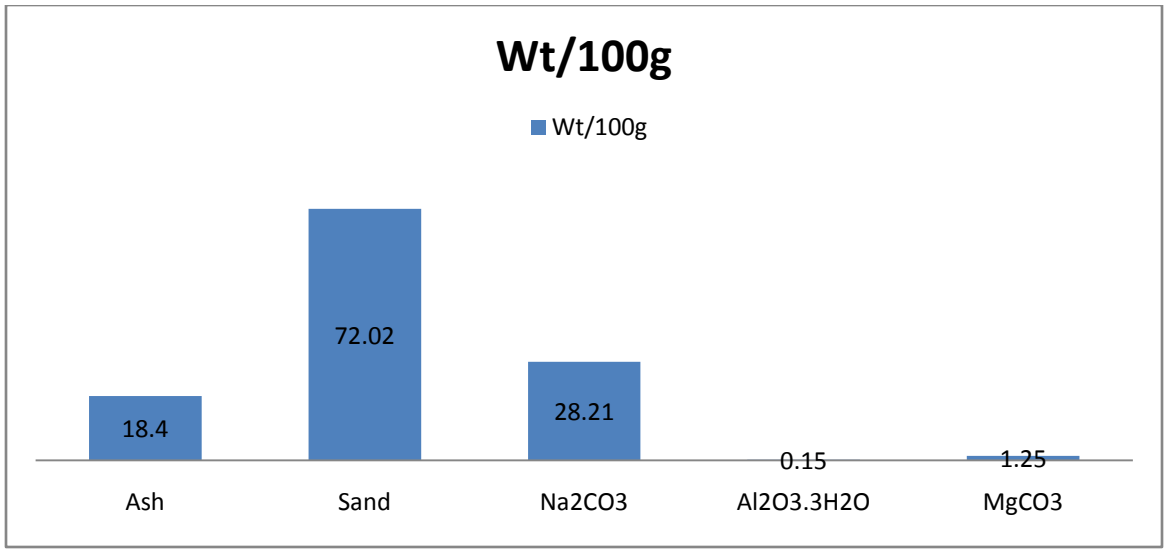



**Figure 4.4: Bar chart Showing Percentage Weight of the Raw Materials to Form Batch 4.**

## 4.4 Result of Test Melting

Glass from sample 1 was found to be transparent, and the one obtained from samples 2 and 3 was found to be white crystal like sintered materials, while the glass obtained from sample 4 was found to be light brownish glass.

Varshneya (1994) described that oxide glasses are obtained in the laboratory by melting a well mixed batch of appropriately weighed quantities of raw materials which are the constituent oxides or compound that converts to glass upon heating. Thus, the exact amount of saw dust ashes and supplementary raw materials are weighed and melted in refractory crucibles.

The result of test melting shows that batches 1 and 4 melted and formed glasses, while batches 2 and 3 sintered, this may be due to improper heat distributions within the furnace due to frequent power failure during heating. The light brownish colour obtained in

glass obtained from batch 4 is as a result of high content of $Fe_2O_3$ as shown in the result of chemical analysis in table 4.3.

## 4.5 Result of Density Test

The result density determination was found as follows:

Glass sample 1

Weight of glass before sample = 17.25g

Volume of water in the glass cylinder = $30cm^3$

Volume of water after glass sample was immersed = $37cm^3$

Volume of water displaced = $7cm^3$

$\rho = 17.25/7.00 = 2.46gcm^{-3}$

Glass sample 4

Weight of glass before sample = 21.40g

Volume of water in the glass cylinder = $30cm^3$

Volume of water after glass sample was immersed = $38.70cm^3$

Volume of water displaced = $8.70cm^3$

$\rho = 21.40/8.70 = 2.46gcm^{-3}$

By calculation, the density was obtained as follows:

Density factors of oxides are $SiO_2$ 2.3, $Na_2O$ 2.6, $K_2O$ 2.8, CaO 3.3, $Al_2O_3$ 4.1 and MgO 3.8 (Isa, 2000)

$100/\rho = p_1/d_1 + p_2/d_2$ --------

$100/\rho = 72.5/2.3 + 17.5/2.6 + 0.5/2.8 + 4.9/3.3 + 1.6/4.1 + 3.5/3.8$

$\rho = 2.43 gcm^{-3}$

The densities of glasses obtained by Archimedes principle are little above that of lead crystal glass which has a density of $3.20 gcm^{-3}$, chemical resistant glasses of density $2.25 cm^{-3}$ and fused silica glass of density 2.20 $cm^{-3}$. But the densities are very close to the density of dark green glass bottle having density 2.54 $cm^{-3}$, sheet glass of density 2.50 $cm^{-3}$ to 2.53 $cm^{-3}$ and colourless glass bottle of density 2.54 $cm^{-3}$. The variation in the values of measured and calculated values of density is another indication that the batches might have been contaminated with some impurities within the laboratory environment or from the materials forming the crucibles. As described by Shelby (2005) that the density of a glass sample that contain foreign inclusions will have higher densities than the true density, which might, for example, be due to particles of unmelted batch or crystals formed during cooling, will cause the apparent density to be greater than the true density. It can also be as a result of some ash constituents not covered within the scope of this research.

## 4.6 Result of Chemical Durability Test

After putting concentrated hydrochloric acid on the two glass samples, and allowing it to stay for an hour, little white change on the colour of glasses was observed indicating the occurrence of reaction between the acid and ions from oxides such as Na+ from NaO, Ca+ from CaO within the glasses in a process described by Varshneya (1994) as glass weathering. Weight changes lost was found to be negligible indicating that the attack is slight.

## 4.7 Result of XRD Analysis

XRD patterns of prepared glass samples 1and 4 show no sharp Bragg's peak, a broad diffuse hump which is a clear indication of amorphous nature within the resolution limit of XRD instrument. While that of samples 2 and 3 show sharp peaks indicating low or absence of amorphous body (formation of crystals). The major crystals found in glass sample 1 are Witherite (Barium carbonate), Halloysite (Aluminium silicate hydroxide) and Fluorite (Calcium fluoride). While in sample 4 the crystals found include Kaolinite (Aluminium silicate hydroxide), Talc (Magnesium silicate hydroxide), Illite ( potassium aluminium silicate hydroxide), Vickie (Aluminium silicate hydroxide).

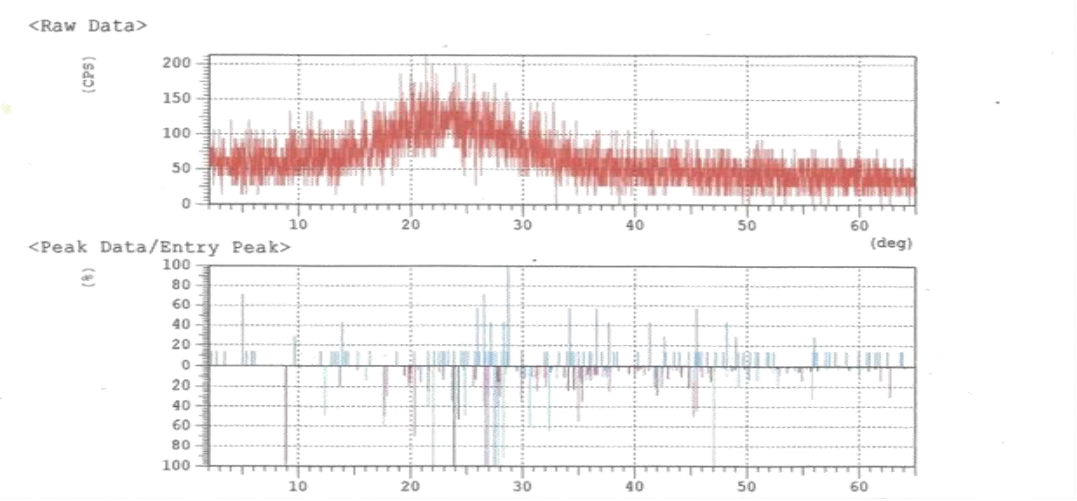



**Fig 4.5a: XRD Spectrum of Glass Sample 1**

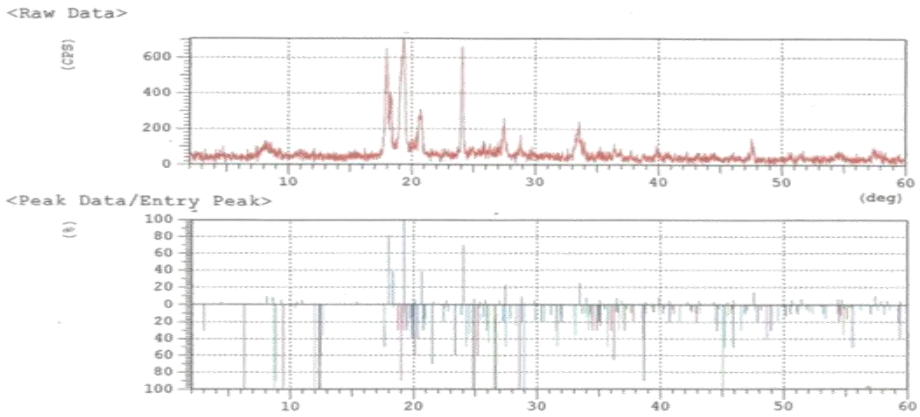



**Fig. 4.5b: XRD Spectrum of Sintered Sample 2**

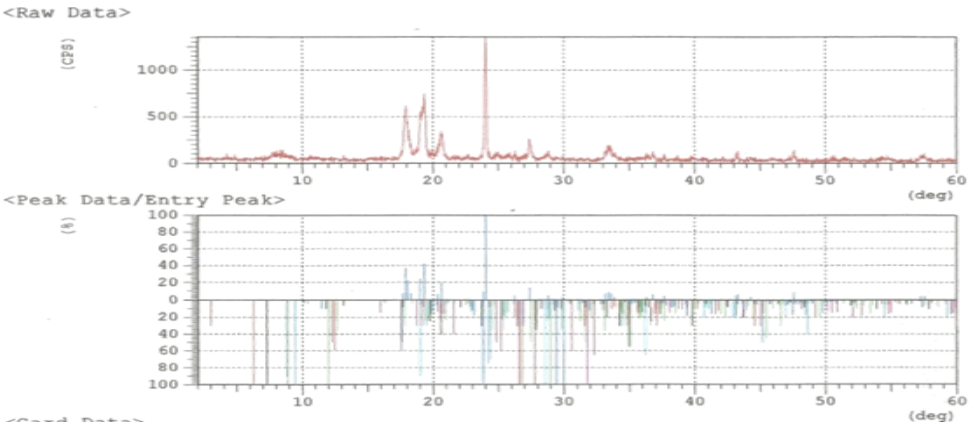



**Fig 4.5c: XRD Spectrum of Sintered Sample 3**

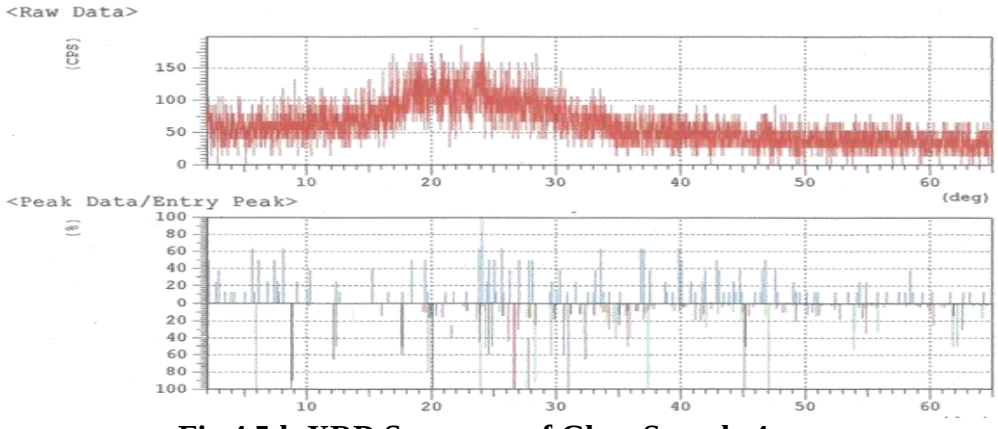



**Fig 4.5d: XRD Spectrum of Glass Sample 4**

The XRD results show narrow diffraction peaks fig. 4.5b and 4.5c in samples 3 and 4 indicating formation of crystals, this type of peaks are similar to those obtained by Ali (2008), who developed glass ceramics from locally obtainable raw materials within Nigeria. While a broad peak (halo) fig. 4.5a and 4.5d in samples 1 and 4 show the formation of amorphous components. These types of broad peak patterns or humps are in accordance with what Ward and French (2005) derived from different types of glassy materials.

The result also show occurrence of crystalline bodies not only within the sintered bodies, (samples 2 and 3) but also within the glasses obtained, (samples 1 and 4). This is in compliance with what made Varshneya (1994) to described glass as an assemblage of finely divided micro crystals as can be seen in the result of scanning electron microscopy, Fig 4.6 and 4.7.

## 4.8 Result of SEM Analysis

Morphology to study the texture of the glass samples, presence of pores and fiber were scanned by back scattered electron while energy dispersive x ray spectroscopy was used to determined elements distributions on various spots of the samples was used.

### 4.8.1 Morphology

The two glass samples obtained were enlarged to 420 times and 1000 times magnifications and scanned using back scattered electron. The surface scanning of glass sample 1 reveals some textures of varying sizes. Glass sample 4 exhibits relatively rougher surface than glass sample 1. It can also be seen, some dark portions on it indicating areas of

brown colourations on the glass surface. Also, the result shows some fiber of different diameters ranging from 905.93nm to 17.74µm. and pore of surface areas ranging from 0.41 $\mu m^2$ to 0.01$mm^2$. Figures 4.6 and 4.7 shows the surface textures of the two glasses while figures 4.8 and 4.9 show fiber and pore histograms of the two glass samples.

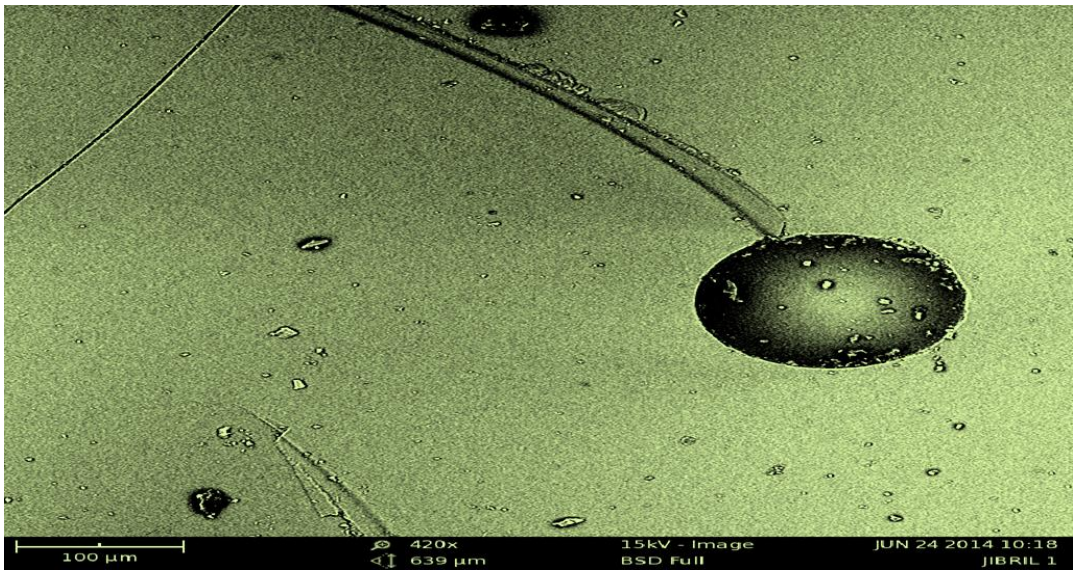



**Figure 4.6a: External morphology of Glass Sample 1, Magnification: x420**

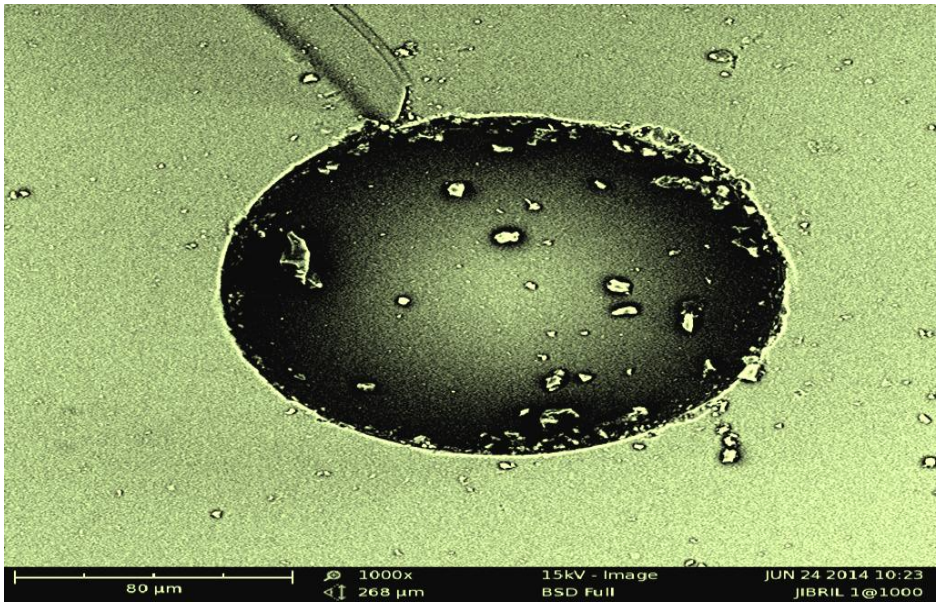



**Figure 4.6b: External Morphology of Glass Sample 1, Magnification x1000**

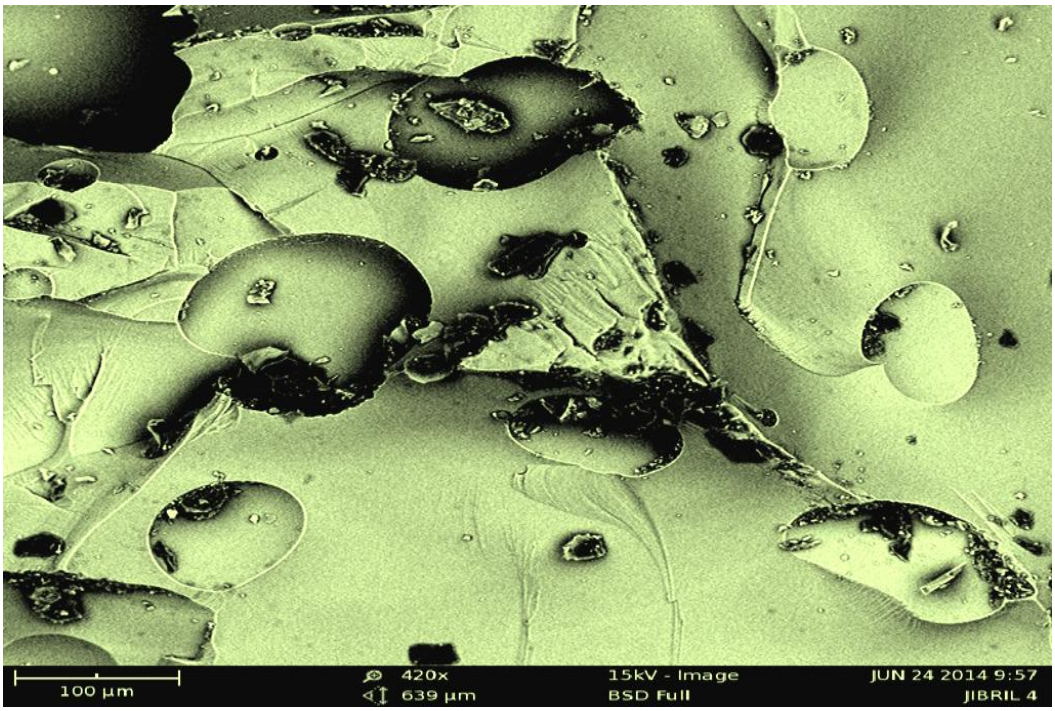



**Figure 4.7a: External Morphology of Glass Sample 4, Magnification x420**

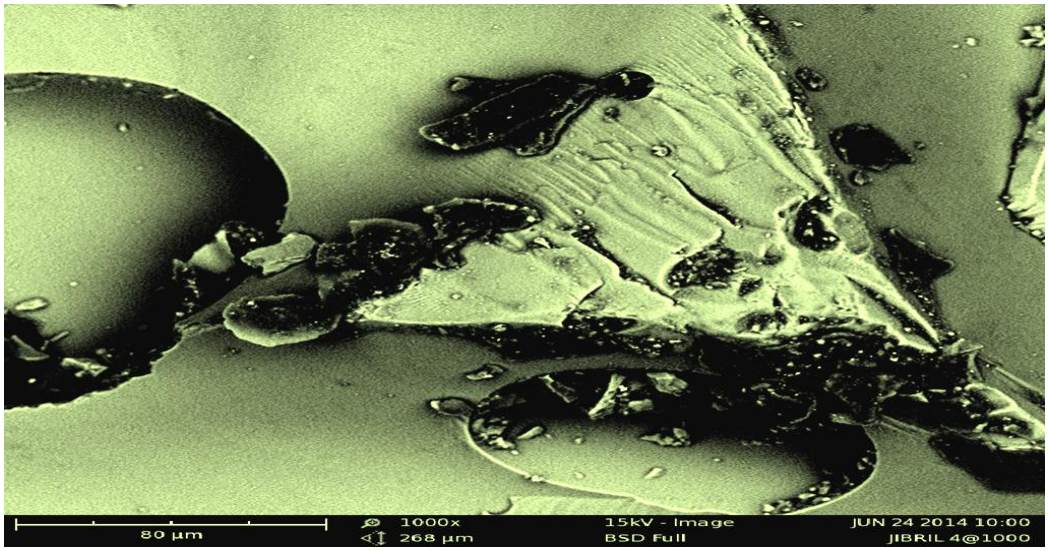



**Figure 4.7b: External Morphology of Glass Sample 4, Magnification x1000**

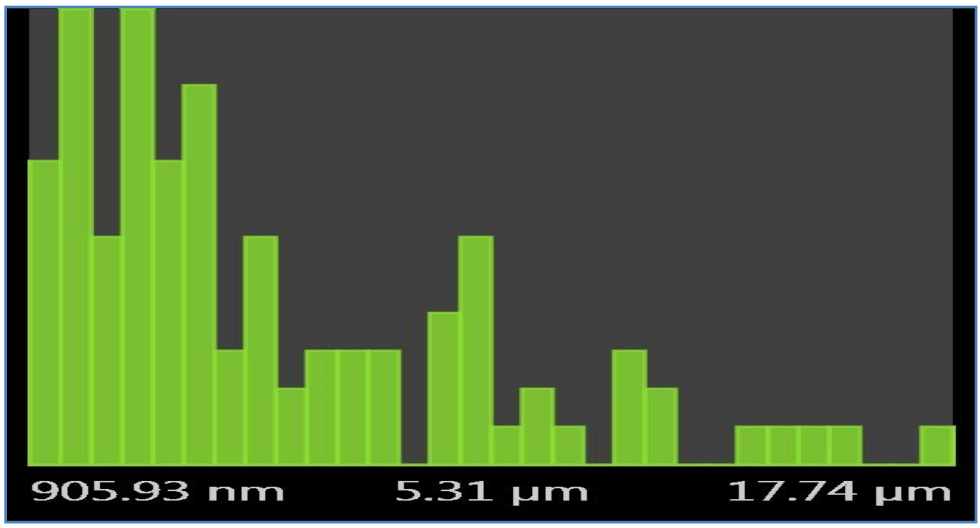



**Figure 4.8a: Fiber Histogram of Glass Sample 1**

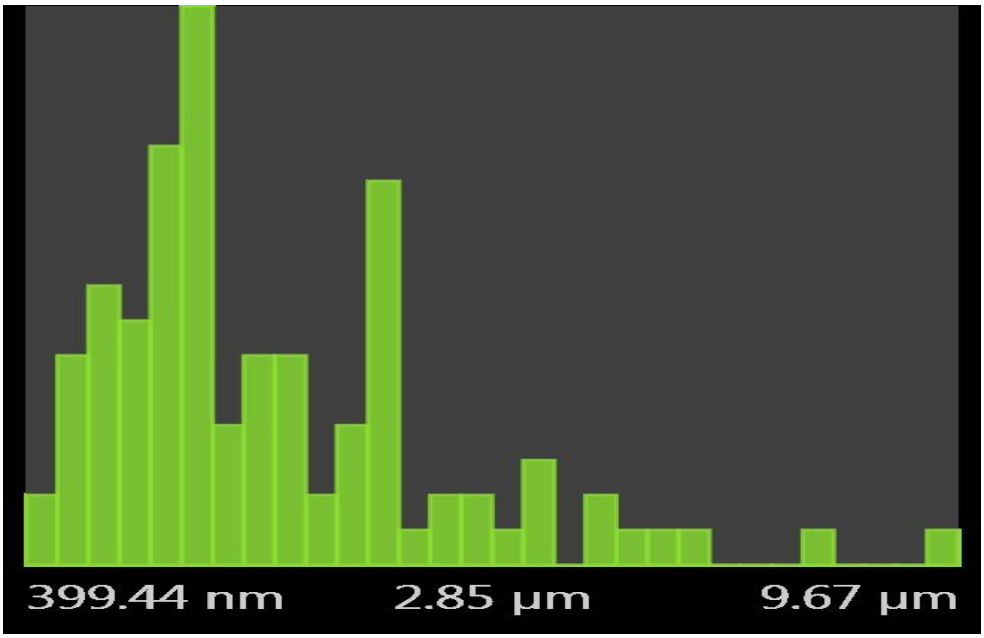



**Figure 4.8b: Fiber Histogram of Glass Sample 4**

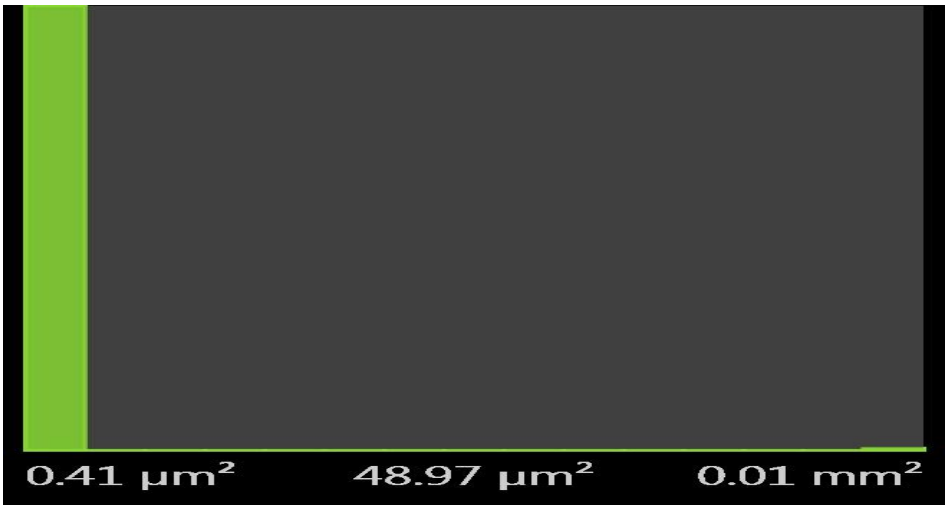



**Figure 4.9a: Pore Histogram of Glass Sample 1**

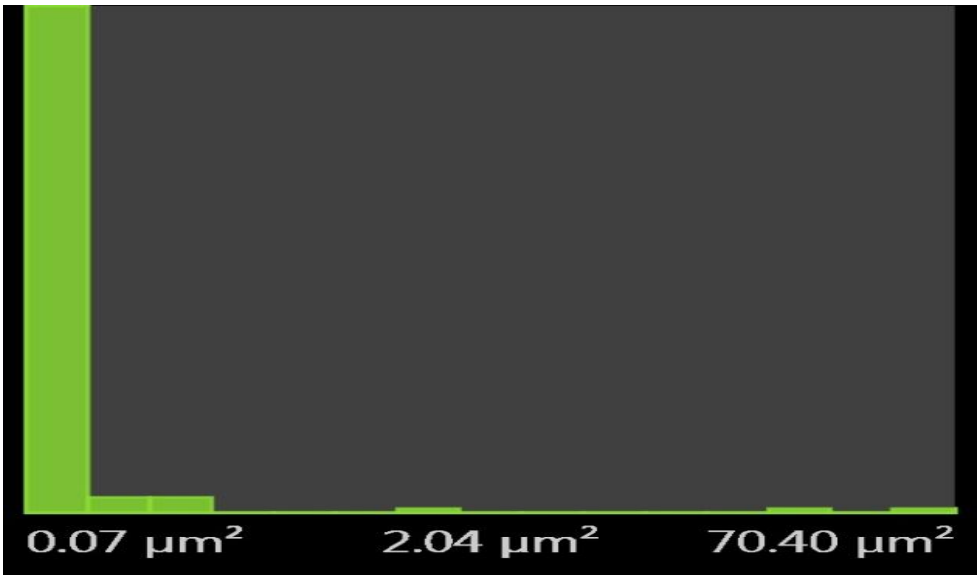



**Figure 4.9b: Pore Histogram of Glass Sample 4**

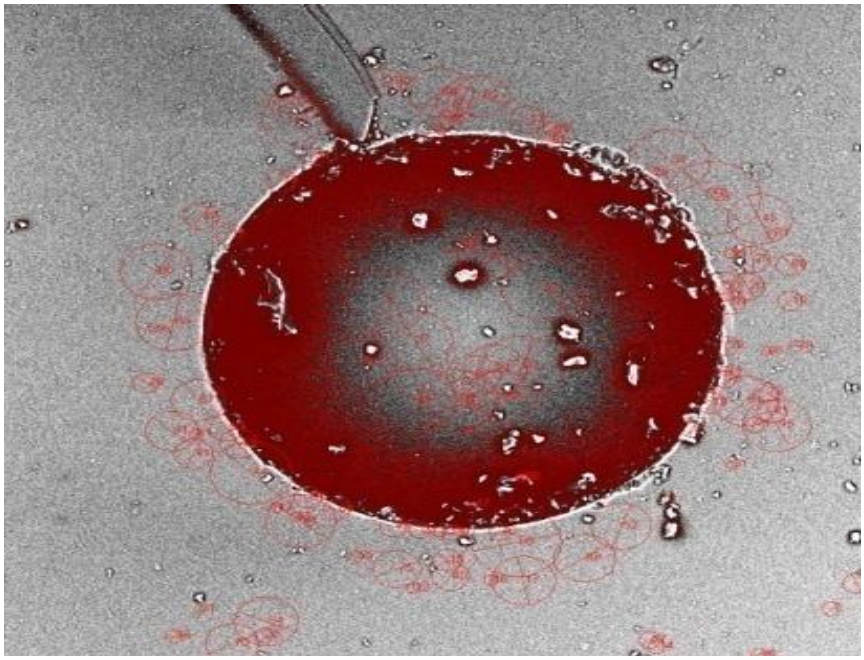


**Figure 4.10a: Fiber Metric of Glass Sample 1**

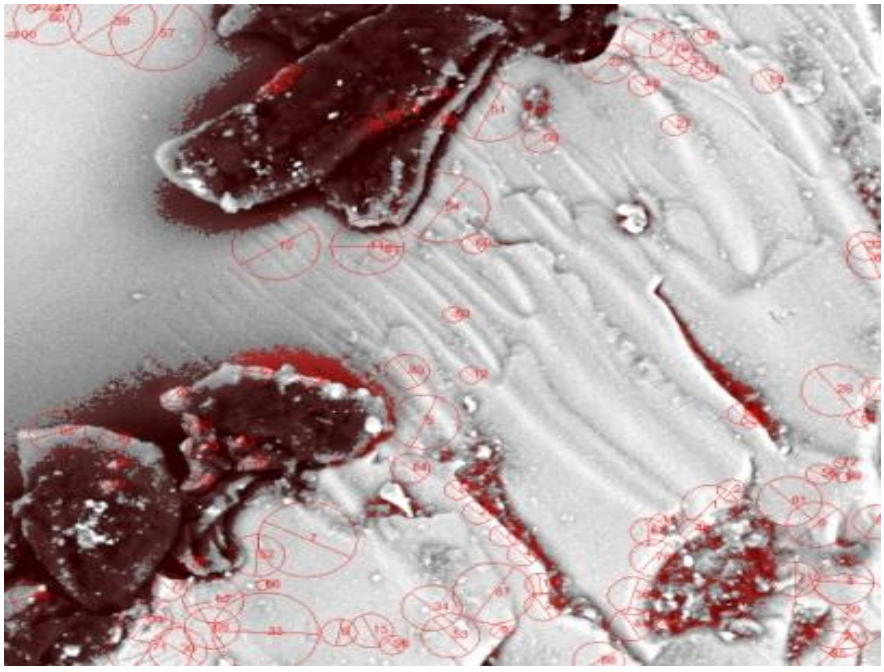


**Figure 4.10b: Fiber Metric of Glass Sample 4**

The external morphology of the glasses revealed the existence of rough surfaces, pores and fibers. The rough surfaces can be seen in Figures 4.6a, 4.6b, 4.7a and 4.7b; it can be attributed to the way the glasses were formed. It was melted and cooled without subjecting it to any mechanical means of fabrications such blowing, pressing etc. Rough in glass surface can be as a result of corrosion of the crucibles or incomplete melting of raw materials.

The pores exist due to presence of trapped air due to incomplete fining of the melt, because during melting there was no stirring. Bubbles of gas are formed during melting period of glass due to poor fining. According to Muijsenberg et *al* (2013), the main initial source of bubbles in industrial glass production is the batch melting. The batch melting reactions release a large amount of $CO_2$ gas that creates an enormous amount of bubbles. The sizes of the pores are not up to the level that can cause any defect to either of the glasses obtained. Their areas range from $0.41\mu m^2$ to $0.01\mu m^2$ which is too small to be considered a defect.

Presence of fibers might be from constituents not cover within the scope of this research; due to incomplete homogenization of raw materials forming the glass batches or due to contaminations from the laboratory environment or corrosion of refractory crucibles. The size of the fibers as shown in fig. 4.8a and 4.8b is so small that it cannot form deformity to the glasses formed.

### 4.8.2 Energy Dispersive X-Ray Spectroscopy (EDS)

EDS was used to identify elements present in each spot and summary of the elements in each spot in each glass samples was derived and presented below.

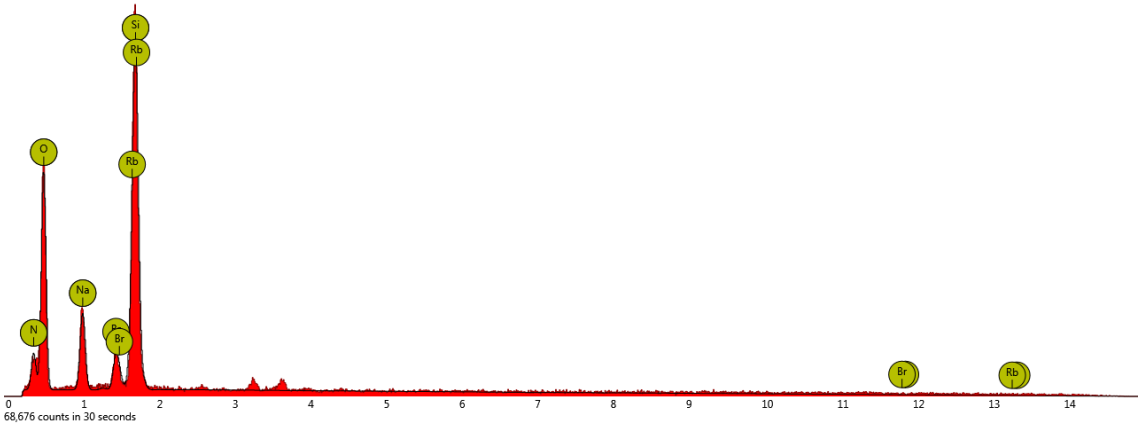



**Figure 4.11a: Spectrum of Elements in Spot 1 of Glass Sample 1**

| | Weight percentage | Certainty |
|---|---|---|
| O | 54.1 % | 98.8 % |
| Si | 16.4 % | 99.1 % |
| Na | 13.4 % | 98.0 % |
| N | 6.2 % | 96.7 % |
| Rb | 5.5 % | 95.3 % |
| Br | 4.4 % | 97.0 % |

**Figure 4.11b: Histogram of Elements in Spot 1 of Glass Sample 1**

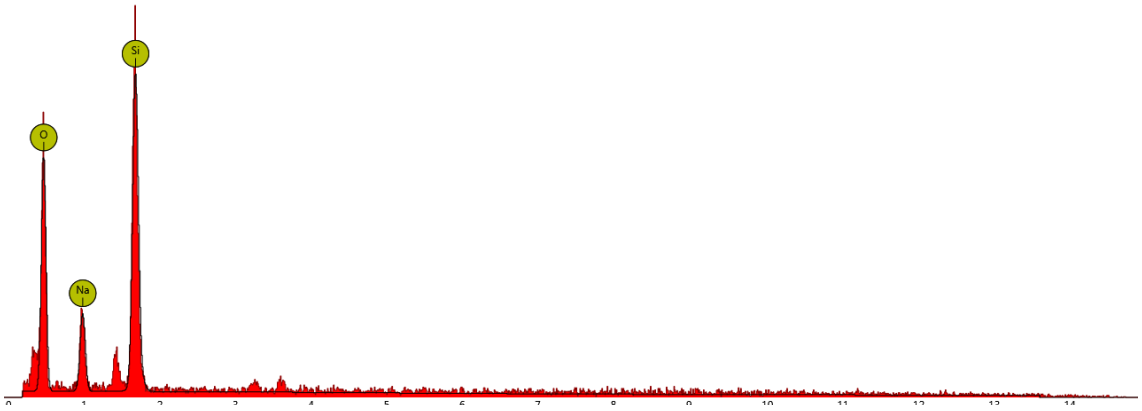



**Figure 4.12a: Spectrum of Elements in Spot 2 of Glass Sample 1**




**Fig 4.12b: Histogram of Elements in Spot 2 of Glass Sample 1**

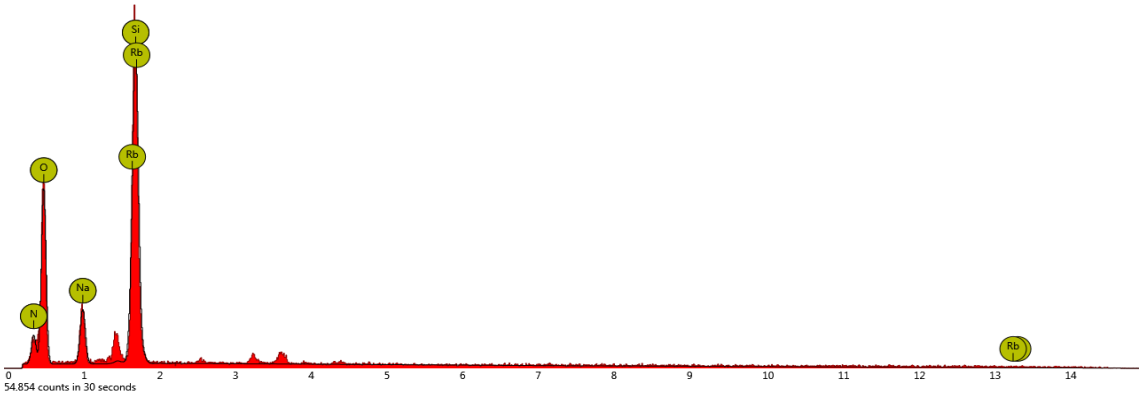



**Figure 4.13a: Spectrum of Elements in Spot 3 of Glass Sample**

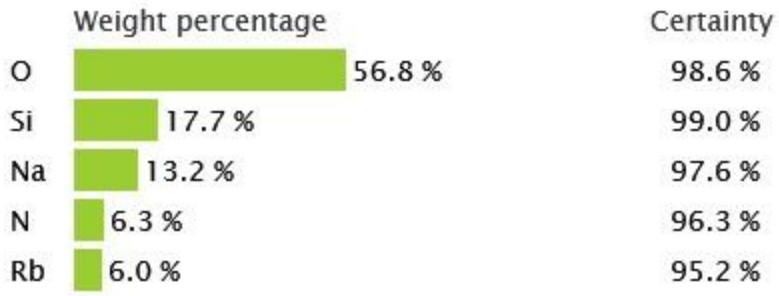



**Figure 4.13b: Histogram of Elements in Spot 3 of Glass Sample 1**

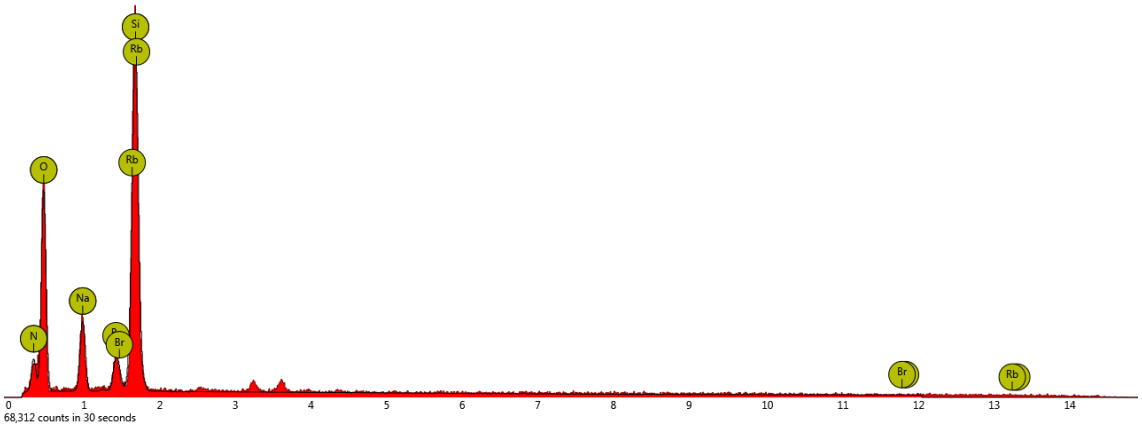



**Figure 4.14a: Spectrum of Elements in Spot 4 of Glass Sample 1**

| | Weight percentage | Certainty |
|---|---|---|
| O | 53.7 % | 98.8 % |
| Si | 17.1 % | 99.1 % |
| Na | 12.8 % | 97.9 % |
| Rb | 6.1 % | 95.7 % |
| N | 6.0 % | 96.6 % |
| Br | 4.3 % | 97.0 % |

**Figure 4.14b: Histogram of Elements in Spot 4 of Glass Sample 1**

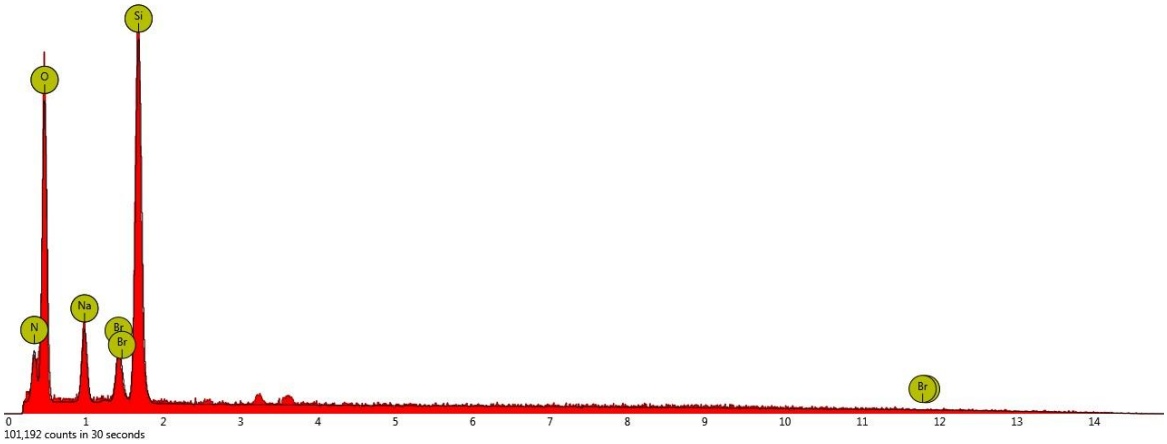



**Figure 4.15a: Spectrum of Elements in Spot 5 of Glass Sample 1**

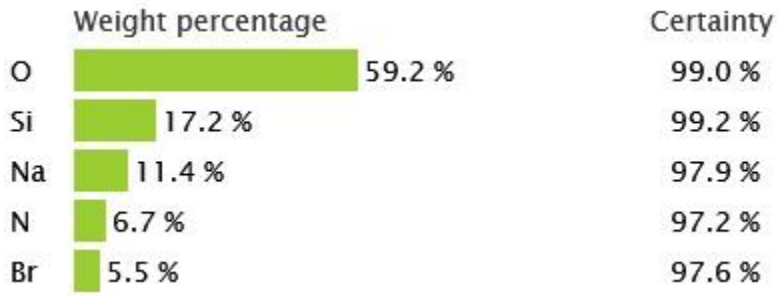



**Figure 4.15b: Histogram of Elements in Spot 5 of Glass Sample 1**

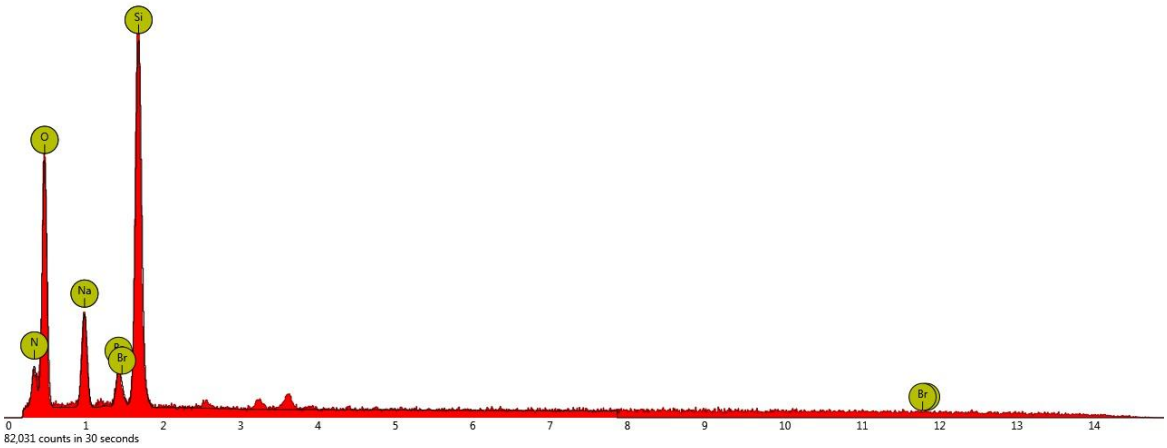



**Figure 4.16a: Spectrum of Elements in Spot 6 of Glass Sample 1**

| | Weight percentage | Certainty |
|---|---|---|
| O | 54.8 % | 98.8 % |
| Si | 19.2 % | 99.1 % |
| Na | 15.3 % | 98.0 % |
| N | 6.3 % | 96.5 % |
| Br | 4.4 % | 96.7 % |

**Figure 4.16b: Histogram of Elements in Spot 6 of Glass Sample 1**

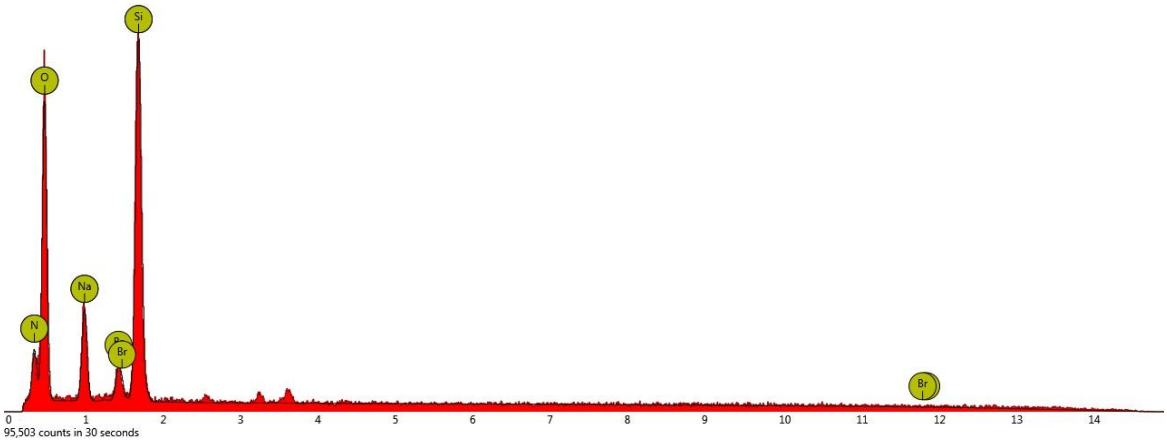



**Figure 4.17a: Spectrum of Elements in Spot 7 of Glass Sample 1**

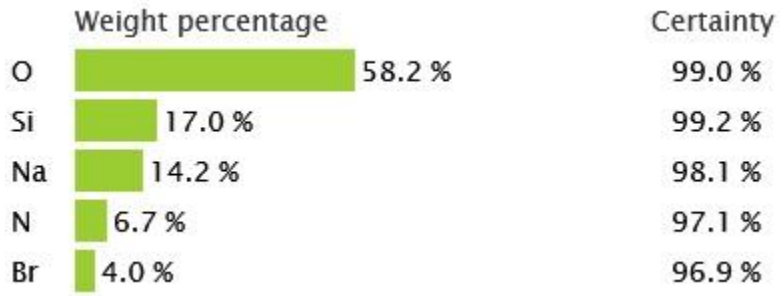



**Figure 4.17b: Histogram of Elements in Spot 7 of Glass Sample 1**

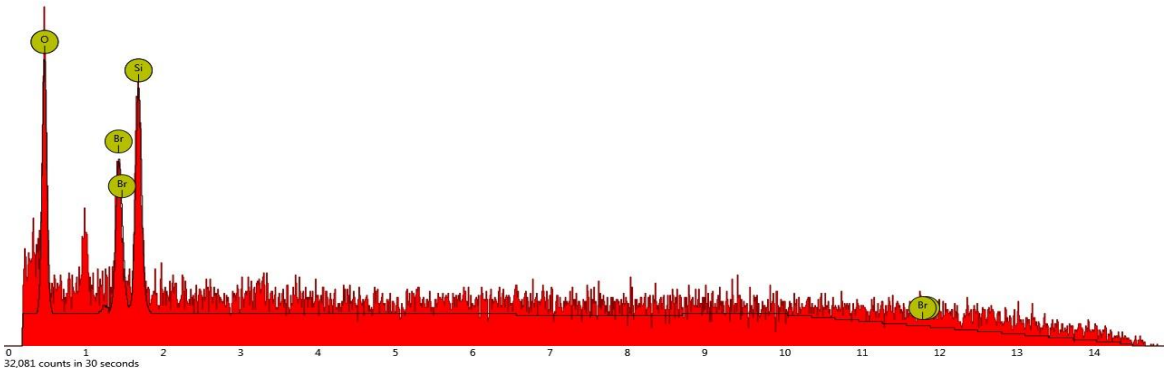



**Figure 4.18a: Spectrum of Elements in Spot 1 of Glass Sample 4**

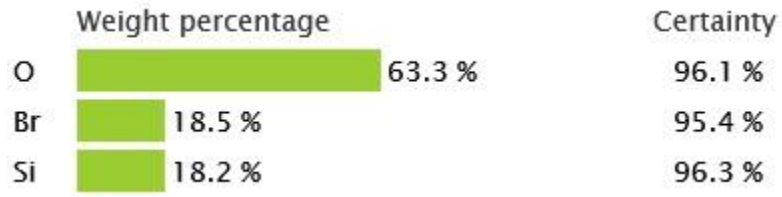



**Figure 4.18b: Histogram of Elements in Spot 1 of Glass Sample 4**

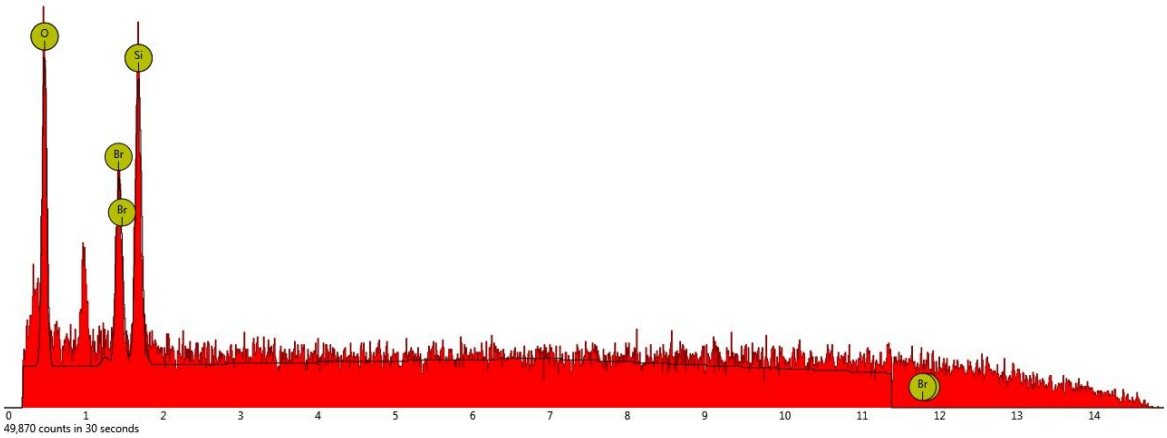



**Figure 4.19a: Spectrum of Elements in Spot 2 of Glass Sample 4**

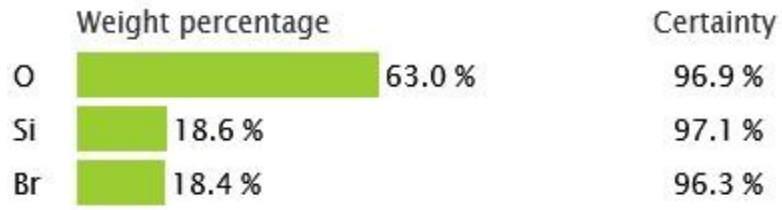



**Figure 4.19b: Histogram of Elements in Spot 2 of Glass Sample 4**

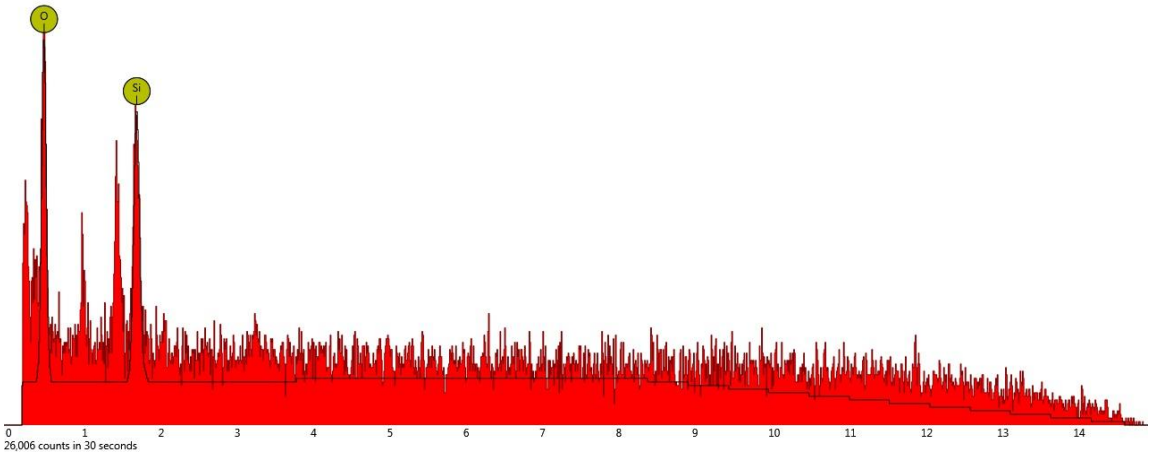



**Figure 4.20a: Spectrum of Elements in Spot 3 of Glass Sample 4**




**Figure 4.20b: Histogram of Elements in Spot 3 of Glass Sample 4**

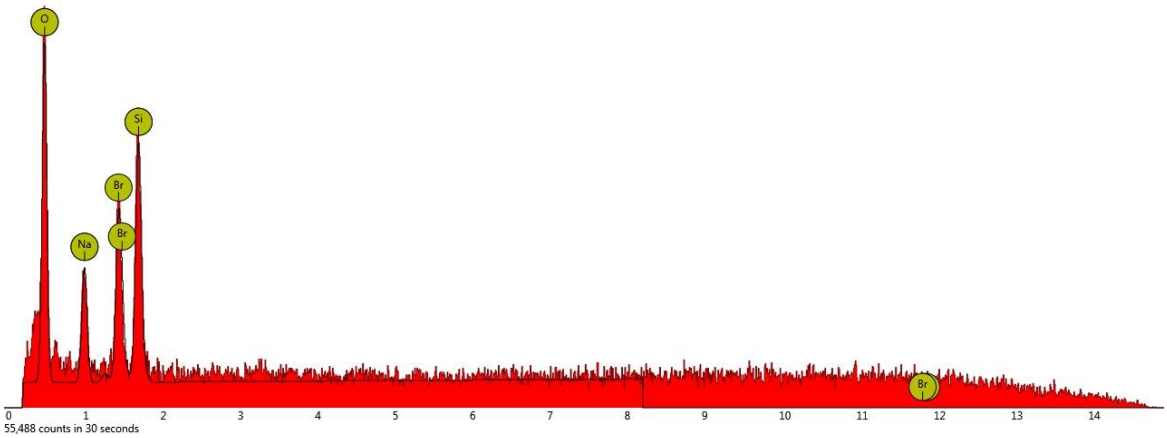



**Figure 4.21a: Spectrum of Elements in Spot 4 of Glass Sample 4**

| | Weight percentage | Certainty |
|---|---|---|
| O | 54.4 % | 97.8 % |
| Br | 16.9 % | 97.1 % |
| Na | 14.7 % | 96.1 % |
| Si | 14.0 % | 97.6 % |

**Figure 4.21b: Histogram of Elements in Spot 4 of Glass Sample 4**

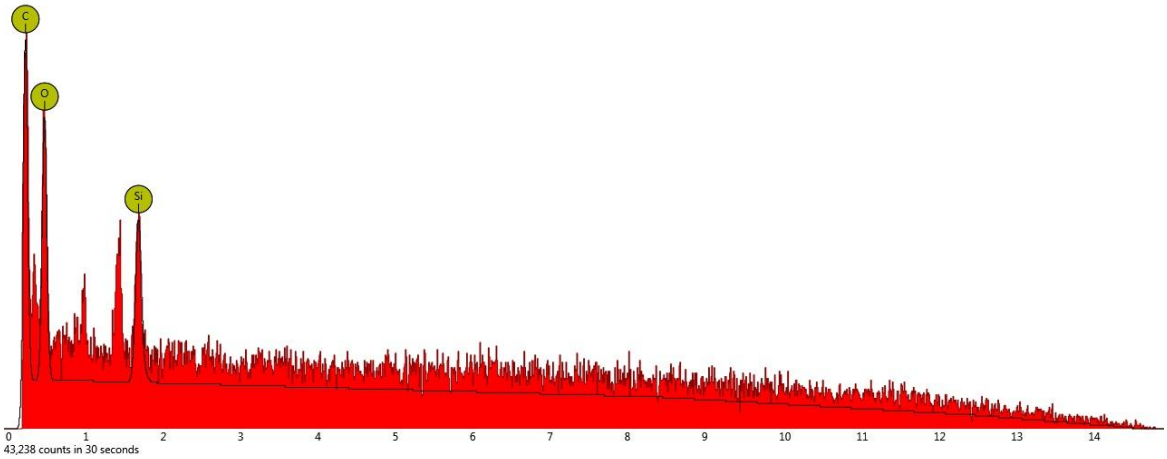



**Figure 4.22a: Spectrum of Elements in Spot 6 of Glass Sample 4**

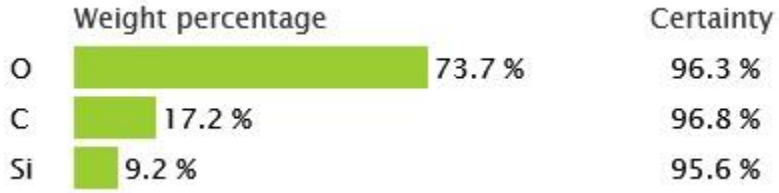



**Figure 4.22b: Histogram of Elements in Spot 6 of Glass Sample 4**

The result of Energy dispersive x-ray spectroscopy (EDS) shows presence of Si, Na, Br, O and N in the two glasses obtained. Occurrence of Si and Na in all the spots scanned is an indication that the raw materials were homogenously mixed. Azour *et al* (2006) explained that if analysis using SEM revealed good consistency of elements with that of starting elements with that of start of composition is an indication that there is good compositional control and also a confirmation that heating temperature, time of heating are adequate. Presence of oxygen and nitrogen in most of the spot examined is as result of trapped air in the pores. GTS described that gaseous components of glass bubbles in most cases are water ($H_2O$), nitrogen ($N_2$), oxygen ($O_2$), argon ($Ar_2$), carbon dioxide ($CO_2$), sulphur dioxide ($SO_2$), carbon oxy-sulphide (COS), hydrogen sulphide ($H_2S$). Also, occurrence of carbon in spot 6 of glass sample 4, (fig. 4.22a) might be as a result of unborn carbon from supplementary raw materials as they were all introduced in form of carbonate.

The overlapping of some elements in the spectrum as in sample where there is an overlapping of Si and Rb as shown in Fig. 4.13a and Fig.4.14a according to Goodge (2013) occur due to energy peak overlap of the elements, and element that is most expected in the sample is consider, in this sturdy, Si is considered.

## 4.9 Findings

1. The ash yield from saw dust is so small that high amount of saw dust will be required if it is to be used as glass making raw material. Its utilization as glass making raw material will contribute immensely in tackling environmental pollution caused to the saw mills and neighboring communities.

2. Saw dust ash from white Afara (Terminalia superba) is good source of CaO having percentage concentration of 24.80%. While saw dust from Marke (Anogeissus leiocarpus) and mixed saw dust are good sources of $K_2O$ with percentage concentrations of 22.80% and 29.90% respectively. Saw dust from isoberlinea doka is a good source of MgO with 24.21% as percentage concentration. These ashes can supply high percentage or whole of such oxides in some glass batches. Therefore, it can be consider a cheaper source of stabilizer and in some cases modifier due to the dual role of $K_2O$, which can serve the two purposes.

3. The temperature of $300^0$C is not sufficient enough to melt glass containing saw dust ash, this might be the reason why two out of the four batches prepared was not melted. The roughness of the two glasses obtained would have been reduced, if the melting temperature was raised above $300^0$C.

4. The glasses obtained are good enough to be use as soda lime silica glass because the densities and chemical durability of the glasses are within the range of most glasses of this type.

# CHAPTER FIVE

# CONCLUSION AND RECOMMENDATIONS

## 5.1 Conclusion

From the result of chemical analysis and glasses prepared, it can be concluded that the saw dust ash can be used in glass preparation as a source of CaO and $K_2O$, that is glass stabilizer. The amount of $SiO_2$ is so negligible for any of the ash to be considered as a glass former. Also considering the availability and cheap cost of saw dust, it can serve a cheaper source of these oxides (CaO and $K_2O$).

## 5.2 Recommendations

Based on the analysis of ash and properties test on the glasses prepared, the following recommendations are made:

1. More research should be made on saw dust of different woods by taking note of where the woods are sourced, for better understanding of environmental impacts on the chemistry of the woods.

2. More research should be conducted on other properties such as mechanical, optical etc of glasses containing different forms of ash for its practical applications in our day to day activities.

3. There is also need to further investigate the causes of occurrence of fibers in the glass prepared, as wood in its natural form contain some fibers, which in most cases destroyed, at high temperature.

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