THE FORMULATION OF GLAZES USING RAW MATERIALS FROM SELECTED STATES IN NORTH EASTERN NIGERIA

BY

MAMZA, JIDAI ALEWAI
Ph.D/ENV-DES/10798/2008-2009

A DESERTATION SUBMITTED TO THE POSTGRADUATE SCHOOL, AHMADU BELLO UNIVERSITY, ZARIA.

IN PARTIAL FULFILMENT OF THE REQUIREMENTS FOR THE AWARD OF DOCTOR OF PHILOSOPHY IN INDUSTRIAL DESIGN, DEPARTMENT OF INDUSTRIAL DESIGN, AHMADU BELLO UNIVERSITY, ZARIA.

SUPERVISORY COMMITTEE:

PROF. A.M. AHUWAN - Chairman
PROF. U.A. SULLAYMAN- Member
DR. A.D. GARKIDA- Member

DECEMBER, 2014

DECLARATION

I hereby declare that the dissertation titled "The Formulation of Glazes using Raw Materials from Selected States in North Eastern Nigeria" was written by me in the Department of Industrial Design of the Faculty of Environmental Design under the supervision of Prof. A.M. Ahuwan, Prof. U.A. Sullayman and Dr. A.D. Garkida. The information derived from the Literature has been duly acknowledged in the text and the list of References provided. No part of this dissertation has been previously presented for another degree or diploma at any Institution.

Mamza, Jidai Alewai

_____ _____

Signature Date

CERTIFICATION

This dissertation titled "The Formulation of Glazes using Raw Materials from Selected States in North Eastern Nigeria" by Mamza Jidai Alewai meets the regulations governing the award of Doctor of Philosophy in Industrial Design, Ahmadu Bello University, Zaria, Nigeria, and it is approved for its contribution to knowledge and literary presentation.

_____	_____
Prof. A.M. Ahuwan Chairman, Supervisory Committee	Date
_____	_____
Prof. U.A. Sullayman Member, Supervisory Committee	Date
_____	_____
Dr. A.D. Garkida Member, Supervisory Committee	Date
_____	_____
Dr. C.V. Alkali Head of Department	Date
_____	_____
Prof. A. Z. Hassan Dean, Postgraduate School	Date

DEDICATION

This dissertation is dedicated to the memories of my father, late Mallam Jidai Nandarayu Mamza; my sweet mother, late Anabaryu Jidai Nandarayu Mamza (Gaji Ngwadir) my brother, late Mr Jafia Jidai Mamza and Mr. Tsimiri Jidai Mamza. I am very grateful for everything they have done, especially their prayers. I thank them very much, knowing that their souls are resting in perfect peace. Amen.

ACKNOWLEDGEMENTS

Glory, honour and adoration are to God. I am very grateful to God Almighty for His wonderful love, direction and protection throughout this study. I am ever grateful to Him.

I am grateful and appreciative of the tremendous efforts of my supervisors, Professor A.M. Ahuwan, Professor U.A. Sullayman and Dr. A.D. Garkida for their unlimited illuminating tremendous assistance and concern towards the completion of this study. I am grateful to Dr. S. Maiwada, the former Head and Dr. C.V Alkali, the current Head, Department of Industrial Design, Ahmadu Bello University, Zaria for their concern and encouragement. I am also grateful to Dr. S.A. Dutsenwai, Mr. M. S. Aluwong and Mr. E. V Opoku for their assistance. My appreciation goes to Professor I.A. Olaofe of the Department of Education, Ahmadu Bello University, Zaria, and also present and former Heads of Department Fine Arts; Dr. K. Danjuma and Professor A.R. Salihu respectively, for their encouragement and assistance.

I appreciate the support and encouragements of the University of Maiduguri: Professor M.M. Daura the former Vice Chancellor, Professor I.A. Njodi, the current Vice Chancellor, Professor Y. Karta former Deputy Vice Chancellor Administration, Professor A. Abubakar, Chairman of Committee of Provosts, Professor Ahmed Dean Faculty of Arts, Dr Peter Saleh, Head of Department, Industrial Design, Professor S.O. Ododo Head of Performing and Theatre Arts, as well as members of staff of Geology Department of the University of Maiduguri.

I extend my gratitude to Rev. Engr. C.M. Gonah, Dr. P.O. Ada, Dr. J.I, Azi, Dr. S.Dakyes, Professor B.A. Kparevzua, Professor B.K. Olorukooba , Professor T. Okpe, Professor M.P.Mamza, Professor M.M. Sani, Dr P.O. Gushem, Dr. D.G. Gambo, Dr. A. Muhammad, Dr. Ken Okoli, Mr. I.S.Chafe, Mr.E.M.Alemaka, Mr.S. Gyams, Late Mrs. E.E. Ewule,S.O.Ogunwole (sunnybest), E.S. Jamson, L. Lamidi, B. Mohammed, A. Aminu, A.

Usman, J.A. Ali, all of the Department of Industrial Design and Fine Arts, Ahmadu Bello University, Zaria.

I also appreciation Professor K. Y. Musa of the Department of Pharmacology and Drug Development, Faculty of Pharmaceutical Science, Ahmadu Bello University, Zaria, Dr. P.A.P. Mamza of the Department of Chemistry Ahmadu Bello University, Zaria, Professor K. Bala, Professor I.K. Zubairu, and Dr. I.H. Mshelgaru of the Department of Building, Dr. Y.A. Yawas and Dr. D.M. Kula, of the Department of Mechanical Engineering, Mrs. B. Afuwai, J. Gadzama, A.A. Haruna, C. Burbur, Sarah Joel, Paul Joel, A.P. Ogboli, N.A Jesse, Obed Pascal, Paul Agbo, Ishaku Shamaki for their assistance.

I am also grateful to the librarians of Industrial Design, Fine Arts Departments and Kashim Ibrahim Library (KIL), A.I. Abdullahi former Administrator Amina Hall, Ahmadu Bello University Zaria, all of Ahmadu Bello University Zaria. To Tertiary Education Trust Fund (TETFund), through the University of Maiduguri, I thank them for their worthy assistance of scholarship offered me and encouragement for the study.

To my husband Mr. J.M. Burbur, my beloved children, Janada, Munakur, Abbas, Charles and Sarah, my brothers and sisters: Kellari Jidai Mamza, John Jidai Mamza, Midaryu Jidai Mamza and Esther Tumba. I thank you for all your moral support and encouragement. I am also grateful to all my colleagues and friends. These names are in exhaustible, but I will ever remain grateful to all, especially those whose names were not mentioned. Above all human beings, I once again thank the Almighty Father, God for the life and strength to forge ahead. The Lord has been my strength for ever and will continue to be, Amen.

ABSTRACT

The formulation of glazes using raw materials from selected states in north eastern Nigeria. This study explored some raw materials from North Eastern Nigeria suitable for the development of glazes for use in pottery studios and tertiary institutions. The study was set to: explore, identify and obtain some suitable raw materials for development of glazes. The raw materials were Beneficiated. The raw materials obtained were analyzed by means of X-ray fluorescence (XRF). Batches were formulated for the glazes. Maturing temperature suitable for the glaze developed was determined. The raw materials used for the study are ball clay, feldspar, kaolin and quartz. Triaxial and quadaxial blends of the materials were made for the tests of the glazes as the basis for glaze development. Physical property tests conducted on the glazes included vitreous crazing and imperviousness. The imperviousness of the glazed blend was determined. The bisque fired samples of the tiles were at 900℃? The experimental samples which were numbered in accordance to the glazes were then glazed after biscuit firing for the different tests of glazes. Nine differently blended batches were formulated for the glaze development, making a total of 390 samples. These glazes were fired and the following showed the best results: Samples 4 blend 25 (75% feldspar, 25% ball clay) and 32 of batch blends of 121 (8% ball clay, 72% feldspar, 2% kaolin, 18% quartz) and sample 20 of blend 121 (18% ball clay, 72% feldspar, 2% kaolin, 8% quartz). The glaze sample 4 of 25 member blend, samples 20 and 32 of 121 member blend proved to be suitable glazes developed from the materials of the study. They have met the necessary characteristics expected of good glazes such as smooth and shiny surface, hardness resistance and resistance to corrosion. These batches were tested on thrown products successively that were fired at a temperature of 1260^{o}C. Stoneware glazes have been formulated using raw materials found in Bauchi, Borno and Yobe States.

TABLE OF CONTENTS

Title page - - - - - - - - - i

Declaration - - - - - - - - - ii

Certification - - - - - - - - - iii

Dedication - - - - - - - - - iv

Acknowledgements - - - - - - - - - v

Abstract - - - - - - - - - vii

Table of Contents - - - - - - - - - viii

List of Tables - - - - - - - - - xiii

List of Plates - - - - - - - - - xiv

List of figures - - - - - - - - -

Definition of Terms - - - - - - - - xvi

Abbreviation - - - - - - - - - xix

CHAPTER ONE: INTRODUCTION

1.1 Background of Study - - - - - - - 2

1.2 Statement of Problem - - - - - - - 7

1.3 Aim of the Study - - - - - - - - 8

1.4 The Objective of the Study - - - - - - 8

1.5 Scope and Delimitation of the Study - - - - - 9

1.6 Research Questions - - - - - - - 9

1.7 Hypothesis of the Study - - - - - - - 9

1.8 Significance of the Study - - - - - - - 9

CHAPTER TWO: REVIEW OF RELATED LITERATURE

2.1 Introduction - - - - - - - - 12

2.2 Ceramic Raw Materials - - - - - - - 12

2.3 Glaze Raw Materials - - - - - - - 13

2.4 Glazes - - - - - - - - - 15

2.4.1 Importance of Glazes - - - - - - - 17

2.5 Sources of Clay - - - - - - - - 18

2.5.1 Ball Clays - - - - - - - - 19

2.5.2 Kaolin - - - - - - - - - 20

2.5.3 Feldspar - - - - - - - - - 20

2.5.4 Quartz - - - - - - - - - 21

2.6 Glaze Calculation - - - - - - - - 22

2.6.1 Limit Formula - - - - - - - - 23

2.6.2 The RO, R_2O_3, RO_2 System - - - - - - 25

2.7 Fluxing Agents - - - - - - - - 26

2.8 The Empirical Glaze Formula - - - - - - 26

2.9 The Batch Recipe - - - - - - - - 31

2.10 Variables in Glaze Formulation - - - - - - 32

2.11 Ceramic Oxides - - - - - - - - 33

2.11.1 Sources of Base (RO) Oxides - - - - - - 34

2.11.2 Sources of Neutral (R_2O_3) Oxides - - - - - 35

2.11.3 Sources of Acids (RO_2) Oxides - - - - - 36

2.12 Glaze Formulation - - - - - - - 37

2.13 Pyrometric Cones - - - - - - - 39

2.14 Temperature Ranges for Firing Glazes - - - - - 40

2.14.1 Very Low - Fire - - - - - - - 40

2.14.2 Low - Fire - - - - - - - - 41

2.14.3 Lower - Mid- Range - - - - - - - 41

2.14.4 High - Fire - - - - - - - - 42

2.15 Glaze Type - - - - - - - - 42

2.16 Glaze Composition with Regard to Glaze Properties - - - 44

2.17 Desired Properties of Glazes - - - - - - 45

2.18 Experimenting on Test Tiles - - - - - - 45

2.19 Developing an Original Glaze - - - - - - 46

2.20Keeping a Log - - - - - - - - 48

CHAPTER THREE: METHODOLOGY

3.1 Sourcing of Materials - - - - - - - 49

3.2 Beneficiation of Materials- - - - - - - 50

3.3 Calcination/ Pre-treatment of Samples - - - - - 52

3.3.1 Feldspar - - - - - - - - - 52

3.3.2 Quartz - - - - - - - - - 53

3.3.3 Kaolin - - - - - - - - - 55

3.3.4 Ball Clay - - - - - - - - 55

3.4 Chemical Analysis of Raw Materials - - - - - 55

3.5 Batch Calculation for Glaze Making - - - - - 57

3.5.1 Formulation of Glaze Batches - - - - - 57

3.6 Determination of Suitable Firing Temperature - - - - 65

3.7 Property Testing - - - - - - - - 65

3.7.1 Determination of Vitreousness and Crazing - - - - 65

3.7.1.1 Vitreousness - - - - - - - - 65

3.7.1.2 Crazing - - - - - - - - 65

3.7.2 The Imperviousness - - - - - - - 65

CHAPTER FOUR: RESULTS AND DISCUSSION

4.1 Results of Chemical Analysis of the Samples - - - - 66

4.2 Results of Test Melting of the Glaze Batches - - - - 67

4.2.1 Test Melting Results of Quadaxial Test Blend of 36(A) Fried at 1200^{o}C -67

4.2.2 Test Melting Results of Quadaxial Test Blend of 36(A) Fired at 1250^{o}C -67

4.2.3 Test Melting Results of Quadaxial Test Blend of 121(B) Fired at 1260^{o}C -68

4.2.4 Test Melting Results of Quadaxial Test Blend of 25(C) Fired at 1260^{o}C -72

4.2.5 Test Melting Results of Triaxial Test Blend of 36(D) Fired at 1260^{o}C -74

4.2.6 Test Melting Results of Triaxial Test Blend of 28(E) Fired at 1260^{o}C -76

4.2.7 Test Melting Results of Triaxial Test Blend of 21(Fi) Fired at 1260^{o}C -77

4.2.8 Test Melting Results of Triaxial Test Blend of 21(Fii) Fired at 1260^{o}C -77

4.2.9 Test Melting Results of Triaxial Test Blend of 66(G) Fired at 1260^{o}C -78

4.2.10 Results of the Vitreous Test - - - - - - -80

4.2.11 Crazing - - - - - - - - - -80

4.3 Results of Tests of Glazes on Thrown Pieces - - - - -80

4.4 Property Testing - - - - - - - - -83

4.4.1 Determination of properties of vitreousness, Crazing and imperviousness -83

4.5 Hardness and Abrasion Resistance Test - - - - - -84

4.6 Chemical Resistance Test - - - - - - - -84

4.7 Findings - - - - - - - - - -84

CHAPTER FIVE: SUMMARY, CONCLUSION AND RECOMMENDATIONS

5.1 Summary - - - - - - - - -86

5.2 Conclusion - - - - - - - - -86

5.3 General Recommendations - - - - - - -87

5.4 General Recommendations - - - - - - -87

Reference - - - - - - - - - -89

Appendixes - - - - - - - - - -93

LIST OF TABLES

Table 2.1: Glaze Batch Weights Calculation - - - 23

Table 2.1.1: Limited Formula of Glaze Types - - - 24

Table 2.1.2: The RO, R_2O_3, RO_2 - - - - - 26

Table 2.2a: Equivalent Weight/ Molecular Weight of Potash Feldspar- 27

Table 2.2b: Glaze Molecular Proportions - - - - 28

Table 2.2c: Glaze Batch Empirical Formula (RO System) - - -28

Table 2.2d: Empirical Formula (RO System for Comparism) - -29

Table 2.2e: Glaze Batch using Empirical Formula - - - -30

Table 2.2f: Batch to Empirical Formula - - - - -30

Table 2.3: Batch Recipe From Empirical Formula - - - -32

Table 3.1a: Quadaxial Test Blend (36) of Materials of the Study in % -57

Table 3.1b: Quadaxial Test Blend of 36(A) - - - - -58

Table 3.2 : Quadaxial Test Blend of 121(B) - - - - -59

Table 3.3: Quadaxial Test Blend of 25(C) - - - - -60

Table 3.4: Triaxial Test Blend of 36(D) - - - - -61

Table 3.5: Triaxial Test Blend of 28(E) - - - - -62

Table 3.6a: Triaxial Test Blend of 21(Fii) - - - - -63

Table 3.6b: Triaxial Test Blend of 21(Fii) - - - - -63

Table 3.7: Triaxial Test Blend of 66 - - - - - -64

Table 4.1: Results of the Chemical Analysis of the Samples - - -66

LIST OF PLATES

Plates I & II: Location (site) of the Raw Materials at Lokodisa Village Gwoza Town,Borno State Nigeria - - - - - 50

Plates III & IV: Collection of Feldspar and Quartz from the Site - - 50

Plates V & VI: Materials Weighing in the Ceramic Section Of Ahmadu Bello University, Zaria, Nigeria - - - - - 51

Plates VII: Packing of Raw Materials (Feldspar and Quartz) into the Kiln for Calculations - - - - - - - 51

Plate VIII: Calcined Materials (Feldspar and Quartz) in the Kiln - - 52

Plate IX: Crushed Material (Feldspar) - - - - - 53

Plate X: Crushed Material (Quartz) - - - - - - 54

Plate XI: Loading of Materials (Crushed) Quartz for ball Milling - - 54

Plate XII: Drying Process of the Materials (Feldspar and Quartz) - - 55

Plates XIII: Test Melt Result of Tiles - - - - - - 67

Plates XIV: Test Melt Result of Tiles 1-36 Tested at 1200^{o}C - - 67

Plate XV: Test Blend Result of Files 1-121 Fired at 1260^{o}C - - - 68

Plate XVI: Test Blend Result of Files 1-25 Fired at 1260^{o}C - - - 72

Plate XVII: Test Blend Result of Files 1-36 Fired at 1260^{o}C- - 74

Plate XVIII: Test Blend Result of Files 1-28 Fired at 1260^{o}C - - 75

Plate XIX: Test Blend Result of Files 1-21(i) Fired at 1260^{o}C - - 76

Plate XX: Test Blend Result of Files 1-21(ii) Fired at 1260^{o}C - - 77

Plate XXI: Test Blend Result of Files 1-66 Fired at 1260^{o}C - - 78

Plate XXII: Sample 4 of blend 25 - - - - - - 79

Plate XXIII: Sample 4 of blend 25 - - - - - - 81

Plate XXIV: Sample 4 of blend 25 - - - - - - 81

Plate XXV: Sample 32 of blend 121 - - - - - - 82

Plate XXVI: Sample 20 of blend 121 - - - - - - 82

Plate XXVII: Sample 20 of blend 121 - - - - - 83

Plate XXVIII: Weighing of materials using digital scale - - - 94

Plate XXIX: Measuring of materials using the digital scale - - - 95

Plate XXX: Display of developed glazes on tiles - - - - 96

Plate XXXI: Good glazes developed from the study - - - - 97

Plate XXXII: Production of pot for glaze testing - - - - 98

Plate XXXIII: Produced pot for glaze testing - - - - 98

Plate XXXIV: Results of tests of glazes on thrown wares - - - - 99

LIST OF FIGURES

Fig 1: Map of North Eastern States showing study Areas - - - - 93

DEFINITION OF TERMS

Ball mill	A drum like, pebble grinding mill, used for the grinding of glaze materials.
Beneficiation	The process of washing and removing impurities form ceramic raw materials.
Biscuit	A porous unglazed fired pottery
Bisque (Bisque firing)	– First firing given to ceramic ware"green ware" usually between 900°C - 950°C.
Body	An admixture of clay and other materials producing a workable material suitable for manufacturing process.
Ball clay	A highly plastic clay, this is the basis of many pottery bodies. It comes from the practice of digging the clay out of the open pits in convenient blocks of about 12kg called ball obtained from Gada- bull and Dambo'a road, Maiduguri, Nigeria.
Bentonite	A highly plastic clay originating in the decomposition of volcanic ash. It is extremely fine and can be dispersed widely through bodies and glazes, in which it is used to impart plasticity and an ability to keep a glaze slop in suspension.
Calcined	A method of softening some ceramic raw materials by the action of heat. For example feldspar and quartz
Crazing	A glaze defect characterized by a network of fine cracks
Cones	Pyrometric cones. Elongated three – sided pyramids, 2cm to 8cm tall, which are placed inside a kiln for the measurement of heat inside the kiln
Earthenware	Low-fired pottery (below 1200°C) usually red or tan in colour with absorbency of between 5 – 10%.
Efflorescent	Substance that loses its water of crystallization to the environment, the powder which appears on the surface of bricks, rocks, etc when water evaporates.
Flux	An oxide which influences ceramic fusion at lower temperature by interacting with other oxides.

Frit	A frit is a combination of a flux like lead that is combined with other materials like quartz, melted in a kiln to form an insoluble glass that is grounded to be used as a base for making glaze.
Feldspar	A group of minerals used in proportions of up to 25% as flux in bodies and up to 100% in glazes.
Fusion	The melting of different materials into homogeneous liquid mass.
Glaze Batch	Composition of glaze that has been formulated
Gada- bull	A river sourced at Mambula hill through Alau Dam area that passed through Madaganari area of Maiduguri to the heart of Maiduguri to Lake Chad
Glaze firing	Gloss firing of up to 1300°C at which fusion of minerals take place to produce glass coating on ceramic wares
Hardness	Physically hard and difficult to scratch or cut
Hygroscopic	Capable of easily absorbing moisture e.g. from air.
Imperviousness	Not allowing a liquid or gas to pass through
Jaw crusher	A machine used for breaking sizeable raw materials into smaller particles, for example feldspar and quartz
Kiln	A structure built to conserve heat. It is used for firing pottery or ceramic products
Kaolin	The purest clay. It is a primary clay, it is high in aluminum oxide content and with little alkaline impurity. It is refractory with a melting point of over 1770°C (3218°F). It is used in glazes to introduce aluminium oxide and silica and to provide bulk in the slop suspension.
Molecular weight	The total of the atomic weights of all the atoms which make up a molecule
Oxidation	Firing materials or pottery in excess oxygen
Oxide	A chemical combination of oxygen with another element. To the potter there are two types of oxides. The metallic oxide and the non-metallic oxide e.g. cobalt, zinc, copper, manganese, nickel and chromium.

Quartz	A mineral rock, used as one of the sources of silica in glazes.
Raw glaze	A glaze compounded of materials that have not been previously fritted.
Tri-axial Blend	A method of blending three materials.
Temperature	A means of describing heat. Temperature is measured in degrees Celsius (°C) or degree Fahrenheit (°F).
Vitreous	Hard, shiny and translucent ceramics like glass, vitreous enamel, etc.
Viscosity	The stiffness of a liquid created by the friction amongst its particles and molecules. Viscous glazes move very little, in the firing.

ABBREVIATIONS/NOTATIONS

ASTM	American Standards for Testing and Materials
MW	Molecular weight
Wt	Weight
^{o}C	Degree Celsius
^{o}F	Degree Fahrenheit

CHAPTER ONE

INTRODUCTION

Nigeria, as a nation, is in need of research efforts that explore and identify locally available raw materials for exploitation and internal consumption (Manzuche, 2006). The development of a nation depends on the quality and quantity of its research output (Olaofe, 2010). Nigeria needs to carry out researches that will contribute to the development of the nation both educationally and industrially.

Bauchi, Borno and Yobe States which are parts of northeastern Nigeria have diverse solid mineral resources. These solid minerals could be utilised for ceramic glazes and can be processed and formulated into useful ceramic materials. Examples of such minerals include; feldspar, kaolin, potash, ball clay, quartz and others. These minerals could be explored from the localities of North Eastern States of Nigeria for economic and aesthetic purposes. Ceramists therefore, need to exploit such raw materials and adopt production techniques of the modern industry to boost the diversification of our industrial sector. Doing this will not only provide the much needed import substitution but the joy of accomplishment and an enhanced standard of living. In this age of specialization, when computers and other technological tools are indispensable, the need to mobilize stakeholders (consumers and producers of innovation) cannot be overemphasized.

These innovation producers include the universities, government laboratories, joint private institutes and private firms. These organizations have different goals and work under different conditions. Researches in the universities generate knowledge that can be transferred as service to firms and used in their innovation process. The universities also produce trained personnel that can be hired by firms and make contributions in their innovation processes. Government laboratories like the Raw

Material Research Development Council also contribute to these technological innovation processes in similar ways (Opoku, 2006).

1.1 Background of the Study

The desire and the will to explore, create and develop glazes for its multipurpose uses to the society is what prompted this study. For example, glazes could be useful to the individual ceramists, industries and higher institutions for teaching and learning. It is a challenge to note that students from the University of Maiduguri travel from Maiduguri to Ahmadu Bello University, Zaria and Abuja in order to obtain glazes due to lack of basic infrastructure, studio and requisite glaze materials and for glaze processing in the institution.

There are problems of inadequate ceramic materials in Colleges of Education in the North Eastern part of Nigeria (Mamza, 2008). This hinders effective teaching and learning processes in the Art and Design Departments of the tertiary institutions of which lack of processed glaze materials is one of the problems.

On the other hand, government is poised to support research and development efforts that would propel Nigeria to a higher level of technology and capability such researches should enable the country to meet up with the demands of the current era of globalization (Manzuche, 2006). This includes the development of ceramic materials for teaching and learning processes in schools as well as for production in industries. This will enhance quality ceramic education (in schools) and quality products in industries. With this, such schools will help to accelerate the development of the North East and Nigeria.

Notably,

1) Abuja initiative for increasing the country's foreign exchange earnings from non oil sector. That these measures are currently being taken by the federal government to ensure that the next eight to ten years, if all be well, the country will be making much from the solid minerals.

2) The government policy at this point in time is of great encouragement and motivation to the researchers.

3) That investing in the solid minerals sector will bring about a problem of indigenous man power, development, a possible reduction of dependence on other countries of the world in form of trade, finance, technology, market and generation of multiplying effect through local manufacture of equipment and technology acquisition (Manzuche, 2006).

However, some of the reasons for the low production and in some cases closure of local ceramic industries are traceable to lack of infrastructural facilities, negative economic policies, lack of adequate funding and management (Fatuyi, 2007). The principal raw materials for ceramic manufacture are available in Nigeria, nevertheless, domestic production is low and imports are large, constituting a major drain of the scarce foreign exchange reserves. Other constraints facing ceramic development are the non availability and inadequate facilities, high cost of technological capabilities in the key areas of the design and fabrication of processing equipment and machines.

Lack of job satisfaction for the few trained ceramic engineers/technologists have led to the migration of available ceramic experts to rich and industrialized countries, mostly due to frustration and poor income which also have further worsened the technical manpower deficiency in the ceramic industry. Up to 1999, the industrial sector of the Nigerian economy was characterized by poor infrastructure and policy inconsistency

resulting in low investments, high production costs and manufacturing of goods that are noncompetitive on both national and international markets. The micro-economic environment in Nigeria is highly negative, witnessing capital flight, high interest rates, negative real growth rates and fiscal excesses (Opoku, 2007).

Some of the problematic factors against the teaching and learning of ceramics are non availability of basic equipment, high cost of production of the materials and exorbitant prices of basic equipment (Ewule, 2006). Many higher institutions responsible for the training of graduates in the country lack even the basic training aids such as kiln, throwing wheels and also glazes and colorants (Ajayi and Chindo, 2005). Other problems include lack of funds to carry out necessary researches especially in the higher institutions of learning of which he advised the government at various levels to make enough funds available to carry out the necessary research that can lead to the advancement of the profession at local and national levels. Some students take ceramics as a stepping stone to other professions such as banking, law and so on. This could be due to high cost of equipment necessary for practicing ceramics and inadequate facilities in schools. Lack of resource centers where there could be research materials, laboratory and research equipment such as high temperature kilns is yet another challenge (Idowu, 2006).

The shortfall in skilled manpower and modern technology, research and development, as well as adequate and reliable ceramic infrastructure is also some of the problems. The present curriculum is too shallow, narrow and lack contents for the development of ceramic technology (Ajayi and Chindo, 2005). Therefore raw materials development need to be considered seriously and immediately for the problems on ground to be resolved for the improvement of ceramic education and industries at the local, national and international levels. This will promote economic and educational strength and development of individuals and communities.

Product design and development in Nigeria requires great improvement in conceptualization and visual appeal. Modeling prototypes and mould making should be approached with the highest level of skill, dexterity and finesse (Sullayman, 2003; Ajayi, 2007). The development of ceramic raw materials for glaze formation, the design concept and methods in ceramic production will tremendously enhance the quality and growth of ceramics in the North Eastern States of Nigeria and internationally.

Agriculture and Pottery productions are labour intensive, thereby engaging more people in productive venture. A number of national development plans in Nigeria spanning from 1962 – 1985 has been formulated to bring about the desired industrialization which was regarded as the beacon of national growth. Government set up import substitution industries with the intention of fundamental development model for attaining self – reliance using the revenue realized from oil. Although most of these industries were established using imported machines and raw materials, their growth has been slow and marginal since they depended on imported inputs such as raw materials, machineries and spare parts. The consequences of such failure of the import substitution policy was the neglect of the agricultural sector resulting in the massive importation of food, and lack of development of local raw materials as input for industries. Research into viable substitutes for imported industrial inputs was not implemented as part of the industrial policy of the government then (Nwanah, 2007). These industries include:

- Federal Institute of Industrial Research, Oshodi (FIIRO)
- Project Development Institute, Enugu (PRODA)
- National Steel Council, Kaduna (NSC)

Among the many challenges confronting the ceramic industries include the following: low level of application of science and technology to the mining and beneficiation of locally available non metallic minerals, dearth of requisite high level

manpower, limited and uncoordinated Research and Development (R & D) of ceramic materials, and lack of government incentive.

Some of the problems of ceramic industries in Nigeria are dependent on importation of raw materials and their possible applications to limited research materials and their development to substitute these imported products. Due to inadequate investment in ceramics in Nigeria, ceramics producers use a number of raw materials whose properties are not known precisely. Prospective Nigerian ceramics producers as a matter of necessity, mine, process and refine their raw materials. In view of this problem, the Nigerian ceramics cannot match imported ceramics in terms of quality standard. In developed economics, there is a specific process of standardization of raw materials and inputs; guaranteeing the quality and consistency of the products. The absence of an established comprehensive industrial test facility for materials, as well as evaluation and characterization is a major challenge of investment in ceramics (Nwanah, 2007).

Ceramic education programme is currently an- inter and multi- disciplinary field which should be reflected in the curriculum. This programme should take into account the country's objective in the area of ceramics, technological level and should be aimed at catching up with nations like China, Korea and others that have successfully transformed their economies.

In Nigeria, nearly all the necessary raw materials for development of ceramic glazes are available in almost all the states of the federation. These materials are found in raw state and cannot be used directly for glaze making. The problems of lack of milling facilities have hindered rapid development of glaze technology in North-Eastern Nigeria. The ceramic raw materials processing sector of our national economy is underdeveloped (Bassey, 2009). Therefore every practicing ceramist and a mining engineer have to quarry his/her own materials from the source. The good intention on the part of the government

needs to be supplemented by assisting in the acquisition of these basic raw materials. The processing industry should be given some local raw materials. This could be among other reasons – the lack of glazes obtained from researches for qualitative products. This situation calls for reflection, creativity and innovations in the ceramic profession. (Ahuwan, 2003)

In contemporary techniques of pottery production, government, individuals, corporate entrepreneurs and tertiary institutions have to become active agents in the process (Ahuwan, 1999). In addition to the suggestions, researchers should put in their best to see that the profession moves forward by contributing in the processing of locally available materials through tests and experiments.

The modern approach to pottery (ceramics) production started as experiments to test the locally available materials and their suitability for production of tableware. However, this was through the trend of available literature. Research should be the main stay of postgraduate programmes in institutions of higher learning especially in the universities. Most of these institutions are ill equipped; most of the equipments are archaic and derelict. Raw materials cannot be accurately tested. Simple processing procedures like calcining, crushing, ball milling cannot be carried out in some of such institutions. Where kiln is available, fuel such as gas, kerosene or diesel may be scarce and prohibitively expensive. There is also the lack of resource centers to supply information and standard materials. These problems put together would discourage the greatest of all zealots in the profession (Ahuwan 1999).

1.2 Statement of the Problem

The lack of glaze materials and glazes in tertiary institutions in the North-Eastern States of Nigeria is slowing down the development of ceramic education. The fact is that learning theory without the practical aspects of ceramics is not a good development for

ceramic education in tertiary institutions. This lack of glaze materials and glazes in tertiary institutions hinders the effectiveness of teaching and learning processes, and this could affect the practical performance of the students negatively. The problem of this study is the exploration of locally available raw materials from the North Eastern States of Nigeria for the production of ceramic glazes for effective teaching and learning processes.

1.3 Aim of the Study

The aim of this study is to identify local raw materials suitable for development of glazes for educational purposes and for studio potteries in North-Eastern States of Nigeria.

1.4 The Objectives of the Study

The specific objectives of the study are to:

1. identify, obtain and explore some raw materials suitable for development of glazes in the North-Eastern States of Nigeria. (Bauchi, Borno and Yobe States)
2. analyze the raw materials obtained by means of x-ray fluorescence Spectrophotometer (XRF)
3. beneficiate the raw materials and formulate glazes by composing the constituent materials.
4. determine the maturing temperature suitable for the glazes developed.
5. carry out some property tests of fired glazes on ceramic wares which include, hardness, imperviousness tests, etc.

1.5 Scope and Delimitation of the Study

This study is delimited only to considering some ceramic raw materials from the North-Eastern States of Nigeria for glaze formulation for use in educational institutions and potteries.

1.6 Research Questions

1) Have the raw materials from the north-eastern states of Nigeria been identified, obtained, explored and found suitable for development of glazes in North Eastern States of Nigeria?
2) Would the X-ray fluorescence method of chemical analysis be adequate for analyzing the chemical properties of these raw materials?
3) Will the quality of the materials improve after being beneficiated for glaze formulated?
4) How would the maturing temperature suitable for the glazes developed be determined?
5) How would the property test of hardness and imperviousness be conducted?

1.7 Hypothesis of the Study

The Hypothesis formulated for this study was that the raw materials from the North-Eastern States of Nigeria will not be suitable for glaze formulation.

1. 8 Significance of the Study

Generally, the developments of local raw materials play very important roles in the socio-economic life of the people of any nation. The development usually starts through education by educating the students in schools. In this regard, through teaching and learning processes. The study will be of importance to the tertiary institutions in

North Eastern Nigeria that offer ceramics in their curricular because the outcome will serve as a reference material to such institutions. It will make it easier for the students and staff to follow the procedure involved in processing ceramic materials for teaching and learning in institutions. It will also broaden the knowledge of the students to be more creative when searching for similar materials for their practical work. This study will also ease the problem of traveling with the student's project to other institutions and training centers like Ahmadu Bello University, Zaria and Abuja. It will be of economic benefit to the local communities and the nation. If pottery producers are going to make any meaningful contribution to the economy, human development and compete with other professions in this country and beyond, then their products must be standardized.

These could be achieved through the use of a variety of glazes and clay bodies for the production of their wares (Alkali, 2004). Ceramic products are not all about functionality and durability but aesthetics as well. These are features that one looks for in any work of ceramics. The raw materials in ceramics are the major elements that contribute in bringing out all these features. Thus the introduction of a new material and its unique characterization is expected to create a new outlook in terms of its finishing and beauty. Perhaps this new outlook or unique appearance could be an added advantage that will promote marketability of ceramic product (Alkali, 2009) In line with the above points, development of raw materials for the formation of glazes is of great importance in ceramic industries and in institution for better quality products in the industries and institutions.

Undoubtedly previous researches conducted on Nigeria's raw materials such as feldspar, quartz, limestone, kaolin and a host of others have revealed that these raw materials found in Nigerian soil are of high industrial quality and could compete

favourably with the imported types if they are properly beneficiated using standardized methods (Datiri, *et al* 2008).

There is a constant search for new and relevant sources of materials so as to enhance the supply of materials, the cost of production and the quality of products. Its future will depend largely on the national will and determination to succeed (Ahuwan 1999). This would be possible if the government would facilitate the appropriate channel of establishing manpower that can contribute positively in processing of local materials for glaze making in tertiary institutions.

The exploitation and processing of ceramic (glaze) raw materials are enough to influence development and capacity building among the local miners around the communities where they are located (Kashim, 2011).

CHAPTER TWO

REVIEW OF RELATED LITERATURE

2.1 Introduction

This chapter reviews available related literature relevant to the following areas; (1) Ceramic Raw Materials, (2) Glaze Raw Materials, (3) Glazes, (4) Glaze Calculations, (5) Fluxing Agents, (6) The Empirical Glaze Formula, (7) Variables in Glaze Formulation, (8) Ceramic Oxides, (9) Glaze Formulation, (10) Pyrometric Cones, (11) Temperature Ranges, (12) Glaze composition with Regard to Glaze Properties, (13) Desired Properties of Glazes, (14)Experimenting on Test Tiles, (15) Developing Original Glazes, (16) Keeping a Log.

2.2 Ceramic Raw Materials

Since clay materials are available, the locations, relevance, types and its characteristics should be taken into account when formulating for the development of glazes. With the present day advanced technology, one can achieve almost any effect one wishes with ceramic raw materials. But first one must know something about these materials and how to use them. A potter must have practical knowledge about the elements or metallic oxides, their characteristics, how they work with other elements, under what heat conditions they produce best results, and in what compounds they are readily available. Then one will be able to devise glaze recipes intelligently (Count, 1974). In order to gain an understanding of the qualities imparted to the glazes, the beginner should make several tests with varying proportions of the ceramic materials (Nelson, 1978). The basic materials needed in glaze formulation are available all over Nigeria (Emodah, 2006).

2.3 Glaze Raw Materials

Glaze raw materials are the solid minerals that are milled and mixed to form glazes. Common materials that are used in glazes include the following: Calcium oxide – (CaO), Zinc oxide – (ZnO), Barium oxide – (BaO), Kaolin (Al_2O_3 $2SiO_2$ $2H_2O$), Potash Feldspar - (K_2O, Al_2 O_3 $6Si$ O_2), and so on and so forth. Glazes are important factors in ceramic materials. Its application to pottery products dignifies the products. It also allows the products to be fired at high temperature, which guarantees the hygienic aspect of the products (Emodah, 2006).

Some of the materials or chemicals used in the ceramic process are plastic- clays, adhesives, cohesive, while some are non-plastic: fillers, silicate, openers, modifiers, deflocculants, colouring oxides, toxics, hygroscopic, efflorescent and fritted materials. These are to be used in clay bodies or glaze preparations.

Since these materials are mostly natural substances, they are bound to change if stored under wrong conditions. Some materials need quick utilization while some may have a long storage life (Udoh and Ekong, 2008). For example metals can change after long storage (rust), Calcium chloride will absorb water or moisture from air or other substances. Many of the ceramic (glaze) raw materials pose a great danger to the worker's health (Sullayman, 2004)

Ceramists should be educated about safety in ceramics, as wrong handling can inject toxins into the body resulting possibly in fatal consequences. Keep simple rules of hygiene and good observatory knowledge of handling of materials. Do handle ceramic materials with care, knowledge, and intelligence.

Some glaze problems arise from formulating application method, drying methods, loading in the kiln. The loading of gloss firing should be done with extra caution since the

body expands more than it shrinks and may stick on or pull wares. The consideration of the types of materials in the glazes should be paramount as some materials that are run or fall short of expectation thereby. One example of the fluxing materials combination is that calcium oxide which melts about 1200^0C and feldspar about 1000^0C will combine about 25% calcium with 75% feldspar to melt at 900^0C or even 850^0C and thereby exhibiting eutectic characteristics (Zakin in Udo and Ekong, 2008).

Another example is that experience shows that Barium Oxide or carbonate which is a refractory; at about 1280^0C can flux down seriously if there is borax in the glaze. In considering quantity therefore, glazes tests should be made first before application to a big kiln load.

Assumptions should be avoided in ceramics, especially in materials preparation and in firing reactions of materials. It is possible to conclude that wares' position, timing and sintering conditions of the ceramic firings, dissimilar glazes/wares or materials contents could cause effects. For example, chrome oxide which gives a hard green in firing, if zinc glaze ware is put in the kiln, the expected green would be brown as fumes of zinc diffuses freely. It is advisable that in studio firing, even with industrial glazes application, warning should be given on the glaze contents, medium, temperature to be fired and atmosphere so as to identify the undesired effects of some materials and remove it before the "magic of the kiln" is accepted instead of ignorance of standardizing materials in studio ceramics. The standardization is in form of observing, recording and thereby being able to repeat the result if need be (Udoh and Ekong, 2008). 'Practice makes perfect and becomes mastery'.

Glaze materials are ground finely in a ball mill and sieved very fine using 40-80 meshes. In advanced ceramics, ceramic processes go beyond decoration to include sol-gel, nitride ceramics, and composites etc., which are used purely to get a particular type

of effect. This is the application sol-gel to ceramic surfaces or the deposition of sol-gel to ceramic surface or the deposition of powder on surface known as Chemical Vapor Deposition (CVD) (Emodah, 2006).

2.4 Glazes

The art of making glazes may be traced back to ancient civilization. The traditional potter used the burning method and locally brewed stains to give pots good finishing. This was achieved by treating the fired pots with a mixture made from bark of trees. This acts as glaze to render the pot impervious and attractive. Glaze materials are: feldspar, quartz/silica, limestone, kaolin, ball clay and bentonite (Bassey, 2009). Glaze is a layer or coating of a vitreous substance which has been fused to a ceramic object through firing and oiling.

Glaze is "a thin, glass like coating that is fused to a clay surface by the heat of the kiln" Glaze is a continuous adherent layer of glass or glass crystals on the surface of a ceramic body." The glaze is usually applied as suspension of glaze brining ingredients in water, which dries on the surface of the piece in a layer. On firing, the materials react and melt to form a thin layer of glass (Norton, 1956; Nelson, 1971; Alasa, 2005, and Counts 1974).

There are three main or basic elements required in glaze formation:

1. Silica, the glass former; (2) A Flux, which lower the fusion point of the silica; and (3) Alumina, a refractory element that gives added toughness and hardness to the glaze, allows a higher maturing temperature, increase viscosity and prevents diversification (Hogan, 1973 and Nelson, 1971).

There are different types of glazes. For example ash glaze, Temmoku, chun, crakle, raku and others. Each batch of glaze is being prepared or formulated depending on

the products the potter is going to glaze. The firing temperature will also determine the type of glaze to be formulated. For example, low fire glaze materials could be of varying proportions of feldspar, kaolin and borax fired to cone 04. As suggested beginning formula for a cone 04 glaze test is three parts borax, three parts feldspar, and four parts kaolin. A total sample of 100 grams is advisable. Experience is required in glaze formulations (Nelson, 1971). The glaze should be tested on tiles before applying on large products to avoid materials and time wastage.

Glaze formulas are available in books and craft magazine and from the potters. Containers for storing glazes, measuring cups and spoons, a 60-mesh screen, a brush for applying the glaze, and an under glaze pencil for marking test tiles would be required and so is a weighing scale for very accurate ingredient measurement. The colleges should have these facilities in their stores (Hogan, 1973).

To mix a glaze, adequate water in proportion to the dry ingredients should be used so that the result is about the consistency of light cream: The glaze is poured through a screen to remove lumps and transferred to a labeled container when it is ready. Only by experimentation will one gain understanding of the interrelationship of glaze materials, the particular effects of each ingredient and how to adjust glaze formulas to fit a clay body.

Glazes may be coloured, transparent or opaque. The main purpose of the glazes is to provide a surface that is hard, non absorbent, and easily cleaned. At the same time glazes provide aesthetically attractive coatings, variety of surface colours and textures (Bassey, 2009).

Rhodes in Alasa, (2005) describes glaze as a thin glass-like coating which is fused into the body of a ceramic ware. As asserted, the normal glaze is transparent and has a high gloss surface which is a result of the complete fusion of the ingredients of the glaze

and the formation of a smooth layer of clear glass on the surface of the piece. Norton in Alasa (2005), also described glaze as a continuous adherent layer of glass, or glass and crystals, on the surface of a ceramic body. The layer of glass or crystal is applied as suspension of glaze forming ingredients, which dried on the surface of the piece in a layer and on firing, react and melt to form a thin layer of glass providing a surface that is hard, non-absorbent, and easy to clean.

Generally speaking, glazes are a type of glass. Glazes are vitreous or glassy coatings, fired on to the surface of wares. They are produced as a result of the fusion or melting of silica and other fluting materials. The raw glaze solution is first applied to the ceramic ware to create a powdery layer composed mainly of glass forming material as quartz, fluxes and stabilizes (Pius, 2008).

2.4.1 Importance of Glazes

They are compounded in order to provide covering for ceramic bodies, which shall among other things be:

- Insoluble in water and the use of acids and alkalines likely to be met in everyday use.
- Glaze can serve to colour, decorate, strengthen or waterproof a ceramic ware.
- Resistant top scratching.
- As far as possible, impervious
- Resistant to crazing, peeling and similar faults.
- Suitable for producing certain decorative effects such as colour, crystal development e.t.c. and
- Fusible at predetermined temperature ranges (Ajala and Munai, 2007).

Generally, Glaze is functionally important for earthen ware vessels, which would otherwise be unsuitable for holding liquids due to porosity. In addition to the functional

aspect of glazes, aesthetic concerns include a variety of surface finishes, including. Glazes may also enhance an underlying design or texture which may be either the "natural" texture of the clay or an inscribed, carved or painted design.

2.5 Sources of Clay

Clay materials abound in most localities in Nigeria. The garden clay pits and construction sites may yield us variety of clay materials. Some knowledge of the local geology or consultation with geology teachers, also local potters can be sources of valuable information. Ball clays are usually more plastic and have greater shrinkage. These clays can range in colour anywhere from a light tan to a dark reddish-brown after firings (Aluwong, 1988).

Clays are found nearly everywhere on the earth's habitable surface. Clay is the earth's crust; it results from decomposition by weathering of the older rocks of the feldspar type (Norton, 1965). Clay is obtained when a granite-type rock is broken down and decomposed into fine particles. When water is added to these particles, they become plastic and can be forced into shapes (Counts, 1974). Clay is derived from the disintegration of granite and other feldsparthic rock which, as they decompose, deposit alumina and silica particles (Nelson, 1978). Clay is formed as a result of the decomposition of an igneous rock. Clays vary according to their location that is indicative of where they are found and the process responsible for their formations. Primary clays are residual clays and secondary clays are clays that have been transported from their places of formation by such natural forces as wind, glacier and water, and have been further deposited in strata in lower areas (Gukas and Datiri, 2001).

Having access to the clays will enable students to mix different clay bodies and understand their behaviours while in use. Also understanding of the chemical aspects of the clays would be easier for them and they will know which clays are suitable for

particular purposes. A good knowledge about these by the students is essential since they are going to transfer such knowledge to the younger students in the secondary and primary schools.

Nearly all clays contain some impurities, that is why processing of clay before working is important (Umar, 2003).

2.5.1 Ball clays

These are sedimentary plastic refractory clays which are dark in the unfired state because of organic impurities but burn white or cream coloured as long as they are not vitrified fully. They have a large proportion of kaolinite but also contain a variety of impurities and probably have some montmorillonite attached to the edges of the kaolinite platelets. The name is derived from the English mining method of cutting the clay out in cubes or 'balls'. Ball clays are used in white-wares (earthenware, porcelain etc) to make the body more plastic and workable (Singer and Singer, 1971).

Ball clays improve the plasticity and working properties of clay bodies and increase mechanical strength of casting slips. In glazes they are used to introduce alumina and to a lesser extent silica. They also act as suspenders and hardness (Fulham, 1611). It also furnishes the glaze with silica and alumina, serves as a flux, a binder as well as a medium keeping the glaze components in suspension. It's use was however, limited due to the possibilities of high iron content that could colorate the glaze to give brown or tan colour (Alasa, 2005).

Ball clays or plastic clays are examples of sedimentary or secondary clays. These are the clays which over the years have been removed from their place of formation by natural forces and deposited elsewhere. Such removal allows the clay to get in contact with other materials as organic matters, iron, oxide, debris, etc (Pius, 2008).

2.5.2 Kaolin

China clays are highly refractory primary clays (Kaolin) which fire white. In glazes they are used to introduce alumina and, to a lesser extend silica. They also act as suspenders and hardness to the unfired glaze. A good general purpose clay for all types of work put especially for slip casting. Above 1200°C it may caose slight discoloration (Fulham, 1671).

Kaolin (china clay) is the important source for alumina and silica in the glazes. When start to order some from a list, one may find that calcined kaolin is given. This means that the clay has been heated to drive off chemical water, carbon dioxide, or volatile gases. Kaolin could be calcined in bisque bowls when bisque-fired. Using calcined kaolin helps to prevent "crawling" and some other glazing detects. Usually, however, there are no problems and the clay content of glazes serves as a binder to make the glaze stick to the bisque were better before firing (when it counts because it is in the hands) (Counts, 1974).

This was introduced into the glaze as a major source of alumina (Al_2O_3) and silica (SiO_2) in the glaze. The former serves a refractory elements importing higher firing temperature, strength, hardness, increase viscosity, prevent detrification, effect mattness in the glaze and unit with the silica to form a needle-like mulite crystal that creates a bond more resistance to abrasion and thermal shock. Silica on the other hand, is the major source of glass in the glaze. The clay (kaolin) also serves as a floatative – helping to suspend the various particles in the glaze composition (Alasa, 2005).

2.5.3 Feldspar

Feldspar is a word derived from "fels", meaning rock, and spar, one of the various crystalline minerals. Potters soon learn that feldspars are a source of potassium, sodium or calcium, as well as alumina and silica. These rocks are common all over the face of the

earth. In America, potters usually remain dependent on mining operations to supply their materials. Suggested the selection of one or two domestic feldspar in your shop to simplify procedures. Feldspar also is useful sometimes in a clay body (Counts, 1974).

It is used in almost all glazes and in high-fired glazes. It is often the principal flux, it is made up of an alkaline portion sodium, potassium, calcium etc, in combination with alumina and silica. Feldspars high in potassium are favoured for addition to clay bodies while those high in soda are preferred for glaze making because of their relating lower fusion point. When a glaze recipe calls for feldspar without specifying which, trial may have to be carried out to determine which feldspar is really required.

Feldspar is the chief source of potash and soda in an insoluble form. It is a kind of flint. Its melting point is 1280°C. The fusible nature of feldspar is due to its content of sodium, potassium or lithium. Glaze with high feldspar can craze following the present of high sodium or potassium content (Alasa, 2005).

Feldspars are universal fluxes used in all types of bodies and affect practically all characteristics of the fired ware, even translucency. Their alkaline properties are such that they may affect the casting and working properties of slips, depending on the other constituents. Potash feldspar is generally preferred for use in bodies (Up to 15%) as it produces a stronger material than soda feldspar which reduces the firing temperature. Feldspar combine all the principal glaze ingredients in an insoluble form; in effect they are natural flits used in all most all glazes, particularly stone ware (Up to 50%); those incorporating potash feldspar are more durable, those containing soda feldspar mature at a lower temperature (Fulham, 1671).

2.5.4 Quartz

Quartz (Silica) is an alternative to flint as a source of silica (Fulhan, 1671). Silica SiO_2, potter's flint or quartz) is one of the essential materials. Some pottery supply houses

offer it for sale both 200 mesh and 325 mesh. Prefer the finer mesh for glazes. Clays and feldspars already contain silica. Sometimes a potter needs additional silica in the clay body. A glaze mixture often is half silica since this material is the main glass-forming oxide (Counts, 1974).

Flint (SiO_2) is otherwise known as silica. Silica in its pure and crystalline state is referred to as quartz. Quartz is usually gray, black, or brown and has extremely small crystalline structure. Grinding any form of crystal into fine powder may make the potter's flint. Flints is insoluble and chemically inert, it is abundant, cheap and also the main sources of silica the major glass former in glazes silica only begins to melt at about 1700°C (Alasa, 2005). It is a hard mineral, often in crystal form (Wehmeier, 2002).

2.6 Glaze Calculations

All matter composing the earth is made up of approximately one hundred chemical elements in the form of atoms. These elements do not occur in nature in a pure form but in compound, which are groups of atoms held together by electrical attraction or bond. The atom is much too small to be weighed, but it is possible to determine the relative weight of one type of atom to that of another. Oxygen, symbolized by the letter O, has been given the arbitrary atomic weight of 16; other atoms are attributed weights in relation to oxygen. Silica is a major ceramic compound found on clay and glaze. The molecular formula for silica (more precisely silicon dioxide) is SiO_2. The weight of silica is 28. Since two oxygen atoms weight 32, the total molecular weight of the compound silicon dioxide is 60. Silica can be added to a clay body or glaze in the silicon dioxide form, but it is most commonly found as part of more complex compound such as kaolin (Al_2O_2. $2H_2O$) or potash feldspar K_2O. Al_2O_3. $6SiO_2$) (Nelson, 1971).

Glaze calculation is by atomic weight. The chemical equation of each material is involved. Atoms and molecule of substances taking part in chemical changes also react

qualitatively between the molecular involved. This is determined when calculating a glaze. For example, the molecula weight of calcium carbonate is 100. i e Ca (40) +(12) $+0_3$ (16x3=100 so 100 grams of calcium carbonate is heating to yield 56 gms Cao and 44 gms of CO_2 while the later evaporate. So kaolin is analysed as: $A1_2 O_3$ $2SiO_2$ >-$A1_2 O_3$. $2SiO_2$+ $2H_2O$ molecular weigh 258 = 22+ 36 water is evaporated and lost on ignition (Emodan, 2006). The Table (2.6) one is showing an example of glaze batch weights calculation as stated.

Table 2.1 Glaze Batch Weights Calculation

Raw material	Equivalents	Molecular weights	Batch weights
Soda feldspar	0.03 x	524 =	15.72
Borax	0.16 x	382 =	61.12
Soda ash	0121 x	106 =	22.26
White lead	0.153 x	775 =	118.38
Red iron oxide	0.07 x	160 =	11.20
Flint	1.91 x	60 =	114.60

(Nelson, 1971) and (Alasa, 2005).

2.6.1 Limit formula

In glaze experiments, no definite limits. The major purpose of these glaze tests was to gain familiarity with the qualities of the various glaze chemicals. The firing temperature and the type of flux are also important factors. In the several limits formula that follows, a number of possible fluxes are indicated. The total amount used must add up to a unit of one. In general, a higher proportion of alumina will result in a matt surface, provided the kiln is cooled slowly. Table (2.6.1) two is showing examples of limit formula of glaze types as indicated in the table.

Table 2.1.1 Limit Formula of Glaze Types

Cone	**08-04**	**Lead**	**Glaze**
PbO	0.2-0.60	Al_2O_3	0.15-0.20
KNaO	0.1-0.25	B_2O_3	0.15-0.60
ZnO	0.1-0.25		
CaO	0.3-0.60	SiO_2	1.5-2.50
Boas	0.-0.15		
Cone	**08-04**	**Alkaline**	**Glaze**
PbO	0-0.5	Al_2o_3	0.05-0.25
KNaO	4-0.8		
CaO	0-0.3	SiO_2	1.5-2.5
ZnO	0-0.2		
Cone	**2-5**	**Lead**	**Calcium Glaze**
PbO	0.4-0.60	Al_2O_3	0.2-0.28
CaO	0.1-0.40		
ZnO	0-0.25	SiO_2	2.0-3.0
KNaO	0.1-0.25		
Cone	**2-5**	**Lead**	**Borax Glaze**
PbO	0.2-0.3	Al_2O_3	0.25-0.35
KNaO	0.2-0.3	B_2O_3	0.20-0.60
CaO	0.35-0.5		
ZnO	0-0.1	SiO_2	2.25-35
Cone	**2-5**	**Colemanite**	**Glaze**
CaO	0.2-0.50	Al_2O_3	0.2-0.28
ZnO	0.1-0.25	B_2O_3	0.3-0.6
Boas	0.1-0.25		
KNaO	0.1-0.25	SiO_2	2.0-3.0
Cone	8-12	Stoneware	Glaze
KNaO	0.2-0.40	Al_2O_3	0.3-0.5
CaO	0.4-0.70	B_2O_3	0.1-0.3
MgO	0-0.35		
ZnO	0-0.30		
Boas	0-0.30	SiO_2	3.0-5.0

(Nelson, 1971)

2.6.2 The RO, R_2O_3, RO_2 SYSTEM

There are three principal components in glaze. One of these is silica (SiO_2), which gives the glaze its glassy, transparent quality. The second is alumina (Al_2O_3), a refractory element, which contributes toughness and abrasion resistance to the glaze. Since neither of these oxides will melt at temperatures below 1704°C(3100°F), the potter must add a third ingredient, a flux, such as lead oxide or sodium oxide, that will lower the melting point and combine with both silica and alumina to form, after firing, a hard, glassy coating on the ceramic body.

Glazes are usually mostly comprised of a glassy material. In the case of opaque and matt glazes the glassy matrix contains crystalline mater. Certain glazes which contain large amounts of crystalline matter, deliberately grown in-situ to develop special properties, are described as glass ceramic glazes.

Glazes are usually mostly comprised of a glassy material. In the case of opaque and matt glazes the glassy matrix contains crystalline mater. Certain glazes which contain large amounts of crystalline matter, deliberately grown in-situ to develop special properties, are described as glass ceramic glazes.

In order to compare glazes or to understand the effects of these varied chemical, it is necessary to separate the component oxides into the three major parts of the glaze formula. The symbol RO refers to the fluxing agents, which are chiefly metallic or alkaline elements that form their oxides by combining with an atom of oxygen, such as CaOor PbO. Similarly, alumina (Al_2O_2) can be symbolized by R_2O_3 and silica (SiO_2) by RO_2.

The RO, R_2O_3, RO_2 system indicates the three principal components in glazes. That is RO stands for bases, R_2O_3 stands for neutrals while RO stands for acids (See table 2.6.2).

Table 2.1.2: The RO, R_2O_3, RO_2 SYSTEM

RO	R_2O_3	RO_2
Bases	Neutrals	Acids
Li_2O *	Al_2O_3	SiO_2
K_2O *	B_2O_3	TiO_2
CaO	Fe_2O_3	ZrO_2
MgO	Sb_2O_3	Glass formers
Boas	Cr_2O_3	
ZnO	(Refractory elements)	
FeO		
PbO		
CdO		

(Nelson, 1971) and (Alasa, 2005).

2.7 Fluxing agents

In the first column are the RO oxides that have the effect of fluxes in either a glaze or a body. They are also called the base oxides. Some of the neutrals or amphoteric oxides can function as either a base or an acid oxide. However, the major oxide in this group, alumina, (Al_2O_3), always has refractory effect in a glaze. On the other hand, red iron oxide (Fe_2O_3) is an active flux as well as colorant. Boric oxide (B_2O_3) can react as either a base or an acid. Silica is the major oxide in the acid column. The contribution of TiO_2 and ZrO_2 to a glaze is opacity, not their glass-forming effect with minor exceptions, the RO, R_2O_3 and RO_2 method of categorising the main components of a glaze works very well, (Nelson, 1971).

2.8 The Empirical Glaze Formula

In the interests of simplifying calculations, glaze batches are usually reduced to an empirical formula. This is a formula in which the various active ingredients are expressed in molecular proportions. By contrast, the batch recipe is a proportion expressed in the

actual weights of the raw chemical compounds making up the glaze. Since the materials used in the average glaze batch are rather complex compounds, it is distinct advantage to be able to define the glaze in terms of single oxides that bear the same proportional relationship to each other as when in their more complex form in the glaze batch. These oxides are grouped into RO, R_2O_3 and RO_2 units.

Before a batch recipe is converted into an empirical formula, it is first necessary to know the chemical formulas of the individual raw materials, as well as their equivalent weights. Many compounds are designated by more than one name, such as whiting and calcium carbonate.

The molecular weight of a compound is the sum of the atomic weight of its constituent elements. The equivalent weight will often be the same as the molecular weight of the compound, but in many cases it is smaller. This is true of potash feldspar. (See table 2, 8a).

Table 2.2a: Equivalent weight/molecular weight of Potash feldspar

			Equivalent weight		
Raw material	Formula	Molecula weight	RO	R_2O_3	RO_2
Feldspar (Potash)	$K_2O.Al_2O.6SiO_2$	556.8	556.8	556.8	92.9

According to the given formula, when one molecular unit of potash feldspar is added, the glaze will have one unit of potassium oxide, one unit of alumina, and six units of silica. Since one unit each of potassium oxide and alumina form the compound, their individual equivalent weights are 556.8. And since six units of silica are necessary to form the compound, the equivalent weight of silica is $^1/_6$ of the compound weight, or 92.9. The equivalent weights of the oxides of a compound are the same as its molecular weight if the oxide in question appears only once. However, if more than one unit of an

oxide occurs, then its equivalent weight will be found by dividing the compound molecular weight by the number of times the oxide in question appears. Check over the raw material formulas for determination of the oxides appropriately.

The batch weight of the raw materials are divided by the equivalent molecular weights of the particular oxides concerned, giving the molecular proportions in the form of single oxides. By arranging these oxides with the amounts calculated into their appropriate RO, R_2O_3 and RO_2 groups, we find that the glaze batch has the following empirical formula: see tables 2.8b and 2.8c.

Table 2.2b Glaze Molecular Proportions

Glaze recipe	**Raw materials**	**Parts by weight**
A lead – borax	White lead	128
Glaze maturing at cone 04	Whiting	15
	Borax	103
	Feldspar (Potash)	83
	Kaolin	38
	Flint	85

Table 2.2c Glaze Batch Empirical Formula (RO System)

RO	**R_2O_3**	**RO_2**
.495 PbO	.295 Al_2O_3	2.599 SiO_2
.150 CaO	.540 B_2O_3	
.149 K_2O		
.270 Na_2O		

Source: Nelson 1971

The total RO oxides come to 1.064 instead of the desired unit of one, because round numbers have been used in the batch recipe. By dividing each figure in table2.8c by 1.064, it will result to the following empirical formula, of RO now 0.998, a figure that is accurate enough for comparative purposes (See table seven 2.8d).

Table 2.2d Empirical Formula RO System for Composition

RO	**R_2O_3**	**RO_2**
.465 PbO	.277 Al_2O_3	2.442 SiO_2
.140 CaO	.507 B_2O_3	
.140 K_2O		
.253 Na_2O		

Source: Nelson 1971

Considerable research has been done on the properties of ceramic materials, and some reasonable predictions can be made about the probable change that a particular chemical will cause in a known type of glaze. There are, however, many variables, such as the length of the glaze grinding time, the thickness of application, the reactions between glaze and body, the kiln atmosphere, and the rate of temperature rise and fall. Since each or all of these conditions can markedly affect a particular glaze, glaze experimentation is something less than a true science. Successful work depends largely upon the overall experience and care of the operator in controlling these factors, (Nelson, 1971).

Calculating for a glaze using empirical formula; the sum basic oxides must be equal to unity. The basic oxide is PbO and dividing through by 0.287 the formula becomes: 1 PbO: 0.202 Al_2O_3 : 1.756 SiO_2 (See tables 2.8e and 2.8f).

Table 2.2(e) Glaze Batch using Empirical Formula

Materials	Mol. Wt	Mol. parts	Part by Wt	PbO	$A1_2O_3$	SiO^2
White lead	775	0.33	258	1		
Kaolin	258	0.2	52		0.2	0.4
Flint	60	0.35	81			1.35
			391	1	0.2	1.75

(Emodah, 2006)

Table 2.2(f) Batch to Empirical Formula

Raw material & formula	**Batch weight**	**Equivalent weights**	**PbO CaO B_2O_3 K_2O Al_2O_3 SiO_3**
White lead [$2PbCO_3.Pb(OH)_2$]	128 ÷(RO)	258.5	0.495.5
Whiting ($CaCO_2$)	15 ÷ (RO)	100	0.15
Borax	103 ÷ (RO)	181.04	0.270
($Na_2O.2B_2O_3.10H_2O$)	(R_2O_3)	190.7	0.540
Feldspar (Potash)	83 ÷ (RO)	556.8	0.149
	(R_2O_3)	556.8	0.149
($K_2O.Al_2O_3.6SiO_2$)	(RO_2)	92.9	0.890
Kaolin ($Al_2O_3.2H_2O$)	38 ÷ (R_2O_3)	258.1	0.146
	(RO_2)	129	0.294
Flint (SiO_2)	85 ÷ (RO_2)	60.06	1.415
Total	452		0.495 0.15 0.270 0.54 0.149
			0.295 0.599

Source: Emodah 2006

2.9 The Batch Recipe

To convert an empirical formula to a batch recipe, it is necessary to reverse the procedure. This type of conversion will require slightly more familiarity with raw chemical compound, because one must select those compounds containing the proper oxides without adding any unwanted elements.

Convert the following empirical glaze formula into a batch recipe:

0.40 Na_2O 0.03 Al_2O_3

0.46 PbO 0.32 B_2O_3

0.14 FeO

The general procedure is as follows: first, if any alkaline fluxes are present (Na_2O or K_2O) it is best to include as much as possible in a soda or potash feldspar. This is a cheaper source of flux and equally important, it is non-soluble and not as likely to become lumpy in a glaze as borax will. We then fill out the other oxides, trying to save the alumina for next to last and taking out the silica at the end. Alumina and silica are often included in other compounds, and the balance can easily be taken care of last in the form of either kaolin or silica. Since silica always presents in large amounts more than alumina and available in a cheap, pure form, it can safely be saved for the last.

In the first addition, when we take 0.03 equivalent of soda feldspar, we get six times as much SiO_2 as either Na_2 or Al_2O_3, because the formula for soda feldspar is Na_2O_3. Al_2O_3. $6SiO_2$. similarly, borax has twice as much B_2O_3 as Na_2O.

Now that we have the need molecular equivalents of the chemical compounds, we can find the gram batch weights (or pounds) by multiplying each equivalent by the molecular weights of the chemical compounds. Thus the following set of calculations: see table 2.9.

Table 2.3 Batch Recipe from Empirical Formula

Raw material	Equivalents	Molecular weights	Batch weights
Soda feldspar	0.03 x	524 =	15.7261.12g
Borax	0.16 x	382 =	61.12g
Soda ash	0121 x	106 =	22.26g
White lead	0.153 x	775 =	118.38g
Red iron oxide	0.07 x	160 =	11.20g
Flint	1.91 x	60 =	114.60g

Barium will also tend to create a mat surface. A study of these formulas indicates how the ratio of alumina and silica rises as the temperature increases. While not intended to take the place of glaze formulas, the listing should be a helpful guide to those seeking to change the temperature range of a favourite glaze. Some glaze chemicals are quite complex, and under certain conditions an addition to a glaze will not have the desired effect. By converting the batch recipes to empirical formulas and comparing them with the suggested limits, one ought to be able to detect the direct of the error.

2.10 Variables in Glaze Formulation

Potters are frequently disturbed and disappointed when a glaze obtained from a friend or from a text book produces quite unexpected results. Several factors may be involved in this problem. Ingredients that are more finely ground than usual will melt at as slightly lower temperature. In the same way fritted glaze additions melt more quickly than do raw compounds of a similar composition. Occasionally, the thickness of application or reactions with clay body will produce slightly different results. More distressing and troublesome is the variable nature of such major glaze ingredients as corn wall stone and the feldspar.

In attempting to alter a glaze that has proved unsatisfactory, the potter has several simple remedies. If the glaze is too runny, add flint or kaolin, either of which usually raises the melting points. If it is too dry, increase the flux to develop a more fluid glaze. Sometimes, however, the effect is quite the opposite, and in such cases student, quite justifiable, may feel that the instructor who suggested the change doesn't know much about glaze chemistry.

The use of triaxial diagram for planning experiments has many applications in ceramics. It takes much time to complete the entire sequence, and considerable effort can be saved by concentrating on those areas in which meaningful results can be expected. This system is especially helpful in gaining knowledge about the relative fluxing power of various compounds, their effects on colorants, and the blending colorants and opacificiers, (Nelson, 1971).

2.11 Ceramic Oxides

Oxides are formed when oxygen combines with an element which can be metallic or non-metallic (Cotton and Wilkinson, 1980, Menham, 2000). Glaze components are not single refined oxides but complex compounds that are commercially available at reasonable costs. The oxides may act as a base, neutral or acidic oxide serving as fluxes or colorants (Wheeler et al., 1973). Each oxide used in pottery is discussed under the entry for each element (Fournier, 1973). Metallic oxides and stains are used to give colours to the clay body or glazes and can be used under or over a glaze. One of such oxides commonly used as a colorant is cobalt. The basic oxides for glazes include: colouring oxide, zinc oxide, tin oxide, talc and others (Hogan, 1973).

The chemicals used by the potters are generally not pure single oxides but more complex compounds that are available for industrial uses in a moderately refined form at low cost. Because of differences in either the original mineral or in refining methods, the

ceramic chemicals available from different dealers can vary slightly. Even items from the same suppliers can change minutely from year to year. Commercial potteries constantly check new shipments of chemicals, but this is seldom done in the small studio. If a favorite glaze reacts strongly, one should consider this factor, provided, of course, there was no deviation from the normal glazing and firing procedures. The feldspar, in particular vary in their fluxing power, (Nelson, 1971).

Glazes are glasses and hence their most indispensable constituents are glass – forming oxides seconded by network modifying and intermediate of glasses (Singer and Singer, 1971).

Base Glaze

Fluxes	**Alumina**	**Silica**
(Lower melting point)	(Makes less runny)	(Makes the glass)

A base glaze will usually be (not always) colourless. Colours in glazes can be achieved by addition of oxide. Usually add a colourant to our taste using colouring oxide (or carbonate). Some flux materials contain alumina and silica as well. The source of variable alumina is kaolin which contains some silica as well. Some colouring oxide act as fluxes. One glass former (boric oxide) is also flux. Some materials behave quite differently in oxidation and reduction, Glazes are the protective coatings that make ceramics safe and useful.

2.11.1 Sources of Base (RO) Oxides

Barium oxide, magnesia, potash, calcia and lead oxide, among others. Barium oxide (BaO) is very active flux under some conditions. Its glass formation has brilliancy second only to the lead silicates. Barium's effect on the thermal expansion or the glaze is less than that of the alkaline and calcia. The best source for Barium is Barium carbonate ($BaCO_3$).

Calcium oxide (CaO), in comparison with the alkaline oxides, produces a glaze more resistant to abrasion, mild acids, and weathering. It also lowers the coefficient of thermal expansion, thereby increasing tensile strength. Although it is often used in small amount with other fluxes in low-fire glazes. It is the most common flux used at porcelain temperatures. As with alumina, an excess of calcia tends to produce mat textures.

Sources of calcium are: calcium carbonate ($CaCO_3$),

Calcium borate ($2CaO.3B_2O_3.5H_2O$), Dolomite [$CaMg(CO_3)$]

Bone ash, refined, [$Ca_3(PO_4)_2$]

Sodium oxide (Na_2O) is one of the more common low fire fluxes. It has the highest coefficient of expansion of all the bases and generally gives a lower tensile strength and elasticity to the silicates formed than do most other fluxes. The sources are: sodium chloride (NaCl), Borax ($Na_2O.\ 2B_2O_3.\ 10H_2O$), Soda feldspar $NaO.\ Al_2O_3.\ 6SiO_2$).

2.11.2 Sources of Neutral R_2O_3 Oxides

Alumina, antimony, borax, Chrome, red iron oxide. Unlike the RO group, which has numerous similar compounds, the R_2O_3 group is almost limited to alumina (Al_2O_3) and a few oxides that have the same oxygen ratio. The greatest different between a glass and glaze is the presence at alumina in the glaze. The alumina content is a most important factor in a successful glaze. It controls the fluidity of the melting glaze and enables it to withstand the temperatures needed to mature the body. Greater amounts of alumina increase the hardness of the glaze and its resistance to abrasions and acids.

Alumina (Al_2O_3) in a glaze can vary from 0.1 to 0.9 molecular equivalents, depending upon firing temperatures. The equivalent ratios between the alumina and the silica groups can be from 1:4 to 1:20. For glossy glazes the ratio is about 1:10. An increase of alumina will tend to produce mat textures. A glossy porcelain glaze firing

from cones 10-12 will have an alumina – silica ratio between 1:7 and 1:8, whereas the mats will be 1:3.2 to 1:3.8 Alumina also has an effect on the colours developed. The normal blue of cobalt oxide will become a rose pink in the absence of alumina. Chromium oxide, which usually gives various green tones, will tend to become reddish in the presence of excess alumina. Source of alumina are: Alumina hydrate [Al $(OH)_3$], Kaolin (China clay) Al_2O_3 .$2SiO_2$. $2H_2O$), (Nelson, 1971).

2.11.3 Sources of Acid (RO_2) Oxides:

The important oxide in this group, silica, has a refractory effect on glaze, while the others function largely as opacifiers or coloring agents. Silica (SiO_2) Combines readily with bases to form glossy silicates. It is the most common element in glaze, comprising about 50 percent of it by weight. In glaze it has the effect of raising the melting point, decreasing fluidity, increasing resistance of the glaze to water and chemicals, increasing hardness and tensile strength and reducing the coefficients of thermal expansion of the glaze. The amount of silica used depend upon the flux and the maturing point of the glaze, but it is generally between 1 and 6 molecular equivalents.

Silica (SiO_2) is commonly obtained from sand, stone, quartz sands, or flint pebbles. Silica is found combined with many ceramic materials. The following are the more frequently used silica compounds: Ball clay ($Al_2O_3.2SiO_3.2H_2O$), Kaolin ($A_2O_3.2SiO_2.2H_2O$), Soda feldspar ($Na_2O.Al_2O_3.6SiO_2$), Potash feldspar ($K_2O.Al_2O_3.6SiO_2$) (Nelson, 1971).

Cobalt oxide (Co_2O_3) is the major blue colorant. It is extremely strong and therefore is often fritted with alumina and lime or with read for lower-fire under glaze colors. The frit allows a lighter and more even color dispersion. Color stains made of cobalt, alumina, and zinc are uniform at all temperature ranges. Small amount of cobalt in

combination with MgO, SiO_2, and B_2O_3 will produce a variety of hues in the pink and lavender range, (Nelson, 1971).

2.12 Glaze Formulation

Glaze can be made from only three necessary elements: Silica, the glass former, a flux or glass; and alumina, the refractory element. It is not economically practical to use refined and pure oxides to make up glazes (Nelson, 1976). The compounds employed by the potter are usually those mineral that are abundant in nature in a relatively pure state. Nelson added that lower fire glaze will consist of: Kaolin ($A1_2O_3$. $2SiO_2$. $2H_2O$) potash and feldspar (K_2O. $A1_2O_3$. $6SiO_2$) and red lead (Pb_30_4) or borax (Na_20-$2B_203.10H_20$) as the flux. Nelson continues that in order to gain an understanding of the qualities imparted to the glaze by silica, alumina and the flux, the beginner should make several tests with varying proportions of feldspar, kaolin and borax fired to cone 04 (Nelson, 1978).

Even though two materials blended together could produce a good glaze, richer and better glazes are usually produced when more materials are blended for the purpose of glaze making. Due to the advantage of producing rich glazes, the study will adopt the use of triaxial blend. This model requires thirty-six members test slabs, which four different glaze material will be required to react with each other in various proportions on each slab. The slab at the three extreme corners had one each of the materials to be blended, while the slabs in between (within the model) percentages of either two or four materials depending on their position in the blend (Gonah, et al., 2003).

Most glaze materials are supplied from the manufactures in form of finely grounded powder. Glaze materials are usually attached with a supply label for easy identification and to avoid mistaken identification since most glaze materials look alike. Also some glaze materials are toxic, such as lead compound and Barium. The raw

materials used in glaze should be insoluble in water. Some eventually turned out to be soluble and caustic, with such materials, extreme care should be taken to avoid prolong contact with the skin (Alasa, 2005). The presence of powdered glass in a glaze batch will hasten the formation of the glassy state (Garkida, 2004).

The basic composition of most glass and glaze batches comprise of silica, potash/soda, alumina and calcium oxide/magnesium oxide acting as principal glass former, modifiers, intermediate oxide and stabilizers respectively. Feldspar is one singular raw material that contains three categories of these oxides. The most important members to glass and ceramics are potash, soda and lime. Feldspar occurs hardly in pure form. They carry impurities such as iron, calcium oxide and magnesium oxide and clay, which also depending on the glass/glaze type, these impurities can be allowed in the batches (Garkida et al, 2003).

Although several glass-forming systems exist ceramic glazes are almost exclusively based on alumino silicate glass system. The main glass forming oxide, silica (SiO_2) is modified by the addition of a range of other oxides. The modifiers act to alter thermal, chemical, and physical properties.

The glaze materials are usually applied as a water - based suspension by spraying or dipping methods. Mechanization is common though many craft and manual techniques persist. In some cases, dry and electrostatic methods are used for application of glazes on wares. Glazes are applied to many substrates including:

- Sanitary ware – toilets, basins, bath tubs
- Tiles – wall and floor tiles
- Table ware – crockery.
- Onaments – giftware, figurines
- Electrical porcelains

- Engineering ceramics
- Refractories.

2.13 Pyrometric Cones

Pyrometric cones are thin pyramids of ceramic materials that will not only deform or bend at a particular time-temperature but rather as a measure of the work done by heat on the pieces being fired(Hogan,1973).

Pyrometric Cones provide the potter with his most accurate method of measuring the work heat in the kiln. It is tetrahedron or pyramid shape one and three inches high with a base that is one half (1/2") inch across. He also states that they are compounds of materials similar to glaze. When softened by the heat they bend and when a complete arc is formed, the temperature in the kiln is the name at that of the cone number (Nelson, 1966). Pyrometric cone is a small pyramid shaped piece as nose composition which can be so adjusted to melt at a definite temperature under standard conditions. Musoke,(1987)This is to indicate the state of the formulated raw materials for suitability of the glaze formation.

Pyrometric cones are shaped like slender pyramids and made of special combinations of clay that melt at known temperatures. Each cone is stamped with a number (Counts, 1974). The number on the cone indicates the temperature at which that cone will become soft and begin to bend at the tip. As the temperature rises, the cone bends and eventually melts completely.

Cones are used in ceramics for recording heat work instead of parameters. A cone is a special piece of clay shaped like a triangle that you put in your kiln. As it heats up it softens, and it is calibrated pend over when one's clay and glaze have received the right amount of heat. However, the cone actually falls based on the total amount of heat it observed, not the maximum temperature it reached, so the temperatures can vary.

Low fire glazes are typically fired at cone 06-04. High fire glazes are typically fired at cone 9-10. Mid fire glazes fall in between and are fired at cone 4-6. Low fire takes less power, but mid-high fire which is fired using stoneware and porcelain clay, is stronger and more durable.

2.14 Temperature Ranges for Firing Glazes

Ceramic glazes each have a temperature range that they should be fired to. If the glazes are fired at too low a temperature, the glazes will not mature. If the temperature goes too high, the glaze will become too melted and run off the surface of the pottery. For success, a potter must know their glazes' temperature ranges at which they become mature.

When potters talk about ceramic firing ranges, they are usually referring to the three most common: low-fire, mid-range, and high fire ranges. In regards to glazes, we need to add two other ranges: very low-fire, and lower mid-range firing ranges.

2.14.1 Very Low-Fire

- From cone 022 (approx. 1112°F - 605°C)
- To cone 013 (approx. 1566°F - 850°C).

(Peterson, 2011)

This range is usually used for luster glazes and very low-firing glazes. Ware must be fired at least once at a higher temperature first, in order for the clay body to mature. The ware will often not only go through a bisque firing, but also a higher temperature glaze firing. Very low-fired over glazes and luster are then applied to the already fired primary glaze. The ware is returned to the kiln for a very low temperature firing in order to fuse the overglazes.

2.14.2 Low-Fire

- From cone 012 (approx. 1623°F - 882°C)
- To cone 02 (approx. 2048°F - 1120°C).

The low-fire range has historically been the most commonly used firing range. In the past this was mainly due to limitations in kiln technology. However, low-fire temperatures allow potters to use a variety of colorants that either burn off or become unstable at higher temperatures.

Low-fired ware can present some difficulties, including

- The clay body may remain overly porous
- Low-fire glaze colors can appear rather harsh and raw-looking.
- The high percentage of flux or stronger-acting fluxes used can result in a softer, less durable glazes, and
- Many of the traditional glaze materials used in this range are quite toxic in their raw state.

2.14.3 Lower Mid-Range

- From cone 01 (approx. 2079°F - 1110°C)
- To cone 3 (approx. 2264°F - 1210°C).

This range is being used more and more as potters become more concerned about energy and fuel usage. Another factor has been the availability of electric kilns that can comfortably reach this range without severely decreasing the kiln's and the kiln elements' lifespans.

Other advantages of firing in the mid-range include

- Ability to adjust and use stoneware clay bodies to this range
- In turn, mid-range stoneware bodies increase the durability of the ware

- Mid-range glazes are also more durable than those fired at lower temperatures, and
- There is still a fairly extensive color range available

2.14.4 High Fire

- From cone 8 (approx. 2305°F - 1260°C)
- To cone 14 (approx. 2530°F - 1390°C)

This range includes the stoneware's and porcelains. Glazes and clay bodies are dense and durable; however, the color range is limited. Because of the varying effects of oxidation and reduction on glaze colorants, the few coloring oxides that are viable at this range can still produce a rich, if much more limited, palette (Peterson, 2011).

2.15 Glaze Type

The earliest glaze dating back to 3,000BC, or earlier were found in Egypt. The discovery of the application of glazes on pottery ware in Egypt resulted accidentally from the melting of potash and sand deposits under bon fire. The glaze is named after the place where it has been formed, that is Egyptian glaze (Alasa, 2005).

The three constituent materials of glaze are silica, flux and alumina. Silica commonly called flint is the essential glaze ingredient. It is also known as quartz in its pure, crystalline state. Flux is a term applied to those compounds that lower the melting temperature of a glaze. Alumina helps to form a stronger glaze that will better withstand the wear and tear of normal everyday use (Ajala and Munai, 2007).

Salt Glaze, is composed from the common salt sodium chloride. There are also Slip Glaze, Ash glaze, low melting glaze, the lead glaze and so on. Glaze composition should be taught to the students of Colleges of Education early enough so that they can gain the necessary experiences before they graduate. The glaze types should be made

readily available in the stores for demonstration and practice in the colleges of education. The lecturers concerned should strongly advise the students on how to handle the glazes because some are toxic (Alasa, 2005).

Glazes are virtually ground glasses, being based upon mixtures of silica and fluxes. The nature of which depends on a number of factors but which allows the glaze to melt at a certain temperature range from 950°C and above. At all lower part of the range, the glaze normally contains manufactured fluxes. It is known as frits which will be either without lead or will contain lead, although in the later event the lead content must be chemically combined with frit usually in the form of a lead bisilicate or lead sequisilicate (Gonah et. al., 2003).

Pottery glazes are made to melt but to be static on the surface of the wares unto which they are applied, even when fired and have fused. Pottery glazes are glass in themselves (alumina). Alumina introduced to a glass batch increases viscosity making it suitable for glazing ceramic wares. Glazing is the best form of ceramic decoration (Datiri and Gukas, 2001).

A functional and attractive glaze can be made from only a few materials. However, several glazes use rather uncommon ingredients or require special firing techniques (Nelson, 1971) Glazes are either group according to the temperature at which they mature or according to the materials involved in the composition (Pius, 2008). The necessary ceramic glaze elements required for glazes formulation need to be in place for the procession of glazes. These necessary elements include silica, flux and a refractory among others to be able to form different type of glazes at various temperatures depending on the potter's intention or the purpose for which it is meant for.

Some of these glazes include Earthenware glazes, Stoneware glazes, Porcelain glaze, Lead glazes, Alkaline glazes, Ash glazes, Feldspathic glazes, Fritted glazes and Salt glazes (Pius, 2008).

2.16 Glaze Composition with Regard to Glaze properties

There are no fixed properties for glazes as their composition and use is so varied. The application of glaze may be purely for aesthetic reasons, functional purposes or both. One of the most common functional reasons is to provide an impermeable barrier to an otherwise porous ceramic body, as is the case with the common tea or coffee cup, while at the same time providing a visually pleasing surface to the article.

The following properties are considered of importance, depending on the application and product/process types.

- Thermal expansion, colour, durability and maturing range. Thermal expansion: Glazes are normally required to be in compression in the fired state to avoid the fault of crazing, a differential of between 0.02% and 0.04% at around 550°C is normally recommended. Excessive compression can lead to the fault known as peeling.

The colours available will depend upon the firing temperature, the chemistry of the base glaze, and the composition of the chosen colour. Durability: Depending on the application, chemical and physical durability may be importance. Various tests are employed for the different product types of assess durability. Maturing range: Glazes most mature within a appropriate temperature and time interval. There must also be a suitable range over which maturity is reached in order to allow for process variables. This dictates the choice of glaze and the materials used to produce the glaze batch for a particular product (http://www.azom.com/article.aspx?)

Glazes are glasses and hence their most indispensable constituents are glass – forming oxides seconded by network modifying and intermediate oxides. The adjustment of the numerous possible glaze constituents to suit particular purposes must be considered.

2.17 Desired Properties of Glazes

1) Fusibilities Must be such that maximum liquid glass is formed at the desired maturing temperature.
2) Viscosity Should be moderate at peak firing temperature so that surfaces even out but no over-all flow occurs down inclined or vertical surfaces.
3) Surface tension Should be low to avoid crawling.
4) Volatilization Of glaze components during firing should be minimized
5) Reaction with the body Should be moderate to give good fit without too much change in composition of either glaze or body.
6) Absorption into the body of glaze Constituents or eutellics formed during firing should not occur
7) Devitrification Should not occur in transparent glassy glazes
8) Expansion coefficient and young's modulus of elasticity Should relate to those of the body in such a way that maximum strength is achieved.
9) Homogeneity, smoothness and hardness To resist abrasion, scratching, etc.
10) Chemical durability
11) Colour for aesthetic or thermal reasons
12) Electrical properties, e.g. low power factor (Singer and Singer, 1971).

2.18 Experimenting on Test Tiles

Experiment freely with these basic glazes. Beginning students try 10 percents additions of every material they can think of: wood ash, local clay (right from the nearest bare spot of earth), pulverized soft – drink bottles, marble dust, slag from nearby foundries as well as the usual ceramic raw materials and commercial glaze stains. Fire these experiments on small, white, unglazed floor tiles from a building supply company

as well as on test tiles made. Pre-test the commercial tiles first to see how they work in the kiln.

To make test tiles, make a tablet of clay as done for slab pots. Let the tablet harden to leather – hard stage; then, cut it in two-inch squares. With the tip of a knife, flat side down, cover – third of the tile with a heavy texture. Bore a hole in the other end for stringing the tile) together so that they can be stored easily for later reference.

Allow the tiles to dry and bisque-fire them before making tests. Apply the experiment with a fully-loaded soft brush, making sure that it covers some of the texture, but does not run off the sides of the tile. On the back of each tile use cobalt – water solution and a fine brush to describe the test. Fire these tiles test – side up, on a kiln shelf that is well – coated with wash. (Note: Kiln wash is a 50-50 combination of kaolin and silica mixed with water to a smooth, brush-able paste).

The white tiles will allow glaze test to be observed easily in a relatively pure form. The tiles made will given an indication of how the glaze reacts with the clay, and can collectively provide a practical sample testing of glazes, however, will be small pots. Save and bisque more successful early throwing exercise for this purpose. The glazes given should be fired at about 2225°C. They were developed to be used on basic clay body which also matures at about this temperature, (Counts, 1974).

2.19 Developing an original Glaze

After experimenting with known glazes, one may wish to develop an "original" glaze.

1) Pick a firing temperature range
2) Decide on the character of the glaze (shiny, matt, blue, etc)
3) Select materials from the chart which one expect to provide that character
4) Mix sample batches and fire at a chosen temperature

5) Analyze the results
6) Make additional tests based on the analysis
7) Repeat the experimentation until obtained the desired glaze.

Simple ingredients can produce effective glazes for potters who through trial-and-error experimentation have determined which materials and how much of each to use. A scientifically – trained ceramist understands that the clay in the "creek settings", and the oxides from the ashes, provide the silica, alumina, and fluxes necessary for glaze formation.

Examine the given glaze formulas, starting with silica use from .02 percent to 25 percent in the pure form, although clays (kaolin and ball clay and feldspar add silica as well as alumina).

Lead has traditionally been the most used flux for low-fired glazes. Because of lead poisoning danger, however, it is best to choose one of the other fluxes, such as borax, for low-temperature firing. Lead does still have important assets, and careful control in firing yields its positive potential.

Fluxes are present in feldspars, predominant flux often giving its name to the compound – potash spars, for example or soda spar. Alumina is also present in feldspars. Because spars already contain so many of the requirements for glaze making, it would be logical to start by choosing a feldspar if you are making a glaze for stoneware, most glazes call for nepheline syenite because it is certain to begin to melt earlier than some other feldspars. Though nepheline syenite is heavy in sodium, but also contains potassium, as well as silica and alumina.

Whiting (calcium oxide) is used for its fluxing action in most stoneware glazes. Zinc and Barium are also fluxes used in glazes, but these materials perform other functions as well. They are matting an agent, which means that they cause opacity in a

glaze. Lithium is a powerful flux, though expensive if used only to cause material fusion, and is used in turquoise matt to aid in the production of a beautiful copper – blue color.

In these glazes, kaolin and ball clay are used as stabilizers. Do not get discouraged when results seem ridiculous or even disastrous. Try to decide why they are as they are and go on from there. Remember the kiln and its heat do miracles for the experimenting (Counts, 1974).

2.20 Keeping a Log

One can get a special notebook for the purpose of keeping a log or record each time firing is done. Note the date, firing time, the weather conditions, the kind of wares that were fired and the results (Count, 1974).

CHAPTER THREE

MATERIALS AND METHODS

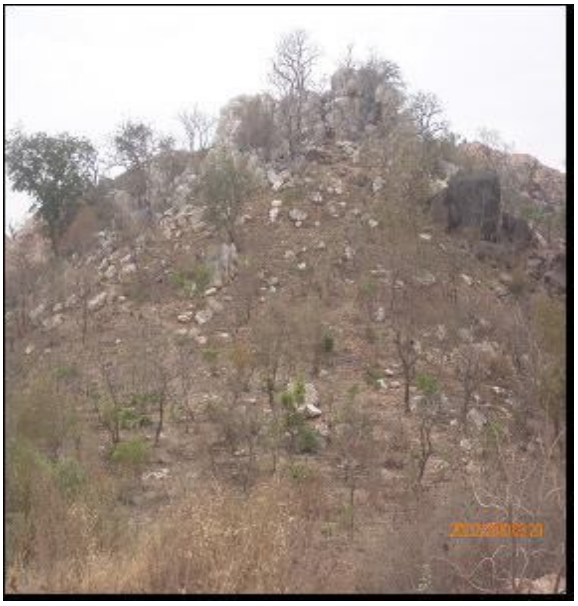
Research is a specific pattern of structuring investigation aimed at identifying variables and their relationships (Asika in Sullayman, 2006). It is used for the purpose of obtaining data to enable the researcher test hypothesis or answer research questions. It is an outline scheme, arrangement or order suitable for a research based on the study parameters or nature of materials or research topic under investigation (Sullayman, 2006). The suitable method for this study therefore is "experimental procedures" among others in the studio/ laboratories.

3.1 Sourcing of Materials

The raw materials, Quartz, feldspar, ball-clays and kaolin have been surveyed collected from the localities of North Eastern States of Nigeria (see plates I & II) and transported to Ahmadu Bello University, Zaria, Department of Industrial Design, Ceramic section studio where they were processed for the glaze formulation.

Quartz and feldspar were obtained from Liga Hill or Dutsen Tangaram in Gwoza Local Government Area of Borno State. The kaolin was obtained from Bauchi, Bauchi State. The ball-clay was obtained from Gadabul which is a small stream located near Madaganari ward of Maiduguri, Borno State. Information on this was obtained from the local potters in the localities of the study, and from staff/lecturers of Geology Department, University of Maiduguri, Borno State of Nigeria were consulted for professional inputs and advice. The raw materials have been obtained from the following locations (see plate III & IV):

Small samples of the materials for the study were sent to the Department of Geology, University of Maiduguri for chemical analysis where the elemental properties were obtained for the glaze compositions.




Plates I & II: Location (site) of the raw materials in Lokodisa village Gowza Town, Borno State, Nigeria




Plates III & IV: Collection of Quartz from the Site

3.2 Beneficiation of Materials

The raw materials feldspar and quartz were washed and dried for two days: After they were dried, they were measured and packed into the kiln for calcination (see plate V, VI, VII).





Plate V & VI: Materials weighing in the ceramic studio of Ahmadu Bello University, Zaria



Plate VII: Packing of materials into kiln for calcination

3.3 Calcinations/Pre-treatment of samples

3.3.1 Feldspar

The colour of the feldspar was white – pink. They were obtained from Liga hill (Dutsen Tangaram) of Gowza local government area of Borno State. They were loaded into the kiln and were calcined in the kerosene fired kiln at 900°C. There were cracks on the surface after calcinations (see plate VIII). The bigger pieces were broken into smaller sizes with the use of metal hammer and jaw crushed into smaller units before ball milling, hand gloves and nose mask were worn for protection against cuts and inhalation of the dust (see plates IX).

It took 4 hours for ball milling. The ratio of feldspar to water was 1:3 in the ball mill. After ball milling it was poured into a big plastic bin and allowed to settle for three days before the excess water was removed and then dried for two days, sieved with 120 mesh and then stored.



Plate VIII: Calcined materials in the kiln



Plate IX: Crushed material (Feldspar)

3.3.2 Quartz

Quartz was pure white, collected from Liga Hill (Dutsen Tangaram) of Gowza Local Government Area of Borno State. They were calcined at 1000°C and after the calcinations, they were broken into chips with the use of metal hammer and then crushed using a jaw crusher (see plate X). The crushed quartz was then bal- milled.



Plate X: Crushed material (Quartz)

The ball milling lasted for six (6) hours. It was then poured into a plastic container and kept to settle for three days. The excess water was removed, dried on a slab for 3 days and sieved (see plates XI, XII).



Plate XI: Loading of material (Crushed) quartz for ball milling. At the Ceramic section, Department of Industrial Design. Ahmadu Bello University, Zaria.




Plates XII: Drying process of the materials

3.3.3 Kaolin

The type of kaolin obtained from Bauchi was white in color. The kaolin was soaked for twenty-four hours (24 hrs). It was stirred, mixed homogeneously and was sieved through a 60 mesh sieve. It was allowed to settle for two days, the excess water was removed, poured into a plaster mould for twenty four hours (24hrs) and when hardened it was dried on a flat slab.

3.3.4 Ball clay

The colour of this plastic material is gray. The clay was brought from Gadabul in Maiduguri of Borno State. The clay was soaked for two (2) days. It was mixed thoroughly, sieved in a 60 mesh sieve. It was allowed to settle for five (5) days, poured into a plaster mould for two days, when hardened it was dried on a flat slab.

3.4 Chemical Analysis of Raw Materials

The raw materials obtained were analyzed by means of x-ray fluorescence method at the Department of Geology, University of Maiduguri, Borno State, Nigeria. A total of 9

blends were produced for the purpose of testing the glaze batches formulated, and were fired at a temperature range of 1200^{o}C – 1260^{o}C. Those blends are:

- 2 types of quadiaxial blends of 36
- 1 quadiaxial blend of 121
- 1 quadiaxial blend of 25
- 1 triaxial blend of 36
- 1 triaxial blend of 28
- 2 types triaxial blend of 21
- 1 traxial blend of 66

3.5 BATCH CALCULATION FOR THE GLAZE MAKING

TABLE 3.1a: QUADAXIAL TEST BLEND (36) OF MATERIALS OF THE STUDY IN PERCENTAGES (%)

1 F Feldspar 100	2 F = 80% Q = 20%	3 F = 60% Q = 40%	4 F = 40% Q = 60%	5 F = 20% Q = 80%	6 Quartz 100%
7 F80% K=20%	8 F=64% Q=16% K=16 % BC=4%	9 F=48% Q=32% K=12 % BC=8%	10 F=32% Q=48% K=8 % BC=12%	11 F=16% Q=64% K=4 % BC=16%	12 Q=80% BC=20%
13 F=60% K=40 %	14 F=48% Q=12% K=32 % BC=8%	15 F=36% Q=24% K=24 % BC=16%	16 F=24% Q=36% K=16 % BC=24%	17 F=12% Q=48% K=8 % BC=32%	18 Q=60% BC=40%
19 F=40% K=60 %	20 F=32% Q=8% K=48 % BC=12%	21 F=24% Q=16% K=36 % BC=24%	22 F=16% Q=24% K=24 % BC=36%	23 F=8% Q=32% K=12 % BC=48%	24 Q=40% BC=60%
25 F=20% K=80 %	26 F=16% Q=4% K=64 % BC=16%	27 F=12% Q=8% K=48 % BC=32%	28 F=8% Q=12% K=32 % BC=48%	29 F=4% Q=16% K=16 % BC=64%	30 Q=20% BC=80%
31 K Kaolin 100 %	32 K=80 % BC=20%	33 K=60 % BC=60%	34 K=60 % BC=40%	35 K=20 % BC=80%	36 BC Ball-Clay 100 %

Source: Riegger in Manzuche (2006)

Batches of materials for glaze formulation were made based on unity formula (Riegger in Manzuche 2006)

3.5.1 Formulation of the Glaze Batches

Three quadrilateral blends and five triaxial blends were formulated as shown in tables 3.4a -3.4h. Individual glaze batch was made for these blends and tested on test slabs.

TABLE 3.1b: QUADAXIAL TEST BLEND OF 36 (A)

1F Feldspar 10g	2 F = 8g Q = 2g	3 F = 6g Q = 4g	4 F = 4g Q = 6g	5 F = 2g Q = 8g	6 Quartz 10g
7 F = 8g K= 2g	8 F = 6.4g Q = 1.6g K = 1.6g BC = 0.4g	9 F = 4.8g Q = 3.2g K = 1.2g BC = 0.8g	10 F = 3.2g Q = 4.8g K = 0.8g BC =1.2g	11 F = 1.6g Q = 6.4g K = 0.4g BC =1.6g	12 Q = 8g BC = 2g
13 F = 6g K = 4g	14 F = 4.8g Q = 1.2g K = 3.2g BC =0.8g	15 F = 3.6g Q = 2.4g K = 2,4g BC =1.6g	16 F = 2.4g Q = 2.6g K = 1.6g BC =2.4g	17 F = 1.2g Q = 4.8g K = 0.8g BC =3.2g	18 Q = 6g BC =4g
19 F = 4g K = 6g	20 F = 3.2g Q = 0.8g K = 4.8g BC =1.2g	21 F = 2.4g Q = 1.6g K = 3.6g BC =2.4g	22 F = 1.6g Q = 2.4g K = 2.4g BC =3.6g	23 F = 0.8g Q = 3.2g K = 1.2g BC =48g	24 Q = 4g BC =6g
25 F = 2g K = 8g	26 F = 1.6g Q = 0.4g K = 6.4g BC =1.6g	27 F = 1.2g Q = 0.8g K = 4.8g BC =3.2g	28 F = 0.8g Q = 1.2g K = 3 .2g BC =4.8g	29 F = 0.4g Q = 1.6g K = 1.6g BC =6.4g	30 Q = 2g BC =8g
31 Kaolin 10 %	32 K = 8g BC = 2g	33 K = 6g BC =6g	34 K = 6g BC =4g	35 K = 2g BC =8g	36 BC Ball-Clay 10g

Source: Riegger in Manzuche (2006)

TABLE 3.2: QUADAXIAL TEST BLEND OF 121 (B)

A	=	Ball Clay	Standard
B	=	Feldspar	121 – tile square blend
C	=	Kaolin	Calculations
D	=	Quartz	

1 A 100	2 A 90 B10	3 A 80 B 20	4 A 70 B30	5 A60 B40	6 A 50 B 50	7 A 40 B 60	8 A 30 B 70	9 A 20 B 80	10 A 10 B 90	11 B 100
12 A 90 B 0 C 10 D0	13 A 81 B 9 C 9 D 1	14 A 72 B 18 C 8 D 2	15 A 63 B 27 C 7 D 3	16 A 54 B 36 C 6 D 4	17 A 55 B 45 C 5 D 5	18 A 36 B 54 C 4 D 6	19 A 37 B 63 C 3 D 7	20 A 18 B 72 C 2 D 8	21 A 9 B 81 C 1 D 9	22 B 90 D 10
23 A 80 B 0 C 20 D 0	24 A 72 B 8 C 18 D 2	25 A 64 B 16 C 16 D 4	26 A 56 B 24 C 14 D 6	27 A 48 B 32 C 12 D 6	28 A 40 B40 C 10 D 10	29 A 32 B 48 C 8 D 12	30 A 24 B 56 C 6 D 14	31 A 16 B 64 C 4 D 16	32 A 8 B 72 C 2 D 18	33 B 80 D 20
34 A 70 C 30	35 A 63 B 7 C 27 D 3	36 A 56 B 14 C 24 D 6	37 A 49 B 21 C 21 D 9	38 A 42 B 28 C 18 D 12	39 A 35 B 35 C 15 D 15	40 A 28 B 42 C 12 D 18	41 A 21 B 49 C 9 D 21	42 A 14 B 56 C 6 D 24	43 A 7 B 63 C 3 D 27	44 B 70 D 30
45 A 60 C 40	46 A 54 B 6 C 36 D 4	47 A 48 B 12 C 32 D 8	48 A 42 B 18 C 28 D 12	49 A 36 B 24 C 24 D 16	50 A 30 B 30 C 20 D 20	51 A 24 B 36 C 16 D 24	52 A 18 B 42 C 12 D 28	53 A 12 B 48 C 8 D 32	54 A 6 B 54 C 4 D 36	55 B 60 D 40
56 A 50 C 50	57 A 45 B 5 C 45 D 5	58 A 40 B 10 C 40 D 10	59 A 35 B 15 C 35 D 15	60 A 30 B 20 C 30 D 20	61 A 25 B 25 C 25 D 25	62 A 20 B 30 C 20 D 30	63 A 15 B 35 C 15 D 35	64 A 10 B 40 C 10 D 40	65 A 5 B 45 C 5 D 45	66 B 50 D 50
67 A 40 C 60	68 A 36 B 4 C 54 D 6	69 A 32 B 4 C 52 D 12	70 A 28 B 12 C 42 D 18	71 A 24 B 16 C 36 D24	72 A 20 B 20 C 30 D 30	73 A 16 B 24 C 24 D 36	74 A 1 B 28 C 18 D 42	75 A 18 B 32 C 12 D 48	76 A 4 B 36 C 6 D 54	77 B 40 D 60
78 A 30 C 70	79 A 27 B 3 C 63 D 7	80 A 24 B 6 C 56 D 14	81 A 21 B 9 C 49 D 21	82 A 18 B 12 C 42 D 28	83 A 15 B 15 C 35 D 35	84 A 12 B 18 C 28 D 42	85 A 9 B 21 C 21 D 49	86 A 6 B 24 C 14 D 56	87 A 3 B 27 C7 D 63	88 B 30 D 70
89 A 20 C 80	90 A 18 B 2 C 72 D 8	91 A 16 B 14 C 64 D 16	92 A 14 B 16 C 56 D 24	93 A 12 B 8 C 48 D 32	94 A 10 B 10 C 40 D 40	95 A 8 B 12 C 32 D 48	96 A 6 B 14 C 16 D 64	97 A 4 B 18 C 8 D 72	98 A 2 B 18 C 8 D 72	99 B 20 D 80
100 A 10 C 90	101 A 9 B 1 C 81 D 9	102 A 8 B 2 C 72 D 18	103 A 7 B 3 C 63 D 27	104 A 6 B 4 C 54 D 36	105 A 5 B 5 C 45 D 45	106 A 4 B 6 C 36 D 54	107 A 3 B 7 C 27 D 63	108 A 2 B 8 C 18 D 72	109 A 1 B 9 C 9 D 81	110 B10 D 90
111 C 100	112 C 90 D 10	113 C 80 D 20	114 C 70 D 30	115 C 60 D 40	116 C 50 D 50	117 C 40 D 60	118 C 30 D 70	119 C 20 D 80	120 C 10 D 90	121 D 100

Source Daly 2013

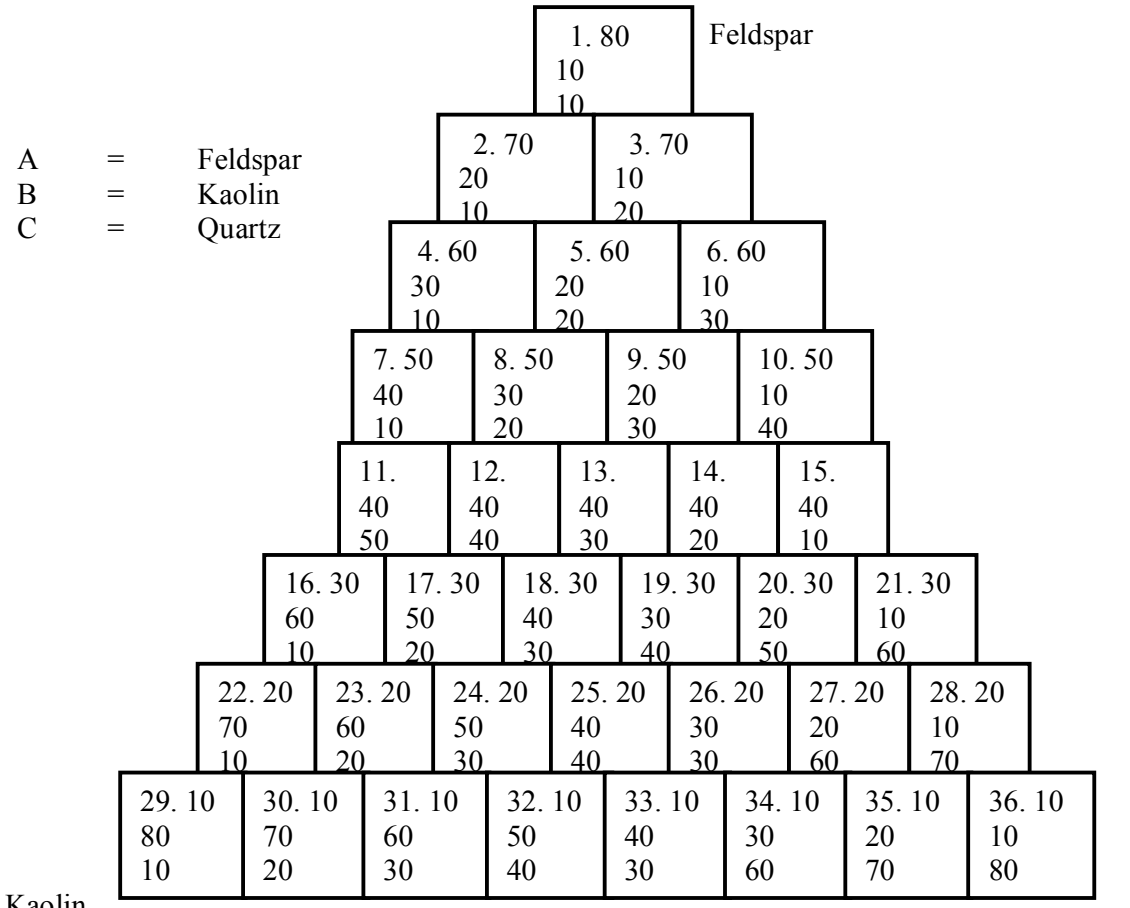
TABLE 3.3: QUADAXIAL TEST BLEND OF 25 (C)

A	=	Ball Clay	Standard
B	=	Feldspar	25 – tile square blend
C	=	Kaolin	Calculations
D	=	Quartz	

1.	A 100	2.	A 75 B 25	3.	A 50 B 50	4.	A 25 B 75	5.	B 100
6.	A 75 C 25	7.	A 56 B 19 C 19 D 6	8.	A 37.5 B 37.5 C 12.5 D 12.5	9.	A 19 B 56 C 6 D 19	10.	B 75 D 25
11.	A 50 C 50	12.	A 37.5 B 12.5 C 37.5 D 12.5	13.	A 25 B 25 C 25 D 25	14.	A 12.5 B 37.5 C 12.5 D 37.5	15.	B 50 D 50
16.	A 25 C 75	17.	A 19 B 6 C 56 D 19	18.	A 12.5 B 12.5 C 37.5 D 37.5	19.	A 6 B 19 C 19 D 56	20.	B 25 D 75
21.	C 100	22.	C 75 D 25	23.	C 50 D 50	24.	C 25 D 75	25.	D 100

Source: Daly 2013

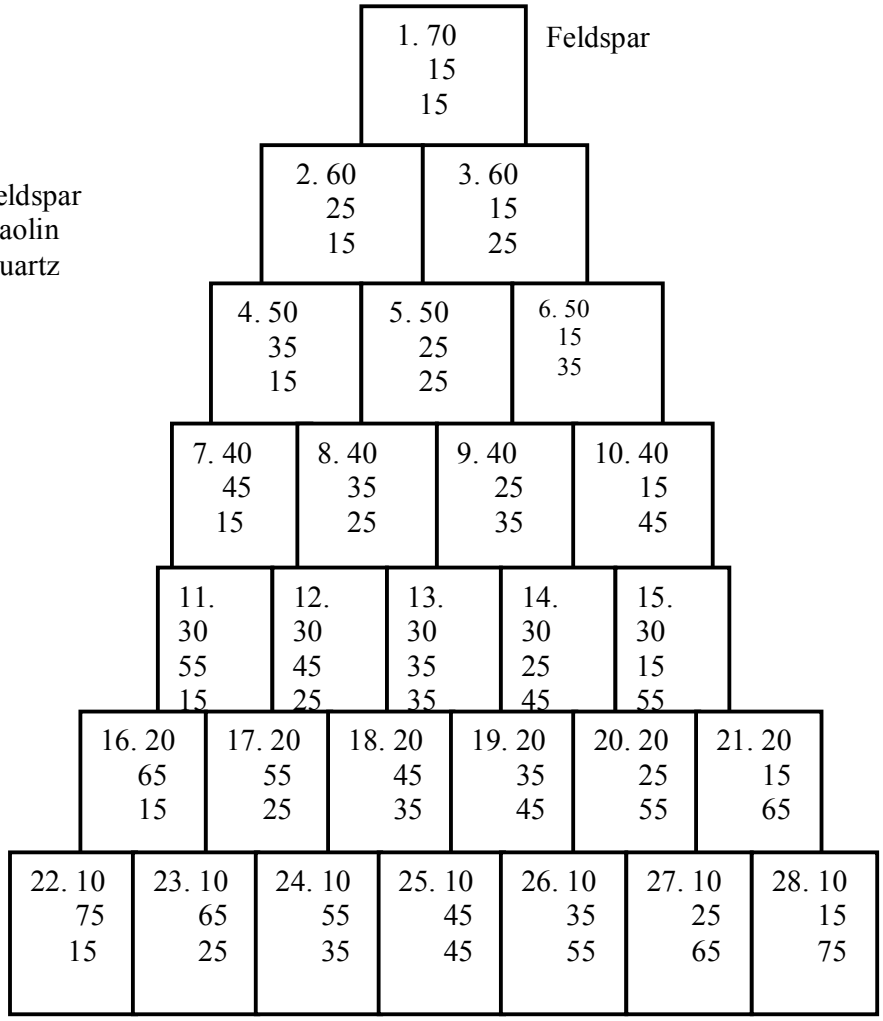
TABLE 3.4: TRIAXIAL TEST BLEND OF 36 (D)



Source: Okoruwa 1986 (Trixial Blend of 36)

TABLE 3.5: TRIAXIAL TEST BLEND OF 28 (E)

A = Feldspar
B = Kaolin
C = Quartz



Source: Okoruwa 1986 (28 Traxial Blend Calculations)

TABLE 3.6a: TRIAXIAL TEST BLEND OF 21 (fi)

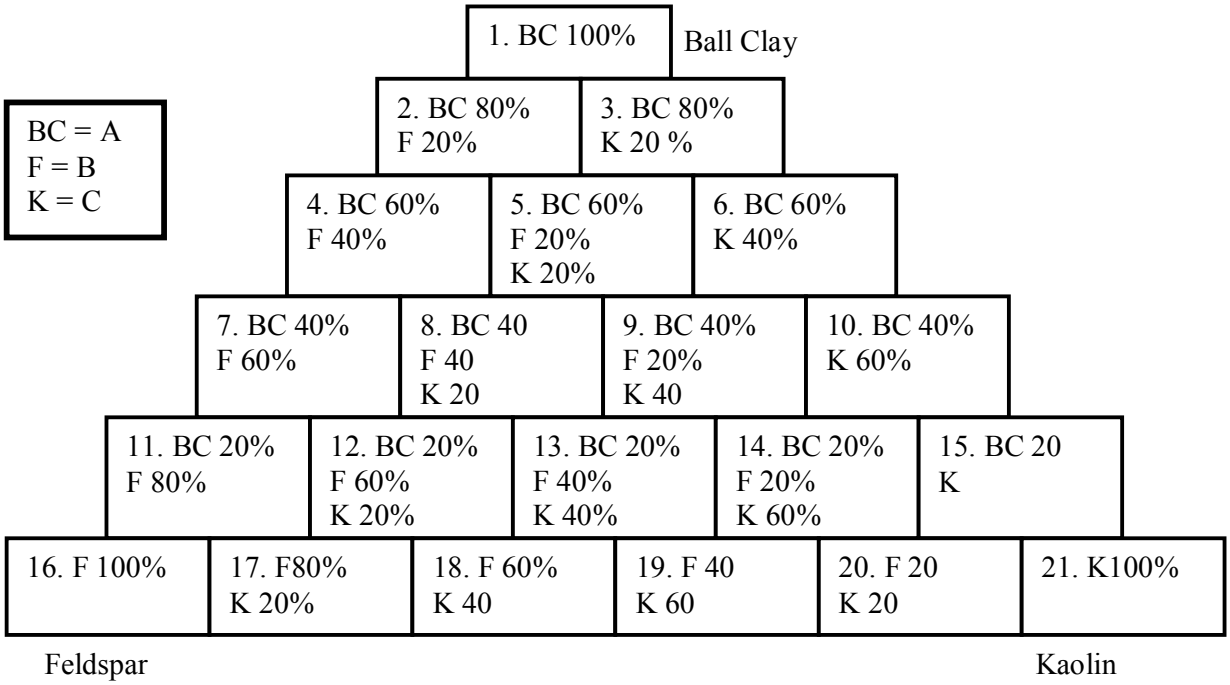


TABLE 3.6b: TRIAXIAL TEST BLEND OF 21 (fii)

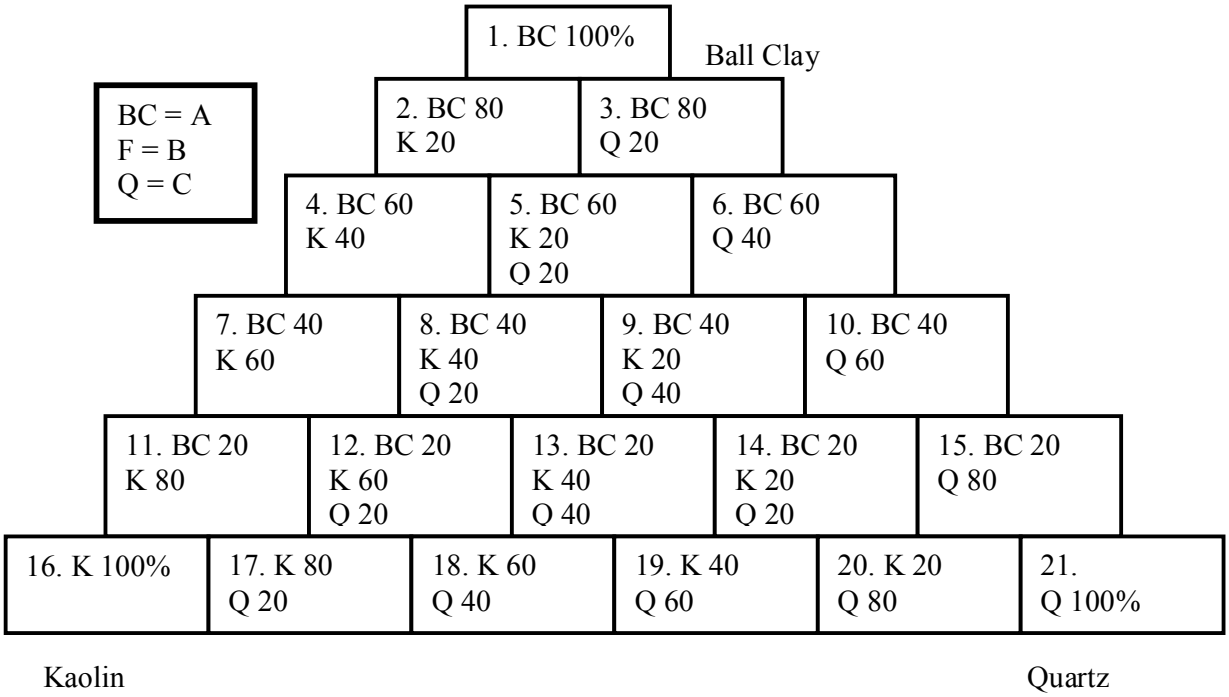
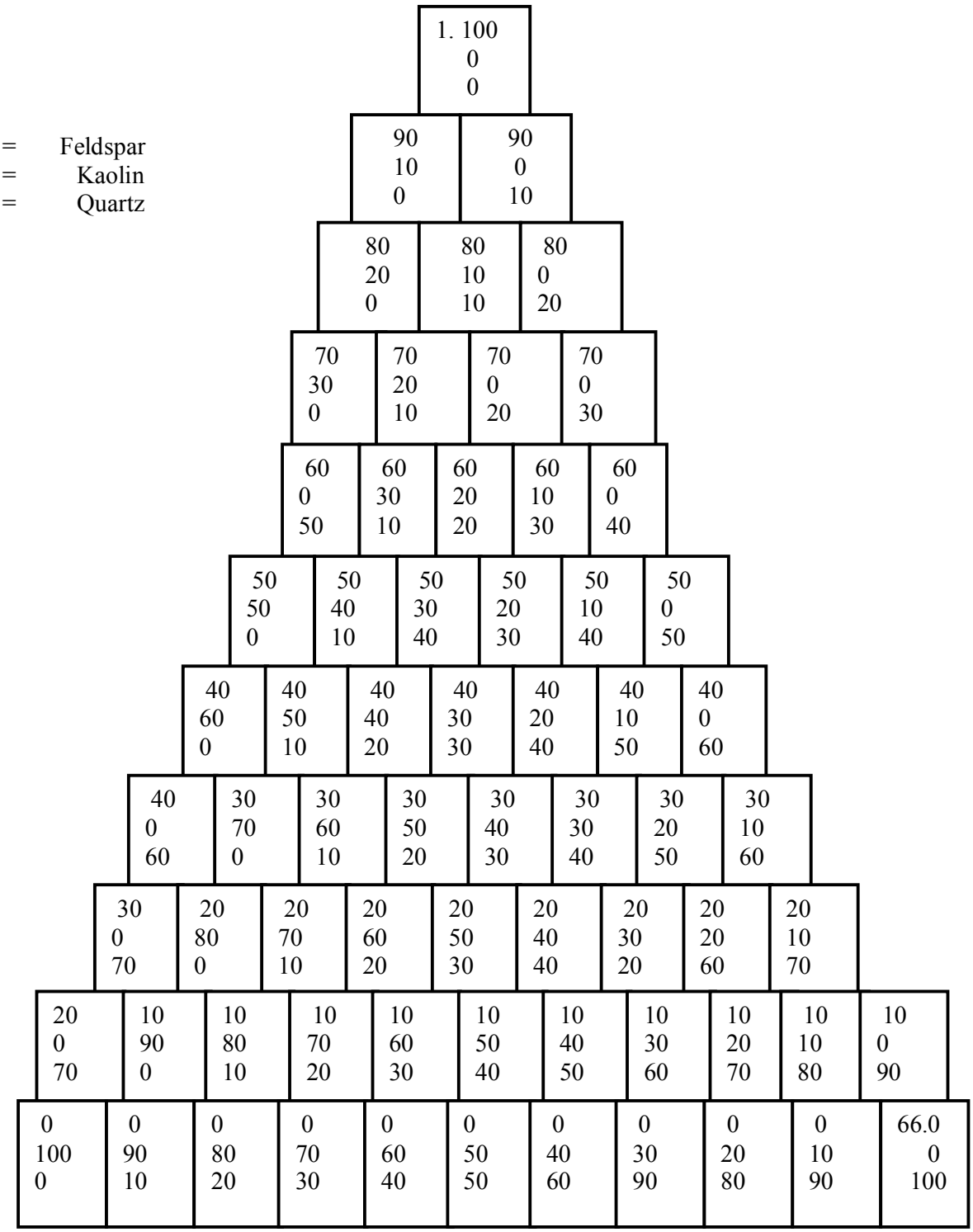


TABLE 3.7: TRIAXIAL TEST BLEND OF 66 (G)

Feldspar

A = Feldspar
B = Kaolin
C = Quartz



Kaolin

Source: Okoruwa, 1986 (Traxial 66)

Quartz

3.6 DETERMINATION OF SUITABLE FIRING TEMPERATURE

Test tiles were made for testing the firing temperature of the glazes. These tiles were glazed and fired at the temperature range of 1200°C, 1250°C and 1260°C.

3.7 PROPERTY TESTING

3. 7.1 Determination of Vitreous and Crazing

To determine the vitreous and crazing ASTM C554 was employed as thus:

3.7.1.1 Vitreousness

Vitreousnes was determined by visible inspection of the glazed specimens.

3.7.1.2 Crazing

The crazing test was carried out by observation of the glazed specimens. The samples were inspected with oblique lighting and application of a dye. This was done by the application of ink on the specimen (test tiles) and cleaned off immediately with clean towel and observation made for fine lines.

3.7.2 The Imperviousness

The imperviousness was tested by application of water on the sample for 10 minutes. Was made and recorded.

CHAPTER FOUR

RESULTS AND DISCUSSION

In this chapter, results of all tests conducted and treatment rendered to the materials of the study at various stages and their discussion are handled. The items in the chapter include results of batches of good glazes and the property tests. The chemical analyses of the materials are also included.

4.1 Result of the Chemical Analysis of the Samples

Table 4.1 Result of the Chemical Analysis of the Samples, by means of X-ray fluorescence method

Parameter	Quartz	Feldspar	Ball Clay	Kaolin
SiO_2	92.40	7.48	69.20	45.73
Al_2O_3	1.470	28.90	21.88	34.50
Fe_2O_3	0.513	2.65	0.310	1.52
MgO	0.162	3.66	0.05	0.64
CaO	0.447	16.36	0.66	0.32
Na_2O	0.532	37.42	0.32	0.05
MnO	0.033	1.50	0.00	0.00
TiO_2	0.017	0.81	0.56	3.74
SO_4	0.563	0.62	0.05	0.075
H_2O^+	0.052	5.47	8.53	12.61

4.2. Result of Test Melting of the Glaze Batches

4.2.1 Test Melting Results of Quadaxial Test Blend of 36 (A) Fired at 1200°C

The Glaze samples 1, 7, 8, 10, 14 16, 20 and 29 fused to the tiles but they were not completely melted. They also had no cracks. Samples 2, 3,4,5,6, 11, 12, 13, 17, 18, and 23 did commenced fusing and did not fuse completely to the tiles. Samples 9, 21 and 22 were fused to the tiles. They had slight cracks. Samples 19, 24, 25, 26, 27, 28, 30, 31, 32, 33, 34, 35 and 36 all fused, they all also had cracks and did not adhere to the tiles. Samples 1, 2, 3, 7, 8 and12 fused and were glossy. Sample 1 proves to be the most glossy which shows the maturity of gloss firing and glaze melting.



Plate XIII: Test Melt Result of Tiles 1-20 fired at 1200°C



Plate XIV: Test Melt Result Of Tiles 21-36 Fused at 1200°C

4.2.2 Test Melting Results of Quadaxial Test Blend of 36 (A) Fired at 1250°C

Glaze compositions in samples 1 and 12 fused to the tiles and gave glossy/ shiny appearance. Samples 2 and 3 fused to the tiles, but they were matty and showed some cracks. Samples 4 and 7-17 fused to the tiles slightly, but they exhibited crack lines on them. Samples 5, 6, and 18 to 36 did not fuse to the tiles, they had cracks and had no shiny appearance.



Plate XV: Test Melt Result of Tiles 1-36 Fused at 1250^{o}C

4.2.3 Test Melting Result of Quadaxial Test Blend of 121(B) Fired at 1260^{o}C

Samples 1, 2, 12, 13, 14, 23, 24, 34 and 45 are of similar characteristics of the test of this study. They had shattered and peeled off from the tile surfaces, while some partially got stuck to the tiles. Their colours are brown and dark brown on the double dipped sides. Samples 3 and 4 are alike. Though Sample 3's cracks are major while sample 4's are minor both on the double dipped sides. Sample 5 has oily surface. It has more cracks on the double dipped side and colour is black. On the single dipped side, it swelled and was about to open at the centre but cracked. The surface is not smooth, while its colour is brown.

Sample 6 has similar characteristics as Sample 5 in some cases. Sample 6 is shinny and has spotty effect on the double dipped side. The colour is deep brown. Samples 7, 8, 9, 11, 19 and 20 are in the same zone of characteristics. They are all very smooth, glossy shinny and the glazes stuck well to their tiles. Samples 7, 8 and 19 had bubbles on the double dipped side.

Samples 15, 16, 17 and 18 of blends 121 are of similar behavior in terms of sticky to their tiles, of which they have, surface are not smooth and shinny. Sample 18 has an exceptional quality of bubbles /fumes on the double dipped side.

Their colours are also the same which are dark brown. Samples 21and 22 are the same in character. They have stuck to their tiles, smooth and shinny, though 21 on the single dipped sides is not as smooth as on the double side and Sample 22 on both sides. Samples 25, 26, 27, 28, 29, 30, 31, 32 and 33 have stuck to their tiles.

Sample 25 have minor cracks on the double dipped side. The surface is not smooth on both sides. Sample 26 have major cracks, though cracks on the double dipped side are bolder than ones on the single dipped side. Sample 27 have swollen up at the centre of the tiles and have cracks on it at the centre. Sample 28 is meek on the tile with rough surface. Sample 29 has swollen on it. Smooth surface on the double dipped side and shinny with bubbles. While on the single dipped side is not as smooth as on the double dipped side of the tile. Sample 30 is smooth and shinny with spotty effects on both sides and of the tiles.

Sample 31 also swollen on the centre smooth and shinny on the double dipped side than on the single dipped side. It has swollen on the centre of the single dipped side. Samples 32 and 33 are both smooth and shinny and only that Sample 33 have bubbles on the double dipped side. In terms of colours, samples 25, 26, 27, 28, 29 and 30 are brown in colour, only that the single dipped side of the 29 is ash in colour as sample 27. Sample 31 and 32 are light brown while 33 is ash in colour.

In the case of Samples 35, 36, 37, 38, 39, 40, 41, 42, 43 and 44, they have all sticked to their tiles. Samples 35, 36 and 37 have cracks on the double dipped side. Samples 38, 39 and 40 have sintered but not smooth nor shinny on the surface in appearance as well as on physical. Samples 41, 42 and 43 have bubbles on the double

dipped sides. Sample 44 has more bubbles on the single dipped side than the double dipped side. In terms of colours, Samples 35, 36 and 37 and 40 are brown. Sample 38 are black. Samples 41, 42 and 43 are light pink, while Sample 44 is glassy ash in colour.

Samples 46, 47, 48, 49, 50, 51, 52, 53, 54 and 55 have stuck to their tiles. Sample 46 have minor cracks on the double dipped side. Samples 49, 50, 51, and 52 have the same smoothness on their surfaces. They are averagely smooth but not shinny. Samples 53 and 54 have smooth though with bubbles on them on the doubled dipped side and have even surface on the single dipped side. In terms of colouration, Samples 46, 4, 48 and 49 are brown. Samples 50 and 51 are dark grey. Sample 52 is brown on the dipped side and grey on the single dipped side. Samples 53 and 54 are grey and have pinkish ash on the double dipped side of Sample 53. Sample 55 on the other hand is black (Oily black).

Along the line, the samples of 56, 57, 58, 59 60, 61, 62, 63, 64, 65 and 66 have stuck to their ties with the exception of Sample 57 which have cave cracks as well as minor cracks on the double dipped side. Sample 56, 62, 63 and 64 have swollen on the centre, though the swollen of 64 is similar in size than those of 62 – 63.

Samples 58, 59 60, 61, 62, 63, 64, 65, 66, 67 and 68 have sintered calmly on their tiles. Their surfaces are averagely smooth, though not shinny. Samples 65 and 66 have spotty distanced glittering, though the double dipped side of Sample 66 is looking dull meek in appearance. In terms of colours, Samples 56, 57, 58, 59 60 and 61, are brown. Samples 62, 63, 64, 65 and 66 are grayish, though 66 look white-grey.

Similarly, Samples 67, 68, 69, 70, 71, 72, 73, 74, 75 76 and 77 have stuck to their tiles. With the exception of 77 which have peeled off on the double dipped side of the blend. Samples 67, 68, 69 and 70, 73, 74 and 75 have sintered mainly. Samples 69, 71 and 72 have swollen on the middle of the tiles. Sample 70 and 71 and 76 have cracks

(major). Their surfaces have appeared averagely meek smooth but not shinny. In terms of colours, Samples 67, 68, 69 and 70 are brown. 71, 72, 73, 74, 75 76 and 77 are grey.

On this axis so far, Samples 78, 79, 80, 81, 82, 83, 84, 85, 86, 87 and 88 have also stuck to their tiles. Samples 78 - 80, 83, 85 and 86 have major cracks. Samples 80 and 85 have swollen up. Sample 87 have peeled off on the double dipped side. Sample 88 have rocky solidified on the double dipped side. Samples 81 and 82 and 84 have meekly sintered. In terms of colours, Sample 78 is cream. Samples 79, 80, 81, 82 and 83 are brown. 84 and 86 are brownish-ash. While 85, 87 and 88 are ash in colour.

In Samples 89, 90, 91, 92, 93, 94, 95, 96, 97, 98 and 99, they have stuck to their tiles with major cracks and pilings. Samples 89, 91 and 92 94 have major cracks. The cracks on sample 89 look hanging on the double dipped side. Samples 94 and 99 have peeled off on the double dipped sides. There are also hanging pilings surface and have sintered. Concerning their colours, Samples 89, 90 and 91 are light brown. Sample 92, 93, 94, 95, 96, 97 and 98 are all pinkish-brown, while Sample 99 is light spotty-whitish.

On Samples 100, 101, 102, 103, 104, 105, 106, 107, 108, 109, 110 and 111 of this blends, have the following characteristic. Samples 100, 101, 104 and 106 have stuck to their tile. Samples 100, 102 and 103 have major cracks. Samples 105 and 109 have oiled on their double dipped sides. Samples 107, 108 and 110 have pilled of their tiles. Regarding their colours Samples 100 and 101 are light cream and 102, 103, 104, 105 and 106 are cream. Samples 107, 108 and 109 are light orange. Sample 110 is purple.

Samples 111, 112, 113, 115, 119 120 and 121 have pilled of their tiles. Samples 114 and 118 have major cracks on their double dipped sides. Sample 118 have swollen also on the double dipped sides. Number 117 have sintered on the tiles on both sides. Their colours are as follows:

Samples 111, 112, 113, 114 and115 are light cream. Samples 116 – 117 are cream. Samples 118 – 119 are light orange. Sample 120 is light-yellow. Sample 121 is purple which is the last of the blend and is the biggest Sample of the blends.



Plate XVI: Test Blend Result of Files 1-121 Fired at 1260^{o}C

4.2.4 Test Melting Result of Quadaxial Test Blend of 25(C) Fired at 1260^{o}C

These blends consist of four (4) materials of the study. These materials are Ball clay feldspar kaolin and quartz. They are indicated as A B C and D in the study. Their results of the test are as follows:

Samples 1, 2, 6 and 7 are similar categories in terms of colour. Sample 1 blend has shattered on the tile and partially stuck to the tiles. Sample 2 have minor cracks while Sample 6 has major cracks on the double dipped sides. Sample 7 though rough, but more rough on the double dipped side and has slight cracks.

Samples 3 and 4 have similar results in the test. They have both fused and stuck to the tiles. They are meek on the single dipped sides and bubble like on the double dipped

side. They are shinny and Sample 3 is not very smooth has Sample 4. Sample 4 is very smooth and is impervious. Their colours are chocolate brown.

Sample 5 is smooth, fused to the tiles and shinny glassy like in characters. On the single dipped side, the glassy light brown and on the double dipped side the colour is glassy, transparent and there are hidden crazing lines. Sample 8 has sintered and fused to the tile. It is not smooth. It has also solidified. The colour is dark ash. Sample 9 has sintered and fused to the tile. The surface is smooth and is smoother and shinny on the double dipped side of which the colour is brown. While on the single dipped side is not very smooth surface and the colour is spot shinny grey in colour. Sample 10 has sintered to the tile on the single dipped side. It has stuck on the double dipped side. It has formed glassy but solid gum like though smooth surface with colour grey. Unlike the single dipped side which is not smooth with colour water like on light ash.

Samples 11 and 12 are the same characteristics. They have stuck to their tiles. They are not smooth, have major cracks Sample 11, minor cracks on Sample 12. Their colours are brown.

Samples 13, 14 and 15 are of also of similar characteristics. They have sintered and stuck to their tiles. They are strong looking but Sample 5 has peeled off on the double dipped side. Their colours are grey.

Sample 16 has sintered to the tiles and has smooth surface. The colour is cream. Sample 17, 18 and 19 are of similar characteristics. They have sintered to their tiles on Sample 17, it has been demarcated by line at the edge of the double dipped side and the single dipped side with no crack. Sample 18 has minor cracks on the double dipped side. Sample 19 has 2 major cracks on the double dipped side and is like piling off the tile but did not. Their colours are cream-brown. Sample 20 has stuck to the tile and sintered surface not very smooth. The colour is grey.

Sample 21, 22 and 23 are of similar characteristics. They have sintered to their tiles, though Sample 21 have shown sign of pilling off but has not at the double dipped side of the tiles. Their colours are white.

Sample 24 has sintered unto the tile. The surface is eve but not smooth at the dingle dipped side. The colour is spotty cream at the double dipped side and spotty spotty-grey at the single dipped side.

Sample 25 which is the last Sample of the blends, has the following characteristics. It is still half way stuck to the tile and in the middle are powdered in form. The surface is smooth but piled and broken. The colour is pink.



Plate XVII: Test Blend Result of Files 25 Fired at 1260^{o}C

4.2.5 Test Melting Result of Triaxial Test Blend of 36 (D) Fired at 1260^{o}C

On these blends, Sample 1, 2 and 3 have their characteristics in common. The sticking to the tiles, fusion, sintering, smoothness and the colours are almost the same positively. The colours are ash. Only that the Sample 1 is shinny ash in colour.

Samples 4, 5, 6, 7, 8, 9 and 10 of these blends are also equally of the same characteristics as of 1 – 3. Only that they are not as smooth as the 1, 2 and 3 blends.

Samples 11 – 15, 20 – 21 and 26 are equally of the same characteristics with the difference of the smoothness. They are not as smooth and shinny as the previously mentioned above Sample. Also with major cracks on double dipped side of 21 made the difference.

Samples 17, 18, 19, 22, 23 24, 25, 27, 32, 33 and 34 are of the same family. They have sintered to the tiles smooth but not shinny and their colours are creamy. Sample 16 is different. It has stuck properly at the single dipped side, half ticked at the double dipped side and major crack on the double dipped side also. The colour is cream.

Sample 26 has sintered to the tiles and the surface is even but not very smooth. The colour is ash.

Sample 18 is has sintered at the single dipped side and has crack at the joint of the double dipped side. The colour is cream.

Sample 29, 30, 31 and 35 are of similar characteristics. They have sintered to the tiles smooth but not shinny. Their colours are white. Though Sample 30 has major cracks on the double dipped side.

Sample 36 has sintered to the tile and the colour is orange at the double dipped side light spotty orange at the single dipped side.



Plate XVIII: Test Blend Result of Files 1-36 Fired at 1260^{o}C

4.2.6 Test Melting Result of Triaxial Test Blend of 28 (E) Fired at 1260^{o}C

Samples 1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 11, 14, 15, and 20 of similar characteristics in these blends. They are not smooth and shinny, though they have sintered to their tiles. Their colours are ash.

Samples 16, 17, 18, 19 and 22, 23, 24, 25 and 26 of this lends are of similar characteristics. They have also sintered as the previous Samples to their tiles. They are calm in action. Their colours are white, although Sample 25 of the blends have major cracks on the double side dipped. Samples 27 and 28 are alike. They have sintered to their tiles and are neither smooth nor shinny. They are cream in colour.



Plate XIX: Test Blend Result of Tiles 1-28 Fired at 1260^{o}C

4.2.7 Test Melting Result of Triaxial Test Blend of 21(Fi) Fired at 1260^{o}C

Samples 1, 3, 9, 10, 12, 13, 14, 15, 16, 17, 18, 19, 20 and 21 did not bond to their tiles. They peeled off their tiles. Sample 11 was bonded to the tile but had major cracks on the surface of the tile. Samples 2, 4, 5, 6, 7, and 8 have melted and bonded on their tiles. Concerning their colours, samples 2, 4, 5, 10, 14, and 17 are light brown. Samples 7, 8, 9, 16 and 18 are white-brown. Samples 11, 12, 13, 16, 19, and 20 are white. Sample 21 is light pink.

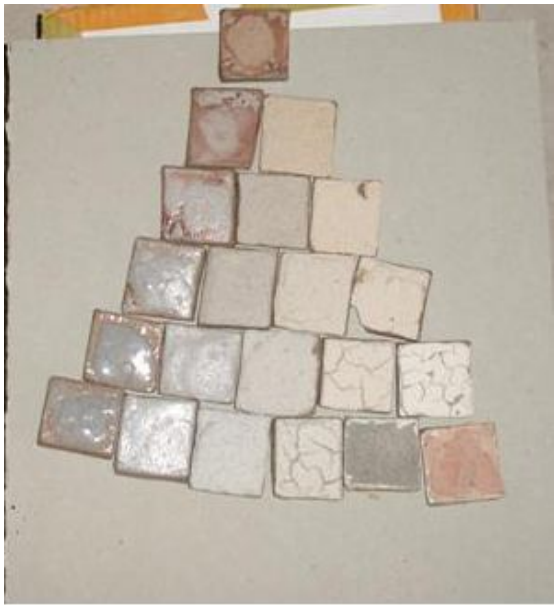


Plate XX: Test Blend Result of Files 1-21(i) Fired at 1260^{o}C

4.2.8 Test Melting Result of Triaxial Test Blend of 21(Fii) Fired at 1260^{o}C

Samples 16, 20 and 21 of this blend did not bond to their tiles. They were shattered and peeled off their tiles. Samples 1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 11, 12, 13, 14, 15, 17, 18 and 19 were melted and bonded to their tiles. Sample 11 and 16 are glass and shinny, though sample16 have swells on the middle of the tile. Their colours are as follows: Sample 1 was brown. Samples 2 and 4 are brownish-grey. Samples 3, 6, 9, 10,

14, 15, 20 and 21 are white. Samples 5, 7, 8, 11, 13, 16, 17, 18 and 19 are all grey in colour.



Plate XXI: Test Blend Result of Files 1-21(ii) Fired at 1260^{o}C

4.2.9 Test Melting Result of Triaxial Test Blend of 66 (G) Fired at 1260^{o}C

Samples 1 – 66 blend of the firing are as follows: Samples 1, 2 3 and 4 and 6 had the same characteristics. They fused to the tiles. They have the same effect of smoothness, glossiness and imperviousness. Their colours are also pink on the single dipped sides and grey on double dipped sides. Sample 5 have melted, stuck to the tile, is smooth and shinny but not as Samples 1, 2 3 and 4 and 6. There are also light or silent cracked.

The colour is light grey. Samples 7, 8, 9, 10, 11, 12, 13, 14, 15, 16, 17, 18, 19 and 20 have similar characteristic. They have melted and sintered, stuck to the tiles but they are not very smooth as Samples 1, 2 3 and 4 and 6. They are also slightly spotty glittering. Their colours are grey. It is only on Sample 19 that have major cracks on the double dipped side. Their colours are grey.

Samples 21, 22, 23, 24, 25, 26, 27 and 28 and 31, 32, 33, 34, 35 and 36 are in the same group. They have melted and stuck to the tiles. They are calm and not smooth in appearance. 32, 33 and 34 have major cracks on the double dipped sides. Their colours are also grey.

In the cases of Samples 29, 30, 37 – 39, 46, 47, 48, 49 and 50, and 56, 57, 58, 59, 60, 61, 62 and 63 are all of the same characteristics. They are stuck to the tiles. Sample 49 have major cracks on the double dipped sides of the same group. Their colours are cream.

Samples 40 and 41 have cracks on the double dipped sides. They have stuck to the tiles and their colours are cream on the double dipped sides and grey on the single dipped sides.

Samples 42, 43, 44 and 45, 51, 52, 53, 54 and 55 and 64 and 65 are of similar characteristics. They have all stuck to their tiles. Only that 45, 52 and 55 pilled off on the double dipped sides. They are all yellowish in colour.

Sample 66 is in rough on the surface though stuck to the tile. It has peeled off on the double dipped side and it is pink in colour.



Plate XXII: Test Blend Result of Files 1-66 Fired at 1260^{o}C

4.2.10 Result of the Vitreous Test

It was observed that the following samples were vitreous as they were hard, shinny and transparent like glass; They include Sample 1 (100g felsdpar) of Quadrilateral Blend 36 (A), Samples 11(100g feldspar), 22(90g feldspar, 10g quartz) and 33 (80g feldspar, 20g quartz) of quadrilateral blend of 121. Samples 1(100g feldspar) and 2 (90g feldspar, 10g kaolin) of triaxial Blend of 66.

4.2.11 Crazing

It was observed that samples 5(100g feldspar) of quadaxial blend of 25; Samples 10 (10 g ball clay, 90g feldspar), 11 (100g feldspar), 22 (10g quartz, 90g feldspar) and 33 (80g feldspar, 20g quartz) of quadaxial blend of 121; Sample 1(80g feldspar, 10g kaolin, 10g quartz) of triaxial blend of 36; Samples 1(100g feldspar) and 2(90g feldspar, 10g kaolin) of traixial blend of 66; Sample 1(70g feldspar, 15g kaolin, 15g quartz) of triaxial blend of 28, all showed varying degrees of crazing.

4.3 Results of Tests of Glazes on Thrown Pieces

The test piece that showed good glaze melt was further tested on thrown pieces. Te result is seen on figures 4.3a- 4.3e. Figure 4.3a and 4.3b show glaze from sample 4 of blend 25. The glaze fused well (smooth and shiny) and is light brown in colour. Samples 20 and 32 are of the blend of 121 as seen in plates XXII - XXVII were also fused properly but they are grey in colour.



Plate XXIII: Sample 4 of blend 25



Plate XXIV: Sample 4 of Blend of 25



Plate XXV: Sample 32 blend 0f 121



Plate XXVI: Sample 20 Blend of 121



Plate XXVII: Sample 20 blend 121

4.4 Property Testing

4.4.1 Determination of properties of Vitreousness, Crazing and Imperviousness

Vitreousness and crazing were determined according ASTM C554. Vitreousness was determined by visible inspection of the glazed specimens. It was observed that the samples were vitreous as they were hard, shinny and transparent like glass. The crazing test was carried out by inspecting the glazed specimens with oblique lighting and application of a dye. This was done by the application of ink on the specimen and cleaned off immediately with clean towel and observation made for fine lines seen.

The property of imperviousness was tested according to the ASTM C126 by the application of black ink to the glazed surface of the samples and allowed to remain for 5 minutes. The surfaces were then washed with a wet cloth and running water and then examined from a distance of 1.52m for staining of the finish. There was no stain observed. Therefore these glazes can be termed to have good finish according to the ASTM C554 and C126.

4.5 Hardness and Abrasion Resistance Test

Glazed specimens resisted scratching by ordinary glass or steel.

4.6 Chemical Resistance Test

The specimens were cut using a tile cutter and were tested with 10% solution of hydrochloric acid (HCl) for 3 hours. Similarly, specimens were also tested with 10% of solution of potassium hydroxide (KOH) for 3 hours.

These solutions were maintained at a temperature of 60^0F to 80^0F (15^0C to 27^0C). They were rinsed, dried and examined for changes of texture and change in colour. There was no change in texture and no change in colour either on the specimens, glazed ceramic samples which indicate a good chemical resistance. This test was conducted in Chemical Engineering Department of Ahmadu Bello University Zaria and is in conformity of ASTM C126.

There was no colour change for the glazed ceramic samples tested using the chemical resistance test, which was conducted in line with American Society for Testing and Materials Standards (ASTM).

The specimens were cut using a tile cutter. This was conducted in the Department of Chemical Engineering.

4.7 Findings

Based on the experiments and tests conducted, the following findings were made:

1) The local raw materials from the north eastern states of Nigeria are capable of adaptation into glazes. These materials include ball clay, feldspar, kaolin and quartz.
2) The chemical analysis results of these materials show that quartz, ball clay and kaolin obtained for the study contained high level silica (92.40%), while feldspar contained the least amount of silica (7.48%), but high amount of Na_2O (37.42%).

Kaolin has the highest amount of Al_2O_3 (34.50%) followed by feldspar (28.90%), then ball clay (21.88%), while quartz has the least (1.470%). All the materials have some quantities of Fe_2O, MgO, CaO, MnO TiO_2, SO_4 and H_2O^+ except ball clay and kaolin that do not have MnO at all.

3) The glaze sample 4 of 25 member blend, samples 20 and 32 of 121 member blend proved to be suitable glazes developed from the materials of the study. They have met the necessary characteristics expected of good glazes such as smooth and shiny surface, hardness resistance and resistance to corrosion. These batches were tested on thrown products successively that were fired at a temperature of 1260^oC.
4) 30 minutes to one hour duration of ball milling the mixed materials produced homogenous batches. And for every 500g of batch, 250ml of water was required.
5) Two types of ball mills were used. The gasoline powered ball mill and the electricity powered ball mill. The electric powered ball mill was used for dry batches. The dry glaze materials were then ball milled for six hours each. After the electrically powered ball milling, 30 minutes ball milling was carried out in the gasoline powered ball milling and they proved effective.
6) The gloss firing using kerosene fuel was found to have varieties of shades on the product at the temperature of 1260^oC on cone 09 (fused flat). The gloss firing using electric kiln showed one shade effect on the test tiles.
7) Since the glazes matured at 1260^oC they can be classified as stoneware glazes (Burleson 2003)

CHAPTER FIVE

SUMMARY, CONCLUSION AND RECOMMENDATION

In line or agreement with the study area, the aim and objectives of the study were centered on the development of glazes using local raw materials from Northeastern States of Nigeria. The chapter represents some of the observations made based on the several tests (experiments) carried out in the course of this study; it also contains some general recommendations.

5.1 Summary

In summary, the result of the study revealed that the local raw materials consisting of ball clay, feldspar, kaolin and quartz obtained from the northeastern states of Nigeria are good for the production of glazes. Feldspar and quartz were obtained from Gwoza Local Government Area of Borno State; ball clay was obtained from Maiduguri ward of Maiduguri Metropolitan Council (MMC), kaolin was obtained from Bauchi and Yobe states. These states are all in northeastern part of Nigeria. The result of testing the materials showed that good glazes can be produced. The gloss firing results showed that oxidation firing produced good quality glazes. The reduction firing also showed varieties of colours on the samples (pots). These tests were conducted at 1250^{o}C and 1260^{o}C.

5.2 Conclusion

Based on methods and results involved in this study such as sourcing, obtaining, processing, formulating, throwing, casting, hand building, bisque firing, gloss firing of some local raw materials for glaze compounding, the following conclusions were made; The test samples numbered 20 and 32 of blend 121 and 4 of 25 were considered the best results for glaze development. This has proved that the local raw materials from the northeastern states of Nigeria are capable of developing quality stoneware glazes.

5.3 Recommendations for Further Studies

1) There is need to carry out further researches on the materials used for this study for the production of glazes.

2) Other materials from the localities should be investigated by other researchers for comparison and upgrading of glazes in the northeastern states in Nigeria.

3) The glazes developed should to be tested with oxides that can impact colours.

4) Glaze(s) development researches also need to be carried out on the materials of study including other materials like; limestone, bentonite, potash and others.

5) Biaxial blends and other types of blends should be conducted on the glazes developed including other materials.

5.4 General Recommendations

The following recommendations are made based on the importance of glazes in schools, potteries and for the ceramist practicing locally in Nigeria;

1) There is the urgent need for the Government to develop glaze processing and production unit of the University of Maiduguri and other tertiary institutions in the North Eastern States for the purpose of teaching and learning.
2) There is need for supply of electric kilns in ceramic sections in Nigerian Universities especially University of Maiduguri and Ahmadu Bello University. Test kilns and electric kilns for gloss firing should be made available to enhance quality research works in the section.
3) Digital scales should be made available for accurate measurement of glaze materials in the Ceramics Section.
4) Postgraduate studio and laboratories are required in Ceramic Section as a matter of urgency.

5) Federal, state and local governments should endeavor to assist students of ceramics by encouraging them on the basis of research for better results for development processes.
6) Ceramic students need to undertake some introductory courses in sciences; particularly in chemistry and geology. This will enable them understand the chemical and mineralogical properties of the materials used in ceramics.
7) Analytical equipment like XRF and XRD machines should be made available in Ceramic Section for the purpose of analyzing the elemental and mineralogical constituents of ceramic materials.
8) The current Ceramic Section of industrial design department should be made a full-fledged department in order to provide a platform for in-depth study of Ceramics in the University.
9) Property testing standards for example American Society for Testing and Materials (ASTM) standard as well as property testing facilities should be provided to the Ceramic section of Ahmadu Bello University Zaria and other Universities across the nation. This will aid researchers in the area to confirm their results for reliability and accurate determinations.

REFERENCES

Adah, N.J (2007): Ceramics As a Way forward to Economic Empowerment and Capacity Building in Nigeria. *Ashakwu Journal of Ceramics,* 4(2):43

Ahuwan, A.M. (1999). Trends in Contemporary ceramics in Nigeria. *Journal of EnvironmentalStudies,*1(2&3): 32 and 46

Ahuwan, A.M. (2003). Contemporary ceramic in Nigeria 1952-2002 Achievements and pitfalls. *Ashakwu journal of ceramics,* 1(1): 19-20

Ajala, O. A and Munai (2007). D.A Pacifying transparent ash glaze using Tin oxide. *Ashakwu Journal of ceramics*, 4(1): 14-15.

Ajayi, B.A (2007). Harnessing Design Concepts and Methods for better Ceramic Products in Nigeria. P. 26

Ajayi, B.A. and Chindo, S.L. (2005). Restructuring Ceramic Education for Technical Advancement. *Ashakwu Journal of Ceramics*, 2(1): 74 – 76.

Alasa S. (2005). *Fundamental of ceramics*. Mora Mon Bros. Ent. Benin City, Nigeria. Pp 6-142.

Alkali, V. (2004). Standardization and Product Differentiation, A means of promoting Appeal and Marketability of pottery products. *Ashkawu Journal of Ceramics*, 1(1): 136-137.

Alkali, V. (2009). *Exploration of the potentials of Granite sawing waste in developing ceramic bodies and glazes*. An Unpublished Ph.D Seminar presentation at Faculty of Environmental Design, Ahmadu Bello University, Zaria, P. 6

American Society for Testing and Materials (1991), ASTM West Conshohocken, United States.

Aluwong, M.S. (1988). *Developing slip Glazes from some Selected Clay Materials in Kaduna state*. An Unpublished M.A Thesis, Ahmadu Bello University, Zaria.

Bassey, E.A (2009). Processing Glaze Formulation using Local Raw Materials CPAN Journal of ceramics NOS 2&3 Craft potters Association Nsukka-Nigeria P. 30

Bassey, E.A (2007). Building in Nigeria. *Ashakwu Journal of Ceramic*, 4(2): Pp43

Burleson, M (2003): *The Ceramic Glaze Handbook; Materials, Techniques and Formulas*. Revised edition by Lark Books New York.p44

Counts, C. (1974). *Pottery Workshop*. Macmillan Publishing Co. Inc. New York. P.130

Cotton, F.A and Wilson G (1980): *Advance Inorganic Chemistry, A comprehensive text* 4th Ed. McGraw- Hill, Inc. New York P. 1

Daly G (2013): *Developing Glazes*. Bloomsbury Plc. 50 Bedford square pp 6, 70-71

Datiri, Y.C., Sadiq and Umar, A.D. (2008) Utilizing pumice from Kerang to develop mid temperature ceramic glazes. *Ashakwu Journal of Ceramics*, 5: 146.

Emodah, C.S. (2006). *An Approach to Ceramic Decorations and Metal Engineering*. Mon Mora Bros. Ent. Benin City, Nigeria. P. 2

Ewule, E.E. (2006) Issues that Hamper the progress of ceramics production in Nigeria Today. Ashkawu Journal of Ceramics, Vol. 3, no 2. Dasma press, Zaria, Nigeria. Pp. 82 – 83

Fatuyi, A.O. (2007). Revitalization of Local Ceramic Industries towards Poverty Alleviation in Edo-North of Nigeria. *Ashakwu Journal of Ceramics,* 4(2): 79

Fournier, R. (1973). *Illustrated Dictionary of Practical Pottery*. London: Van Nostrand Reinhold Company, Inc.P.163

Hammer, F and Hammer, j (2004). *The potter's Dictionary of materials and techniques fifth edition*. A and Black University of Penny/Vania Press London. Pp 18-358.

Garkida, A.D. (2003). Ijero Feldspar: A good source of potassium Alumino-Silicate For the Glass and Ceramics Industries. *Journal of Ashakwu,* 1(1):73

Garkida, A.D, Opoku E.V and Ahuwan A.M (2004): Recycling Window Glasses in Ceramics Glazes as an Alternative to Feldspar. *Ashakwu Journal of Ceramics*, 1(2): 39

Gonah, C.M. Yaro, A.S and Ahuwan, A.M. (2003). Design and Fabrication of ceramic glaze materials processing machine. *Ashakwu Journal of Ceramics,* 1(1): 86.

Gukas, H. J and Datiri Y.C (2001). *The Art of Pottery published by C.C Communications*, Jos Nigeria. Pp 3, 9-11

Hogan, E. (1973). *Ceramic techniques and projects*. California: Lane Magazine and Book Company, Menlo Park Carliforna USA. P 9-75.

Idowu, I.O. (2006). Appraisal of Traditional Pottery and the chronological development of modern ceramics in Nigeria: The challenges of the New Millennium. *Ashkawu Journal of Ceramics*, 1(3): 136

Kashim, I.B (2011): Solid Mineral Resources Development in Sustaining Nigeria's Economic and Environmental Realities of the 21st Century. *Journal of Sustainable Development in Africa,* 13(2): 212

Mamza, J.A. (2008). *Evaluation of Ceramic Facilities, Equipment and materials in some selected Colleges of Education in North East Geo-political zone of Nigeria*. Unpublished M.A Thesis in Industrial Design Department, Ahmadu Bello University, Zaria. Pp. 25-38.

Manzuche,S.M.A.(2006). *Suitability of Some Solid Minerals for Industrial Ceramics in Nasarawa State*. Unpublished Ph.D. Thesis, Ahmadu Bello University Zaria. P4

Musoke, W. L. (1978). *The Development of Pyrometric Cones*. Unpublished M. A. Thesis, Ahmadu Bello University Zaria. P. XXIX

Nelson G.C. (1978). *Ceramics: A Potter's Hand Book New York Garrison*. Pp.271-277

Nelson, G.C., (1971) *Ceramics A Potter's Handbook.* Holt, Rinehart and Winston. Inc., New York pp. 219-318.

Norton, T.H. (1956). *Ceramics for the Artist – Potter*. New York: Addison –Wesley Publishing Company.

Nwanah, D.O. (2007). Ceramics and Industrial Development: The Nigerian Situation. *Ashkawu Journal of Ceramics,* .4(1): 52 – 53

Okoruwa, P. E. (1986) The *Utilization of Some Selected Raw Materials for Porcelain Slip Casting and Glazing of Moulded Ceramics*, M. A Thesis Unpublished Ahmadu Bello University Zaria Pp 32-63

Ola ofe, I. A.(2010) *Research Writing for Academic Growth*. Ahmadu Bello University Press Limited, Zaria, Nigeir. Pp 9- 51

Opoku, E.V (2006): The Ceramic Industry: Changing Life Styles and Patterns of Consumption as They Influence the Philosophy of Materials and Design. *Ashaku Journal of Ceramics,* 3: 5

Opoku, E.V. (2007). Contemporary Development in Ceramic Materials: Development of Local Raw Materials for the Ceramic Industry in Nigeria. *Ashakwu Journal of Ceramics*, 4(2): 84

Peterson, B. (2011) *Temperature Ranging for Firing Glazes*. About.com Guide Internet Retrieved- 11/9/2012 11:27AM

Pius, O., (2008) *Ceramics at a Glance.* Otoba Press Limited: Asaba, Nigeria p.128-129.

Samuel, J O. and Adeyemi, M.B. (2004). Effects of Silica/clay types and comparison on the Properties on ceramic tiles. *Journal of raw materials and research*, 1(1): 42-47

Singer, F., and Singer, S.S., (1971) *Industrial ceramics*. Chapman and Hall Ltd II New Fetter Lane, London pp. 27-533.

Sullayman, U. A. (2003). Ceramics and the Society: The Creation and Sustenance of Better Opportunities for the Nigerian Ceramics in the Local and International markets. *Ashakwu Journal of Ceramics*, 1(1): 7

Sullayman, U. A. (2006). *Utilization of local raw materials for the production of Local Raw materials for the production of dense Aluminoslicate Refractory Bricks for Blast Eurnaces using semi dry pressing technique.* An unpublished Ph.D

Desertation, Department of Industrial design, Ahmadu bello University, Zaria, Nigeria pp. 41-50.

Sullayman, U. A (2004). The standardization and control of professional practices in the visual arts; the Nigerian ceramics and the professional standards. *Ashakwu Journal of Ceramics*, 1(2): 105.

The Fulham Pottery (1671) materials and equipment for the potter. Pp.11 – 13.

Udoh, E. N. A. and Ekong, C. E. (2008) Problems and Prospects of Standardizing Ceramic Materials in the Studio. *Ashakwu Journal of Ceramics*, 5: 119

Umar, A. (2003). Ceramics: where Geology ends and where Art begins. *Ashakwu Journal of Ceramics*, 1: 36.

Wehmeier, S., (2002) *Oxford Advanced Learners Dictionary of Current English*, sixth Edition Oxford University Press, Pp. 954-1185.

Wehmeier, S., (2010) *Oxford Advanced Learners Dictionary of Current English*, sixth Edition Oxford University Press. Pp. 1187-1188.

Online sources

www.clay-king.com/retrieved on 11/9/2012

http://www.astm.org/standardslc424.htm. (Retrived on 5/25/2014 3:45pm.p.1of 1)
http://ian.currie.to/originalglaze
http://www.global.ihs.com/news/tem/standards/ASTM-15.htm.(Retrieved on 3/19/2011.6:50am pp.2andand 4of7)
http://www.org/database.cart/historical/E165.htm. (Retrieved on 5/26/2014 12:53pm p1of2)
http://www.azom.com/article.aspx?
http://www.bigceramicsstore.com/information/GlazeExplanation

APPENDICES

Appendix 1

MAP OF NORTH EASTERN STATES SHOWING STUDY AREAS

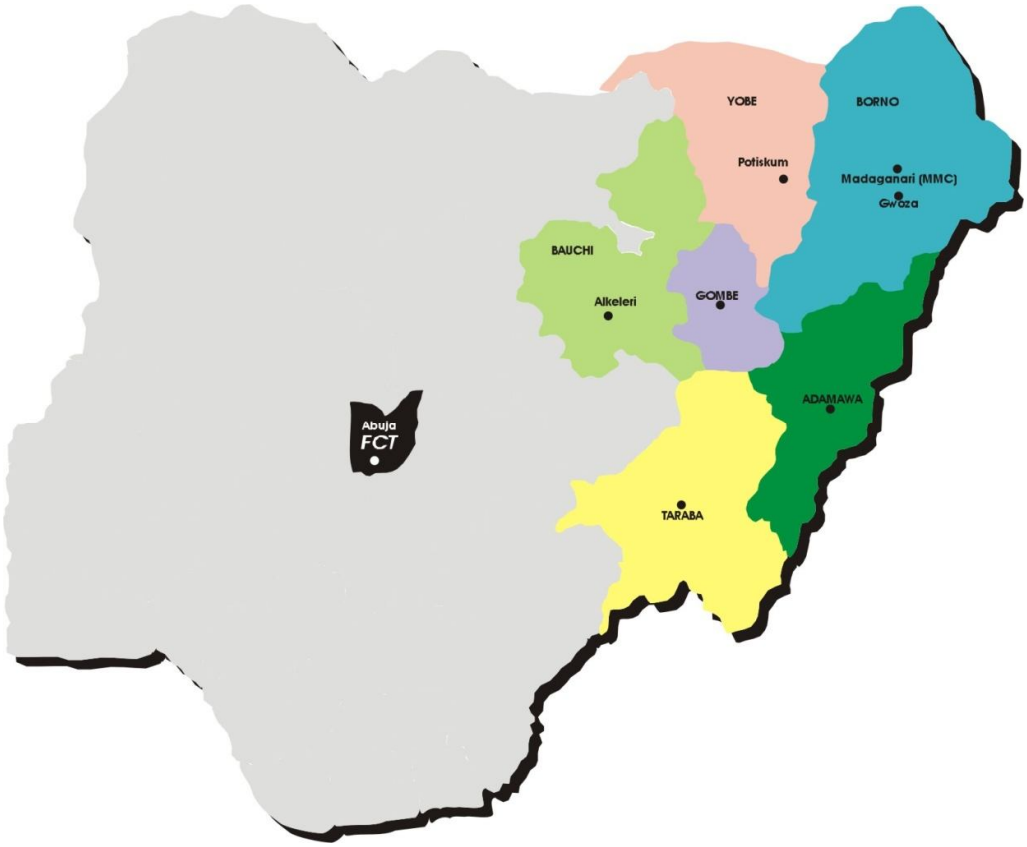


Fig. 1: Map showing study areas

Appendix 2

WEIGHING OF MATERIALS USING DIGITAL SCALE



Plate XXVIII: The researcher explaining the procedure used for weighing the materials to the supervisory team.

Appendix 3

MEASURING OF MATERIALS USING THE DIGITAL SCALE



Plate XXIX: Formulated materials being weighed at the laboratory

Appendix 4

DISPLAY OF DEVELOPED GLAZES ON TILES



Plate XXX: The supervisory team, Prof. Ahuwan, Prof. Sullayman and Dr. Garkida inspecting the developed glazes



Plate XXXI: The researcher explaining the result of the developed glazes to the supervisory team Prof. Ahuwan, Prof. Sullayman and Dr. Garkida

Appendix 5

GOOD GLAZES DEVELOPED FROM THE STUDY



Plate XXXII: Developed glaze samples from the three study areas

Appendix 6

PRODUCTION OF POT FOR GLAZE TESTING



Plate XXXIII: Throwing of pot by the researcher for glaze testing



Plate XXXIV: Thrown pot ready for glaze testing

Appendix 7

RESULTS OF TESTS OF GLAZES ON THROWN WARES



Plate XXXV: Glazed wares (pot) from the study