

**DEVELOPMENT OF LOW-TEMPERATURE GLAZES FROM SELECTED RAW
MATERIALS IN BENUE STATE, NIGERIA**

BY

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**DEPARTMENT OF INDUSTRIAL DESIGN
FACULTY OF ENVIRONMENTAL DESIGN
AHMADU BELLO UNIVERSITY,
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OCTOBER, 2018

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P15EVID8007**

**A THESIS SUBMITTED TO THE SCHOOL OF POSTGRADUATE
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DEGREE IN INDUSTRIAL DESIGN**

**DEPARTMENT OF INDUSTRIAL DESIGN
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ZARIA, NIGERIA**

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DECLARATION

I declare that the work in this Dissertation entitled “The Development of Low-Temperature Glazes from Selected Raw Material in Benue State, Nigeria” was written by me in the Department of Industrial Design of the Faculty of Environmental Design, Ahmadu Bello University, Zaria. The information derived from the literature have been duly acknowledged in the text and in the list of References provided. No part of this dissertation has been previously presented for another degree or diploma at this or any higher Institution.

Tser Helen Ngunengen

Name of Student

Signature

Date

DEDICATION

This Dissertation is dedicated to God Almighty for His goodness, favours, protection and mercies that sustained me throughout the programme.

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ABSTRACT

The development of glazes in the Nigeria is a challenging issue to the ceramic industry and has resulted in the dependence on imported glazes, which is not easy for the ceramic industries and the ceramists. This has become necessary therefore, to explore the possibility of low-temperature glazes using locally sourced raw materials for ceramic production in the Country. The objectives of the study are to: identify and source, for ceramic raw materials in Benue State for, the formulation of low- temperature glazes; carry out chemical analysis of the selected raw materials, formulate low-temperature glazes using locally sourced materials for ceramic production and test; and evaluate the effect of the formulated glazes on ceramic wares. Data collection for the study was from the primary and secondary sources. Dogo Red Clay, Groundnut Shells and Locust Beans Wood were sourced for, from within the area of study; while experimental research design was adopted for the study. The experiments carried out were analysed and discussed. Various stages conducted were documented and their discussions are presented. Among the findings of the study; the chemical analysis carried out shows that the major element found in Dogo Red Clay is iron oxide Fe_2O_3 which acts as flux in bodies and glazes at high temperature. Groundnut Shells Ash and Locust Beans Wood Ash contain potassium oxide (K_2O) and calcium oxide (CaO) which acts as flux to lower the melting temperatures from $1200^{\circ}C$ down to $900^{\circ}C$. The low-temperature glazes formulated using locally sourced raw materials shows that tri-axial blend tested fails to meet the desire to produce low-temperature glaze, the line blend was successful in achieving low-temperature glazes. The study found that there is a similarity in the temperature range of between $900^{\circ}C$, $1000^{\circ}C$ and $1100^{\circ}C$, indicating varying shades of black, dark brown, yellow, lemon green, while at $1160^{\circ}C$ the reddish colour was found. The study recommends that, the Dogo Red Clay should be tested on other bodies with other fluxes to set different effect of colour. Also, there is need to carry out further studies using Dogo Red Clay for the development high temperature glazes. Through this study, it is observed that this research was able to establish that the Dogo Red Clay, Groundnut Shells Ash and Locust Beans Wood Ash, line blend at 900%, 1000%, 1100% and 1160% was successful for use on ceramic wares. This research was able to fire at low – temperature range of 900%, 1000%, 1100% and 1160% to achieved diverse colouration of glossy, some reddish, black, yellowish brown, dark with gold and purple colour

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ABBREVIATIONS /NOTATIONS

ASTM: American Society for Testing and Number

AD - After Death

BC - Before Christ

°C; Degree Centigrade

°F; Degree F

Fig: Figure

ISO: International Organization for Standardization

Nd: No Date

NOS: Numbers

POP: Plater of Paris

Wt: Weight

SIGN

% - Percent

DEFINITION OF TERMS

1. Alkali; The opposite of acid. Potters call the glaze and body fluxes their alkalis. These are the nation- colouring metal oxides which react with the acids in the presence of heat to produce silicates (glasses).
2. Aluminum oxide: Corundum. Dialumimiumtrioxide. Aluminumsesquioxide. Al_2O_3 . An important oxide in ceramics: It is second only to silica in important.
3. Analysis: A method of describing a substance by its different parts.
4. Ashes: The non-combustible remains of animal (bone) and vegetable matter used by the potter as a source of body and glaze fluxes.
5. Ball clay: Blue clay, A highly plastic clay, usually light in colour, which is the basis of many potting bodies. Alone, it tends to be too fine and slippery for use, but additions of sand, grog and coarser and less- plastic clays actually improve workability.
6. Barium Oxide: BaO ; An auxiliary flux in earthenware frits and high temperature stoneware glazes.
7. Biscuit: To fire ware unglazed in preparation for glazing: also the unfired ware.
8. Body: A clay for a special purpose. It is created by blending different clay or adding to clays other minerals, such as feldspar and flint.
9. Borax: Tincal. $Na_2B_4O_7 \cdot 10H_2O$. or $Na_2O \cdot 2B_2O_3 \cdot 10H_2O$. A crystalline mineral which is the source of boron oxide with sodium oxide. It is used in making low-melting glaze fruits without lead oxide.
10. Clay: Hydrated Silica of Alumina. A heavy, damp, plastic material that 'sets' upon drying and can be changed by heat into a hard, waterproof material.
11. Clay body: A mixture of different types of clays and minerals for a specific ceramic purpose.
12. Crawling: Creeping, Rolling, Beading; the glaze defect characterized by the way the glaze part and rolls back on itself leaving a bald patch of body.
13. Crazing: Crackle. Craquelle. The glaze defect characterized by a network of fine cracks.
14. Chemical element: A substance which is so pure that it contains only atoms of the same atomic number.

15. Colours: Ceramic colours. Metal oxides and prepared stains used to colour bodies, slips, glazes and enamels.
16. Deflocculation: The action of dispersing the fine clay particles in a slip so that the slip becomes more fluid.
17. Dewatering: Taking water out of clay or slip. This is done by leaving the clay, slurry e.t.c outside in pits to dry in the sun and wind: by putting the clay on to an absorbent surface such as plaster, wood or bricks sometimes heated from below: or by filter pressing which is forcing the slurry into previous cloth bags.
18. Dogo Red Clay: is ball clay that has minerals with the high chemical Iron oxides (Fe_2O_3 39.70), (SiO_2 27.40) and (Al_2O_3 12.00) found in Buruku local government of Benue State Nigeria.
19. Dry clay: Clay which has dried completely and is therefore as dry as the surrounding air.
20. Drying: The removal of moisture. In pottery this usually means the drying of wet clay or newly –glazed ware.
21. Earthenware: Pottery made of a porous body which is waterproof.
22. Element: Chemical element .A substance which is so pure that it contents only atoms of the same atomic number.
23. Feldspar: A group of minerals used in proportions of up to 25% as flux in bodies and up to 10% in glazes.
24. Firing: The process of conversion from clay to pot. It involves heat of at least 600°c (1112°f).
25. Firing Range: The range of temperature at which clay becomes mature or a glaze melts.
26. Frit: SiO_2 . Cryptocrystalline native silica. Flit is almost pure silica containing less than 5% impurity in the form of calcium carbonate.
27. Flocculation: The action of altering the physical properties of fine particles in a suspension so that they no longer repel one another but aggregate into larger particles, called flocs, and settle by gravity.
28. Flux: An oxide which promotes ceramic fusion by interaction with oxides,
29. Glaze; A layer of glass which is fued into place on a pottery body.
30. Glass-former: An oxide which is able to retain the amorphous property of its molten state when setting to a solid.

31. Green ware: ware which is complete from the making but has not yet dried sufficiently to be ready for firing.
32. Groundnut Shells Ash: is a tree mineral with the chemical composition potassium oxide (K_2O 17.20w %), calcium oxide (CaO 16, 19w %) located along Kwande Local Government of Benue State of Nigeria.
33. Heat: Energy used by the pottery to achieve the necessary chemical and physical changes which convert clay into pot and minerals into glaze.
34. Iron oxide: FeO or Fe_2O_3 or Fe_3O_4 . The general name for compounds of iron and oxygen.
35. Kankara Clay (Kaolin): This is an industrial clay mineral with the chemical composition 44.56%, Al_2O_3 and 31.45%, SiO_2 found in Kankara town and named after the town and it comes from Katsina State of Nigeria.
36. Kaolin: China clay $.Al_2O_3 \cdot 2SiO_2 \cdot 2H_2O$. Synonymous with China in North America.
37. Line blend: A method of blending two or more materials.
38. Locust Beans Wood Ash: This is a tree mineral with the chemical composition potassium oxide (K_2O 20.59w %) and calcium oxide (CaO 66.87w %) located along Vandeikya Local Government of Benue State of Nigeria.
39. Low-temperature glaze: One which matures in the range up to $1050^\circ C$ ($1922^\circ F$).
40. Majolica: English majolica. A late 19th-century ware with shiny colourful glazes.
41. Mould: A plaster shape designed to pour slip cast into and let dry so the shape comes out as an exact replica of the mould.
42. Maturing: Developing and improving. Clays are said to mature if they are stored in wet plastic condition.
43. Melting point: The temperature which divides the solid state from the liquid state of a material.
44. Mesh: A fabric of interwoven threads producing theoretically square holes between them.
45. Nepheline: $K_2O, 3Na_2O, 4Al_2O_3, 9SiO_2$. A feldspathoid mineral variable in colour from white to dark greenish brown.
46. Oxidation: In pottery this refers to the combination of oxygen with an element or compound.
47. Oxide: A chemical combination of oxygen with another element.
48. Plastic clay: Clay in plastic state, that is, in a state in which it can be formed easily by moderate pressure and yet retain the new form with collapse.

49. Plasticity: The unique property held by clays which combines the strength of a solid with the fluidity of a liquid.
50. Preparation of clay: Pockets of clay of consistency and ideal composition for a particular product can be found.
51. Range: The variation of tempura /time that a clay or glaze can withstand without being over or under fired.
52. Raw: Uncooked, unfired, in original state.
53. Reduction: The action of taking oxygen away from metal oxides.
54. Secondary clay: Sedimentary clay. Clay which has been removed from its place of origin by natural forces and deposited elsewhere.
55. Sedimentary: Settling. The sinking of particles in a suspension.
56. Shrinkage: Decrease in size due to drying and firing.
57. Silica: Silicon dioxide. SiO_2 . The glassy substance which is the most important constituent of pottery glazes and which is an integral part of clay and many potters' minerals.
58. Silicate: A compound involving silica as an important part.
59. Slip: A homogenous mixture of clay and water.
60. Slip-casting: A pottery –forming process which uses moulds to give the form and use liquid clay (slip).
61. Slurry: An uneven mixture of clay or glaze and water.
62. Soak: A term used in kiln firing schedules when the temperature is maintained at the same level for a given period.
63. Soaking: is also done during the cooling cycle to allow the growth of crystals in crystalline glaze.
64. Temperature: A means of describing heat.
65. Tra-axial blend: A method of blending three materials
66. Verification: The furthest stage to which a body can be taken without deformation.

CHAPTER ONE

1.0 Introduction

Nigeria is a country blessed with abundant raw materials for ceramic production. Despite the availability of these raw materials, and the attempts being made to utilize these local raw materials, there is still much to be done especially in the area of low-temperature glazes. The trend of research into ceramic raw materials today is assuming a dynamic one. This has never been so in the past. It is believed that if this trend continues, it may soon be heading for an industrial ceramic revolution considering the abundant natural ceramic resources available all over the country and Benue State in particular. It is noted by Aluwong (1988) that, the most important issues in ceramics today are the replacement of expensive imported materials for glaze formulation by ideal raw materials and how to improve their qualities. The complaint of most practicing ceramists is that there is no equipment for raw processing. Countries noted for high-quality products of ceramics today however, started from the scratch and developed with time.

It is a challenge for the Nigerian ceramists to brace up and tackle the problem of ceramic development in the country in order to put the country on the world map of notable ceramic producers. According to Ahuwan (2003), the first move to modern pottery in Nigeria was in 1902 by D. Roberts, though this was not successful until 1952 when Michael Cardew arrived. Different ceramic industries have folded up and part of the problem is attributed to low or lack of technical knowledge production of local glazes to sustain ceramic production.

In addition Chigbo (2009) observed that the ceramic industry covers up to 7,000 products and processes, and Nigeria has enough raw materials in this field to support a vigorous ceramic industry and to make a great difference in the productivity of those industries that need the ceramic products. The ceramic industry in modern times is a very large one that holds

potentials for the growth and development of the nation's economy, but the issue of glaze is another problem confronting ceramic industries, because a great deal of energy is needed for firing the products in the kilns. There are various ways of trying to further reduce the consumption of energy within the ceramic industry. One possible way is by modifying the ingredients and recipe that can be achieved by using specified additives that cause the clay or glaze to sinter effectively at a lower temperature. Additives that lower the sintering temperature are natural or compounds containing chemical elements that already exhibit a flow-type or sintering type of behaviour at a lower temperature as noted by Hans and Hans (2011).

Earthenware glazes are based on low melting materials. Most of such materials for low-temperature glazes earthenware can be obtained from commonly available indigenous sources. These include clays, wood ash, rice husk ash, and limestone. Earthenware glazes are fired in the range of 900°C-1100°C, as stated by Norsker and Danisch(1993).Materials used Boron is a naturally occurring element. In nature, it is found combined with oxygen and other natural elements forming several different compounds called borates. Borates are widely distributed in nature and found in the oceans, sedimentary rocks, and coal shale and some other soils.

Nigeria, as a nation, is in need of research efforts that will explore and identify locally available raw materials for industrialization. Importantly, the industrial development of a nation could likely be realized with effective and efficient utilization of the available natural resources. According to Ahuwan (1999), there is a constant search for new and relevant sources of materials so as to enhance the supply of materials to reduce the cost of production and improve the quality of products. Thus, the need to take up research work on the abundant locally available raw materials for low-temperature glaze production that will lead to low cost of production is paramount.

It is a fact that the past decades of harsh global economic meltdown, did not help the ceramic industries in Nigeria. Most ceramic production units went underground. They were either totally abandoned or they were being run skeletally at high production cost. Even government ceramic establishments were not spared. Now that the national economy is in recession, it will not be easy for ceramic industries to embark on any serious developmental activities. It was for the reason that, the study was embarked upon for possible contribution to national development in an immeasurable way for ceramics to be in the mainstream of potential industries for national development in Nigeria. The basic rudiments of ceramics development which are raw material research, identification, characterization, exploitation, and formulation of low-temperature glazes from the local sources of ceramics raw materials are essential.

The development of low-temperature glazes from Dogo Red Clay in Buruku, Locust Bean wood from Vandeikya, and Groundnut Shells from Kwandewill be beneficial to the development of many potters, studio and ceramic industries. In view of the industrial benefit derivable from the development of ceramic raw materials in the country, the researcher explored locally sourced raw materials from Benue state in the North-Central Zone of Nigeria which has been found to be endowed with ceramic raw materials such as ball clay, silica and other related minerals in abundance. Therefore, this research focused on exploring suitable ceramic raw materials in Benue state for the production of ceramic glazes.

1.1 Statement of the Problem

The development of glaze in the country is a challenging issue to the ceramic industry and has resulted in the dependence on imported glazes which is not easy for the industries and the ceramists. Such glazes are high temperature in nature which consumes more power or fuel. It has become necessary therefore, to explore the possibility of developing low-

temperature glazes using locally sourced raw materials which will reduce the enormous money paid for light or fuel before wares are fired.

1.2 Aim and Objectives of the Study

The aim of the study is to formulate low-temperature glazes using selected local raw materials from Benue State;

While the objectives of the study are to:

- i. identify and source for ceramic glaze raw materials in Benue State for formulation of the low-temperature glazes;
- ii. carry out chemical analysis of the selected raw materials,
- iii. formulate low-temperature glazes using the locally sourced materials for ceramic production, and
- iv. test and evaluate the effect of the formulated glaze on ceramics wares.

1.3 Research Questions

The following research questions were generated from the objectives:

- i. What are probable raw materials identified in Benue State for the formulation of low-temperature glazes?
- ii. What are the chemical compositions of the materials suitable for the low-temperature glazes?
- iii. How can glazes be formulated for ceramics production using the sourced raw materials?

- iv. How effective is the formulated glaze on ceramic wares comparison with imported glazes?

1.4 Justification of the Study

Available literature from the Raw Material Research and Development Council (2010) shows that Nigeria as a country has a significant reserve of inorganic and non-metallic raw materials. These raw materials buried in the ground can only become wealth when mined, processed and put to industrial use. It is observed that the processing aspect of ceramic raw materials has been a major challenge faced by ceramists and ceramic industries in Nigeria. Alkali (2003) is of the opinion that, when local raw materials are developed, they can spur industrial development and self-reliance, thus maximizing the use of local materials, instead of depending on imported ones with their attendant adverse effects on the economy. Glazes are usually imported for glazing ceramic wares as surface finish. Many people associate fragile, low-fired pottery as earthenware, and mistakenly believe that it cannot be a durable body for tableware. This study, therefore is justified, to developed glazes from selected local materials that will assist the ceramists, studio potters and local industries in Nigeria

1.5 Significance of the Study

The absence of locally processed raw materials for finishing ceramic wares is a major issue that needs attention. This has affected the development of indigenous ceramics in Nigeria. This has also affected the country by acquiring high duties on the importation of foreign glazes. The study is therefore of significance by providing indigenous glazes relevant to the aesthetic and economic growth in ceramic production. This also encourages creation of room for internal development and self-sufficiency, which depends to a large extent on the mobilization of human and natural resources.

1.6 Scope of the Study

The scope of this study is concerned with the use of selected raw materials that were locally sourced from Benue State.

1.7 Delimitation to the Study

The study was delimited to the use of selected raw materials namely Dogo Red Clay, Locust Beans Ash, and Groundnut Shells Ash to formulate earthenware glazes for ceramic production. These materials were sourced from three Local Government Areas of Benue State namely: Buruku, Vandeikya, and Kwande.

CHAPTER TWO

REVIEW OF RELATED LITERATURE

2.1 Introduction

This chapter contains reviewed literary works related to the development of low-temperature glazes. These are written materials from secondary sources such as textbooks and journals among others. The review covered the following: the historical background of glazes, clay, earthenware, earthenware clays, clay bodies, glazes, glazes composition, types of earthenware glazes, low-temperature glazes, production of earthenware, correcting a formulated earthenware body, characteristics and properties of earthenware clay, earthenware glazes, fluxes for earthenware, frits, fluxes used in low-temperature glazes, glaze application and firing.

2.2 Historical Background of Glazes

The knowledge of how glazes are formulated for pottery is of great antiquity. Rhodes (1957) traces this development to about 5000BC long before the scientific knowledge of chemistry. Also, Rhodes (1957) notes that the Egyptians were said to have made the first glazed ware, utilizing soda compounds which were abundantly found in the desert area of the near East. Parmalee (1973) traces the beginning of this development to the Barbarian period of Egyptian history.

According to Parmalee (1973), the art of pottery is one of the oldest forms of art that dated back to 6500BC. However, this was well developed as commercial in about 4000BC. It is observed by Cooper (1971) that, the early pottery appears to have come from Anatolia, and is associated with cave-dwelling communities of the late Mesolithic period.

Most pottery from the earliest times onward was earthenware. Pit fired earthenware dates back to as early as 29,000–25,000 BC. Outside of East Asia, nearly all European pottery up to

the seventeenth century, most of the wares of Egypt, Persia and the Near East; Greek, Roman and Mediterranean, and some of the Chinese; and the fine earthenware form the greater part of available tableware today.

2.3 Clay

Clay is a common name for a number of fine-grained, earthy materials that become plastic when wet. Clay is one of the three principal types of soil, the other two being sand and loamy soil. A certain amount of clay is desirable in the constitution of soil since it binds other kinds of particles together and makes the whole retentive of water. Moreover, clay is the product of the breakdown of the earth's rocky surface (primarily feldspar rock) through the action of wind, water, and temperature, Skutt(2016). These small particles are typically washed collection into deposits known as clay. Clay varies according to its location that is indicative of where it is found and the processes responsible for its formation. Primary clays are residual clay and secondary clay are clay that have been transported from their place of formation by such natural forces as winds, glacier, and water, and have been further deposited in status in the lower areaGukas and Datiri,(2001). Developing raw materials for ceramic work is one of the effective means of promoting homemade ceramic products.

Clay, referred to as a naturally occurring material, composes primarily of fine-grained minerals, which are generally plastic with appropriate water content and becomes hard when fired or dried. The clay formed by this action usually lies near or on top of the granite and is known as primary clay. It is popularly called kaolin or china clay as observed by Singer and Singer (1963) and that it is of low plasticity and highly refractory. Green (1963) reported that, only a few deposits of primary clay have been left undistributed. The majority have been washed away by water or wind and other agents of erosion and re-distributed over the earth, collecting on their journey all manners of materials and vegetables or animal waste. These re-depending clays are known as secondary clays.

Clay materials abound in most localities in Nigeria. Aluwong (1988) is of the view that, garden clay pits and construction sites may yield a variety of clay materials. Ball clay is usually more plastic and has greater shrinkage. This clay can range in colour from a light to a dark reddish-brown after firing.

Plastic raw material clay is characterized by heavy, damp plastic material and becomes hardened when dried. In other words, clay can be changed with the application of heat to a hard and impervious material. Ahuwan (2002) stated that clay as a hydrated silicate of aluminum has a definite chemical formula of clay $-Al_2O_3 \cdot 2SiO_2 \cdot 2H_2O$.

Clay is indisputably the oldest raw material for ceramic and possibly the earliest article produced generally from natural clay. Worall (2007) also explains that clay normally becomes sticky mass if mixed with water. At a wet stage, it can be used to make items of different shapes and if dried, the clay turns hard brittle and maintains its shapes but cannot resist the action of water even at this stage. Clay that no longer changes because of the action of water is heated to the state of redness.

Clay is derived from the disintegration of granite and other feldspathic pegmatite rock which, as they decompose, deposit alumina and silica particles Nelson, (1978). Clay is formed as a result of the decomposition of igneous rock impurities, the reason why processing of clay before working is important. It is observed by Umar (2003) that plasticity of clay depends upon the fineness of particle size. Characteristics of clay make it to be formed in different ways. If the clay is indeed the most widely available material, then it is probably the most widely used for ceramic purposes. The author notes further that, all clay contains impurities and the impurities are responsible for the plasticity of clay. It is noted by Hansen (2015), and Norsker (1990) that high-iron clay can remain anti flux at oxidizing atmosphere of between 700-900°C, and the fluxing action starts at above 1200°C.

2.4 Earthenware Clays

According to Arbuckle (n.d), earthenware clay is low-firing clay that is typically found in creek beds and other deposits that have been transported by wind and water from their location of origin. Earthenware usually means a porous clay body maturing between cone 06 – cone 01 (1873°F - 2152°F). Earthenware clay is usually not fired for verification. Rhodes(1957) states that most of the usable clay found in nature might be called earthenware clay. These clays contain iron and other impurities insufficient quantities to cause the clay to become tight and hard-fired at about 950°C to 1100°C. In its raw state, such clay is red, brown, greenish, or grey, as a result of the presence of iron oxide. When fired, the colour changes may vary from pink to buff to tan, red, brown, or black, depending on the clay and the condition of the firing. Most of the pottery of this world over the years has been made of earthenware clay, and it is also the commonest raw material for brick, tile, drain tile, roof tile, and other heavy clay products.

2.5 Earthenware

Earthenware is glazed or unglazed material. Earthenware comprises all primitive pottery whatever the colour, all terra-cottas, and most building bricks. The Combined Nomenclature of the European Communities describes it as being made of selected clays, sometimes mixed with feldspars and varying amounts of other minerals and white or light-colored Combined Nomenclature of the European Union (1987). Earthenware can be strengthened by glazing. Glazing hardens the surface of the pottery, making it non-porous, thereby making it useful for drinking water, tea, and other liquid substances. Unglazed earthenware such as traditional pots is utilized for cooking purposes (Combined Nomenclature of the European Union 1987).

2.6 Clay Bodies

A clay body Rhodes (1968) defined as a mixture of clays or clays and other earthy mineral substances that are blended to form a specific ceramic purpose. Any clay found in nature serves very well just as they are. Clays can be dug out of the ground, kneaded with the right amount of water, and made into pottery without making any additions. Such clays might be called natural clay bodies. The potters, in pre-scientific times, relied largely on such clays for their raw materials and made their products with little or no additions to them.

Later, however, adjustments were made for better working properties. The demands which are made of clay as a material usually make it necessary to blend two or more materials in order to achieve the desired results. Such demands may be, for example, extreme plasticity to make the clay suitable for throwing, or complete density at a given firing temperature, or whiteness and translucency when fired, or the property of casting, as a fluid slip, or the development of certain desirable colours and textures. The author further states that, in order to arrive intelligently at suitable mixtures for a given use, one must understand the physical properties of clays and their response to firing, and also the physical and thermal properties of other materials used in clay bodies.

According to Opoku (2003), the discovery of industrial minerals by man has been of value in many ways since the dawn of history. The durability of pottery and ceramic-object made from the industrial minerals like clay, kaolin, feldspar, gypsum and the like and used by man has provided an index to our heritage; a mirror to our civilization with which the modern man has able to find out more about himself and his environment at any point in recorded history. The raw materials of ceramic glazes include: silica which is the main glass former, various metal oxides such as sodium potassium and calcium that act as fluxes to lower the melting temperature and Aluminium oxide often derived from clay which stiffens the molten glaze to prevent it from running off the piece. Colourants such as iron oxide (black, green and brown),

copper carbonate(green) or cobalt carbonate(blue) and sometimes opacifiers such as tin oxide(milky white colour) or zirconium oxide(brilliant colours and white) are used to modify the visual appearance of the fired glazes.

2.7 Glazes

A glaze is a layer of thin glass coating which is fused on a pottery body Daly (1995). The glaze provides a hygienic covering on pottery because it is smoother than the body it covers and it is non-porous. It is also decorative, providing colour, shine, and textural strength of the ware by the creation of a body-glaze layer Daly (1995). Also ceramic glaze is defined as an impervious layer or coating of a vitreous substance which has been fused to a ceramic body made through firing Wikipedia (n.d). A glaze is a mixture of various materials, which are ground into powder and mixed with water Rhodes (1968). The glaze on the ware serves as a colourant, decorative pigment, waterproof, and so on. It can also form a variety of surface finishing Wikipedia(n.d). A glaze is also defined as a continuous adherent layer of glass or glass and crystals on the surface of a ceramic body that is hard, non-absorbent, and easily cleared. Glazes can be thought of as a glass coating, either clear transparent or with the addition of a metal in any of several forms, in a rainbow of colours. They may also be glass or matte, translucent or opaque, smooth or even textured. The effects are limited depending on formulation, application and combinations Lakeside (n,d). Glazes are formulated as a mixture of ground, powdered ingredients which, by their nature, do not dissolve but are merely suspended in water Lakeside (n.d). Glaze is usually applied as a suspension of glaze-forming ingredients in water. After the glaze layer dries on the surface of the piece, it is fired, whereupon the ingredients melt to form a thin layer of glass. Typical glaze can be made to mature from 900°C to 1160°C, depending on the items on which they are applied. It is also observed by Otimayin (2015) that, alkaline glazes are glazes with alkaline flux as the main

constituent, such alkaline fluxes are Borax, Colemanite, and soda ash and so on. They have firing range to melt at the temperature range similar to that of lead between 900°C -1100°C.

According to Leach (1976), raw glazing not only saves time and money but importantly, enables one to make and glaze wares in a natural rhythm without the interruption of biscuit firing. Moreover, there are certain decorative processes which can only be used with raw glazing. Glazes on earthenware can be transparent and glossy after firing. Mankind must have realized the importance of minerals in early times. Thus that man used cinnabar ochre and red hematite as pigments long before smelting was devised. Opoku (2007) and Mathias (2015) affirmsto this claim, and further states that both cinnabar ochre and hematite were derived and used for body decorations.

2.8 Glaze Composition

Every glaze is made of the following materials:

- i. Silica- which creates glass. Examples are quartz, flint, pure silica.
 - ii. Alumina, – it stiffens the glazes so it does not slide off the clay. Examples of clay (kaolin, ball clay, or fireclay), alumina hydrate.
 - iii. Flux – this causes the glaze to melt at a low temperature enough to be used in ceramic. Examples: Feldspar, whiting plus a glaze may include one more additives.
 - iv. Opacities, - which make the glaze opaque instead of transparent. Examples: tin oxide, zirconium or zircopax, titanium, zinc.
 - v. Suspenders – they keep the glaze on suspension instead of settling out. Example: bentonite.
 - vi. Colourants – provide various colours. Examples: cobalt oxide, copper oxide.
- (Understanding Glazen.d)

2.9 Types of Earthenware glazes

The following types of earthenware glaze have been identified: Pottery (n.d)

- i. Lustre-a thin film of metallic salts that are reduced either by the medium that holds them when being painted on, or introduced in very low-temperature firing. It is usually applied to an already glazed surface.
- ii. Majolica -traditionally an opaque tin-based glaze with very colourful painted designs. It was first introduced on the island of Majorca in Spain and was later adopted in Italy. It was called Faience from Italian city where it thrived.
- iii. Overglaze and China Paint-ceramic enamel colours painted on a fired glaze surface and fired a second time (at a lower temperature than the first glaze firing, allowing bright red and orange that would burn out at a higher temperature.)
- iv. Raku-a firing process that usually includes a post-firing reduction to alter the soft glazes with an exaggerated crackle, metallic lustre, or opalescent effect.
- v. Terra Sigillata-a very soft slip glaze originated by the ancient Greeks.
- vi. Under glaze-oxides or commercial glazes or stains, painted under the glaze layer before firing.

Leach (1976) states that those types of glazes are composed of either natural stone or clay or a mixture of one or both with a small quantity of wood ash. Leach (1976), also notes that the addition of plastic contracting clay to any glaze recipes turns the glaze into a slip glaze.

2.10 Low-Temperature Glazes

Low-temperature glazes can be applied to any type of clay body, although they are generally used on red clay, earthenware, and stoneware. These glazes are divided into two groups; lead glazes and alkaline glazes. Low- temperature glaze has high amount of boron, lead, soda, or lithium fluxing agents Conrad (1980).

2.11 Production of Earthenware

According to Rhodes (1966), in formulating bodies for earthenware, the best plan is to rely largely on one natural clay. Most of the common red clays are fired to a fairly dense and hard state in the range from cone 06 to cone 1. The first step is to locate a good source of supply for such clay. Many ceramic supply stores sell common red clay which is serviceable for earthenware. However, the cost of such clay, especially if it will be freighted for a long distance, may make it advisable to look into local sources. If one is willing to work, red clay can be located and dug in most localities, a local brickyard or flower-pot factory may have excellent earthenware clay ready for use. The author notes further that, once the clay has been located, the next step is to test it to find out what its characteristics are. Tests for plasticity, shrinkage, and absorption should be made at the various temperatures. It will then be known what additions must be made to the clay to make it work well with the intended processes and firing temperature.

2.12 Correcting a Formulated Earthenware Body

The following procedures are used to correct earthenware body according to Rhodes (1967):

- i. If the clay is too refractory, that is, if it does not become hard enough at the temperature at which it must be fired, some flux must be added to it. This flux might be iron oxide, talc, or a frit.
- ii. If the clay is too fusible and becomes too dense at the intended firing temperature, refractory materials are added, such as kaolin, ball clay, stoneware clay, flint, fine grog, or fire clay.
- iii. If the clay is too sticky and shrinks too much, it will need the addition of more non-plastic material such as flint, kaolin, grog, or fire clay.
- iv. If the clay is mealy and not sufficiently plastic, it will need the addition of some more plastic materials such as ball clay or bentonite.

- v. If the colour of the clay is to be changed, iron or other colouring metallic oxides may be added.

2.13 Characteristics and Properties of Earthenware Clay:

Plasticity: When clay is wet with the proper amount of water, it will tend to hold any shape which is given to it. This property is known as plasticity. This is the property which has made possible the fabrication of the endlessly varied shapes of ceramic objects. Without a very high level of flexibility, the clay will not be workable on the wheel. Earthenware bodies exhibit higher plasticity than most white ware bodies and hence are easier to shape by ram press, roller-head or potter's wheel than bone china or porcelain Rhodes, (1957).

Porosity: Porosity is the amount of water fired clay can absorb earthenware, with water absorption of 5-8%, and is glazed to be watertight. **Strength and Durability:** Earthenware has lower mechanical strength than bone china, porcelain or stoneware, and consequently articles are commonly made in thicker cross-section, although they are still more easily chipped. They are often not durable for serving food.

Dark-coloured "terracotta" earthenware, typically orange or red, due to a comparatively high content of iron oxide is widely used for flower pots, tiles decorative and oven wares. It is used industrially for many products such as bricks and flower, and when glazed, it is no longer porous and is used for domestic wares.

2.14 Earthenware Glazes

They are glazes that vitrify below 1150° C and are usually smooth and shiny (Pottery n.d). They are mixed with products to make an opaque white glaze. According to Mussi (2010) this is known as a base-glaze and can be used for decoration in many different ways, forming very bright colours. Luster, Majolica on-glazing and slips are some of the earthenware glazes.

2.15 Fluxes for Earthenware

According to Arbuckle (n.d) fluxes are added to lower the melting point and help a body mature at the desired temperature. Flux is considered as a non-plastic addition to a clay body. With some clay, the addition of sufficient flux to make the body mature at low-fire temperatures will reduce plasticity and require the use of more ball clay. The author notes further that, Feldspars do not melt at a low temperature to be useful for low-fire fluxes. Nephelinesyenite, talc, frit, or combinations are used as low-fire body fluxes. Other higher-melting fluxes may be tested as supplementary body fluxes that may form eutectics with other body fluxes like whiting and strontium.

2.16 Frits

In view of Rhodes (1957) frits act as fluxes but care must be taken in selecting same as frits, as some are partially soluble and may deflocculate clays (making the body difficult to work) and andor accumulate soluble salts on the clay body during drying that will fuse during firing leaving areas of reduced absorption. The author further suggested countering the deflocculation with additions of 5% aluminium sulfate or magnesium sulfate (Epsom salts). Frit also tends to have a narrower melting range than other fluxes. Rhodes suggested is of the view that range may be within two cones vs. the range of a natural clay body of 4-5 cones.

According to (Rhodes 1957), Talc: Talc ($Mg_3Si_4O_{10}(OH)_2$) has a long firing range, but needs to be used in large amounts if it is the only flux, example , low-fire white wares bodies may be 60% ball clay, 40% talc for cone 05. This may produce a chalky, powdery body that is not very plastic but could be adjusted for casting. (Note: talc according to Arbuckle (n.d) works at low-fire temperatures because its refractory silica and magnesium are in proportions that form a eutectic that depresses the melting point).

Nephelinesyenite ($K_2O.3Na_2O.4Al_2O_3.9SiO_2$) in the view of the author, is similar to feldspar, but with a higher ratio of flux to alumina and silica so that it melts at a lower temperature than spar. Talc in be partially soluble and deflocculates the clay body, leading to working and drying problems.

2.17 Fluxes used in Low-Temperature Glazes

According to Peterson (2016), Boron is the commonly used low –fire flux, other than lead. Boron fluxes include; the following;-

Gerstley Borate; no longer mined, but some limited amount is still available. Synthetics replacement is available from many suppliers. Example of synthetic are as follows; Gillespie Borate –a synthetic product produced by Hammill’s and Gillespie of New Jersey.

Cadycal, and Murrays Borate, Laguna Borate, Frit CC-298C, Frit 439, UlexEmpresaMinera, Imco Borate, Ulexite mention but few.

Colemanite

According to (Peterson 2016)Boron; often used in raku glazes and smooth out higher firing temperature.

Boron-containing frit such as Ferro 3110,324, and 334.

Talc; used as a flux in low-temperature clay, clay bodies and as a flux in both low and high-fire glaze.

2.18 Firing

It is important for the wares to be subjected to heat by means of firing. Zauro (2007) asserts that heat is necessary for the development of a vitreous bond. Normally, the ceramic body undergoes two types of firings. The first firing stage is known as biscuit or bisque a name

is attached to a once fired ceramic body which is not glazed. Biscuit firing refers to the body which has had only one firing unglazed. The second phase of firing is called gloss or glaze firing. It is a stage in which the glaze is fired onto the wares. The firing commences slowly and the temperature in the kiln gradually rises to attain the derived temperature (Zauro 2007).

2.19 Conclusion

From the review of literature made, the historical background affirms the benefits of developing low- temperature glazes for the ceramist using locally sourced materials from Benue just as it is in several locations.

CHAPTER THREE

METHODOLOGY

3.1 Materials and Methods

Research methodology as stated by Sullayman (2006) is an outline, scheme, arrangement or order suitable for research based on the study parameter or nature of materials or research topic under investigation. The experimental research design was adopted for this study. The following steps were followed: Sourcing of raw materials, beneficiation, and chemical analysis of the raw materials blends composition, glaze formulation, firing and property tests. A qualitative research design was used with studio base led process of ceramic production and glaze formulation.

3.1.1 Sourcing of raw materials

Dogo Red Clay, Groundnut Shells, and Locust Beans Wood were used for this study. The samples were collected from the three Local Government Areas namely: Buruku (plate I – II), Kwande (plate III – IV) and Vandeikya (plate V – VI) of Benue State. The samples were transported to the Ceramic Section, Department of Industrial Design, Ahmadu Bello University Zaria December (2016).



Plate 1: Sourcing of Dogo Red Clay, Buruku
Source: Research Photograph, Tser (2017)



Plate 1I: Sample of Dogo Clay
Source: Research Photograph, Tser (2017)



Plate 1III: Locust Beans Tree, at Vandeika
Source: Research Photograph, Tser (2017)



Plate 1V: Sample of Locust Beans Log of Wood
Source: Research Photograph, Tser (2017)



PlateV: Sample Groundnut Shells, Kwande
Source: Research Photograph, Tser (2017)



Plate VI: Drying of Groundnut Shells Ash
Source: Research Photograph, Tser (2017)

3.2 Population of the Research

The entire populations of Samples of ceramic Tile produce were twenty two (22) in number. Samples Tile Nos 6 and 7 were chosen to produces sixteen (16) wares, which out Of 22, twelve 12 were ceramic decorative pieces and twelve 12 were ceramic sculptures were produced.

3.3 Beneficiation of the raw materials

The raw materials were processed through Drying, Burning,sieving.

3.3.1 Dogo Red Clay

The Dogo Red Clay was soaked in a container for two days. This was followed by blend the clay, sieved through 200 (75um) (Plate VII) and dewatered the slurry in the plaster mould. After the water was removed, it was allowed to dry (Plate VIII) and then crushed into a powdered form.



Plate VII: Sieving Process of Dogo Red Clay
Source: Research Photograph, Tser (2017)



Plate VIII: Drying Process of Dogo Red Clay
Source: Research Photograph, Tser (2017)

3.3.2 Groundnut Shells Ash

The sourced Groundnut Shells washed to remove the soil particles attached to the shells and then dried under the sun. The dried shells was heaped in an enclosure (see Plate IX) where it will not be easily blown away by wind due to its light weight and then set on fire in the open air to burn into ash. The ashes collected were soaked in a container for 10 days Sieved through 200 mesh (75 μ m) (Plate X) and dewatered the slurry in the plaster mould. After the water was removed, it was allowed to dried and then crushed into a powdered form.



Plate IX: Burning Process of Groundnut Shells
Source Research Photograph, Tser (2017)



Plate X: Sieving Process of Groundnut Shells Ash
Source: Research Photograph, Tser (2017)

3.3.3 Locusts Beans Wood Ash

The sourced Locust Beans Wood was dried and heaped in an enclosure, before burned down into ash form in the open air (Plate XI). The ash collected was soaked in a container for 10 days. The soaked Locust beans wood ash was then sieved through 200 (75 μ m) (Plate XII) and dewatered the slurry in the plaster mould. After the water was removed, it was allowed to dry and then crushed into a powdered form.



Plate XI: Burning Process of Locust Beans Wood
Source: Research Photograph, Tser (2017)



Plate XII: Sieving Process of Locust Bean Wood Ash
Source: Research Photograph, Tser (2017)

3.4 Chemical analysis of a sample

The chemical analysis of the samples was done using X-Ray Fluorescence Spectrometer to determine the chemical elements present in the samples so as to ascertain their suitability as a ceramic material for low-temperature glazes production.

3.5 Formulation of Low-Temperature Glazes using Tri-axial Blend

The tri-axial blend method was adopted for blending the three materials utilised to formulate low – temperature glazes. These materials are Dogo red clay, Groundnut shells and Locust beans wood ash. It is observed by Pitelka (2001) in Satsi (2016) that, tri-axial blend is a method for testing three– way combinations of glaze or materials, where proportional amounts vary through a series of quantitative allotment of materials between three limits.

It may involve change in glaze materials, or addition of colorants. The tri-axial is one of the two methods used in the formation of low – temperature glazes. The tri-axial blend usually consists of a 21, 36, and 66 blend members. The 36 blend members was adopted from (Zauro 2007) for the study. The chart was drawn and the three materials in various rations were inserted. The first figure in each rectangle represents Dogo Red Clay, the second is Groundnut Shells and the last is Locusts Beans Wood Ash (fig 1 below). That made up the chart, small quantities were physically mixed. The samples were fired to 900°C, 1000°C, 1100°C and 1160°C in the electric kiln available at the Department of Chemical Engineering, Ahmadu University, Zaria to determine their suitability as low – temperature glazes(plates XIII- XVI). Researchers who successful used the tri-axial blend method are(Okoruwa 1986) in Zauro (2007). In the study, Zauro mentioned that suitable body formulation can be achieved through step by step substitution of one ingredient by another which goes on until a desired outcome is accomplished.

Dogo Red Clay (A)

													(1)													
													80													
													10													
													10													
													(2)		(3)											
													70		70											
													20		10											
													10		20											
													(4)		(5)		(6)									
													60		60		60									
													30		20		10									
													10		20		30									
													(7)		(8)		(9)		(10)							
													50		50		50		50							
													40		30		20		10							
													10		20		30		40							
													(11)		(12)		(13)		(14)		(15)					
													40		40		40		40		40					
													50		40		30		20		10					
													10		20		30		40		50					
													(16)		(17)		(18)		(19)		(20)		(21)			
													30		30		30		30		30		30			
													60		50		40		40		20		10			
													10		20		30		40		50		60			
													(22)		(23)		(24)		(25)		(26)		(27)		(28)	
													20		20		20		20		20		20		20	
													70		60		50		40		30		20		10	
													10		20		30		40		50		60		70	
(29)		(30)		(31)		(32)		(33)		(34)		(35)		(36)												
10		10		10		10		10		10		10		10												
80		70		60		50		40		30		20		10												
10		20		30		40		50		60		70		80												

(B)

(C)

Groundnut Shells Ash

Locust Beans Ash

Fig 1: Tri-axial Test Blends of 36- Member Materials

Source: Okoruwa in Zauro (2007).

3.5.1 Formulation of Low-Temperature Glazes Using Line Blend

The second method of glaze formulation was line blend, was used in measuring two or more ceramic materials. Rhodes (1968) states that, some beautiful glazes may result from the addition of single colouring oxides to a glaze, the most exciting glaze colours are usually the result of addition of two or more colorants to the base glaze. Such addition may be arrived at by methodical blending, or by adding combinations and quantities of oxides which are known to be congenial and promise to give good results. The testing usually proceed on the spirit of experimentation, by adding various materials to the glazes just to see what will happen at the end. In methodical blending, the ceramist may anticipate that a certain percentage of his test will prove to be unsatisfactory and will only produce muddy and unpromising colours. According to Harmer (2004) line blend is a method of determining requirements in practical test of materials. In this case the obvious way is to start with the material and add increasing amount of another material. Any convenient number of tests can be decided upon. It is also usual, especially with glazes, to decrease one test ingredient as the other increases. In this way the total bulk remains constant and calculations in percentages are more easily composed.

The line blend of 11 members was adopted from (Rhodes 1968) for the study. The chart was drawn and the two materials in various rations were inserted. The table represents Dogo Red Clay and Borax (table 1 below), that made up the chart, and small quantities were physically mixed. The samples were fired to 900°C, 1000°C, 1100°C and 1160°C in the electric kiln available at the Department of Chemical Engineering, Ahmadu University, Zaria to determine their suitability as low – temperature glazes. (Plates XVII –XX), then two test pieces were selected for the formulation of low – temperature glazes they are number six and seven of the samples.

Table: 3.1 Line Blend Test 11 membersChars.

Samples	1	2	3	4	5	6	7	8	9	10	11
Borax	0	10	20	30	40	50	60	70	80	90	100
Dogo	100	90	80	70	60	50	40	30	20	10	0
Red Clay											

Source: Research, Tser(2017)

3.5.2 Production of Test Bars

Glaze compositions were measured for each of these blends and tested on biscuit fired test slabs. The test slabs were fired at 900°C using the Electric Kiln available at the Department of Chemical Engineering, Ahmadu Bello University, Zaria.

3.6 Determination of Maturing Temperature

The biscuit fired test tiles were glazed and fired to 900°C, 1000°C 1100°C and 1160°C so as to determine the suitable blends for low-temperature glaze production.

3.7 Property Test

The following tests were carried out according to ASTM-C126-13, ASTM-424 and ISO 10545-6 standard to determine the properties of the low-temperature glazes that were developed by this study.

- a. Crazeing Test
- b. Imperviousness Test
- c. Hardness and Abrasion Resistance Test

3.7.1 Craziing Test (C424)

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

3.7.1.1 Imperviousness Test (C126-13)

The imperviousness test was done by the application of water on the sample for 10 minutes, was made and recorded.

3.7.1.2 Hardness and Abrasion Resistance Test (ISO10545-6)

Successfully glazed surfaces were scratched using ordinary glass or steel and observations were made and recorded.

3.8 Preparation of Slip for Casting

Two clay bodies were prepared, the first one was made up of 40 % ball clay and 60 % kaolin as used in the studio, but 2% sodium silicate was added. The second body had 30% ball clay and 70% Kaolin with an addition of 2% Sodium Silicate. The addition of sodium silicate was necessary so as to avoid slip setting. This serves as a diffloculant, as the process of casting required a fluid suspension of clay in water, which will flow readily but will not settle in the mould. Rhodes (1968) states that, ‘casting then would not be a practical way of making pot unless there were some ways of cutting down on the amount of water required to make a fluid slip’. The process used to achieve this result is known as deflocculation. When clay and water are mixed together to form a slip, they are said to be in a flocculent condition. In order to decrease the amount of water needed in the clay slip, it is necessary to disperse the clay particles, to break up the flocks so that each particle of clay floats by itself.

CHAPTER FOUR

4.0 RESULT AND DISCUSSION

In this chapter, results of the experiments carried out are analysed and discussed. Also various stages conducted were documented.

4.1 Chemical Analysis of Dogo Red Clay, Groundnut Shells Ashes, Locust Beans Ashes Rafin - Kukura and Kankara.

Table 4 .1: Results of chemical Analysis on Dogo Red Clay, Groundnut Shells Ash, Locust Beans Ash, Rafin-tukurwa and Kankara

Element	Dogo Red Clay (wt %)	Groundnut Shells Ash (wt %)	Locust Beans Ash (wt %)	Rafin-tukurwa (wt %)	Kankara(wt%)
Fe ₂ O ₅	39.70	-----	-----	-----	0.00
SiO ₂	27.40	-----	-----	49.00	44.56
Al ₂ O ₃	12.00	-----	-----	17.85	31.45
Fe ₂ O ₃	-----	2.003	0.023	10.34	1.07
K ₂ O	1.10	17.20	20.59	3.50	0.30
TiO ₂	1.26	2.287	0.18	4.21	0.14
Na ₂ O	0.06	ND	ND	2.54	0.17
CaO	0.55	16.19	66.87	1.21	0.28
MgO	0.003	-----	-----	0.14	0.87
V ₂ O ₅	0.047	-----	-----	0.20	-----
MnO	-----	0.47	0.22	0.10	-----
MnO ₂	0.001	-----	-----	-----	0.01
Cr ₂ C ₃	0.042	-----	-----	0.025	-----
ZnO	0.058	0.117	ND	0.055	0.00
CuO	0.081	0.067	0.023	0.089	0.00
Rb ₂ O	0.074	-----	-----	-----	0.00
ZrO ₂	0.28	0.243	0.011	0.09	-----
BaO	0.320	0.100	0.59	0.62	-----
Eu ₂ O ₃	0.38	-----	-----	-----	-----
Re ₂ O ₇	0.04	-----	-----	-----	-----
PbO	0.58	-----	-----	-----	-----
Pb ₂ O ₃	-----	-----	-----	-----	0.00
HfO ₂	-----	-----	-----	0.25	-----
SrO	-----	0.142	0.72	0.20	-----
Y ₂ O ₃	-----	0.007	ND	0.067	-----
AS ₂ O ₃	-----	-----	-----	0.001	-----
SO ₄	-----	-----	-----	-----	0.05
CeO ₂	-----	0.0049	ND	-----	-----
P ₂ O ₅	-----	ND	1.583	-----	-----
SO ₃	-----	1.60	1.775	-----	-----
SiO ₂	-----	59.69	7.24	-----	-----
L.O.1	16.02	0.12	1.23	8.21	20.21

Source: National Geosciences Research Laboratory (NGRL) Aernyi 2016, Tser 2017 and Zauro2007

From the result of the chemical analysis Table 4.1 above, it shows that the major element found in Dogo Red Clay is iron oxide which acts as a flux in both bodies and glazes at high temperatures. Once it reduces, (Fe_2O_3 39.70) begins fluxing and forms a glass. Dogo Red Clay also contains silica (SiO_2 27.40) which is a major glass former. It also contains alumina oxide (Al_2O_3 12.00) which stiffens glazes so that it does not slide off the wares during firing. Groundnut Shell Ash and Locust Beans Ash contain potassium oxide (K_2O), calcium oxide (CaO) which acts as a flux to lower the melting temperatures from 1200°C down to 900°C . Rafin-Tukurwa which is (Ball Clay) and Kankara (Kaolin) contains silica (SiO_2) which is a major glass former and alumina oxide (Al_2O_3) which stiffens the glaze so that it does not slide off the wares.

4.2 Result of Body Formulation

A 900°C Body A (70% kaolin and 30% ball clay) came out glossy with a little deformity from the glaze because it did not adhere well to the ware thereby forming a matty effect. For Body B (60% kaolin and 40% ball clay) after being fired to about 900°C , the glaze appeared without any form of deformity. The glaze adhered suitably to the body without any form of crazing or running effect. The glaze came out beautiful and attractive.

4.3 Calculation for the Low-Temperature Glaze Development

Table 4.2: Triaxial Blend Test (36) Of Materials of the Study in Percentages 10-100 (%)

1 D = 80% G = 10% L = 10%	2 D = 70% G = 20% L = 10%	3 D = 70% G = 10% L = 20%	4 D = 60% G = 30% L = 10%	5 D = 60% G = 20% L = 20%	6 D = 60% G = 10% L = 30%
7 D = 50% G = 40% L = 10%	8 D = 50% G = 30% L = 20%	9 D = 50% G = 20% L = 30%	10 D = 50% G = 10% L = 40%	11 D = 10% G = 50% L = 40%	12 D = 40% G = 40% L = 20%
13 D = 40% G = 30% L = 30%	14 D = 40% G = 20% L = 40%	15 D = 40% G = 10% L = 50%	16 D = 30% G = 60% L = 10%	17 D = 30% G = 50% L = 20%	18 D = 30% G = 40% L = 30%
19 D = 30% G = 30% L = 40%	20 D = 30% G = 20% L = 50%	21 D = 30% G = 10% L = 60%	22 D = 20% G = 70% L = 10%	23 D = 20% G = 60% L = 20%	24 D = 20% G = 50% L = 30%
25 D = 20% G = 40% L = 40%	26 D = 20% G = 30% L = 50%	27 D = 20% G = 20% L = 60%	28 D = 20% G = 10% L = 70%	29 D = 10% G = 80% L = 10%	30 D = 10% G = 70% L = 20%
31 D = 10% G = 60% L = 30%	32 D = 10% G = 50% L = 40%	33 D = 10% G = 40% L = 50%	34 D = 10% G = 30% L = 60%	35 D = 10% G = 20% L = 70%	36 D = 10% G = 10% L = 80%

Source: Tser(2017) Batches of materials for glaze formulation were made based on unity formula. The key below:-

Key D - DogoRed Clay

G- Groundnut Shells

L - Locust Beans Ash

4.4 Result of Tri-axial Blend Test of 36 Members Fired at 900°C

The tri-axial blend test was fired at 900°C. Blend sample of Tiles Nos 1-5 did not fuse due to the quantity of Dogo Red Clay which was 80%, Locust Beans Wood Ash 10% and Groundnut Shells Ash 10%. Sample Tiles Nos 6 – 10 was 60% Dogo Red Clay, 20% Locust Beans Wood Ash and 20% Groundnut Shells Ash, the sample Tiles have the same characteristics; they did not fuse due to the high contents of the material Dogo Red Clay 60% which dominated the batches. Sample Tiles Nos 11 – 15 possessed the same characteristics and their colours are the same. Sample Tiles Nos. 16 – 29 had the effect because of the same percentages of 50%, 60%, 70% in the Sample tiles Nos. 30 – 35 behave the same way on their colours and the characteristics are with dark spot, not glossy and not shiny. Sample Tile No 36 did not fused but flacked off as a result of the percentage 80% Locust Beans Wood Ash, 10% Groundnut Shells Ash and 10% Dog Red Clay.



Plate XIII: Tri-axial Blend Test at 900⁰ C Fired
Source: ResearchPhotograph,Tser, (2017).

4.5 Result of Tri-axial Test Blend of 36 Fired at 1000°C

TestBlendsamples Tiles Nos. 1, 2, 3, 4 and 5 are similar of quantity, they had Ash and no Shiny effect surface. Sample tiles Nos. 6-10 of this study they had similar features. Their colours are dark and mattly because they were dominated with Locust Beans Wood Ash and Groundnut Shells Ash. Samples Nos 11-20 had similar characteristics because of the high quantum of Locust Beans Wood Ash and Groundnut shells ash; which are of high melting temperature on the account of alkaline present. While the Dogo clay is a low melting temperature, the two ashes dominance made the body to be under fired. Similarly, samples Nos 21-36 also behave as same as Nos 11-20 because of the obvious dominance of the two ashes from Locust Beans Wood and Groundnut shell. This also did not mature but under fired.



Plate XIV: Tri-axial Blend Test fired at 1000°C
Fired
Source: ResearchPhotograph, Tser, (2017).

4.6 Results of Tri-axial Blend Test of 36 Fired at 1100°C

Tested blend sample Tile Nos. 1- 5 adhered to the Tiles and were found un-useable. The Tiles are of the same characteristics; their colours were same and were without any shine. Samples Tiles Nos 11-15 also behave same as Nos 1- 5. These maintain the same colour when it was applied raw on the tile before firing. Sample Tiles Nos. 16 - 20 were dark in colour without any shine; which was as a result of the predominance of Locust Beans Wood Ash and Groundnut shells Ash. Sample Tiles Nos. 21 – 25 in their category also adhere to the test Tiles, but could not give a good effect of colours. Sample Tiles Nos. 25 – 30 also stock to the Tiles and showed nothing different in terms of colour effect and other properties. Sample Tiles Nos. 31 – 36 did not show anything different, but were full of spots.



Plate XV: Tri-axial Blend Test at 1100⁰ C Fired
Source:Research Photograph, Tser, (2017).

4.7 Results of Tri-axial Blend Test of 36 Fired at 1160°C

Tested Blend samples TileNos 1 – 6 did not fuse to the test Tiles, Sample Tiles Nos. 7 – 10 were completely melted to not have glossy effects, while Nos 11 – 15 were made black and ash in nature. The sample Tiles Nos 16 – 21 which had Groundnut Shells Ash, Locust Beans Wood Ash, Dogo Red Clay dominated fused, with ash black colours, while sample Tiles Nos 22 – 36 did not fuse and flacked off the test Tiles.



Plate XVI: Tri-axial Blend Test at 1160⁰ C Fired
Source:Research Photograph, Tser, (2017).

4.8 Results of Line Blend Test of 11 (Eleven) Members at 900°C

Line Blend Test Sample of Nos 1 and 2 were flaked off from the Tile due to the high quantity of Dogo Red Clay. Sample Tile No 3 was fused with spots due to the same features, while samples Tile No 4 -5 were fused but not glossy. Samples Nos 6 and 7 fused with glossy effect of black and some golden shiny brown and attractive effect. Sample Tile No 8 came out with a golden brown, orange and black, while Nos 9 to 11 had yellow, orange, lemon green and gloss with shiny colour.



Plate XVII: Line Blend Test 11 member at 900⁰ C
Source: Research Photograph, Tser, (2017).

4.9 Results of Test Line Blend of 11 Members at 1000°C

Line blend Test samples Nos 1 and 2 was flaked off, Sample Tile No 3 was fused with black colour but some parts still peeled off, while sample Tiles Nos 4 – 5 were did not mature. Sample Tiles Nos 6 and 7 came out black with golden, gloss and shiny colour and matured well without craze effect but attractive in colour. Sample Tile No 8 was fused with golden brown and orange colour, while Nos 9-11 had yellow, orange, lemon green and gloss with shiny colour.



Plate XVIII Line Blend Test 11 member at 1000°C
Source: Research Photograph, Tser, (2017)

4.10 Results of Test Line Blends of 11 Members at 1100°C

Line Blend Test Sample Tile Nos 1 and 2 were flacked off while sample Tile No 3 was fused but some parts still peed off, sample tiles Nos 4 and 5 not mature. Sample Tile Nos 6 and 7 fused with golden shiny brown, glossy and attractive colour. It did not craze while sample Tile No 8 came out with golden brown colour, orange and black. Sample Tile Nos 9 to 11 had yellow, orange, lemon green but had crazing effect.



Plate XIX: Line Blend Test 11 member at 1100° C
Source: Research Photograph, Tser, (2017).

4.11 Results of Test Line Blend of 11 (Eleven) Members at 1160°C

Line Blend Test Samples Tile Nos 1 and 2 flacked off, while sample tile Nos 3 was fused but still removed some part off, samples Tile Nos 4 and 5 was fused but it did not matured. Sample Tile Nos 6 fused with black and shiny with gold dish colour while sample Tile Nos 7 fused, gloss and shiny. Sample Tile No 8 was fused with yellow, orange colour but was

crazewhile samples Tile Nos 9 -11 has yellow, lemon green and some brown colour on it but was still craze.



Plate XX: Line Blend Test 11 member at 1160⁰ C
Source: Research Photograph, Tser, (2017).

4.12 PlasterMould Making and Production of Wares

The researcher made sketches and designed decorative wares as the model for the formulation of mould. Clay model was produced thereafter, the seam line marked and then the already existing model was placed on a ceramic work table. Cottle was built around the model to prevent the liquid mixture of Plaster of Paris from flowing away. The separator was applied to both the body of the model and the cottle to make the separation of the pieces easy. Water was measured to mix with the plaster of Paris in a ratio of 100:130 as asserted by Sullayman (1999). The total quantity of water and plaster of Paris which was 10kg water and 13kg Plaster of Paris were mixed in two deference parts. Then all the molds were taken in two different pieces. The first mold was taken into two different parts. The first was of measured 4kg water and 5.20kg Plaster of Paris, and waited for 2 hours before taking the second piece which was measured the same amount of 4kg water and 5.20kg plaster of Paris. Then the viscous plaster was poured over the designed clay model, and allowed to dry at ambient temperature for about two weeks before the commencement of slip casting of the samples.

Casting method was adopted for the production of the wares. The two-piece cast mould were coupled and held together with firm rubber bands, and then prepared slip was poured into the two pieces mould. It was allowed to absorb the water for 5 minutes at that stage. The researcher was observing the slip thickness inside of the mould to make sure it replicates the shape side, before it was allowed to stay in the mould for four hours before de-mould and it was de-molded and removed from the mould and left on the table for three hours before they were covered with polythene bag for 7 days. At the leather stage, seam lines on each cast were eliminated and wares were allowed to dry for 14 days under room temperatures before the green-wares were further fettled out, ready for bisque firing as shown in (plates XXI to plate XXX).



Plate XXI: Pouring Slip inside the Mould
Source: Research Photograph, Tser, (2017).

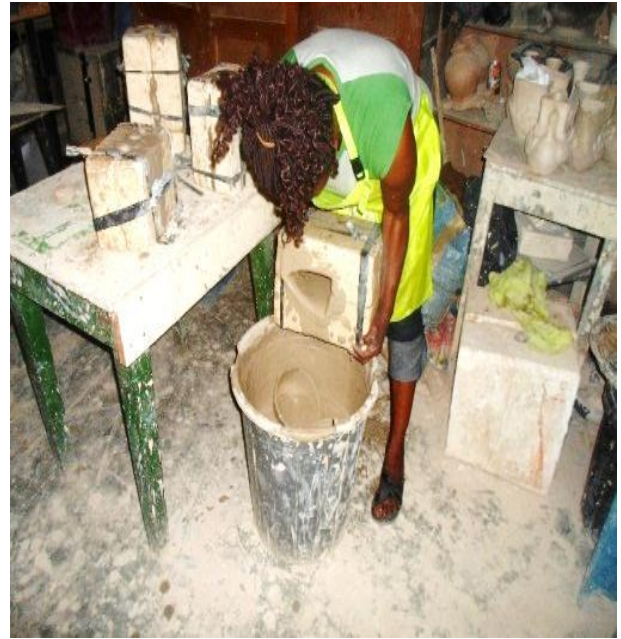


Plate XXII: Poured out Excess Slip
Source: Research Photograph, Tser, (2017).



Plate XXIII: Cast Work in the Mould
Source: Research Photograph, Tser, (2017).



Plate XXIV: Cast Work in the Mould
Source: Research Photograph, Tser, (2017).



Plate XXV: Cast Work in the Mould
Source: Research Photograph, Tser, (2017).



Plate XXVI: Cast Work Removed out of the Mould
Source:Research Photograph, Tser, (2017).



Plate XXVII: Bone Dry Cast Work
Source: Research Photograph, Tser, (2017).



Plate XXVIII: Leather Hard Cast Work
Source:Research Photograph, Tser, (2017).



Plate XXIX: Bisque Fired Wares
Source: Research Photograph, Tser, (2017).



Plate XXX: Leather Hard Cast Work
Source: Research Photograph, Tser, (2017).

4.13 Procedures Used for Measurement of Low-Temperature Glaze Materials

The instrument used for measuring the low – temperature glazes are shown in (Plates XXXI to XXXIV) below:



Plate XXXI: Measuring of Barax Using Digital Scale
Source: Research Photograph, Tser, (2017).



Plate XXXII: Measuring of Barax Using Analogue Scale
Source: Research Photograph, Tser, (2017).



Plate XXXIII: Measuring of Dogo Red Clay Using Digital Scale
Source: Research Photograph, Tser, (2017).



Plate XXXIV: Measuring of Dogo Red Clay Using Analogue Scale
Source: Research Photograph, Tser, (2017).

4.14 Result of Glaze Formulation

The mixing of materials for glazes formulation was carried out through the processes shown in (plates XXXV-XXXVIII) below. This enabled the researcher to obtain the result of glazes formulation usually; glazes are prepared by mixing them with water to form a fluid slip. Intimate intermingling of the various materials of the glaze helps promote melting, and this is necessary for materials for glaze to be thoroughly mixed. The dry glaze and sufficient water are mixed and put in the jar and the cover is secured in place. The jar is then placed on the device which rotates it, and it is allowed to grind for about an hour. In the ball mill the action of the pebbles falling over one another thoroughly mixed and grinds the glazes slip. Rhodes (1959) is of the view that, mixing and grinding can be accomplished on the ball mill.

Glaze materials are used in the finely grounded powdered form. The materials used for this research were milled to pass through the 200-mesh screen that is about 75-micron size.

Glazematerials were developed from the raw materials using line blend of nine (11) member samples which are Dogo Red Clay and Borax. The developed glazes started from 10% Borax to 100% Dogo Red Clay. As 10% Borax increases, 10% Dogo Red Clay decreases. From the test glaze tiles, number 6 which is 50% Borax and 50% Dogo Red Clay, and number 7 which is 60% Borax and 40% Dogo Red Clay was chosen and used for the development of low-temperature glazes. It was observed that the glaze sample on the Test Tiles of numbers 6 and 7 did not craze on the test tiles and on the body of the produces wares,(plates XVII to XX) above.



Plate XXXV: Mixing of Glaze
Source:Research Photograph, Tser, (2017).



Plate XXXVI: Pouring of Mixed Glaze in Ball Mill
Source:Research Photograph, Tser, (2017).



Plate XXXII: Fixing of Ball Mill
Source:Research Photograph, Tser, (2017).



Plate XXXIII: Starting the Ball Mill to mixes the materials for low-temperature glazes
Source:Research Photograph, Tser, (2017).

4.15 Glaze Application

A glaze may be applied on ceramic wares using different methods. Some of the applications involve the suspension of glaze materials in liquid form. They are applied wet to allow the glaze particle adhere on to the surface of the ware. Otimeyin (2015) states that,glazing could be dipping method, spraying method, pouring method, brushing method, waterfall and many others. In this study, the researcher used only four methods which are discussed below:

1. Dipping method: This is when the ceramic ware is immersed or dipped in the glaze suspension stored kept in a suitable container, then removed and shaken off. This method ensures good coating and also the fastest way of glaze application because the consistency of the glaze is thinner, which allows the pot longer time to absorb the glaze(PlateX XXIX).
2. Spraying method: This is a conventional method of glaze application. It is usually done by using a spraying gun or airbrush. This involves filling a thinner

consistency of a glaze and spraying on the ware. This method is a better option especially for items that cannot easily be dipped or poured. In other words, spray glazing has the advantage of using a very little reserve of glaze. An average-size piece can be easily glazed with a cupful of glaze(Plate X LII).

3. Brushing: With this method, a paintbrush can be used in applying the glazes on pottery. This method needs special skills and attention to obtain an even coat because more time is wasted in allowing each coat to dry before applying the next coat in the event where more than one coat is needed. The consistency of the glaze at this stage is always thicker than in dipping and pouring, and can also determine the intensity of the colour(Plate X LI).
4. Pouring: In this fast method, glazes can be quickly poured into a pot, left for some seconds and then quickly returned to the glaze container. A thinner coat of glaze can also be poured quickly over the outer surface of the pot(Plate XL).



Plate XXXIX: Dipping Method



Plate XL: Pouring Method
Source: Studio Research, Tser, (2017).



Plate X LI: Brushing Method
Source:Research Photograph, Tser, (2017).



Plate XLII: Spraying Method
Source: Studio Research, Tser, (2017).

4.16 Result of Firing

The produced wares were first bisque fired at a temperature of 800°C(see plate XXIX and XLIII). At that temperature, the works became hard, white and porous. After bisque firing, the prepared glaze was applied before the final firing at different temperatures of 900°C, 1000°C, 1100°C and 1160° C respectively. At this stage, the fired waves became hard, glossy, some reddish, black, yellowish brown, dark brown, dark brown with gold and purple colour(see plates XLV –XLVII).



Plate XLIII: Loading Process of Glaze Firing
Source:Research Photograph, Tser, (2017).



Plate XLIV: Finished Glazed Work at 900° C
Source: Research Photograph, Tser, (2017).



Plate XLV: Finished Glazed Work at 1000° C
Source: Research Photograph, Tser, (2017).

Plate XLVI: Finished Glazed Work at 1100° C
Source: Research Photograph, Tser, (2017).



Plate XLVII: Finished Glazed Work at 1160° C
Source: Research, Tser, (2017).

4.17 Findings

From the studio based experimental study conducted, the followings findings were made:

1. The chemical analysis carried out shows that the major element found in Dogo Red Clay is iron oxide (Fe_2O_3 , 39.70w %) which acts as a flux in clay bodies and glazes at high temperature. Groundnut Shells Ash has potassium oxide (K_2O 17.20w %), calcium oxide (CaO 16, 19w %) and Locust Beans Wood Ash contains potassium oxide (K_2O 20.59w %) and calcium oxide (CaO 66.87w %) which acts as a flux to lower the melting temperatures of the glazes from 1200°C down to 900°C.
2. The found out that the Dogo Red Clay, Groundnut Shells Ash and Locust Beans Wood Ash through line blend at 900%, 1000%, 1100% and 1160% was successful for use on ceramic wares as low temperature glazes.
3. The low-temperature glazes formulated from locally sourced raw materials analyzed, showed that the tri-axial blend tested failed to meet the desire to produce low-temperature glazes. Rather, the Line blend was successful in achieving low-temperature glazes.
4. The line blend at 900°C, the body A (70% kaolin and 30% ball clay), came out glossy with a little deformity from the glaze because it did not adhere well, thereby forming a matt effect. For Body B (60% kaolin and 40% ball clay), after being fired to about 900°C the glaze appeared without any deformity.
5. The study found out that, there is a similarity in the temperature range of between 900°C to 1100°C indicating varying shades of black, dark brown, yellow, lemon green; but at 1160°C, the reddish and purple colour were found.
6. The study found out that at 900°C the glaze could not adhere to body A comprising B, (30% ball clay+70% kaolin) but was suitable on body B, (Ball clay 40%+kaolin 60%) at 900°C to 1160°C respectively.

7. The gloss firing using electric kiln showed the colour shade effect on the tested tiles from 900°C to 1160°C.
8. The results proved the achievement of developed glazes since it can mature at 1000°C to 1160°C thus they can be classified as low-temperature glazes.
9. The study found out that the low-temperature glazes was not crazing.
10. The imperviousness test of the glazed surfaces was successful without water absorption.
11. The study found out that the hardness and Abrasion Resistance tested achievement was successful on low-temperature glazes.

CHAPTER FIVE

5.0 SUMMARY, CONCLUSION AND RECOMMENDATIONS

5.1 Summary

The study set out to develop low-temperature glazes using locally sourced raw materials from Benue State of Nigeria. The aim of the study was to formulate low-temperature glazes, while the specific objective were to; identity and source for ceramic raw materials; to carry out chemical analysis of the selected raw materials; to formulate low- temperature glazes using locally soured materials for ceramic production; test and evaluate the effect of the formulated glazes on ceramic wares.

The locally sourced materials include; Dogo red clay, Groundnut shells and Locust beans wood ash. The literature review was premised on earthenware clays, clay bodies, glazes, production of earthenware glazes among other topics. The method utilized involved using an experimental research design. Among other findings, the results proved the achievement of developed glazes since it can mature at 1000°C to 1160°C and they can be classified as low-temperature glazes. The imperviousness of the glazed surfaces was successful without water absorption. The chemical analysis carried out shows that the major element found in Dogo Red Clay is iron oxide (Fe_2O_3 , 39.70w %) which acts as a flux in clay bodies and glazes at high temperature. Groundnut Shells Ash has potassium oxide (K_2O 17.20w %), calcium oxide (CaO 16.19w %) and Locust Beans Wood Ash contain potassium oxide (K_2O 20.59w %) and calcium oxide (CaO 66.87w %) which acts as a flux to lower the melting temperatures of the glazes from 1200°C down to 900°C. The low- temperature glazes formulated from locally sourced raw materials analyzed, showed that the tri-axial blend tested failed to meet the desire to produce low- temperature glaze. Rather, the Line blend was successful in achieving low-temperature glazes.

5.2 Conclusion

The aim and objectives of this research were set to evolve a low- temperature glaze premised on relevant experimental research design using locally sourced materials. This is with the aim of producing low-temperature glazes that will benefit the ceramist and industries. The result of the study has been the possibility of making this research become a model for the productions of low-temperature glazes.

The development of glazes starting from 10% addition of Borax increases 10% decreases Dogo red clay. From the test glaze tiles, number 6 which is 50% Borax and 50% Dogo red clay and number 7 which is 60% Borax and 40% Dogo red clay which has the most differentials was chosen and used for the development of low-temperature glazes. Test results indicated that glaze sample on the tiles of No 6 and No 7 did not craze both on test tiles and on the body of the produced wares (table 1). The low- temperature glazes formulated from locally sourced raw materials analyzed showed that the tri-axial blend tested failed to meet the desire to produce low- temperature glaze. Rather, the Line blend was successful in achieving low-temperature glazes. The temperature range between 900°C, 1000°C ,1100°C and 1160°C indicate varying shades glossy, some reddish, black, yellowish brown, dark with gold and purple colour (Plates XLV to Plate XLVII).

5.3 Recommendations

1. There is need to carry out further researches using Dogo Red Clay for the development of stoneware glazes.
2. There is need to carry out further studies using Dogo red clay and Locust beans wood ash with other fluxes for achieving high temperature glazes.
3. The Dogo Red Clay should be tested on other bodies with other fluxes to get different effect of colours.

5.4 Contribution to Knowledge

The study established that:

1. this research was able to establish that the Dogo Red Clay, Groundnut Shells Ash and Locust Beans Wood Ash through line blend at 900%, 1000%, 1100% and 1160% was successful for use on ceramic wares as low temperature glazes.
2. this produced ceramic wares produced in research was fired at low – temperature range of 900%, 1000%, 1100% and 1160% to achieved diverse colouration of glossy look, on some the colouration were reddish brown, black shades, yellowish brown, with gold and purple colours.
3. groundnutshells and locust beans ash contains potassium oxide (K_2O) and calcium oxide (CaO) which acts as the flux to lower the melting temperatures from $1200^{\circ}C$ down to $900^{\circ}C$.
4. potassium oxide (K_2O) produced bright glossy glazes, that cannot be used alone as a flux.
5. glazes materials that contains iron oxide can produces wide range colors (blacks, brown, yellow, green) effects in glazes, during firing in an oxidation atmosphere.

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