Title

DEVELOPMENT OF ZIRCON-BASED OPAQUE GLAZE LOW-TEMPERATURE GLAZE USING RAW MATERIALS FROM KOGI

Abstract

A ceramic glaze is an impenetrable covering or coating
been fired into fusion. The term "glaze" refers to both the thin glassy layer tha
is fused to the surface. Zircon, a white crystalline zirconium
maintain colors and to increase opacity. by also aiding in
lowering the melting. Additionally, glazes defense against c
abrasive. Also mentioned are contemporary potters whose

greatly impacted me. There is also a brief explanation of the kind of clay, fire techniques, and application. The goal of the research was to develop a variety of opaque glazes made from zirconia from transparent recipes in order to increase the range of locally accessible and practical glazes for both domestic and commercial application. Based on the result of the investigation carried out during the course of the study, the following conclusions were drawn, The development of zirconia –based opaque Glazes for table ware Decorations can be utilized in the composition of body glaze used for the manufacture of table wares by adding it to serve as a glass former and as a flux.

Key Words: Zirconia, Opaque, Ceramics practice in Nigeria, Glazes and ceramic glaze recipes.

1.0 Introduction

A significant step toward the creation of high-quality, locally produced ceramic products with strong commercial potential may be seen in the scientific investigation and application of ceramic materials. Today, ceramics are used strategically in a wide range of applications where hardness, high temperature stability, aesthetic appeal, practicality, and corrosion and wear resistance are sought. These factors mentioned make ceramic materials important in the lives of human beings- which are applicable as objects of sanitary wares and table wares amongst others. Linda, (2013) defines tableware as dishes or dishware used for setting tables which may include cutlery, glassware, serving dishes or other items for utility as well as decorative purposes using different glazes to beautiful the table wares.

A glaze is a particular kind of glass used to coat ceramic items. It forms a hard, long-lasting, impenetrable vitreous coating after being melted at a high temperature in the kiln. A glaze is a glassy coating applied to ceramic items made from earthy mineral compounds that have been refined, combined with water, and then coated (greenware or bisque ware) before being heated to a high temperature in a kiln. It is also the best form of decorations which render the porous pottery vessels impermeable to water and other liquids.

There is an increase in the amount of both locally made and imported ceramic table wares of varied types gaining acceptance into the markets on regular basis especially well glazed products, most of which lacked critical information about the product composition, thus making it difficult to ascertain their safety, composition and quality for day to day use. However, with the use of local raw materials the quality of locally produced wares can be determined and improved by adding some certain materials one of which is zirconia.

Zirconia in particular have recently attracted special attention as high-technology thermal, electrical, chemical and optical properties. Zirconia is primarily used in ceramic to opacify glazes, increase glaze stability and to generally improve overall performance of glazes. The opacity of a glaze is dependent on the amount of zircon in it. Small doses (1-3%) may not make a difference at all, however they are occasionally used to increase glaze hardness. Since zirconia is extremely reflective, the more of it added will have a greater impact on the degree of glaze melting (and melt viscosity). Hence, zircon affects glaze melt viscosity, surface smoothness, thermal expansion and colour development of traditional pottery knowing fully well that colour tone is influenced by the firing temperature; the firing atmosphere, the calcinations temperature and fineness of the colour particles, the type of filler or flux used; the compound used for introducing the metal oxide concerned; and the mode of application i.e. whether applied as on glaze or under-glaze (Paolo, 2017). zirconia has a way of stabilizing glazes for quality results.

The goal of the research is to discover a few local raw materials from Kogi state in Nigeria that are acceptable and practical for the production of clear glazes. Utilizing established compositions and formulae for glaze formulations developed by previous researchers, sourced materials were created utilizing zirconia as an opacifier. For stoneware decoration, mid-temperature glaze formulas that underwent heat treatment between (1100°C and 1140°C) were developed.

Research Problem

Clay bodies are composed primarily from the common secondary clay with fillers and fluxes to improve the body strength making it dense, hard and vitreous. Likewise, glaze compositions are made out of the same bodies with few additions of glass former, metallic oxides, carbonates and oxides of certain metals that introduce more colours to the base hue. These hues can be experimented with and improved by adding opacifier. Glaze materials are readily available within the country- a good number of mineral deposits within Kogi state offer differs materials that can be worked on for ceramic production.

Fadairo (2008) admits that successful attempts have been recorded at producing glazes from indigenous materials, yet there is hardly a place in Nigeria where locally made glazes are sold in commercial quantities for potters to purchase. Likewise, there is room more for glaze exploration, experimentation and proper documentation which may help improve the standard of living of potters by making glazes available in large quantity and sold at commercial quantities as it is in the developed countries. Finally, the use of zirconia would give ceramic

artist the platform that would allow paintings on ceramic wares, using ceramic oxides that could be attractive and competitive which is rarely used in the country.

The purpose of the study is to source for some selected raw materials, process them for experimental analyses and subject them to ceramic production such that the research outcome can assist government, investor, private individuals and potters to know the potentials inherent in the mineral deposits in North Central Nigeria for the production of different categories of ceramics productions in the country.

Research Questions

The study will address the following research questions:

1. What are the materials required to produce good transparent glaze?
2. what are other basic raw materials required to formulate workable low-cost transparent and opaque glaze?
3. What technique were adopted to opacify the developed glazes composition with zirconia?
4 What are the resulting outcome of formulated glaze in firing in terms of physical properties?

Methodology

A 40kg bag of each sample was collected for each mineral because the nature of certain of the components (powdery) prevents them from being tallied. To finish the research, these were gathered from their various places. Feldspar, clay, kaolin, quartz, tin oxide, barium carbonate, and quartz were all purchased from the Kogi State and were identified in samples; they were then processed, ground into a fine powder, and characterized to determine their level of impurity and suitability for stoneware glaze recipes, while zirconia was bought from a reputable ceramics supplier in the state of Lagos, Nigeria.

After characterization, bulk of the raw materials were then processed by crushing, pulverising, ball milling, sieving, weighing and then measuring into desired composition to formulate transparent glazes. After a successful composition, Some test were carried out. line blend technique was adopted to opacify the already formulated transparent glazes in order to determine the most suitable glaze-opacifer ratio. Samples of bisque were experimented upon with developed mid-temperature transparent glazes before introducing opacity from zirconia. Thereafter, the lowest maturation temperature range achievable were determined, followed by experimentation with different metallic oxides and ceramic stains on tableware. Different materials and compositions were assessed and sampled at various percentage distributions,

carried out to determine the viability, efficiency and quality of the minerals for ceramic productions.

Beam balance were used to weigh all the materials for glaze compositions before the glaze batching, while pyrometric cones and thermocouple were inserted into the kiln chamber during firing for the measurement of the temperature in the kiln. In addition, digital camera was adopted as an instrument of data collection for taking photographs of all the processes involved in the formulation of glazes from excavation to sieving, milling, production of stoneware bisque samples, batching of glaze recipes and firing of the test samples to the final process.

The pulverized kaolin, limestone, quartz, feldspar, and ball clays were taken to Dangote Cement Company in Obajana, Kogi State for X-Ray Fluorescence analyses. Also, refractive index were conducted at Physics Department, Federal University of Technology, Akure.

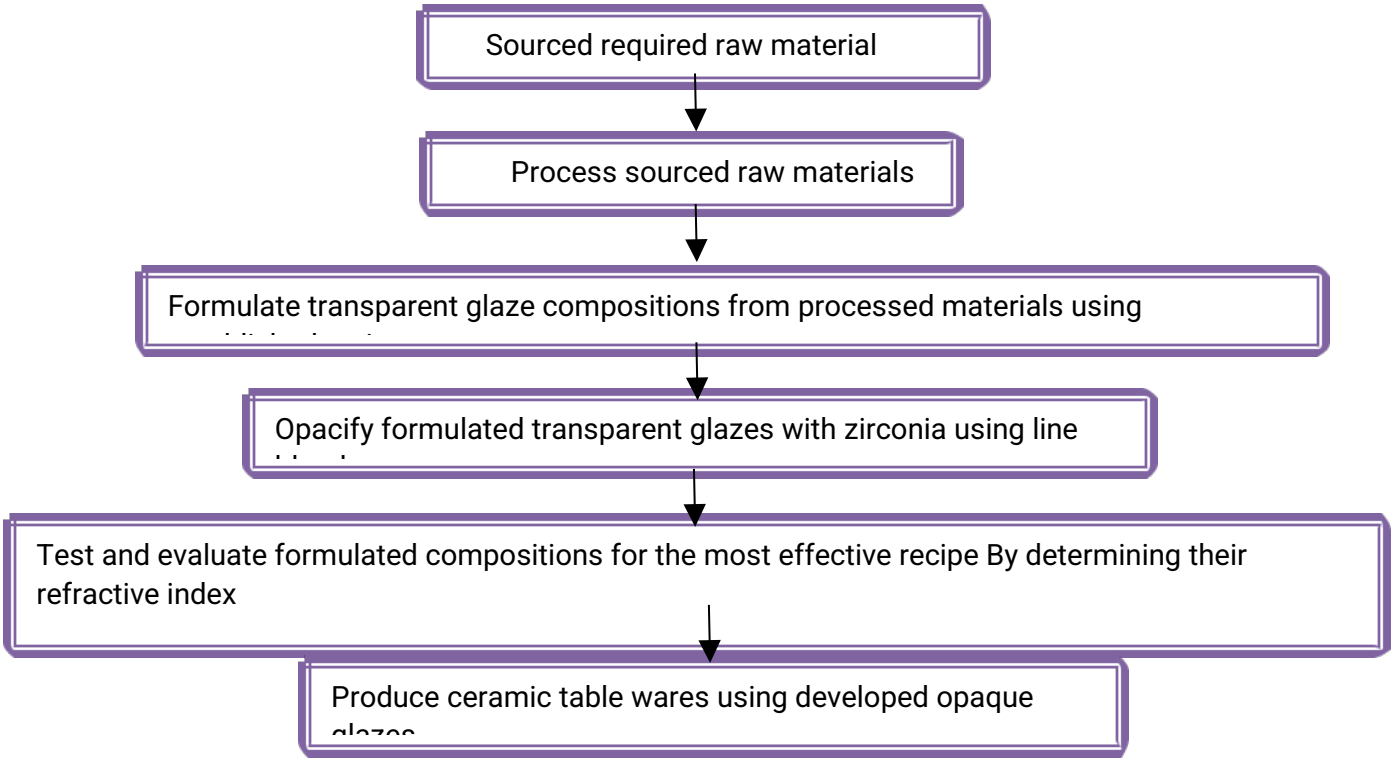

Figure 1: chart showing the experimental procedure in the study.

In order to achieve the aim and objectives of the research, the following steps were undertaking in gathering the data for the study:

i) **Sourcing of materials:** this includes ball clay, kaolin, limestone, feldspar, quartz

and zirconia,

ii) **Chemical analysis of the selected materials:** this were done using Efelex model machine of X-ray Fluorescence (XRF) in order to identify metallic oxides of the materials in the chemical compositions and their suitability for the development of ceramic production.

iii) **Materials processing:** this involves crushing, pulverization and wet sieving.

iv) **Materials composition and mixes:** These involve mixture proportion of the materials selected within the study area by weighing each sample, use it to produce, dry and fire samples produced. Causes and effects of these processes as it enhances the viability of the materials selected for ceramic productions was also be examined.

v) **Evaluation test:** this involves physical observations and carrying out most important property test such as: index of refraction- is the measurement of the test.

Objective 1: source for glaze raw materials constituents from selected locations to compose transparent glazes.

A bag of 50kg was collected for each sample. The quantity of each sample taken was large enough to ensure sufficiency. In taking the samples, the topsoil was scrapped off to about 3 feets before collecting samples; this is to ensure that only relatively pure materials were collected. The minerals obtained were independently crushed, milled, dried, sieved and labelled then taken to Laboratories for chemical analysis. Quality Control Department of West Africa Ceramics Limited also known as Royal Ceramics Ltd. There samples were analysed for their chemical properties, test analyses and comparison. Beam balance was used to weigh all the materials for glaze composition.

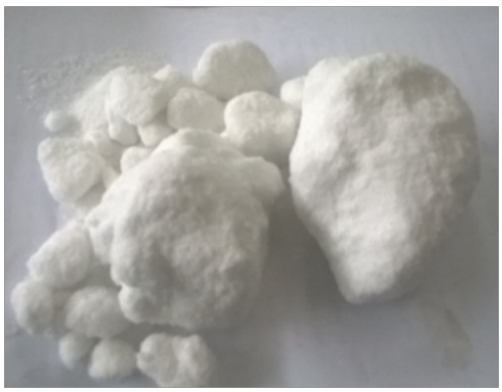

Plate 1: Zirconia in Dekina in Kogi State (Source: Author's Fieldwork, 2021)

Plate 2: **Borax Frit** in Dekina in Kogi State (Source: Author's Fieldwork, 2021)

Observation: it was observed during the visits to these mentioned locations in Northern Nigeria that many useful raw materials sites have been unexploited. Kashim (2011) states that the exploitation and processing of ceramic raw materials are enough to influence development and capacity building among the local miners around the communities where they are located.

Objective 2: Process Sourced Glaze Material For Transparent Glaze Formulation;

In order to achieve this objective; selected ceramic raw materials sourced from the Kogi state were prepared and taken to laboratories, in order to evaluate the industrial and cottage suitability of each material using X- Ray Fluorescence (XRF) machine at Dangote Quality Control Laboratory in Dangote Cement Plc, Obajana.

All the raw material were Ball mill, in a jaw mill inside the Industrial Design laboratory Federal University of Technology Akure, to a mesh size 80 to 120. Advantages of milling are to; make the raw material homogenous and make the material melt easily during firing. This is because the smaller the surface area the faster the melting point of the material.

Processing the raw materials sourced for the composition of the body

All the raw materials used have passed through materials processing and treatment.

The materials processing includes:

i soaking of all the raw materials in water and its surface impurities washed off.

ii hand crushed with hammer and crushed using jar crusher, and it was also ball mill

to smaller size and fine particles.

iii it was sieved to fine powder using mesh before use.

There are hundreds of different ball clays available and they vary widely in plasticity, particle size, raw colour, and drying properties. A typical ball clay powder is light grey (from lignite) or cream colour and fires to a buff or cream white colour with some soluble salt deposits on the fired surface. Ball clays are very plastic and much finer grained than kaolin's. They are typically unverified at cone 10. Ball clays are used in ceramic bodies (porcelains, stoneware's and earthenware's, casting slips, pressing bodies) because of their plastic nature combined with high firing temperature.

These samples were subjected to firing using gas kiln under an oxidised firing atmospheric condition, the same temperature and the same kiln condition.

Objective 3 opacify formulated transparent glazes with zirconia for ceramic ware production using line blend technique.

This objective is in two phases. The first phase is formulation of transparent glaze from established composition while the second phase is opacifying formulated transparent glazes with zirconia oxide. For the first phase, three different transparent glaze bodies named (Glaze A,B and C) were developed using established glaze compositions and they are:

Table 1: Using line blend glaze composition A, B and C were blended thus;

Zirconia %	0	10	20	30	40	50	60	70	80	90	100
TGlaze A %	100 ZTa1	90 ZTa2	80 ZTa3	70 ZTa4	60 ZTa5	50 ZTa6	40 ZTa7	30 ZTa8	20 ZTa9	10 ZTa10	0 ZTa11
TGlazeB %	100 ZTb1	90 ZTb2	80 ZTb3	70 ZTb4	60 ZTb5	50 ZTb6	40 ZTb7	30 ZTb8	20 ZTb9	10 ZTb10	0 ZTb11
TGlaze C %	100 ZTc1	90 ZTc2	80 ZTc3	70 ZTc4	60 ZTc5	50 ZTc6	40 ZTc7	30 ZTc8	20 ZTc9	10 ZTc10	0 ZTc11

Table 2: Compositions of Glaze Recipes Using Established Recipes Measured In Percentage(%)

RECIPE	KAOLIN	FELDPAR	LIMESTONE	SILICA	CLAY SLIP	TALC	FRIT
Glaze 1	20	15	21	32	-	-	12
Glaze 2	7	28	14	21	-	-	30

Glaze 3	10	24	16	35	5		5

Source: Researcher's field work, 2021.

For each composition a batch of 1kg was prepared. Each composition was thoroughly jar milled for 18hrs with 100ml of water. This was done in order to have a uniform quality with small particle sizes. It must be noted that Clay, Feldspar, Quartz, Limestone, Talc and Kaolin used were sourced from the kogi state in line with established recipes list for transparent glaze composition in order to test the viability of Kogi State raw materials for glazes production. Pouring and dipping method was used for glaze application. Each of composed transparent glaze were then fired in a gas text kiln together. The samples were subjected to firing using gas kiln under an oxidised firing atmospheric condition. The recipes formulated were subjected to test firing at temperature range between (1100^{o}C-1140^{o}C) and the results were recorded. The firing was monitored with a thermocouple and pyrometric cones to ascertain the maturing temperature for each glaze formulated.

Therefore Thirty (30) different test tiles were produced to blend the three formulated transparent glazes at ten (10) per composition. The formulated opaque compositions were subjected to heat treatment at their required temperatures ranging from 1100^{o}C to 1140^{o}C. The results of firing were properly documented. After firing the blended composition on thirty different test tiles observations were made.

Observation: From the physical examination conducted on the samples after they were composed and fired at 1140^{0}C, it was observed that composition ZTa5, ZTa4 and ZTa7 came out best from opacified glaze sample A. while ZTb6, ZTb4 and ZTb5 came out outstanding in the sample B composition. The third sample had text number ZTc3, ZTc54 and ZTc6 as its best. However only three test samples could be used for mass production as these three compositions came out with outstanding performance without the common glaze defects like crawling or crazing. More importantly, sample ZTc6, ZTb4 and ZTa7 were selected for production and decoration of ceramic wares because they had the best opacity as recorded by refactometer from the Physics department, FUTA. The opaque effect of zirconia was not visible in sample ZTa1, ZTa2, ZTa3, ZTa4 as they were still very transparent as the base of these test tile were still very visible, while ZTa8,ZTa9 were very mat. ZTa10 and ZTa11 were not only matte but stiff as they did not mature due to high presence of zirconia in the compositions. The same is applicable to opacified glazes from sample B and C.

Objective 4 Determine their physical qualities and also produce ceramic tablewares with opaque glazes Developed.

To achieve this objective, ceramic wares were produced using three ceramic forming techniques: casting, throwing and hand built methods. Throwing was used to produce more wares amongst these techniques because it has better chance of producing bigger wares than the rest than slip casting. Clay body was formulated using kaolin and ball clay sourced from kogi State in ratio 2:1 for stoneware ceramics as the clay body will have the same mineralogy with glazes formulated.

Glazing: opaque glaze sample ZTc6, ZTb4 and ZTa7 were reproduced in batch of 3kg each and were thoroughly wet milled for 8hrs using the department jar mill. After milling, dipping and bruising method of glaze application were applied to evenly coat the lightly-wetted bisque with the composed glazes. Excesses were scrapped off and glazes at the base of each ware were thoroughly removed to prevent wares from sticking to the shelf of the kiln during gloss firing.

4.2 Result of Test Melting of the Glaze Batches

The glaze sample are 1,2,3,4,5,6,7,8,9,10. Only glaze sample 1.4.5.6.7.8 melted completely at 1140^0C and have cracks. Glaze sample 1,7 and 8 was matt after firing to 1120^0C. Sample 1, 4, 7, and 8 was fired to higher temperature of 1130^0C, the glaze sample fired melted well but glaze sample 7 was transparent while sample 4 was translucent. Glaze sample 5 and 6 are clear transparent and melted well at 1100^0C but have cracks. Glaze sample 2,3,10 did not melt. Glaze sample 9 melted well under reduction firing.

Plate 1: Test Blend Result of Files glaze number 1 -10

(Source: Author's Fieldwork, 2021)

Test melting result of glaze calculator formulated blend fire to 1080^0C

The glaze sample are 11, 12, 13, 14, 15, 16, 17, 18, 19, 20, 21, 22, 23, 24, 25 and 26. Only sample, 11, 12, 14, 15, 17, 22, 23, 25, and 26 melt very well at 1050^0C to 1080^0C, while glaze sample 13, 16, 18, 19, 20, 21 and 24 did not melt on the test tiles. Sample, 23, 25 and 26 are

transparent after firing to 1080^0C. Glaze sample 12, 14, 15 and 17 are matte after firing to 1080^0C. Sample 11 and 22 are opaque after firing to 1080^0C. Glaze sample 11, 12, 23, 25, and 26 craze on the test tile body. Glaze sample 12, 14, 15 and 17 did not craze on the test tile. Sample 22 and 26 run on the test tile.

Plate 2: Test Blend Result of Files glaze number 11-26

(Source: Author's Fieldwork, 2021)

Four batches of glaze sample 5 and 6 are prepared. 5 and 10 percent talc is added to one of each glaze batch. 10 percent ball clay and 5 percent silica is added to each glaze sample

Five batches of glaze sample 7, 21, 22, and 26 are prepared. 5 and 10 percent talc, 10 percent ball clay, 5 and 10 percent feldspar are added to each glaze sample batch.

After firing all this glaze sample the crazing only reduce and not eliminated, because of this more test ware carried out on the glaze body while keeping the glaze constant,

Plate 3: Test Result after Adjusting Crazing

(Source: Author's Fieldwork, 2021)

Glaze runny

Only Glaze sample 22 and 26 run on the test tile. For Glaze sample 22, the borax was

constant at 20% and the kaolin was vary by 5% increase, making the kaolin 26%, 31.4%, 36.4% in each batch of glaze sample to control crazing and runny in the glaze.

Glaze sample 26, the borax was vary by 5% decrease making, the borax, 37.6%, 32.6%, 27.6%, 22.6% and 17.6% borax while the kaolin was constant at 15% , control crazing and runny.

Plate 4: Test Result after Adjusting Runny

(Source: Author's Fieldwork, 2021)

Result of glaze sample on thrown wares

Out of all the glaze sample only glaze sample 6 and 7 were picked out of glaze formulated by seger, while sample 22 and 26 are picked out of glaze formulated by glaze calculator.

Glaze sample 6 and 7 are food safe, which means they can be used on table wares, this sample are transparent and melts good on thrown wares, at 1120^0C. The wares was bisque fired to 900^0c in a gas kiln.

Table 13: Gloss firing of ZTc6, ZTb4 and ZTa7fired on Stoneware at 1140^{o}C

Date: 2nd July, 2021

Temperature: 1250^0C

Measuring Instruments: Thermocouple and Orton cones (3, 4 and 5)

Kiln Used: Gas Kiln

Firing Log

Time	Firing Reading(Thermocouple)	Remark

09:20am	27°C	Firing started with preheating
09:50am	52°C	Preheating continues
10:20am	119°C	Preheating continues
10:50am	138°C	Gas pressure was slightly increased
11:20am	168°C	Preheating continues
11:50am	190°C	Gas pressure was slightly increased again
12:20pm	218°C	Preheating continues(black)
12:50pm	450°C	Full firing started with red glow colour
1:20pm	510°C	Firing continues
1:50pm	560°C	Gas pressure increased with Cherry red colour
2:20pm	648°C	Gas pressure increased with Cherry red colour
2:50pm	684°C	Gas pressure increased with Cherry red colour
3:20pm	747°C	Gas pressure increased with Cherry red colour
3:50pm	850°C	Gas pressure increased with Cherry red colour
4:20pm	1015°C	The glaze looks red-yellowish in mirror-like in the kiln
4:50pm	1085°C	Cone 3 bents to 2 O'clock
5:20pm	1109°C	Cone 3 bents to 4 O'clock
5:50pm	1130°C	Cone 4 begins bending

Source: Researcher's fieldwork, 2021

The research outcomes shows that

That Kogi state is bless with numerous solid materials that cannot be overemphasised. These materials are useful in many regards ranging from pharmaceutical, engineering, cosmetic, and ceramics productions. Trip was made to Kogi State were different ceramic solid minerals were excavated for ceramic production. These raw materials were processed- sorted, crushed, sucked and dried, pulverized ball milled and jar milled, chemically analysed and weighed for transparent glaze formulation. Using existing transparent glaze compositions, recipes were measured accordingly and test fired. Raw materials from Kogi State came out fine and the test outcome was promising with little addition of frit. Thereafter, compositions were mixed with zirconia as an opacifier using line blend technique which shows that only three test samples ZTc6, ZTb4 and ZTa7could meet the international standard out of thirty other test samples produced as analysed by a refractometer. These three samples were mass produced on stoneware bisques that were modelled using throwing, hand built and casting method of body formation and were fired in oxidation at 1140^0C in a gas kiln.

The first stage of this investigation was directed towards the collection and various processes of the raw materials used for the study. The Ball clay, feldspar, Kaolin, Zirconia, opaque glaze and transparent glaze used for this study went through various beneficiation processes. The process of soaking and sieving helped in washing out the mineral salt and impurities as well as the removal of the organic matters. The gloss firing was carried out in a gas kiln at temperatures varying from 1050^0C to 1140^0C

This project has open up areas for investment opportunities from minerals exploration, processing, ceramic body specialist and glazes to ceramic manufacturing for ceramic prationiers. All these areas mentioned above are business and employment opportunities for our teeming unemployed youths and revenue generations for the state. If properly ultilized, it will salvage the state from her present state of federal allocation dependency to the state of wealth afluence.This will reduce crimes and kidnapping if government can create enterpreneurial skills acquisition centres for pottery, clay body packages for sale even glazes indicating their maturing temperature and their state of firing – oxidation or reduction firing, even colour reaction after firing for glazes would be of economy values

Conclusion

Based on the result of the investigation carried out in the course of this study, the following conclusions were drawn;

i. The development of zirconia –based opaque Glazes for table ware Decorations can be utilized in the composition of body glaze used for the manufacture of table wares by adding it to serve as a glass former and as a flux. This means that it should be possible to obtain quality tableware product in the country.

ii. The zirconia can be used as a product with higher added value with respect to environmental and economic issues. It is therefore an attractive alternative source of ceramic raw materials.

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