FORMULATION OF LOW-TO-MEDIUM TEMPERATURE GLAZES USING VARYING PROPORTIONS OF GROUNDNUT SHELL ASH AS A BASE MATERIAL

Ogunwole V.B[1], Balogun A.[2]

1. Department of Fine and Applied Arts, Kaduna State College of Eduation, Gida-Waya
2. Department of Industrial Design, Ahmadu Bello University, Zaria

Corresponding Author: Veronica Ogunwole Benedict
Email: ogunwolevic21@yahoo.com **Phone:** 08069677782

Abstract

This study investigates the use of groundnut shell ash (GSA) as a sustainable base material in the formulation of low to medium-temperature ceramic glazes using the triaxial blend method. The research was aimed at determining the effects of varying proportions of GSA in combination with feldspar and kaolin on the physical and aesthetic properties of glaze coatings fired between 1000°C and 1180°C. The experiment was conducted using a systematic triaxial blend approach in which GSA, feldspar, and kaolin were mixed in varying ratios across 15 formulated glaze samples. The oxide composition of GSA revealed high levels of CaO (22.8%), K_2O *(10.4%), and* SiO_2 *(38.7%), making it suitable as a flux-rich material. Results showed that increasing GSA content significantly enhanced glaze maturity, reduced melting temperature, and produced glossy surfaces with color variations from light brown to greenish tones, depending on the concentration. Samples with higher kaolin content yielded matt surfaces, while feldspar-rich compositions produced smoother glossy textures. The study concludes that GSA can effectively serve as a primary flux material for sustainable glaze development in ceramic production, providing a low-cost, eco-friendly alternative to imported fluxes. The triaxial blend method proved valuable for predicting compositional effects and guiding glaze optimization. The research contributes to the advancement of green ceramics and circular materials use in the ceramic industry. This will serve as a step in the right direction for waste materials reutilization and its productive use in sustainable ceramic glaze production.*

Keywords: Groundnut shell ash, triaxial blend, glaze formulation, low–medium temperature glazes, ceramic materials, sustainability

Introduction

The formulation of ceramic glazes involves combining silica, alumina, and fluxing agents in specific proportions to achieve desired surface and aesthetic qualities upon firing. In traditional ceramic industries, feldspars, borates, and other industrial minerals are commonly used as fluxes due to their ability to lower the melting point of silica and promote vitrification. However, dependence on imported flux materials has raised production costs and environmental concerns. In recent years, increasing attention has been directed toward the use of agricultural waste ashes as alternative raw materials for glaze formulation (Onwuka, 2019; Isah & Eze, 2021).

Groundnut shell ash (GSA) is a promising candidate among biomass ashes. Nigeria, being one of the world's leading producers of groundnuts, generates large quantities of shells that are often discarded as waste. When properly calcined, these shells yield an ash rich in silica, potassium oxide, and calcium oxide—constituents essential in ceramic glaze chemistry.

Utilizing such waste products supports sustainable production, minimizes environmental impact, and promotes local material development (Ogunlana, 2020).

The triaxial blend method has long been recognized as a systematic experimental design for glaze formulation. It allows ceramic researchers to vary three materials simultaneously and observe compositional interactions within a triangular matrix (Rhodes, 1973). This method helps identify optimal compositions with desirable surface, color, and melting properties.

Despite progress in the use of agro-waste ashes for ceramics, limited research exists on the integration of groundnut shell ash in triaxial glaze systems for low to medium temperature applications. This study therefore explores the use of GSA as a base flux material, combining it with feldspar and kaolin to produce glazes fired between 1000°C and 1180°C.

Studies conducted outside Africa provide useful comparative perspectives on the use of biomass ashes in ceramic glaze formulation. Research in Asia has examined rice husk ash as a silica rich and flux contributing material in stoneware and earthenware glazes. For example, Singh and colleagues reported that rice husk ash based glazes fired at mid-range temperatures exhibited good gloss and chemical durability when blended with feldspar and clay minerals (Singh et al., 2016). Similar work in China demonstrated that potassium rich plant ashes could partially replace conventional fluxes without compromising glaze stability, while reducing material costs and supporting waste reutilization strategies (Li et al., 2018). In Europe and North America, investigations have focused on wood ash and other agricultural residues as sustainable glaze materials. Hamer and Hamer documented the long standing use of wood ash in traditional and contemporary glaze recipes, noting its variable but beneficial fluxing behavior depending on combustion conditions and botanical origin (Hamer & Hamer, 2004). More recent experimental studies in the United States explored the use of corn cob ash and wheat straw ash in low and mid temperature glazes, reporting acceptable melting behavior and diverse surface qualities when systematic blending methods were applied (Carty & Senapati, 2019). These international studies support the broader viability of agro waste ashes in glaze development and provide a relevant context for evaluating groundnut shell ash within controlled triaxial systems.

Aim

To develop and characterize low to medium temperature glaze samples using groundnut shell ash as a base material through the triaxial blend method.

Glaze Composition and Function

Ceramic glazes are composed primarily of silica (SiO_2), alumina (Al_2O_3), and fluxes (CaO, K_2O, Na_2O, etc.). Silica provides the glassy network, alumina imparts stability and viscosity, while fluxes reduce the melting temperature. The balance among these oxides determines the glaze's surface finish, gloss, and color response (Hamer & Hamer, 2015).

Agricultural Ashes as Glaze Materials

Research has demonstrated the potential of various agricultural ashes in ceramics. Rice husk ash, plantain peel ash, and palm kernel shell ash have been successfully used as flux substitutes (Nwankwo et al., 2018). These ashes contain high silica and alkali content, facilitating the glass formation process. According to Adebowale (2017), such materials enhance local raw material utilization and reduce import dependency.

Groundnut Shell Ash and its Ceramic Potential

Groundnut shell ash typically contains silica (35–45%), calcium oxide (20–25%), potassium oxide (8–12%), and trace amounts of iron oxide (1–3%) (Adewumi et al., 2019). These oxides mirror the composition of conventional feldspars and wollastonite, both common in glaze formulations. GSA's chemical similarity makes it a promising material for flux replacement.

Triaxial Blend in Glaze Research

The triaxial blend method was pioneered as a systematic means to explore the interrelationship between three glaze components. Each vertex of the triaxial diagram represents 100% of a single material, with incremental mixtures forming intermediate compositions. This experimental grid enables efficient evaluation of numerous compositions in a single firing (Chappell, 2008). Triaxial blending offers a visual and analytical tool for identifying compositions yielding ideal glaze qualities.

Research Gap

While biomass ashes have been extensively studied in clay bodies and cements, limited literature addresses their use in glazes through triaxial methods. Studies on GSA have mostly focused on refractory and pozzolanic applications. There is a need for experimental evidence demonstrating its glaze-forming potential, particularly at low to medium firing ranges.

Materials and Methods

Materials

The primary materials used were groundnut shell ash GSA, feldspar, and kaolin, each sourced locally in Nigeria. The groundnut shells were obtained from a local groundnut processing site in Zaria, within Kaduna State, where they were air dried and later calcined to produce the ash. Feldspar and kaolin were sourced from mineral deposits in Alkaleri Local Government Area of Bauchi State, a region known for its abundance of ceramic raw materials.

Preparation of the Raw Materials

Characterization of the groundnut shell ash, feldspar, and kaolin was carried out using standard analytical equipment. Chemical composition was determined using an X ray fluorescence spectrometer PANalytical Axios MiniPal 4 manufactured by Malvern PANalytical Netherlands. Phase identification was conducted with an X ray diffractometer PANalytical X Pert PRO manufactured in the Netherlands. Microstructural features were examined using a scanning electron microscope JEOL JSM 6480LV manufactured by JEOL Ltd Japan. These instruments provided reliable data on oxide composition, mineral phases, and particle morphology of the materials. Prior to characterization and formulation, all raw materials were pulverized and sieved to achieve uniform particle size distribution. A set of standard laboratory test sieves was used, with the final particle size passing through a 200 mesh sieve corresponding to 75 micrometres. This mesh size was selected to ensure adequate homogeneity and reactivity of the materials during subsequent ceramic processing stages.

Since the groundnut shell ash was sieved through a 100 µm mesh, the feldspar and kaolin were prepared using the same particle size to maintain consistency among the raw materials. The groundnut shells were washed thoroughly, sun dried, and then incinerated in a muffle furnace. The furnace temperature was increased from room temperature to 800 °C at a controlled heating rate of 5 °C per minute. The material was held at 800 °C for a soak time of four hours to ensure complete incineration. After cooling, the resulting ash was ground and sieved through a 100 µm mesh. The ash sample was subsequently analyzed using X Ray Fluorescence to determine its oxide composition.

Table 1.0 shows the chemical composition of GSA compared with feldspar and kaolin.

Table 1.0: Oxide Composition of Raw Materials (wt%)

Oxide	GSA	Feldspar	Kaolin
SiO_2	38.7	65.2	45.4
Al_2O_3	4.2	18.6	38.7
Fe_2O_3	2.6	0.5	0.8
CaO	22.8	0.9	0.2
K_2O	10.4	12.7	0.1
Na_2O	1.3	2.6	0.1
MgO	3.5	0.3	0.2
LOI	16.5	1.4	14.5

Triaxial Blend Design

A triaxial diagram was constructed with the three materials positioned at each vertex: GSA (A), Feldspar (B), and Kaolin (C). Fifteen blend points were selected across the grid (Figure 1). Each mixture was calculated to total 100 g dry weight.

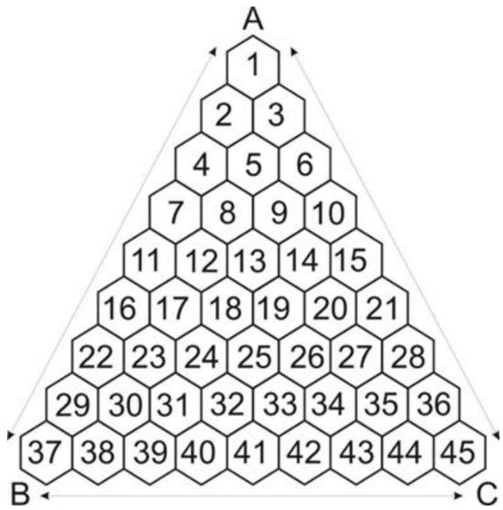

Figure 1.0: Schematic Representation of Triaxial Blend Design; Descriptive representation: each vertex corresponds to 100% of one component

Source: Researcher, 2026

Glaze Preparation

Each mixture was ball-milled with 50 mL of distilled water for 30 minutes to ensure uniform dispersion. The slips were sieved through 100 mesh and applied on bisque-fired stoneware tiles (5 × 5 × 0.5 cm). Glazes were fired in an electric kiln at temperatures ranging from 1000°C to 1180°C, increasing in 60°C increments. Firing cycles lasted 8 hours including soaking time.

Evaluation Criteria

i. After firing, the samples were evaluated based on:

ii. Surface texture (glossy, semi-matt, matt)

iii. Color tone (visual observation under daylight)

iv. Crazing or pin holing (defect assessment)

v. Melting behavior (degree of flow and maturity)

vi. Observations were recorded using both naked eye and 10× magnification lens.

Results

Glaze Appearance and Maturity

Glazes with higher GSA content (above 50%) displayed excellent melting at 1060°C, forming smooth glossy surfaces. Samples with higher kaolin content remained underfired at 1080°C, developing semi-matt textures. Feldspar-rich glazes achieved full gloss at 1140°C.

The Table 2.0 presents the summarized glaze appearance results across the triaxial samples.

Table 2.0: Summary of Glaze Surface Appearance at 1080°C

Sample code	GSA	Feldspar	Kaolin	Surface texture	Colour	Defects	maturity
G1	70	20	10	Glossy	Amber brown	None	Mature
G2	60	30	10	Glossy	Greenish-brown	None	Mature
G3	50	30	20	Semi-gloss	Light brown	Few pinholes	Near mature
G4	40	40	20	Semi-matt	creamy	none	underfired
G5	30	50	20	matt	Pale brown	Slight crazing	underfired

Firing Temperature Response

The glazes matured between 1020°C and 1140°C depending on composition. Samples with higher feldspar required higher temperatures, while GSA-dominant samples achieved fluidity earlier.

Table 3.0 below summarizes the firing behavior.

Table 3.0: Firing Temperature and Maturity Range

Composition ratio	Maturity temperature (^oC)	Surface description	Defects
70% GSA, 20% F, 10% K	1020	Glassy	None
60% GSA, 30% F, 10% K	1040	Glossy	None
50% GSA, 30% F, 20% K	1080	Semi-gloss	Few pinholes
40% GSA, 40% F, 20% K	1120	Semi-matt	None
30% GSA, 50% F, 20% K	1140	Matt	Slight crazing

Discussion

The research demonstrates that groundnut shell ash possesses strong fluxing potential, attributable to its high CaO and K_2O contents. These oxides promote early melting and enhance glaze gloss at lower firing temperatures. The progressive increase in gloss with GSA concentration aligns with the findings of Adewumi et al. (2019), who noted similar results using rice husk ash in glaze compositions. The triaxial blend effectively revealed compositional trends, highlighting a transition from glossy to matt finishes across the compositional field. Samples with excessive kaolin produced high viscosity melts, preventing complete flow and resulting in underfired surfaces. The feldspar contributed to smoothness and stability, while GSA induced glass formation. Color variations from amber to greenish tones were linked to minor iron oxide content in GSA. No significant crazing was observed in well-balanced compositions, suggesting compatibility between glaze and clay body. From a chemical perspective, defects such as pinholing and crazing arise from imbalances in reactions occurring during firing and cooling. Pinholing is primarily associated with the release of gases from the glaze or clay body during firing. Chemically, this occurs when volatile components such as carbonates, sulfates, organic residues, or chemically bound water decompose at temperatures near or above the point where the glaze becomes viscous. If the glaze melt stiffens before gas evolution is complete, bubbles become trapped and rupture at the surface, leaving small holes. High alumina content, insufficient fluxes, or incomplete oxidation of the clay body can increase melt viscosity and hinder gas escape, increasing the likelihood of pinholing.

Crazing is caused by a mismatch in thermal expansion between the glaze and the clay body. From a chemical standpoint, this mismatch is governed by glaze composition, particularly the ratio of network formers such as silica, network modifiers such as alkali and alkaline earth oxides, and intermediates such as alumina. Glazes with higher concentrations of alkali fluxes typically have higher thermal expansion coefficients. Upon cooling, if the glaze contracts more than the clay body, tensile stress develops in the glaze layer, leading to crack formation. The absence of significant crazing in well balanced compositions indicates that the chemical formulation produced compatible thermal expansion behavior between the glaze and the clay body, allowing stresses to be accommodated without fracture.

The results affirm that agricultural waste ashes can substitute conventional fluxes in glaze formulations without compromising aesthetic quality or firing performance. The triaxial method provided a comprehensive visualization of compositional interactions, confirming its reliability for exploratory glaze research.

Conclusion

The study established that groundnut shell ash can serve as an effective fluxing component for low to medium temperature glazes. The triaxial blending approach enabled systematic examination of GSA-feldspar-kaolin combinations and revealed that:

1. Glaze maturity and gloss increase with higher GSA content due to flux oxides (CaO, K_2O).
2. Feldspar enhances smoothness, while kaolin controls viscosity and surface stability.
3. Optimal compositions (60–70% GSA, 20–30% feldspar, 10% kaolin) produce fully matured glazes at 1040–1080°C.
4. GSA-based glazes exhibit satisfactory color, gloss, and defect-free surfaces suitable for studio and industrial use.

These findings support the inclusion of agro-waste ashes in ceramic production and promote sustainable material utilization in Nigeria's ceramic industry.

Recommendations

Expanding the triaxial system to incorporate colorants or opacifiers such as iron oxide, cobalt oxide, and tin oxide could broaden application potentials.

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