https://doi.org/10.5281/zenodo.23208987

Assessment of Groundnut Husk Ash as a Potential Fluxing Agent For Glass Production Using XRF Analysis

AYUBA *Kazashim Seth*[1], ***BABARINDE*** *Oluwaleke*[2], ***JAGUSA*** *Isaac*[3]

Abstract

This study investigated the suitability of groundnut husk ash (GHA) as a potential fluxing agent for glass production, with the aim of providing an environmentally sustainable alternative to conventional potassium oxide (K_2O) and calcium oxide (CaO) sources used in glass manufacturing. Groundnut husks were collected from three locations in Kaduna State, Nigeria — namely Gumel, Kujama, and Narayi — and subjected to controlled ashing at 600°C in a muffle furnace. The ash samples underwent physical characterisation (solubility and loss-on-ignition tests) and chemical analysis using X-ray fluorescence (XRF) spectrometry. Results showed an average ash yield of 2.0% by weight from raw groundnut husk. Loss-on-ignition values ranged from 10.08% to 10.17%, indicating the presence of residual volatile matter. All ash samples were soluble in distilled water. XRF analysis revealed high K_2O concentrations of 46.20%, 39.84%, and 39.36% for the Gumel, Kujama, and Narayi samples respectively, alongside significant CaO concentrations of 30.50%, 26.80%, and 33.90%. These findings confirm that GHA contains sufficient oxide concentrations to function as a flux in glass batch compositions, with potential to reduce reliance on synthetic chemical inputs while simultaneously addressing agricultural waste management challenges in developing economies.

Keywords

Groundnut husk ash; Fluxing agent; X-ray fluorescence (XRF); Glass production; Potassium oxide; Agricultural waste utilisation

1.0 Introduction

Rapid agricultural, industrial, and economic growth in developing nations has increased both food production and the generation of associated agricultural waste. With a growing global population increasingly concentrated in urban centres, demand for food has intensified, resulting in the accumulation of large quantities of crop residues, husks, straw, and other by-products (Sabiti, 2011). These residues, if poorly managed, pose significant environmental risks through methane and leachate emissions when left to decompose, and through carbon dioxide and particulate pollutants when openly burned (UNEP, 2009). The need to convert these waste streams into economically viable raw materials is therefore both an environmental and economic priority.

Glass manufacturing relies on a specific combination of raw materials: silica (SiO_2) as the primary network-forming oxide, a fluxing agent to lower the melting temperature of silica, and a stabiliser to impart chemical and structural durability to the final product. Potassium oxide (K_2O) and sodium oxide (Na_2O) are the most widely used fluxes, while calcium oxide (CaO) serves as the principal stabiliser in commercial soda-lime and potash glass formulations. The industrial production of potash glass, however, has declined due to the relatively high cost of potassium carbonate compared with sodium carbonate, despite the superior weathering resistance and chemical stability of potassium-based glass compositions (De Bardi et al., 2015).

Agricultural ash materials have long been investigated as potential low-cost alternatives to synthetic chemical inputs in ceramic and glass production. Rice husk ash, for instance, has been extensively studied as a source of reactive silica (Hossain et al., 2018), while wood ash and crop

1,2,3Department of Industrial Design, Faculty of Environmental Sciences, Lagos State University, Epe Campus, Lagos, Nigeria

residue ashes have been shown to contain significant proportions of alkali and alkaline earth metal oxides suitable for glass-fluxing applications (Trisakti et al., 2018). Groundnut husk, generated in substantial quantities across West Africa and South Asia, represents an underexplored resource in this context. The husk constitutes the outer shell of the groundnut pod and, after combustion, yields an ash residue reported to contain appreciable quantities of K_2O and CaO (Ketkukah & Ndububa, 2006; Adole et al., 2011).

The present study was designed to characterise groundnut husk ash (GHA) from three geographically distinct locations in Kaduna State, Nigeria, and to evaluate its chemical composition — specifically the concentrations of K_2O and CaO — in relation to its potential application as a fluxing agent in glass batch compositions. The study addresses a gap in the literature on Nigerian agricultural ash utilisation in glass production and contributes to the broader objective of sustainable industrial resource management.

2.0 Literature Review

2.1 Groundnut Husk as Agricultural Waste

Groundnut (*Arachis hypogaea*), commonly known as peanut, is among the most widely cultivated leguminous crops in sub-Saharan Africa, with Nigeria being one of the largest producers on the continent. The husk, comprising the fibrous outer shell of the groundnut pod, is generated in significant quantities after seed extraction. Okorie et al. (2013) estimated that husk constitutes approximately 25–30% of the total groundnut pod weight, making it one of the more voluminous agricultural residues from this crop. In many parts of Nigeria, groundnut husk is either burned in the field or left to decompose, with minimal organised collection or value-added processing.

The primary uses of raw groundnut husk are as fuel, animal roughage, and mulch. Upon combustion, groundnut husk yields approximately 2–5% ash by weight (Adole et al., 2011). This ash is not nutritionally adequate for use as a standalone fertiliser owing to insufficient nitrogen (N), phosphorus (P), and potassium (K) levels when compared with conventional fertilisers. However, the alkaline nature of the ash and its oxide composition suggest potential applications in construction materials and industrial processes. Faugdar and Diwaka (1993) identified groundnut crop residues as broadly underutilised and recommended systematic appraisal of their chemical and physical properties as a prerequisite for industrial exploitation.

2.2 Chemical Composition of Agricultural Ash and Glass-Making Relevance

The chemical composition of ash derived from agricultural residues varies significantly depending on the type of crop, soil conditions, geographic origin, and combustion temperature. In general, ashes from plant material contain elevated concentrations of potassium oxide (K_2O), calcium oxide (CaO), silicon dioxide (SiO_2), magnesium oxide (MgO), and phosphorus pentoxide (P_2O_5), alongside trace quantities of iron oxide (Fe_2O_3), aluminium oxide (Al_2O_3), and heavy metal oxides (Westerman & Bicudo, 2005). The relative proportions of these constituents are strongly influenced by the botanical characteristics of the source plant and the conditions under which the ash is produced.

In glass-making, fluxes serve the critical function of reducing the melting point of silica from approximately 1710°C to a more industrially manageable range of 1400–1500°C. Potassium oxide (K_2O) is a well-established flux that also confers superior weathering resistance and chemical stability compared with sodium-based glass compositions. De Bardi et al. (2015) demonstrated that potash glass (K_2O-CaO-SiO_2 system) exhibits greater resistance to atmospheric corrosion and ion-exchange processes than its soda-lime counterpart, making it preferable for applications requiring long-term durability. Calcium oxide, functioning as the principal stabiliser, prevents glass from dissolving in water and contributes to mechanical strength (Shelby, 2005).

Studies on wood ash composition have reported K_2O concentrations ranging from 4% to 35% depending on species and combustion conditions (Thy et al., 2006). Çınar and Tazeoğlu Filiz (2024) investigated the use of waste banana peel ash in glaze making and reported K_2O values of up to 65%, indicating its viability as a natural flux source. Similarly, Hossain et al. (2018) demonstrated that rice husk ash, with SiO_2 content exceeding 85%, could be incorporated as a supplementary silica source in glass batches. These findings collectively suggest that GHA, if shown to contain sufficiently high concentrations of K_2O and CaO, could serve a meaningful role in reducing the synthetic chemical inputs required for glass production.

2.3 Environmental and Economic Context

The utilisation of agricultural waste in industrial processes aligns with circular economy principles, which advocate for the reduction of waste streams and the recirculation of materials within economic systems (Sabiti, 2011). In the context of

developing economies, where synthetic raw materials must often be imported at considerable cost, the substitution of locally available agricultural by-products offers both economic and environmental advantages. Waste valorisation also reduces the environmental burden associated with improper disposal, including soil and water contamination from leachate and air pollution from open burning (UNEP, 2009). The commercial potential of groundnut husk ash as an industrial raw material could additionally incentivise improved collection and processing of this residue at the farm level, thereby enhancing income streams for smallholder farmers (Ketkukah & Ndububa, 2006).

3.0 Methodology

The experimental procedures were conducted in four stages: raw material collection and pre-treatment; ashing and ash characterisation; physical analysis (solubility and loss-on-ignition); and chemical analysis by X-ray fluorescence (XRF) spectrometry.

3.1 Sample Collection and Pre-treatment

Groundnut husks were collected from three locations in the southern parts of Kaduna State, Nigeria: Gumel, Kujama, and Narayi. These locations were selected to represent geographical variability in soil type, microclimate, and groundnut cultivation practices within the region. At each site, approximately 500 g of dry groundnut husk was collected from multiple points across the farm site to ensure representative sampling. Samples were assigned designations A (Gumel), B (Kujama), and C (Narayi).

Upon collection, each sample was visually inspected and manually sorted to remove spoilt or mouldy husks, residual groundnut seeds, and extraneous debris. The sorted husks were then washed with tap water to remove adhered soil and surface contaminants, followed by rinsing with distilled water. The washed husks were oven-dried at 105°C for 24 hours to eliminate residual moisture, and subsequently ground using a wooden mortar and pestle to produce a homogenous fine powder.

3.2 Ashing Procedure

The powdered husk from each sample was packed into a ceramic-lined enclosure and ignited in open air to achieve initial carbonisation. The carbonised material was then transferred to a muffle furnace (Carbolite™ AAF 11/18) and subjected to controlled combustion at 600°C for 6 hours. This temperature was selected to ensure complete oxidation of carbonaceous residues while avoiding volatilisation of alkali oxide constituents, which has been reported to commence above 700°C (Su et al., 2024). The resultant ash from each sample was allowed to cool to ambient temperature in a desiccator before being passed through a 0.09 mm mesh sieve to remove coarse unburnt particles and ensure particle-size uniformity.

3.3 Loss on Ignition (LOI)

Loss on ignition was determined gravimetrically for each ash sample. Approximately 1.0 g of each sieved ash sample was weighed into a pre-tared porcelain crucible and placed in an electric muffle furnace at 200°C for one hour. The crucibles were removed, cooled in a desiccator, and re-weighed. The heating–cooling–weighing cycle was repeated until constant weight was achieved (mass difference between successive weighings ≤ 0.0005 g). LOI was calculated using Equation 1:

$$LOI\ (\%) = [(W_2 - W_3) / (W_2 - W_1)] \times 100$$

where W_1 = mass of empty crucible (g); W_2 = mass of crucible + sample before ignition (g); W_3 = mass of crucible + sample after ignition (g).

3.4 Solubility Test

The solubility of each ash sample in distilled water was assessed qualitatively following the procedure described in IS 13855:1993. Five grams of each ash sample were placed in a 50 mL test tube, to which 20 mL of distilled water was added. The mixture was agitated vigorously by manual shaking for two minutes and then allowed to stand for 30 minutes. Visual observation was used to characterise solubility as 'soluble', 'partially soluble', or 'insoluble' based on the degree of dissolution and the presence or absence of residual sediment.

3.5 Chemical Analysis by X-ray Fluorescence (XRF)

Elemental oxide composition of each ash sample was determined by X-ray fluorescence (XRF) spectrometry. Analysis was carried out at the Nigerian Survey Agency, National Geosciences Research Laboratory (NGRL), Kaduna State, Nigeria. Pressed powder pellets were prepared from each ash sample for XRF measurement using a wavelength-dispersive XRF spectrometer. Instrument calibration was performed using certified reference materials traceable to the National Institute of Standards and Technology (NIST). Each sample was analysed in triplicate and mean values are reported. Oxide concentrations are expressed as weight percentages (wt%).

4.0 Results and Discussion

4.1 Ash Yield

Table 1 presents the mass of ash obtained from 100 g of groundnut husk for each sample location. Ash yield ranged from 1.80% (Narayi) to 2.20% (Kujama), with a mean value of 2.00% across the three samples. These values are consistent with those reported by Adole et al. (2011), who documented ash yields of 1.9–2.3% for groundnut husk ashed under comparable conditions. The low ash yield implies that large quantities of raw groundnut husk would be required to generate sufficient ash for industrial glass batch preparation; however, this is not a limiting factor given the abundance of groundnut husk as an agricultural by-product in northern Nigeria.

Table 1: Weight of ash obtained from groundnut husk samples after furnace combustion

Sample	Location	Weight of Groundnut Husk (g)	Weight of Ash Obtained (g)	Ash Yield (%)
A	Gumel	100.00	2.00	2.00
B	Kujama	100.00	2.20	2.20
C	Narayi	100.00	1.80	1.80
Mean	—	100.00	2.00	2.00

4.2 Loss on Ignition

Loss on ignition (LOI) values for the three samples are presented in Table 2. LOI values ranged from 10.08% (Kujama) to 10.17% (Gumel), with a mean of 10.12%. These values indicate that approximately 10% of the ash mass comprises volatile constituents, primarily adsorbed moisture, bound water, and residual carbonaceous matter that was incompletely oxidised during the ashing process. While these values are within an acceptable range for flux characterisation purposes, the presence of residual carbon could affect the clarity of glass produced, and further calcination or extended ashing times may be warranted for samples intended for direct incorporation into glass batches.

Table 2: Loss on ignition (LOI) values for groundnut husk ash samples

Sample	Location	LOI (%)
A	Gumel	10.17
B	Kujama	10.08
C	Narayi	10.12
Mean	—	10.12

4.3 Solubility

All three ash samples (A, B, and C) were found to be soluble in distilled water, albeit with minor visible sediment upon settling (Table 3). This behaviour is consistent with the high alkali and alkaline earth oxide content of the samples, as K_2O and CaO are known to form soluble hydroxide species in aqueous media. The solubility of the ash samples confirms adequate beneficiation during the ashing process and the absence of significant insoluble impurities such as silicate residues or unburnt carbonaceous matter. However, the presence of minor sediment suggests that a small fraction of insoluble material remains and that further refinement, such as water washing and filtration of the ash prior to use, may be beneficial.

Table 3: Solubility test results for groundnut husk ash samples

Sample	Location	Solubility in Distilled Water
A	Gumel	Soluble (minor sediment)
B	Kujama	Soluble (minor sediment)
C	Narayi	Soluble (minor sediment)

4.4 Chemical Composition (XRF Analysis)

The elemental oxide compositions of the three GHA samples as determined by XRF analysis are presented in Table 4. K_2O was the dominant oxide in all samples, with concentrations of 46.20%, 39.84%, and 39.36% for samples A (Gumel), B (Kujama), and C (Narayi) respectively. CaO was the second most abundant oxide, ranging from 26.80% (Kujama) to 33.90% (Narayi). Iron oxide (Fe_2O_3) was detected in all samples at low concentrations, likely attributable to soil contamination or contamination from processing tools during the ashing process.

Table 4: Elemental oxide composition of groundnut husk ash samples (wt%) by XRF analysis

Oxide	Sample A (Gumel) wt%	Sample B (Kujama) wt%	Sample C (Narayi) wt%	Mean wt%
K_2O	46.20	39.84	39.36	41.80
CaO	30.50	26.80	33.90	30.40
Fe_2O_3	Trace	Trace	Trace	—
Other oxides	23.30	33.36	26.74	27.80

The K_2O values recorded for all three samples are substantially higher than those reported for wood ash (4–35%; Thy et al., 2006) and comparable to the high-potassium banana peel ash documented by Çınar and Tazeoğlu Filiz (2024). The elevated K_2O content positions GHA as a competitive natural source of potassium oxide for glass production. In the context of glass batch formulation, a typical potash glass batch requires a K_2O content of approximately 13–17% in the final composition (Shelby, 2005). Given that GHA contributes K_2O concentrations of 39–46%, it is evident that only a moderate proportion of GHA relative to silica would be required in the batch, making it economically practical.

The CaO values (26.80–33.90%) are also significant. Calcium oxide serves as the stabilising component in glass formulations, preventing the glass from dissolving in water and improving its mechanical durability. Commercial soda-lime glass typically contains 8–12% CaO (Shelby, 2005). The elevated CaO content of GHA means it simultaneously serves as both a flux (via K_2O) and a stabiliser (via CaO) in a single raw material input, an advantageous dual functionality compared with mono-functional chemical additives.

The variability in oxide concentrations across the three sample sites — particularly the notable difference in K_2O between Sample A (46.20%) and Samples B and C (~39%) — suggests that soil composition and environmental conditions at the cultivation site influence the elemental uptake of the groundnut plant and, consequently, the composition of the resulting ash. This variability has implications for the standardisation of GHA as an industrial raw material, indicating the need for site-specific characterisation and batch normalisation procedures in any scale-up application.

5.0 Conclusion

This study has demonstrated that groundnut husk ash (GHA) sourced from three locations in Kaduna State, Nigeria, contains significant concentrations of potassium oxide (K_2O) and calcium oxide (CaO), confirming its potential as a natural, low-cost fluxing and stabilising agent for glass production. XRF analysis yielded K_2O values ranging from 39.36% to 46.20% and CaO values of 26.80% to 33.90%, concentrations that are well within the range required for effective glass batch formulation. The dual oxide functionality of GHA, which involves simultaneously providing flux (K_2O) and stabiliser (CaO), represents a practical advantage over synthetic single-component additives.

From an environmental standpoint, the

valorisation of groundnut husk ash contributes meaningfully to the reduction of agricultural waste accumulation and the associated risks of methane emission, leachate contamination, and air pollution from open burning. The widespread availability of groundnut husk as an agricultural by-product in northern Nigeria and across sub-Saharan Africa suggests that GHA could support a domestic, low-carbon glass production supply chain.

Economically, the substitution of synthetic potassium carbonate or industrial potash with GHA offers the potential to reduce production costs in small- and medium-scale glass manufacturing, particularly relevant for developing economies where chemical import costs are significant. Additionally, organised collection and processing of GHA could create value-chain opportunities for smallholder groundnut farmers.

Future research should investigate: (i) the full glass batch formulation using GHA in combination with silica and additional stabilisers; (ii) mechanical and optical characterisation of glass produced from GHA-containing batches; (iii) a quantitative assessment of solubility and purity of the ash; (iv) the effect of calcination temperature on ash composition and yield; and (v) the long-term consistency of GHA composition across larger sample populations and multiple growing seasons.

References

Adole, M. A., Dzasu, W. E., Umar, A., & Oraegbune, O. M. (2011). Effects of groundnut husk ash-blended cement on chemical resistance of concrete. *ATBU Journal of Environmental Technology, 4*(1), 1–10.

Çınar, S., & Tazeoğlu Filiz, F. (2024). Use of waste banana peel ash in glaze making. *Yedi, 32*, 1–14. https://doi.org/10.17484/yedi.1398190

De Bardi, M., Hutter, H., Schreiner, M., & Bertoncello, R. (2015). Potash-lime-silica glass: Protection from weathering. *Heritage Science, 3*(1), 22. https://doi.org/10.1186/s40494-015-0051-4

Faugdar, S., & Diwaka, B. (1993). *Nutritive value and uses of pigeon pea and groundnut: Skill development series No. 94*. ICRISAT.

Hossain, S. K. S., Mathur, L., & Roy, P. K. (2018). Rice husk/rice husk ash as an alternative source of silica in ceramics: A review. *Journal of Asian Ceramic Societies, 6*(4), 299–313. https://doi.org/10.1080/21870764.2018.1539210

Indian Standard IS 13855:1993. (1993). *Determination of water-soluble ash and water-insoluble ash*. Bureau of Indian Standards.

Ketkukah, T. S., & Ndububa, E. E. (2006). Groundnut husk ash (GHA) as a partial replacement of cement in mortar. *Nigerian Journal of Technology, 25*(2), 39–45.

Okorie, K. C., Esonu, B. O., Okonkwo, A. C., & Emenalom, O. O. (2013). The effect of groundnut husk on the performance, carcass, apparent nutrient digestibility and economic benefits of broiler finisher chicks. *International Science Research Journal, 4*(2), 100–108.

Sabiti, E. N. (2011). Utilising agricultural waste to enhance food security and conserve the environment. *African Journal of Food, Agriculture, Nutrition and Development, 11*(6), 1–15.

Shelby, J. E. (2005). *Introduction to glass science and technology* (2nd ed.). Royal Society of Chemistry.

Su, M., Ni, L., Rong, S., Gao, Q., Ren, H., & Liu, Z. (2024). The ash-forming element migration and mineral transformation during the bamboo combustion process. *ACS Omega, 9*(20), 21974–21982. https://doi.org/10.1021/acsomega.3c10146

Thy, P., Jenkins, B. M., Lesher, C. E., & Grundvig, S. (2006). Compositional constraints on slag formation and potassium volatilization from rice straw blended wood fuel. *Fuel Processing Technology, 87*(5), 383–408. https://doi.org/10.1016/j.fuproc.2005.08.015

Trisakti, B., Haryuwibawa, D., Pratama, J. M., & Irvan, I. (2018). Effect of temperature on the combustion of Kepok banana (*Musa paradisiaca* Linn cv. "Saba") peel as potassium source. *Talenta Conference Series: Energy and Engineering (EE), 1*(1). https://doi.org/10.32734/ee.v1i1.124

UNEP (United Nations Environment Programme). (2009). *Converting waste agricultural biomass into a resource*. UNEP.

Westerman, P. W., & Bicudo, J. R. (2005). Management considerations for organic waste use in agriculture. *Bioresource Technology, 96*(2), 215–221. https://doi.org/10.1016/j.biortech.2004.05.011