Volume 10, Issue 12, December – 2025 International Journal of Innovative Science and Research Technology
ISSN No:-2456-2165 https://doi.org/10.38124/ijisrt/25dec1406

Effect of Calcination Temperature and Time on the Physicochemical Properties of Rice Husk Ash and Eggshell Ash for Zeolite Synthesis

M. S. Ayuba[1]; S. Y. Goji[2]; L. H. Njila[3]; C. U. Zang[4]; S. N. Atsen[5]; K. G. Tachio[6]

[1;2;3;4;5;6]Department of Science Laboratory Technology, University of Jos, Nigeria

Publication Date: 2026/01/09

Abstract: The conversion of agricultural and biogenic wastes into functional materials offers a sustainable pathway for resource recovery. Rice husk ash and eggshell ash, which are rich in silica and calcium, respectively, are promising raw materials for zeolite synthesis. However, their effectiveness depends strongly on calcination conditions. This study investigated the influence of calcination temperature and calcination time on the physicochemical properties of rice husk ash and eggshell ash. Calcination was carried out at temperatures between five hundred and eight hundred and fifty degrees Celsius for durations ranging from two to six hours. The calcined materials were characterized using x ray diffraction, scanning electron microscopy, Fourier transform infrared spectroscopy, and surface area analysis. Results showed that rice husk ash calcined at seven hundred degrees Celsius produced highly reactive amorphous silica with minimal carbon residues, while higher temperatures promoted partial crystallization. Eggshell ash exhibited progressive calcium carbonate decomposition with increasing temperature, leading to calcium oxide rich phases at higher temperatures, although prolonged heating resulted in particle coarsening. The study demonstrates that controlled calcination is essential for tailoring the reactivity and structural integrity of rice husk ash and eggshell ash, thereby supporting their application as low cost and sustainable precursors for zeolite synthesis.

Keywords: *Agricultural Waste; Biogenic Waste; Rice Husk Ash; Eggshell Ash; Calcination Conditions; Zeolite Synthesis.*

How to Cite: M. S. Ayuba; S. Y. Goji; L. H. Njila; C. U. Zang; S. N. Atsen; K. G. Tachio (2025) Effect of Calcination Temperature and Time on the Physicochemical Properties of Rice Husk Ash and Eggshell Ash for Zeolite Synthesis. *International Journal of Innovative Science and Research Technology*, 10(12), 2773-2779. https://doi.org/10.38124/ijisrt/25dec1406

I. INTRODUCTION

The increasing demand for sustainable materials has intensified research into the conversion of agricultural and biogenic wastes into value-added products. Zeolites are crystalline aluminosilicates widely applied in catalysis, adsorption, and ion exchange due to their high surface area and well-defined porous structures (Chandrasekhar et al., 2003; Yadav et al., 2021). Conventional zeolite synthesis relies on commercial silica and alumina sources, which are expensive and energy-intensive. Consequently, the use of waste-derived precursors offers both economic and environmental advantages [15].

Rice husk is an abundant agricultural by-product, particularly in rice-producing regions of Africa and Asia. In Nigeria, large quantities of rice husk are generated annually and are often disposed of by open burning, leading to environmental concerns. Studies by [5] and [1] have reported that rice husk ash obtained under controlled calcination conditions contains high proportions of reactive amorphous silica suitable for advanced material synthesis. Similarly, eggshell waste is widely generated from domestic and commercial food activities across Nigeria. [6] and [13] demonstrated that calcined eggshells can serve as effective calcium sources after thermal decomposition of $CaCO_3$ to CaO. These studies highlight the potential of locally available biogenic wastes as sustainable raw materials for industrial applications.

The physicochemical properties of rice husk ash and Eggshell ash are strongly influenced by calcination temperature and time. In Rice Husk Ash, insufficient calcination results in residual carbon and poorly condensed silica, whereas excessive heating promotes crystallization of amorphous silica into quartz, reducing reactivity [11]. Similarly, Eggshell ash (ESA) requires sufficiently high temperatures to achieve complete $CaCO_3$ decomposition, although prolonged heating can induce sintering and loss of surface area [8]. Despite their importance, comparative studies examining the combined effects of calcination temperature and duration on both RHA and ESA remain limited.

This study, therefore, investigates the influence of calcination temperature (500–850 °C) and time (2–6 h) on the physicochemical properties of Rice husk ash (RHA) and Eggshell ash (ESA). Using X-ray diffraction (XRD), scanning electron microscopy (SEM), Fourier transform infrared spectroscopy (FTIR), and surface area analysis, optimal calcination conditions for producing reactive silica and calcium precursors suitable for zeolite synthesis are identified.

II. MATERIALS AND METHODS

➢ *Materials*

Rice husk was obtained from a local rice mill, while eggshells were collected from poultry waste within Jos, Nigeria. The raw materials were thoroughly washed with distilled water to remove adhering impurities, oven-dried at 105 °C for 24 h, and ground into fine powders before calcination.

➢ *Calcination Procedure*

The dried rice husk and eggshell powders were calcined in a muffle furnace at temperatures ranging from 500 to 850 °C for 2–6 h. The heating rate was maintained at 10 °C min^{-1}, and the samples were allowed to cool naturally inside the furnace to room temperature. The calcined samples were labeled according to material type, temperature, and calcination time.

➢ *Characterization*

The physicochemical properties of the calcined samples were evaluated using standard techniques. X-ray diffraction (XRD) was used to identify crystalline phases and assess structural evolution. Scanning electron microscopy (SEM) coupled with energy-dispersive X-ray spectroscopy (EDX) was employed to examine surface morphology and elemental composition. Fourier transform infrared spectroscopy (FTIR) was used to monitor chemical bonding and phase transformations. Brunauer–Emmett–Teller (BET) surface area analysis was conducted to determine textural properties relevant to zeolite synthesis.

III. RESULTS AND DISCUSSION

➢ *Morphological Evolution Scanning Electron Micrscopy*

SEM analysis revealed distinct temperature-dependent morphological changes in both materials. RHA calcined at 500 °C retained partially collapsed biogenic structures with residual carbon, consistent with incomplete combustion reported for low-temperature [14]. Increasing the temperature to 700 °C produced highly disordered, amorphous silica particles with minimal organic remnants, in agreement with previous studies on thermally treated rice husk ash [11]; [12].

In contrast, ESA displayed dense, block-like particles at 500 °C, characteristic of undecomposed calcite. At higher temperatures, particularly 700–850 °C, progressive particle fragmentation and surface roughening were observed, reflecting $CaCO_3$ decomposition and CaO formation. Similar morphological evolution has been reported for biogenic calcium carbonate subjected to high-temperature calcination [1]. SEM micrographs revealed distinct morphological transformations in RHA and ESA with increasing calcination temperature (Fig. 1). The corresponding morphological features and their implications for zeolite synthesis are summarized in Table 1.

Table 1 Summary of SEM-observed morphological changes in rice husk ash (RHA) and eggshell ash (ESA)

Sample	Calcination condition (°C)	Key SEM morphological features	Implications for zeolite synthesis
RHA	500 (4-6 h)	Partially preserved biogenic silica framework; collapsed honeycomb structures; irregular flake-like particles; residual carbon patches; limited pore opening	Incomplete carbon removal; low silica reactivity
RHA	600 (4 h)	Greater framework disruption; cleaner silica surfaces; increased pore opening; reduced carbonaceous residues	Improved silica activation; transitional calcination stage
RHA	700 (4-6 h)	Highly disordered amorphous silica; rough surfaces; well-developed interparticle voids; carbon-free; slight agglomeration at longer times	Optimal amorphous silica reactivity; suitable precursor for zeolite synthesis
ESA	500 (4-6hr)	Dense, block-like $CaCO_3$ particles; smooth fracture surfaces; microcracks with prolonged time; minimal porosity	Incomplete $CaCO_3$ decomposition; low CaO reactivity
ESA	700 (4-6 h)	Fragmented and granular particles; increased surface roughness; microvoids; partial $CaCO_3 \rightarrow CaO$ transformation	Enhanced surface activation; suitable calcium source
ESA	850 (2 h)	Highly fragmented CaO-rich particles; rough surfaces; well-developed porosity; minimal sintering	Highly reactive CaO; optimal high-temperature condition
ESA	850 (5 h)	Coarser particles; localized necking and sintering; reduced interparticle voids	Complete CaO formation with reduced surface area due to sintering

RHA = Rice Husk Ash; ESA = Eggshell Ash; C = Carbonaceous Residue

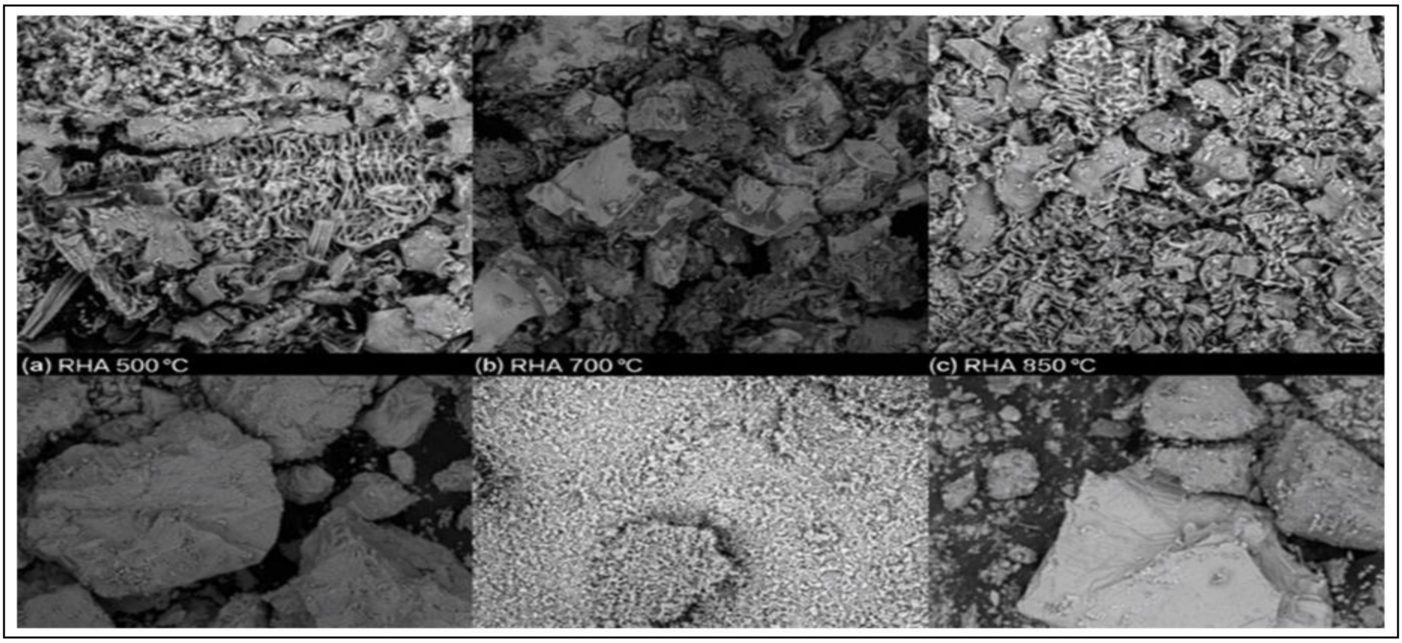

Fig 1 SEM Micrographs Showing Morphological Evolution of Rice Husk Ash (RHA) and Eggshell Ash (ESA) at Different Calcination Temperatures: (a) RHA 500 °C, (b) RHA 600 °C, (c) RHA 700 °C, (d) ESA 500 °C, (e) ESA 700 °C, and (f) ESA 850 °C

➢ *Chemical Evolution (FTIR)*

- *FTIR Analysis*

Fourier transform infrared (FTIR) spectroscopy was employed to examine the evolution of functional groups and bonding environments in rice husk ash (RHA) and egg*[shell ash (ESA) as a function of calcination temperature and time (Figs. 3 and 4). For RHA calcined at 500 °C, broad absorption bands around 3400 cm^{-1} and 1630 cm^{-1} were observed, corresponding to O–H stretching and bending vibrations associated with adsorbed moisture and surface silanol groups. The presence of a strong band in the region 1090–1100 cm −1, together with bands near 800 and 470 cm^{-1}, confirms the formation of an amorphous siliceous network.

With increasing calcination temperature to 600 and 700 °C, the intensity of hydroxyl-related bands progressively decreased, indicating dehydroxylation and improved condensation of the silica framework. At 700 °C, the dominance of Si–O–Si vibrational bands suggests the formation of highly condensed, amorphous silica, which is desirable for zeolite synthesis due to its high reactivity.

In contrast, ESA calcined at 500 °C exhibited strong absorption bands in the range 1410–1450 cm^{-1} and around 870 cm −1 characteristic of carbonate (CO_3^{2-}) groups and confirming the dominance of calcium carbonate. Upon calcination at 700 °C, these carbonate bands weakened significantly, while a new band near 3640 cm^{-1} emerged, attributed to Ca–O–H stretching vibrations. This indicates progressive $CaCO_3$ decomposition and formation of CaO/Ca $(OH)_2$ species. These FTIR observations are consistent with SEM and XRD results and confirm that higher calcination temperatures promote chemical transformations favorable for zeolite precursor preparation.

The evolution of functional groups and bonding environments in RHA and ESA with calcination temperature was examined using FTIR spectroscopy (Fig. 3 and 4). The major absorption bands and their structural interpretations are summarized in Table 2.

➢ *BET Surface Area and Porosity Analysis*

The influence of calcination temperature on the surface area and pore structure of RHA and ESA was evaluated using BET analysis, with representative samples calcined for 4 h (Table 3). RHA calcined at 500 °C exhibited moderate surface area and limited pore development, which can be attributed to incomplete removal of residual carbon and partial collapse of the biogenic silica framework. Increasing the calcination temperature to 700 °C led to a marked increase in surface area and pore volume, reflecting the development of a well-defined mesoporous structure and improved pore connectivity. These textural properties are favorable for zeolite nucleation and crystal growth.

ESA calcined at 500 °C showed very low surface area and negligible porosity due to the dense nature of undecomposed $CaCO_3$ particles. Calcination at 700 °C resulted in increased surface area and pore volume, associated with CO_2 release during $CaCO_3$ decomposition and the formation of porous CaO. However, a reduction in surface area was observed for ESA calcined at 850 °C, which is attributed to particle coarsening and localized sintering at elevated temperatures. Surface area reduction at excessive calcination temperatures due to sintering has also been reported for locally sourced ash materials by [6], reinforcing the need for optimized thermal treatment. Overall, moderate calcination temperatures were found to be optimal for enhancing surface properties relevant to zeolite synthesis, while excessive thermal treatment adversely affected porosity. The influence of calcination temperature on the surface area and pore structure of RHA and ESA, determined from BET analysis, is summarized in Table 3.

Table 2 Summary of FTIR Absorption Bands and Corresponding Chemical Changes in RHA and ESA as a Function of Calcination Temperature and Time.

Material	Calcination condition	Major FTIR bands (cm^{-1})	Band assignment	Structural interpretation
RHA	500 °C (4–6 h)	~3400	O–H stretching	Adsorbed moisture and surface silanol groups
		~1630	H–O–H bending	Residual water associated with incomplete combustion
		~1090–1100	Si–O–Si asymmetric stretching	Formation of amorphous silica framework
		~800, ~470	Si–O–Si symmetric stretching and bending	Silica network vibrations
RHA	600 °C (4 h)	~3400 (reduced)	O–H stretching	Progressive dehydroxylation of silica surface
		~1090–1100 (sharper)	Si–O–Si stretching	Improved silica network condensation
RHA	700 °C (4–6 h)	~1090–1100 (dominant)	Si–O–Si asymmetric stretching	Highly condensed, amorphous and reactive silica
		~800, ~470	Si–O–Si vibrations	Stabilized silica framework
ESA	500 °C (4–6 h)	~1410–1450	CO_3^{2-} asymmetric stretching	Dominant calcium carbonate phase
		~870	CO_3^{2-} out-of-plane bending	Undecomposed calcite
ESA	700 °C (4–6 h)	~1410–1450 (reduced intensity)	Carbonate stretching	Progressive $CaCO_3$ decomposition
		~870 (weak or disappearing)	Carbonate bending	Transition from $CaCO_3$ to CaO
		~3640	Ca–O–H stretching	Formation of CaO/$Ca(OH)_2$ species

Table 3 Summary of BET Surface Area and Porosity Characteristics of RHA and ESA as a Function of Calcination Temperature and Time.

Sample	Calcination condition	Surface area ($m^2\ g^{-1}$)	Total pore volume ($cm^3\ g^{-1}$)	Modal pore width (nm)	Implication
RHA	500 °C, 4 h	53.90	0.06	2.65	Partial pore development
RHA	700 °C, 4 h	75.31	0.09	2.65	Highly mesoporous, optimal for zeolite synthesis
ESA	500 °C, 4 h	39.09	0.05	2.65	Low porosity, incomplete $CaCO_3$ decomposition
ESA	700 °C, 4 h	70.45	0.09	2.65	Enhanced pore formation
ESA	850 °C, 2 h	—	—	—	High CaO purity, some pore collapses due to sintering

- *Phase Transformation (XRD)*

- *Eggshell Ash (ESA)*

The XRD pattern of ESA calcined at 500 °C (4 h) is dominated by sharp diffraction peaks characteristic of calcium carbonate ($CaCO_3$), mainly in the calcite phase. This indicates that calcination at this temperature is insufficient to induce complete thermal decomposition of $CaCO_3$. Similar observations have been reported for eggshell-derived materials calcined below 700 °C, where carbonate phases remain largely preserved due to inadequate thermal energy for CO_2 release [10].

At 700 °C (4 h), a noticeable reduction in the intensity of $CaCO_3$ peaks is observed, accompanied by the appearance of reflections associated with calcium oxide (CaO). This confirms the onset of $CaCO_3$ decomposition, which typically occurs in the temperature range of 700–800 °C according to the reaction $CaCO_3 \rightarrow CaO + CO_2$. The coexistence of carbonate and oxide phases at this temperature suggests partial decomposition, consistent with earlier reports on thermally treated biogenic calcium carbonate sources [8].

For ESA calcined at 850 °C, the diffraction pattern is dominated by CaO reflections, while carbonate peaks are either absent or significantly suppressed. This confirms near-complete decomposition of $CaCO_3$ into CaO, yielding a highly reactive oxide phase. Previous studies have shown that calcination above 800 °C promotes complete carbonate decomposition but may also induce sintering if prolonged, which aligns well with the SEM observations in this study (Rodríguez-Navarro et al., 2009; Wang et al., 2020). The formation of CaO-rich ESA at this temperature is advantageous for zeolite synthesis, where alkaline conditions and calcium availability facilitate framework formation and ion-exchange [15].

- *Rice Husk Ash (RHA)*

The XRD patterns of RHA calcined at 500 °C (4 h) and 700 °C (4 h) are characterized by a broad diffuse halo centered around $2\theta \approx 20–25°$, which is a typical signature of amorphous silica (SiO_2). The absence of sharp crystalline peaks indicates that silica remains predominantly in the amorphous state within this temperature range. This behavior is well documented for rice husk ash produced under controlled combustion conditions [9];[11].

At 500 °C (4 h), the amorphous halo is present but relatively less pronounced, which may be attributed to incomplete combustion of organic matter and the presence of residual carbon. Increasing the calcination temperature to 700 °C (4 h) leads to a more distinct amorphous silica hump, indicating enhanced removal of carbonaceous residues and stabilization of amorphous SiO_2. Importantly, no significant quartz peaks are observed at 700 °C, suggesting that crystallization of silica has not yet occurred. This is critical, as crystallization into quartz typically begins at temperatures above 800–900 °C and significantly reduces silica reactivity [11]. The retention of amorphous silica at 700 °C is particularly desirable for zeolite synthesis, since amorphous SiO_2 dissolves more readily in alkaline media than crystalline silica, thereby enhancing nucleation and growth of zeolite frameworks [7].

Fig. 2 & 3 X-ray diffraction (XRD) patterns of (a) eggshell ash (ESA) and (b) rice husk ash (RHA) calcined at selected temperatures (500–850 °C). ESA patterns show progressive decomposition of $CaCO_3$ with increasing temperature, evidenced by the disappearance of calcite reflections and emergence of CaO phases at higher temperatures. RHA patterns are dominated by a broad amorphous halo centered around $2\theta \approx 22°$, characteristic of amorphous silica, with reduced crystalline contributions at elevated calcination temperatures.

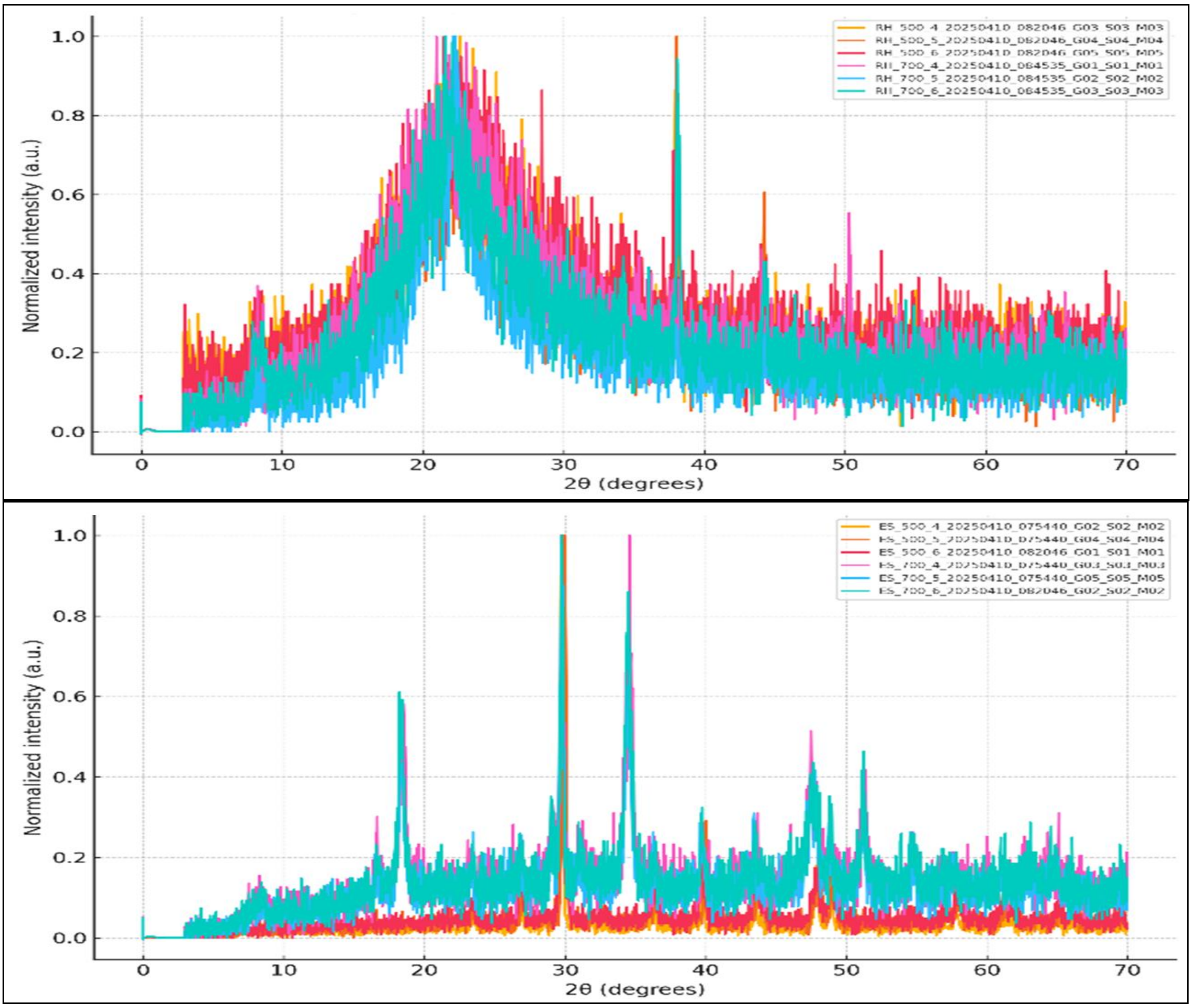

Fig 2 & 3 X-ray Diffraction (XRD) Patterns of (a) Eggshell Ash (ESA) and (b) Rice Husk Ash (RHA) Calcined at Selected Temperatures (500–850 °C).

- *Implications for Zeolite Synthesis*

The combined SEM, FTIR, and XRD results demonstrate that RHA calcined at 700 °C provides highly reactive amorphous silica, while ESA calcined at 700–850 °C yields CaO-rich materials suitable for alkaline activation and ion exchange during zeolite synthesis. These findings align with previous reports emphasizing the importance of controlled thermal treatment when using waste-derived precursors [15].

IV. CONCLUSION

This study systematically investigated the influence of calcination temperature and time on the physicochemical properties of rice husk ash (RHA) and eggshell ash (ESA), with the goal of identifying optimized conditions for their use as precursors in zeolite synthesis. Results demonstrated that RHA calcined at 700 °C for 4–6 h produced highly reactive amorphous silica with minimal carbon residues and well-developed porosity, as confirmed by SEM, FTIR, and BET analyses. In contrast, RHA calcined at lower temperatures retained carbonaceous material, while temperatures above 800 °C tended toward silica densification and reduced surface area.

For ESA, calcination at 700–850 °C effectively decomposed $CaCO_3$ into CaO, with improving surface roughness and porosity as temperature increased. However, prolonged calcination at the highest temperature promoted localized sintering, reducing effective surface area. The combined results indicate that controlled calcination enhances both silica and calcium precursor quality, laying the groundwork for cost-effective and sustainable zeolite synthesis from agro-waste sources. Future work should explore the direct application of these optimized ashes in zeolite formation experiments and evaluate their performance in specific industrial applications.

ACKNOWLEDGMENT

The authors gratefully acknowledge the financial support provided by the Tertiary Education Trust Fund (TETFund) through an institutional-based research grant. The authors also appreciate the Department of Science Laboratory Technology, University of Jos, Nigeria, for providing the laboratory facilities and technical support that made this research possible. Special thanks to Mr Rigat David Mahanan for typesetting this work.

REFERENCES

[1]. Abubakar, A.U., Hassan, L.G. & Dangoggo, S.M. (2019). Effect of calcination temperature on silica obtained from Nigerian rice husk. Nigerian Journal of Basic and Applied Sciences, 27, 55–63.

[2]. Abubakar, A.U., Hassan, L.G. & Dangoggo, S.M (2019). Effect of calcination temperature on silica obtained from Nigerian rice husk. Nigerian Journal of Basic and Applied Sciences, 27, 55–63.

[3]. Adam, F., Chew, T.S. & Andas, J. (2020). The effect of calcination temperature on rice husk ash silica. Journal of Materials Research and Technology, 9: 127–136.

[4]. Adebayo, O.F., Akinola, O.S. & Olatunji, A.O. (2020). Thermal processing of eggshell waste for calcium oxide production. Nigerian Journal of Materials Science, 12: 45–53.

[5]. Adam, F., Chew, T.S. & Andas, J. (2020). The effect of calcination temperature on rice husk ash silica. Journal of Materials Research and Technology, 9: 127–136.

[6]. Adebayo, O.F., Akinola, O.S. & Olatunji, A.O. (2020). Thermal processing of eggshell waste for calcium oxide production. Nigerian Journal of Materials Science, 12: 45–53.

[7]. Adeyemi, A.O., Lawal, I.O. & Balogun, S.O. (2021). Characterization of eggshell-derived CaO for industrial applications. Materials Today: Proceedings, 38: 799–805.

[8]. Adejumo, A.L., Babatunde, M.A. & Ogunbode, E.B. (2012). Production and characterization of rice husk ash as a pozzolanic material. Journal of Engineering and Applied Sciences, 7, 138–144.

[9]. Akinwumi, I.I., Booth, C.A. & Adeyeri, J.B. (2014). Experimental investigation of calcium carbonate derived from eggshell waste. International Journal of Engineering Research in Africa, 11, 23–31. https://doi.org/10.4028/www.scientific.net/JERA.11.23

[10]. Cejka, J., van Bekkum, H., Corma, A. & Schueth, F. (2010). Introduction to Zeolite Science and Practice, 3rd ed. Amsterdam: Elsevier.

[11]. Gupta, S. & Mohapatra, H. (2021). Thermal decomposition and reactivity of biogenic calcium carbonate. Journal of Cleaner Production, 278, 123987. https://doi.org/10.1016/j.jclepro.2020.123987

[12]. Kalapathy, U., Proctor, A. & Shultz, J. (2000). A simple method for production of pure silica from rice hull ash. Bioresource Technology, 73, 257–262.https://doi.org/10.1016/S0960-8524(99)00127-3

[13]. King'ori, A.M. (2011). A review of the uses of poultry eggshells and shell membranes. International Journal of Poultry Science, 10, 908–912.

[14]. Liou, T.H. (2004). Preparation and characterization of nano-structured silica from rice husk. Materials Science and Engineering A, 364, 313–323.https://doi.org/10.1016/j.msea.2003.08.045

[15]. Mansour, A.M., El-Desouky, M.G. & Rashad, M.M. (2022). Effect of calcination on physicochemical properties of agricultural waste-derived silica. Ceramics International, 48, 9876–9886. https://doi.org/10.1016/j.ceramint.2021.12.045

[16]. Onwuka, D.O., Okoye, P.A.C. & Okonkwo, P.C. (2020). Characterization of calcined eggshell as a calcium source for industrial applications. Journal of Applied Sciences and Environmental Management, 24, 1765–1771. https://doi.org/10.4314/jasem.v24i10.14

[17]. Oyekanmi, A.A., Ajao, J.A. & Salami, L.A. (2022). Physicochemical properties of Nigerian rice husk ash. Journal of Applied Sciences and Environmental Management, 26: 455–462.

[18]. Querol, X., Moreno, N., Umaña, J.C. et al. (2002). Synthesis of zeolites from coal fly ash: An overview. International Journal of Coal Geology, 50, 413–423. https://doi.org/10.1016/S0166-5162(02)00124-