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Synthesis, Compositional, Morphological, Structural, and Thermal Properties of Eggshell Calcium Oxide for Solar Photovoltaic Glass

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Abstract

The increasing demand for sustainable materials in engineering applications has prompted the exploration of eco-friendly alternatives. Conventional calcium oxide (CaO) production from limestone is energy-intensive and environmentally detrimental, prompting interest in biogenic alternatives such as poultry eggshell waste, predominantly composed of calcium carbonate. Despite extensive research into eggshell-derived CaO for catalytic and biomedical applications, its potential for photovoltaic glass applications, particularly considering region-specific variations in material properties, has not been adequately investigated. This study comprehensively evaluates CaO extracted from chicken eggshells sourced from Lafia Local Government Area, Nasarawa State, Nigeria, to establish its suitability for photovoltaic glass manufacturing. The extraction process involved thermal calcination at 900°C for two hours, followed by detailed compositional, morphological, structural, and thermal characterizations using XRF, SEM-EDS, XRD, and TGA techniques. The results demonstrated brilliant white powder with high compositional purity (98.58% CaO), nanoscale spherical morphology conducive to uniform integration in glass matrices, robust crystallinity beneficial for structural stability, and strong thermal resilience crucial for high-temperature processing. These findings highlight eggshell-derived CaO as an economically viable, sustainable, and high-performance alternative for PV glass manufacturing. Further studies are recommended to assess practical integration in PV glass formulations, long-term durability, study of farming systems, and lifecycle economic viability.

Keywords: Sustainability, biogenic material, circular economy, eggshell calcium oxide, calcination, eco-friendly, glass

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Introduction

The global transition towards sustainable materials for advanced engineering applications has intensified the search for eco-friendly alternatives. The availability of low-carbon raw materials is crucial for addressing climate change and meeting the Paris Agreement goals [1]. Valorizing agricultural residues into engineering materials adds economic value and mitigates the environmental and health hazards associated with waste disposal [2]. Solid waste generation, particularly food waste, remains a pressing sustainability issue, significantly contributing to pollution [3]. The FAO estimates 1.3 billion tons of edible food waste are generated annually, equating to 3.3 billion tonnes of CO_2, 1.4 hectares of land, and 250 × 10³ m³ of water, with a projected 44% increase by 2025 [4]. Notably, eggshells, generated from domestic sources, pose such a serious environmental concern [5]. While commonly discarded in landfills, eggshells have significant potential for value-added applications, particularly as a major constituent of $CaCO_3$, which serves as a potential source of CaO in numerous applications such as antibacterial composites [6, 7], removal of heavy metals, from aqueous solution [8, 9], photocatalytic application [10], biodiesel synthesis [11], synthesis of gypsum fertilizer [12], solid base catalyst [13–15], fillers [16], Portland cement [17], adsorbent for greenhouse gas (CO_2) [18, 19]. It has also been explored as a modifier in synthesizing glass containing calcium oxide, showcasing a sustainable approach [20–23]. Utilizing eggshell waste as a source of calcium for numerous industrial applications is considered an environmentally friendly approach that converts waste into a valuable resource. This aligns with the principles of sustainability and eco-friendliness [24]. Conventionally, calcium oxide is synthesized from natural limestone through an energy-intensive high-temperature process that generates significant carbon dioxide emissions. In contrast, biogenic calcium carbonate sources like eggshells offer a more sustainable alternative due to their high purity, abundance, and eco-friendly nature [25, 26]. Among various synthesis methods, simple calcination remains the most efficient and scalable technique for producing CaO from eggshells. Unlike hydrothermal, precipitation, or sol-gel methods, which involve complex steps, reagents, and longer durations, thermal calcination provides a cost-effective and environmentally benign route to generate reactive CaO [27, 28].

The extraction of calcium oxide from eggshells through calcination provides a straightforward, cost-effective, and environmentally friendly method. This approach capitalizes on the abundant calcium carbonate content in eggshells to produce high-purity calcium oxide for a wide range of industrial applications, as demonstrated by the comprehensive investigation conducted by Tangboriboon *et al*. [29] into the properties of calcium oxide obtained via this process. To ensure the production of high-purity calcium oxide, the researchers carefully washed, dried at ambient temperature, and finely ground the eggshells into a powder. The powder was then calcined at temperatures ranging from 300 to 900°C at an interval of 1 to 5 h. The study demonstrated that optimal purity (99.6%) was attained at 900°C within one h. TEM characterization indicated excellent dispersion of nearly uniform particles, and TGA confirmed the complete transformation of calcium carbonate into calcium oxide at 900°C. A similar approach was taken by Alobaidi *et al*. [30], where eggshells were cleaned, dried, and ground into powder before calcination at 900°C for one h. This method yielded CaO nanoparticles (NPs) with a cubic crystalline structure, verified by XRD analysis, and SEM images depicted nearly spherical granules with a mean particle size ranging from 20–70 nm. While the study did not specify the percentage purity, results reinforced the efficiency of thermal decomposition in producing nanoscale CaO suitable for catalysis and advanced material applications. Researchers have also explored pre-treatment methods, such as acid treatment. In addition to simple calcination for extracting calcium oxide from eggshells, Hsieh *et al*. [18] developed a sol-gel approach where eggshells were treated with a 1% hydrochloric acid solution, forming calcium chloride, followed by calcination at 900°C for two hours. This combined technique significantly enhanced the purity of the final calcium oxide product by effectively removing organic impurities. Furthermore, the efficiency of the calcination process through thermal decomposition is influenced by several key parameters, primarily temperature and duration, as demonstrated by Razali *et al*. [3]. Their findings indicate that the optimal decomposition of eggshell-derived calcium carbonate occurs at approximately 900°C, with calcination times under three hours. Lower temperatures typically resulted in incomplete decomposition, residual carbonate presence, and reduced purity, while extending the calcination beyond the optimal range offered minimal additional purity improvement. Interestingly, variations in the raw material mass had negligible effects on the calcination outcomes, suggesting that temperature and duration are the primary factors governing the efficiency of the calcination process. Characterization techniques consistently demonstrate near-complete conversion of eggshell-derived calcium carbonate to calcium oxide upon thermal treatment. X-ray diffraction analyses of the calcined powders reveal patterns matching standard calcium oxide diffraction peaks, along with the disappearance of carbonate-related peaks [3]. Scanning electron microscopy generally depicts the presence of white, spherical, and porous calcium oxide particles [6, 10]. Similarly, Bhuvaneshwari *et al*. [31] study observed moderately dispersed, porous calcium oxide particles with narrow size distributions. Thermogravimetric analyses confirm that the decomposition of calcium carbonate predominantly occurs within the temperature range of approximately 600–750°C, with the process being completed at 900°C [14]. Furthermore, the storage duration of the calcined samples significantly impacts the thermal characteristics of calcium oxide due to its hygroscopic nature. Study by Hemmami *et al*. [9], have noted notable weight losses between 300–400°C in thermogravimetric analyses, primarily attributed to the desorption of physically or chemically adsorbed atmospheric moisture. This observation emphasizes the importance of controlled storage environments for calcium oxide intended for precise industrial applications, such as photovoltaic glass manufacturing.

The widespread industrial use of calcium oxide spans glass manufacturing, ceramics, catalysis, and energy sectors. However, this is constrained by the environmental and energy-intensive nature of conventional limestone-derived CaO production. Conversely, recent interest has grown in biogenic alternatives, notably poultry eggshells, due to their high calcium carbonate content, cost-effectiveness, and sustainability as raw materials for thermal decomposition into CaO. Despite extensive application in catalytic and biomedical fields, research investigating eggshell-derived CaO for photovoltaic glass applications remains limited, particularly the impact of regional variability on the physicochemical properties. Therefore, this study presents a sustainable approach towards Calcium oxide synthesis for PV glass with particular emphasis on the source of the eggshell, by comprehensively characterizing CaO synthesized from chicken eggshells sourced in Lafia Local Government Area, Nasarawa State, Nigeria. Furthermore, the investigation evaluates compositional purity, crystallinity, morphology, and thermal stability, aiming to establish the material's suitability for solar PV glass manufacturing and broader industrial applications. By exploring this region-specific biogenic resource, the research contributes significantly to advancing circular economy practices and aligns with UN Sustainable Development Goals 12 and 13, offering a promising pathway towards green material innovation.

Materials and Methods

Calcium oxide extraction

For the extraction of this biogenic calcium oxide, chicken eggshells were collected from a local poultry farm in Lafia Local Government Area, Nasarawa State, Nigeria, and subjected to thermal decomposition as shown in Fig. 1. Initially, the eggshells were thoroughly washed with tap water to remove impurities and organic residues. Subsequently, the internal eggshell membrane was carefully removed, followed by additional rinsing with deionized water. The cleaned eggshells were then oven-dried in a microwave oven, mechanically crushed, and ground into powder. The powdered material was placed in a ceramic crucible and calcined in a muffle furnace at 900°C for 2 h, effectively converting the calcium carbonate to calcium oxide.

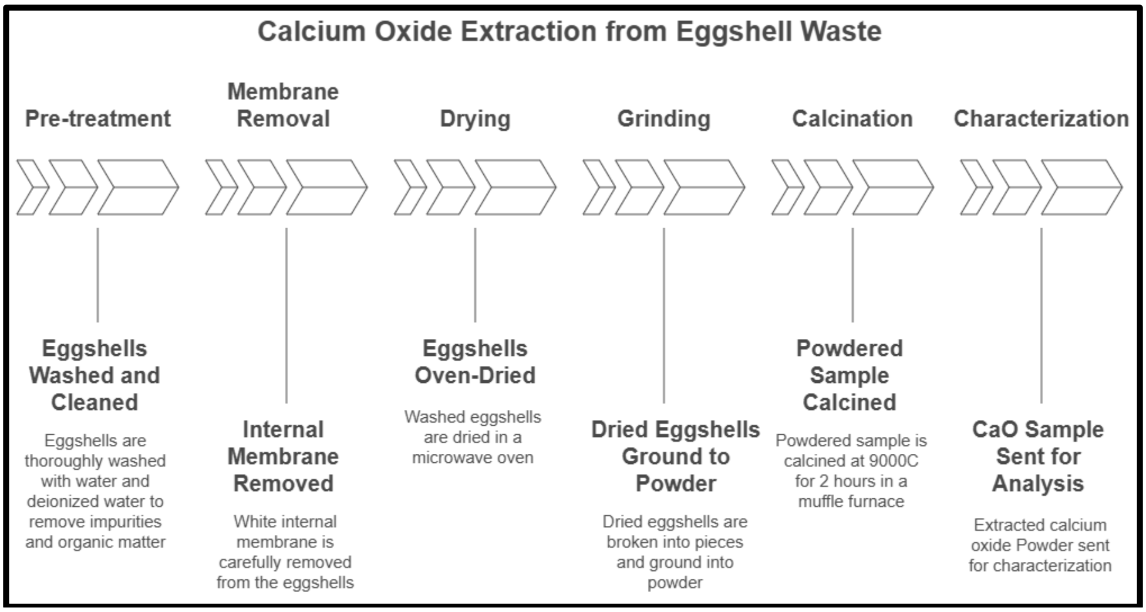

Figure 1: Eggshell calcium oxide extraction process

X-ray fluorescence analysis
To analyze the chemical composition of the samples, X-ray fluorescence analysis was conducted at the central laboratory of Ahmadu Bello University, Zaria, using a Genius IF Xenemetrix XRF system. The prepared Calcium oxide powder was placed into a Prolene film-sealed sample cup and mounted in the XRF system. Furthermore, the X-ray lamp was preheated to stabilize emission intensity, and analytical parameters such as voltage were carefully adjusted. Subsequently, the spectral data were acquired and analyzed using XRS-FP software, referencing the Master Oxide.tfr calibration file for precise quantification of the elemental composition.

Scanning electron microscopy–energy dispersive X-ray spectroscopy
Morphological and elemental characterization was conducted using a Phenom ProX scanning electron microscope at the Central Laboratory of the Umaru Musa Yar'Adua University, Katsina. Calcium Oxide powder sample was mounted securely onto aluminum sample stubs using double-sided adhesive carbon tape and sputter-coated with a thin gold layer using a Quorum Technologies sputter coater. This coating minimized electron charging and improved image conductivity. Mounted samples were then placed into the SEM chamber, where they were initially examined using the Navigation Camera for preliminary focusing and alignment. Subsequently, high-resolution imaging was conducted in SEM mode following automatic brightness and contrast calibration, achieving magnifications of approximately 4607× and resolutions of 127.4 eV. Captured images were digitally stored for detailed morphological analyses. Additionally, elemental compositional analysis via energy-dispersive X-ray spectroscopy was performed on selected zones within the SEM micrographs. The quantitative elemental composition (EDS) was determined as weight percentage and atomic percentage, calculated based on peak intensities generated from characteristic X-ray emissions using integrated mapping software.

X-ray diffraction analysis
The structural characterization was conductedat the Central Laboratory of the Umaru Musa Yar'adua University, Katsina, usingthe X-ray diffraction technique with an ARL'XTRA diffractometer, utilizing monochromatic Cu-Kα radiation. Powdered samples were analyzed in Bragg-Brentano geometry over a 2θ range of 10°–80°, with increments of 0.02° and a scanning speed of 1°/min. Additionally, phase identification and structural characterization were performed via X'Pert HighScore Plus software, employing the ICDD PDF-4+ database and Rietveld refinement to quantify phase compositions, crystallite sizes, and structural parameters.

Thermogravimetric analysis
Thermogravimetric analysis was carried out using a PerkinElmer TGA/DTA 4000 analyzer at the Central Laboratory of the ABU Zaria. Samples were accurately weighed and loaded into alumina crucibles. Furthermore, the analyses were performed under nitrogen flow to maintain an inert environment. Additionally, samples underwent heating from 30 to 950°C at a controlled rate of 10°C/min, allowing a

comprehensive assessment of thermal decomposition, mass loss, and thermal stability. Finally, after cooling, the thermogravimetric data were analyzed to interpret the thermal characteristics and stability profiles of the calcium oxide samples.

Results and Discussion

Calcium oxide extraction

The thermal decomposition of Lafia-sourced chicken eggshells produced a high-purity, brilliant white calcium oxide powder, as shown in Fig. 2, demonstrating suitability for photovoltaic glass applications. Furthermore, the synthesis yielded approximately 60% of the initial raw material weight, aligning with stoichiometric predictions due to carbon dioxide release during calcination.

The resulting white color confirms efficient removal of organic residues, iron oxides, and eggshell membranes, indicative of complete calcination. In contrast, gray or off-white coloration typically denotes incomplete decomposition or contamination [27]. Moreover, the high purity achieved in this work is attributed to rigorous pre-treatment processes, including thorough washing, meticulous membrane removal, precise particle size management, and optimized calcination conditions under oxidizing atmospheres. These factors ensured uniform heat distribution, complete decomposition of calcium carbonate, and oxidation of residual organic compounds, ultimately enhancing CaO quality [32]. Consequently, the synthesized CaO meets the purity standards required for integration into advanced silicate-based photovoltaic glasses.

Figure 2: Extracted eggshell calcium oxide powder

Table 1: XRF analysis of the extracted calcium oxide

Oxides	% Concentration in CaO powder
SiO_2	0.302
V_20_5	0.013
Cr_2O_3	0.001
MnO	0.003
Fe_2O_3	0.034
CoO	0.036
NiO	0.017
CuO	0.035
Nb_2O_5	0.007
WO_3	0.013
P_2O_5	0.004
SO_3	0.02
CaO	98.58
BaO	0.02
Al_2O_3	0.748
Ta_2O_5	0.007
TiO_2	0.036
ZnO	0.012
ZrO_2	0.037
SnO_2	0.075

Compositional analysis

The chemical composition of the extracted eggshell CaO powder characterized via the XRF techniques demonstrates a notably high purity level of approximately 98.58%, affirming the efficacy of the extraction and calcination procedures employed as presented in Table 1.

This high purity is critical, as it confirms the complete thermal conversion of calcium carbonate from eggshells into calcium oxide, effectively removing associated impurities [33]. Furthermore, the minor impurities detected include alumina, silica, and tin oxide, consistent with typical residues attributed to environmental contamination or original feedstock composition [34]. Notably, these low-level impurities are tolerable in industrial glass applications, as they can act beneficially as network formers and stabilizers, potentially enhancing the glass's structural integrity and thermal stability. Moreover, recent studies highlight the importance of maintaining CaO purity above 96–98% for specialized industrial applications, such as photovoltaic glass production, emphasizing the role of purity in achieving desirable optical transparency and chemical stability [35]. Additionally, trace metallic impurities, including iron and cobalt, are typically controlled through precise pre-treatment and optimized calcination conditions. Lastly, the achieved compositional purity aligns well with a similar study of Zia [14], where optimal particle size, membrane removal, and controlled oxidative calcination atmospheres significantly influenced purity levels and compositional profiles. Thus, the high purity of 98.58% obtained confirms the suitability of the synthesized CaO powder for photovoltaic glass and related advanced engineering applications where enhanced optical transparency is required [36].

Elemental analysis

The energy-dispersive X-ray spectroscopy analysis of the synthesized calcium oxide from eggshells demonstrates a compositional profile predominantly comprising calcium, oxygen, and minor carbon, as depicted in Table 2 and Fig. 3.

Table 2: EDS analysis of the extracted eggshell calcium oxide

Element	Symbol	% Weight Concentration
Calcium	Ca	49.85
Oxygen	O	45.65
Carbon	C	2.95
Others		1.55

Specifically, the elemental composition includes approximately 49.85 wt.% calcium and 45.65 wt.% oxygen, which collectively substantiate the formation of calcium oxide. Furthermore, the detected carbon content by the EDS can be attributed to residual carbonate traces or minimal organic residues that may persist following calcinations [37, 38]. Additionally, the presence of other minor elements likely arises from environmental contamination or inherent trace impurities within the eggshell precursor [39]. Importantly, the predominance of Ca and O in near-stoichiometric proportions strongly as depicted by their peaks in the Figure indicates effective conversion from calcium carbonate to CaO, confirming the efficiency of the calcination process. This result is consistent with the XRF analysis presented in Table 1, which shows the dominance of CaO in the composition. Moreover, the result aligns with a similar study by Nath *et al*. [6], where Ca and O were at near proportions after calcination at 900°C. Additionally, the minor carbon and trace impurities identified are within acceptable limits for industrial applications, reinforcing the material's compatibility with advanced manufacturing requirements where chemical purity and thermal stability are essential [39].

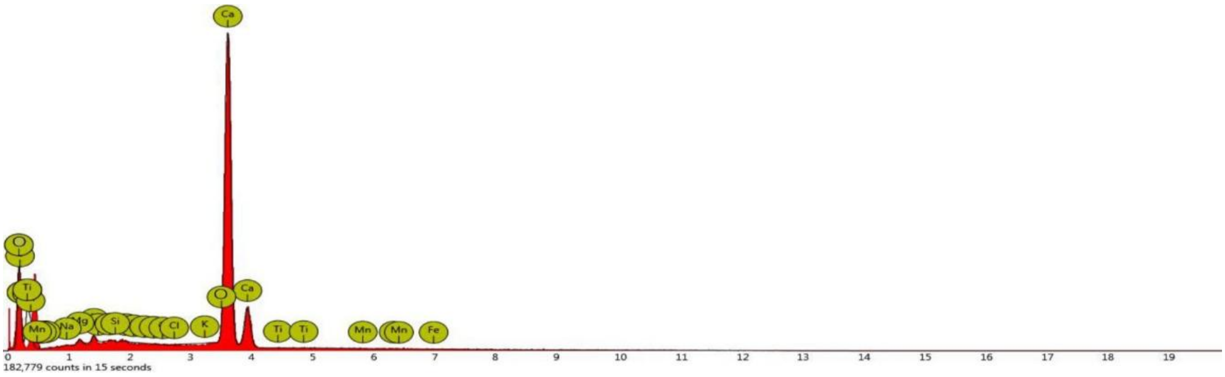

Figure 3: extracted eggshell calcium oxide powder

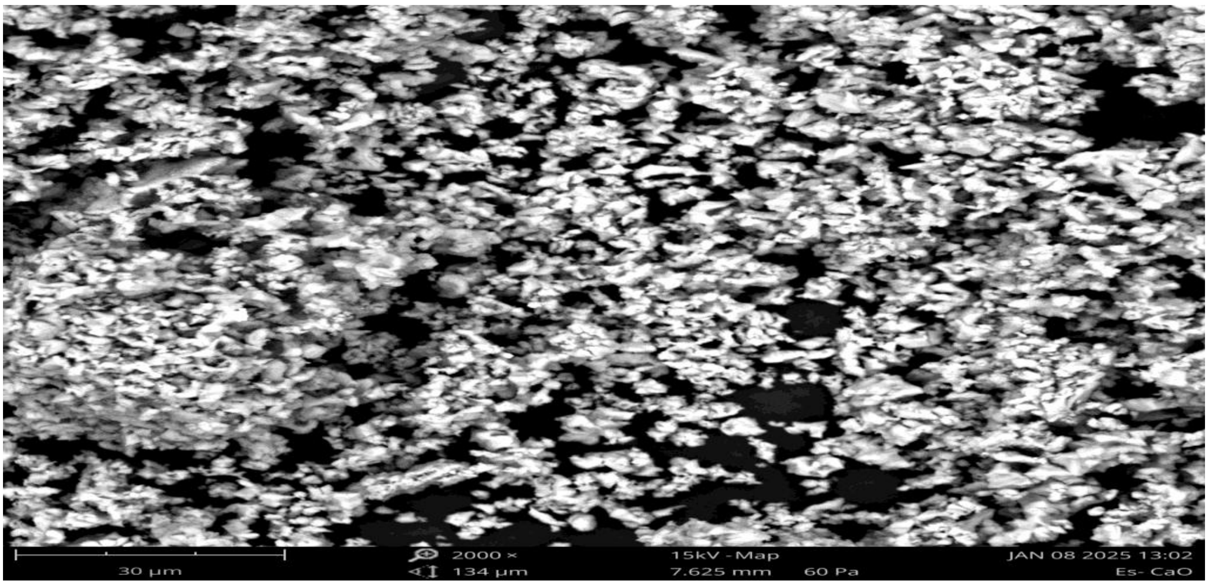

Figure 4: SEM micrograph of the extracted eggshell calcium oxide powder

Morphological analysis

Figure 4 presents the scanning electron microscopy micrograph of calcium oxide synthesized from chicken eggshells calcined at 900°C for the characterization of the sample morphology, revealing distinct morphological transformations.

Notably, the raw eggshell's irregular calcium carbonate particles underwent conversion into finer, near-spherical CaO nanoparticles, consistent with previous findings by Hemmami *et al*. [9], that report eggshell-derived CaO particles in the range of 5–30 nm. These nanostructures likely result from a thermal decomposition process, where the precursor $CaCO_3$ disintegrates under high temperatures to yield nanoscale CaO. Furthermore, the observed clustering or agglomeration of particles can be attributed to their high surface energy and incipient sintering effects during calcination [31]. Additionally, the SEM micrograph reveals morphological diversity, including rod-like or acicular features, reflecting variability due to precursor characteristics and thermal treatment conditions. Interestingly, a notable characteristic is the pronounced porosity of the CaO surface, arising from CO_2 evolution during $CaCO_3$ decomposition. This mesoporous texture, even in the absence of chemical activation, corresponds with prior reports on porous CaO synthesis via direct thermal routes. Moreover, the observed rough and textured topology is attributed to nucleation and growth of CaO crystallites concurrent with carbonate breakdown [40]. Importantly, these SEM-identified features of nanoscale particle size, high porosity, and morphological variation are crucial for photovoltaic glass integration. Specifically, the porous architecture and elevated surface area enhance the dispersion of CaO within the glass matrix, facilitating homogeneous melt mixing [22]. This, in turn, improves optical clarity and mechanical integrity by minimizing structural defects and agglomeration during fabrication [36].

Structural analysis

The structural characterization of synthesized calcium oxide via the X-ray diffraction analysis, as seen in Fig. 5, reveals sharp, well-defined diffraction peaks at approximately 32°, 37°, and 54° 2θ, corresponding to the characteristic reflections of crystalline CaO.

This phase identification confirms the successful decomposition of calcium carbonate and is consistent with the findings of Rinaudo *et al*. [13], who reported comparable peak positions for eggshell-derived CaO calcined at 800–900°C. Furthermore, the diffraction pattern exhibits peaks associated with portlandite, as indexed by ICDD card No. 00–004-0733, indicative of surface hydration due to atmospheric exposure during post-calcination handling [41]. Similarly, the transformation of CaO to $Ca(OH)_2$ under ambient conditions has been reported by Hemmami *et al*. [9], Lanzón *et al*. [42], and Marcos *et al*. [17]. The pronounced crystallinity, as evidenced by the peak sharpness and intensity, reflects a highly ordered lattice structure and affirms the thermochemical efficiency of the calcination process. Additionally, minor diffraction peaks observed near 21° and 27° 2θ correspond to residual quartz and muscovite ($KAl_2(AlSi_3O_{10})(OH)_2$), representing trace aluminosilicate inclusions. These secondary phases are commonly attributed to feedstock impurities or environmental particulates and are consistent with previous observations in eggshell-derived CaO systems [9]. In summary, the crystallographic profile obtained confirms that the synthesized CaO possesses high phase purity and structural definition, which are critical parameters for high-performance applications [43]. Furthermore, in the context of photovoltaic glass manufacturing, such crystallinity is advantageous as it contributes to enhanced thermal stability, predictable chemical reactivity, and improved integration within silicate matrices [44, 45]. These attributes are aligned with the findings of the review study of Adaikalam *et al*. [35], who reported that high-crystallinity CaO significantly improves material performance in energy-related and structural applications.

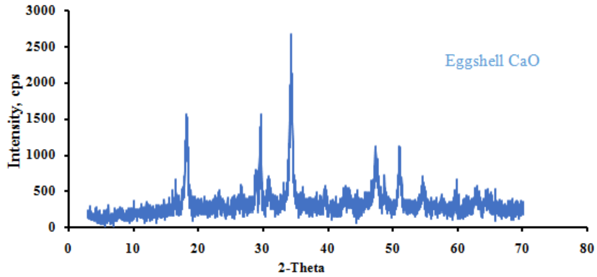

Figure 5: XRD pattern of the extracted eggshell calcium oxide powder

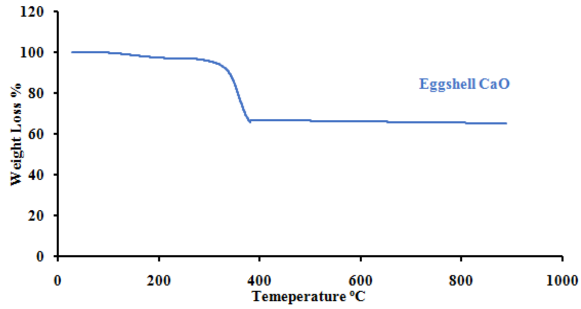

Figure 6: TGA profile of the extracted eggshell calcium oxide powder

Thermal analysis

The thermal properties of the eggshell-derived calcium oxide were characterized using thermogravimetric analysis, as presented in Fig. 6. The TGA profile illustrates a distinct thermal decomposition behavior, highlighting critical weight loss phases indicative of material transformations.

Initially, a minor weight reduction observed below 200°C corresponds to the release of adsorbed moisture and physically bound water from the CaO surface [16]. The most substantial decomposition stage occurs prominently between approximately 300 and 400°C, where significant weight loss is observed. This pronounced reduction is primarily attributed to the desorption of chemically adsorbed water and the decomposition of residual hydroxides formed due to CaO hydration during post-calcination handling and storage, as evidenced in the XRD phases in Fig. 4 and consistent with previous findings by Hemmami *et al*. [9]. Beyond 400°C, the weight loss profile stabilizes significantly, indicating the completion of hydroxide decomposition and confirming the thermal stability of the resultant CaO at elevated temperatures. The thermal behavior exhibited aligns with documented thermal decomposition patterns for biogenic CaO, where post-calcination hygroscopicity leads to hydroxide formation [40]. Such insights are critical for photovoltaic glass manufacturing and other high-temperature applications, as they emphasize the necessity for controlled storage and handling conditions to maintain material integrity and ensure predictable performance. The overall thermal stability and minimal mass loss above 400°C suggest the suitability of this CaO for integration into advanced industrial materials, where thermal robustness and chemical consistency are paramount [35].

Conclusion and Recommendations

This study successfully synthesized high-purity calcium oxide from chicken eggshells sourced from Lafia Local Government Area, Nasarawa State, Nigeria, via thermal calcination at 900°C. The compositional analysis confirmed exceptional CaO purity, satisfying stringent requirements for photovoltaic glass applications by ensuring optimal optical transparency and chemical stability. Morphological characterization via SEM revealed nanoscale spherical particles and a pronounced porous structure, ideal for enhancing dispersion and reducing agglomeration in glass matrices, thereby improving optical clarity and mechanical properties. XRD analysis highlighted high crystallinity and phase purity, essential for achieving predictable reactivity, uniform melting behavior, and structural integrity in high-temperature glass fabrication processes. Furthermore, the thermogravimetric analysis demonstrated favorable thermal stability above 400°C, emphasizing the need for controlled storage conditions to mitigate hygroscopic effects and preserve material quality crucial for precise glass formulation. Collectively, these findings substantiate the suitability of eggshell-derived CaO as an economically viable, environmentally sustainable alternative for advanced photovoltaic glass manufacturing, promoting agro-waste valorization and aligning with circular economy and global sustainability goals. Future studies should focus on integrating eggshell-derived CaO into actual glass formulations, evaluating optical, mechanical, and long-term durability properties in operational photovoltaic modules. Additionally, assessing the economic feasibility, scalability, farming system, and lifecycle environmental impact would provide essential insights for commercial application viability.

Conflict of interest: There is no conflict of interest.

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