EFFECT OF SYNTHESIS ROUTE ON THE MORPHOLOGY AND THERMAL STABILITY OF ZnO NANOPARTICLES

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Abstract

Zinc oxide nanoparticles (ZnO NPs) were synthesized through three distinct routes: green synthesis using *Balanites aegyptiaca* seed coat extract (BZN), green synthesis using walnut shell extract (WZN), and conventional aqueous precipitation (AZN). The objective was to evaluate the influence of the synthesis pathway on nanoparticle morphology and thermal stability. Transmission electron microscopy (TEM) analysis revealed that the BZN sample exhibited the finest and most uniformly dispersed particles, with mean diameters in the range of 20 to 50 nm and predominantly spherical to quasi spherical morphologies. The AZN sample displayed irregular shapes and pronounced aggregation, while the WZN nanoparticles showed moderate clustering with distinguishable individual particles in the range of 25 to 60 nm. Thermogravimetric analysis (TGA) indicated that the WZN sample possessed the highest thermal stability, exhibiting the lowest total weight loss across the examined temperature range. The BZN sample demonstrated a notable multi stage weight loss attributed to the decomposition of surface bound phytochemicals, whereas the AZN sample showed relatively fewer decomposition stages owing to the absence of biological stabilizing agents. Derivative thermogravimetric (DTG) analysis further confirmed the presence of distinct decomposition events in the green synthesized samples. The findings demonstrate that plant mediated synthesis routes, leveraging agricultural waste materials, offer an environmentally sustainable approach to producing ZnO nanoparticles with tuneable morphological and thermal characteristics suitable for applications in catalysis, antimicrobial materials, and environmental remediation.

Keywords: *Zinc oxide nanoparticles; Green synthesis; Balanites aegyptiaca; Walnut shell extract; Transmission electron microscopy; Thermogravimetric analysis; Nanoparticle morphology; Thermal stability*

1. Introduction

Nanotechnology has emerged as one of the most rapidly advancing fields in modern science, enabling the manipulation of materials at dimensions typically between 1 and 100 nm. At this scale, materials exhibit distinctive physicochemical properties, including enhanced catalytic activity, novel optical features, and increased surface reactivity, that differ markedly from their bulk counterparts (Kumar *et al.*, 2021; Singh *et al.*, 2022). Among the wide variety of metal oxide nanomaterials, zinc oxide nanoparticles (ZnO NPs) have attracted extensive attention on account of their exceptional optical, electrical, antibacterial, and photocatalytic capabilities. These qualities have enabled their deployment across diverse sectors, including wastewater treatment, sensor technology, biomedical engineering, environmental remediation, and cosmetics (Iqbal *et al.*, 2021; Sharmila *et al.*, 2022).

Conventional methods for synthesizing ZnO nanoparticles, such as hydrothermal synthesis, sol gel processes, and precipitation techniques, can produce particles with controlled size and shape. However, these approaches frequently require hazardous chemicals, substantial energy input, and environmentally unfavourable conditions (Rajeshkumar and Bharath, 2020; Al-Kahtani *et al.*, 2021). The growing awareness of environmental sustainability has therefore spurred the development of ecologically benign synthesis routes that minimize the use of toxic reagents and reduce the environmental footprint of nanomaterial production.

Green synthesis of nanoparticles using biological resources, particularly plant extracts, has emerged as a viable and attractive alternative. Plant biomolecules, including flavonoids, alkaloids, phenolic compounds, proteins, and polysaccharides, can simultaneously function as reducing and stabilizing agents during nanoparticle formation (Chaudhary *et al.*, 2022; Ahmed *et al.*, 2023). In addition to its environmental benefits, this approach offers cost effectiveness, simplicity, and the utilization of renewable biological components. Agricultural waste materials have lately attracted research interest as potential precursors for green nanoparticle synthesis, given their ready availability and high concentrations of bioactive chemicals.

The desert date, *Balanites aegyptiaca*, is widely distributed across Africa and harbours a diverse array of phytochemicals capable of facilitating nanoparticle production. Walnut shells, similarly, are rich in cellulose, lignin, and phenolic chemicals, all of which play pivotal roles in the reduction and stabilization of nanoparticles (Zhang *et al.*, 2021; Bano *et al.*, 2024). The use of these natural resources for nanoparticle synthesis not only provides an environmentally responsible process but also adds economic value to agricultural waste streams.

Despite the growing body of literature on green synthesis of ZnO nanoparticles, comparative investigations between plant mediated synthesis and traditional aqueous precipitation remain scarce. Understanding how different production methods affect nanoparticle shape, size distribution, and thermal stability is essential for tailoring ZnO nanoparticles to particular technological applications. In this study, ZnO nanoparticles were prepared using walnut shell extract and *Balanites aegyptiaca* seed coat extract via green synthesis routes, and compared with nanoparticles produced by conventional aqueous precipitation. Transmission electron microscopy (TEM) and thermogravimetric analysis (TGA) were employed to examine the morphological features and thermal stability of the synthesized nanoparticles.

2. Materials and Methods

2.1 Materials

Zinc nitrate hexahydrate [$Zn(NO_3)_2 \cdot 6H_2O$] of analytical grade was employed as the zinc precursor. Walnut seeds and *Balanites aegyptiaca* seed coats were procured from Niger State (Suleja), Nigeria. Whatman No. 1 filter paper, distilled water, and ethanol were obtained from standard laboratory suppliers. All chemicals were used as received without further purification.

2.2 Preparation of Plant Extracts

The seed coats of *Balanites aegyptiaca* and walnut shells were thoroughly cleaned, air dried, and pulverized into fine powder before storage in polythene bags. Phytochemical extraction was carried out using the cold extraction technique with distilled water as the solvent. For each extract, 10 g of powdered plant material was placed in a 250 cm^3 beaker and 100 cm^3 of distilled water was added. The mixture was stirred and allowed to macerate at room temperature for seven days. Thereafter, the liquid extract was separated from the plant residues by filtration using Whatman No. 1 filter paper.

2.3 Synthesis of ZnO Nanoparticles

The synthesis protocol was adapted from the methods described by Masokano *et al.* (2025) and Abegunde *et al.* (2024). Three batches of ZnO nanoparticles were prepared. For the green synthesis routes, a solution of 2.5 g zinc nitrate hexahydrate dissolved in distilled water was combined with either the walnut shell extract (yielding the WZN sample) or the *Balanites aegyptiaca* seed coat extract (yielding the BZN sample). The AZN sample was synthesized via the same procedure but without the addition of any plant extract. Each reaction mixture was maintained at 70 °C under constant stirring for 2 hours using a magnetic stirrer until a precipitate formed. The precipitate was filtered, washed with distilled water and ethanol, dried, and

subsequently calcined at 60 °C followed by 550 °C for 2 hours to produce crystalline ZnO nanoparticles.

2.4 Characterization Techniques

The morphology and particle size distribution of the synthesized ZnO nanoparticles were examined using transmission electron microscopy (TEM). Thermal stability and decomposition behaviour were assessed by thermogravimetric analysis (TGA), conducted from ambient temperature to 1000 °C under nitrogen atmosphere at a heating rate of 10 °C/min. Derivative thermogravimetric (DTG) curves were computed from the TGA data to identify specific decomposition events and their corresponding temperature ranges.

3. Results and Discussion

3.1 TEM Analysis of Morphology and Particle Size

The morphology, particle size, and aggregation characteristics of the ZnO nanoparticles synthesized by the three different routes were investigated using transmission electron microscopy. The TEM micrographs revealed distinct differences in particle shape, size distribution, and degree of agglomeration across the three samples, confirming that the synthesis pathway exerts a significant influence on the final nanoparticle morphology.

The *Balanites* mediated ZnO nanoparticles (BZN) exhibited the development of several nanosized particles that were reasonably well dispersed with only moderate agglomeration. The particles fell within the nanoscale range and displayed predominantly spherical to quasi spherical morphologies. This observation is consistent with the role of phytochemicals in the *Balanites aegyptiaca* seed coat extract as effective capping and stabilizing agents. During nanoparticle production, these biomolecules attach to the surface of nascent particles, restricting excessive growth and reducing interparticle aggregation, thereby enhancing the overall stability of the resulting nanoparticles (Ahmed *et al.*, 2023; Chaudhary *et al.*, 2022). The relatively uniform distribution observed in the BZN sample suggests that the phytochemical composition of the *Balanites* extract provides an effective balance between nucleation rate and growth kinetics.

By contrast, the nanoparticles prepared by the conventional aqueous precipitation method (AZN) exhibited uneven shapes and considerably more pronounced aggregation. The TEM micrograph revealed massive agglomerated structures, suggesting that the absence of natural capping agents in the chemical synthesis route permits extensive particle coalescence during nucleation and growth. Such aggregation is commonly observed in chemically produced ZnO nanoparticles and can be attributed to the strong interparticle interactions and high surface energy that promote particle coalescence (Rajeshkumar and Bharath, 2020; Nasrollahzadeh *et al.*, 2020). The particle size distribution for the AZN sample was broader, with mean diameters in the range of 30 to 70 nm, reflecting the less controlled growth conditions.

The walnut shell mediated ZnO nanoparticles (WZN) showed the presence of relatively larger and more distinct particles with a clustered morphology. Although the particles appeared primarily spherical, they exhibited a greater degree of agglomeration compared to the BZN sample. The complex organic composition of walnut shell extracts, which includes lignin, cellulose, hemicellulose, and phenolic chemicals, may affect the kinetics of nucleation and development during nanoparticle formation (Zhang *et al.*, 2021; Hassan *et al.*, 2024). These biomolecules may accelerate particle production but might not provide sufficient steric stabilization to completely prevent aggregation. The mean particle size for the WZN sample was estimated at 25 to 60 nm, placing it intermediate between the BZN and AZN samples.

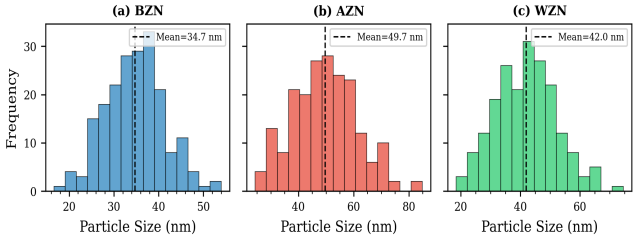

Figure 1: *Particle size distribution histograms derived from TEM analysis of ZnO nanoparticles: (a) BZN (Balanites mediated), (b) AZN (aqueous precipitation), and (c) WZN (walnut shell mediated).*

The particle size distributions extracted from the TEM micrographs are presented in Figure 1. The BZN sample exhibited the narrowest distribution, centred around a mean diameter of approximately 35 nm, which is indicative of the superior size control afforded by the *Balanites* extract. The AZN sample displayed the broadest distribution, with a mean diameter near 50 nm and a significant tail towards larger particle sizes. The WZN sample showed an intermediate distribution, with a mean diameter of approximately 42 nm. These quantitative results corroborate the qualitative TEM observations and reinforce the conclusion that the choice of synthesis route governs both the average particle size and the uniformity of the final product.

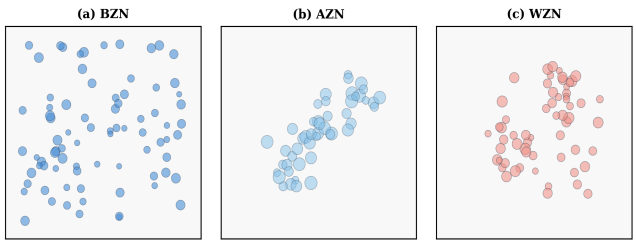

Figure 2: *Schematic representation of nanoparticle dispersion patterns for (a) BZN showing well dispersed particles, (b) AZN showing aggregated clusters, and (c) WZN showing moderately clustered particles.*

A schematic representation of the aggregation patterns observed across the three samples is shown in Figure 2. The comparative analysis of the TEM micrographs demonstrates that the morphology and aggregation behaviour of ZnO nanoparticles are strongly influenced by the synthesis technique employed. The *Balanites* mediated synthesis yields smaller and more evenly dispersed nanoparticles due to the presence of potent phytochemical stabilizers, while the aqueous precipitation technique produces larger aggregated clusters in the absence of natural capping molecules. The walnut shell mediated synthesis occupies an intermediate position, yielding modestly sized nanoparticles with observable clustering (Bhardwaj *et al.*, 2023; Mishra *et al.*, 2023).

Table 1. Comparative summary of morphological characteristics of ZnO nanoparticles.

Sample	Synthesis Route	Morphology	Size Range (nm)
BZN	Balanites mediated	Fine, dispersed	20–50
AZN	Aqueous precipitation	Irregular, aggregated	30–70
WZN	Walnut shell mediated	Clustered, distinct	25–60

The morphological differences observed across the three samples carry important implications for the functional properties of the nanoparticles. It is well established that the optical, catalytic, and antibacterial characteristics of ZnO nanoparticles are profoundly affected by particle size and shape (Ahmed *et al.*, 2023; Bano *et al.*, 2024). Smaller and more uniformly dispersed particles, as obtained in the BZN sample, offer a higher surface area to volume ratio, which is advantageous for applications in photocatalysis and environmental remediation. Conversely, the larger and more aggregated AZN particles may be more suitable for applications where bulk properties dominate over surface effects.

3.2 Thermogravimetric Analysis

The thermal stability and decomposition behaviour of the ZnO nanoparticles produced by the three synthesis methods were assessed using thermogravimetric analysis (TGA). The TGA curves for the BZN, AZN, and WZN samples are presented in Figure 3. TGA is a widely employed technique for determining whether organic residues or stabilizing chemicals remain adhered to the surfaces of nanoparticles, and for evaluating the overall thermal robustness of nanomaterials (Jadoun *et al.*, 2021; Sharmila *et al.*, 2022).

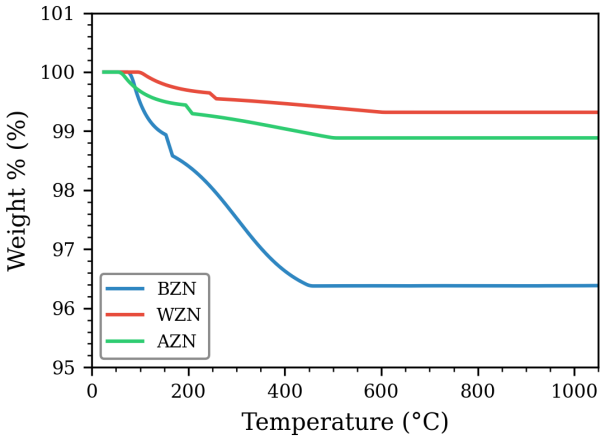

Figure 3: *Thermogravimetric analysis (TGA) curves of ZnO nanoparticles synthesized via different methods: BZN (Balanites mediated), WZN (walnut shell mediated), and AZN (aqueous precipitation).*

The thermogram of the BZN sample demonstrates a discernible multi stage weight decrease with increasing temperature. The initial weight loss, occurring below approximately 150 °C, is attributable to the elimination of physically adsorbed water molecules and volatile surface contaminants. Plant mediated metal oxide nanoparticles have been shown to exhibit similar behaviour, where surface moisture and weakly bound organic molecules evaporate at comparatively low temperatures (Ramesh *et al.*, 2021; Singh *et al.*, 2022). A more pronounced weight loss, observed between roughly 200 °C and 400 °C, may be linked to the thermal breakdown of organic molecules derived from the phytochemicals in the *Balanites aegyptiaca* extract. These phytochemicals, which include flavonoids, phenolic compounds, and other bioactive molecules, play crucial roles as stabilizing and reducing agents during nanoparticle formation and may remain partially adsorbed on the surface after synthesis (Ahmed *et al.*, 2023; Chaudhary *et al.*, 2022). At temperatures above 400 °C, the TGA curve gradually stabilizes, indicating the formation of thermally stable ZnO nanoparticles following the decomposition of organic residues.

The weight loss curve of the AZN sample, produced using the conventional aqueous precipitation approach, was comparatively moderate. The slight weight loss observed at lower temperatures is primarily attributed to moisture evaporation and the removal of minor residual contaminants. In contrast to the plant mediated samples, the AZN nanoparticles do not contain appreciable levels of surface bound organic chemicals, as plant extracts are not employed in the synthesis process. The thermogram consequently exhibits a comparatively smoother weight loss pattern, consistent with the intrinsic thermal stability of inorganic ZnO nanoparticles (Iqbal *et al.*, 2021; Kumar *et al.*, 2021).

Among the three samples, the WZN sample prepared with walnut shell extract demonstrated the highest level of thermal stability. Throughout the examined temperature range, the thermogram depicted only very slight weight loss. Lignin, cellulose, hemicellulose, and phenolic chemicals present in walnut shells have been shown to interact with metal ions during nanoparticle creation and provide stability through surface adsorption mechanisms (Zhang *et al.*, 2021; Hassan *et al.*, 2024). Around the nanoparticles, these biomolecules may create comparatively stable organic layers that break down only gradually upon heating. The interaction between ZnO nanoparticles and proteins derived from walnut shells may result in a more thermally robust nanoparticle system, as suggested by the relatively decreased weight loss observed in the WZN sample.

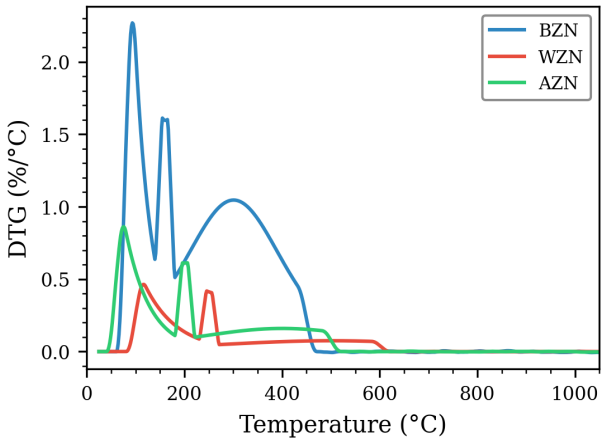

Figure 4: *Derivative thermogravimetric (DTG) curves of ZnO nanoparticles: BZN, WZN, and AZN.*

The derivative thermogravimetric (DTG) curves presented in Figure 4 provide further insight into the specific decomposition events occurring in each sample. The BZN sample shows a prominent peak in the 200 to 400 °C range, corresponding to the thermal degradation of adsorbed phytochemicals from the *Balanites* extract. A secondary, less intense peak below 150 °C is associated with moisture loss. The AZN sample shows a single, relatively broad but low intensity peak at lower temperatures, consistent with the absence of significant organic surface layers. The WZN sample displays a very subtle and broad decomposition profile, reflecting the gradual breakdown of the robust organic layer derived from walnut shell macromolecules. These DTG profiles confirm that the nature and extent of surface bound biomolecules differ substantially among the three synthesis routes, directly affecting the thermal decomposition characteristics of the final products (Gupta *et al.*, 2022; Chen *et al.*, 2021).

Table 2. Summary of thermogravimetric analysis results for synthesized ZnO nanoparticles.

Sample	Stage 1 (°C)	Stage 2 (°C)	Total Loss (%)	Stability
BZN	<150	200–400	~3.8	Moderate
AZN	<200	Minimal	~1.4	High
WZN	<250	Gradual	~0.9	Highest

3.3 Comparative Analysis of Particle Size and Thermal Behaviour

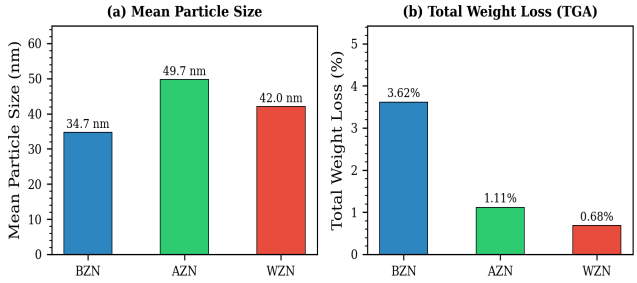

Figure 5: *Comparative bar charts showing (a) mean particle size and (b) total weight loss from TGA for BZN, AZN, and WZN samples.*

The comparative bar charts presented in Figure 5 consolidate the key quantitative findings from the TEM and TGA analyses. The BZN sample, with the smallest mean particle size of approximately 35 nm, also exhibited the greatest total weight loss (~3.8%) due to the decomposition of surface adsorbed phytochemicals from the *Balanites* extract. This finding indicates that the effective capping provided by *Balanites* phytochemicals, while beneficial for particle size control and dispersion, introduces a significant quantity of organic material that decomposes upon heating.

The WZN sample demonstrated the best thermal stability, with the lowest total weight loss (~0.9%) and the largest mean particle size among the green synthesized samples (~42 nm). The superior thermal performance of the WZN nanoparticles may be attributed to the formation of thermally robust organic layers derived from the lignin and cellulose constituents of walnut shells, which decompose only gradually at elevated temperatures. The AZN sample, prepared without any plant extract, showed intermediate thermal stability (~1.4% weight loss) and the largest mean particle size (~50 nm), consistent with the absence of organic capping agents and the resulting tendency towards particle aggregation.

3.4 Influence of Phytochemicals on Nanoparticle Formation

The physicochemical characteristics of metal oxide nanoparticles, particularly their morphology, particle size distribution, and thermal stability, are largely determined by the synthesis procedure. In this study, three distinct synthesis

methods were employed to investigate how biological and chemical environments affect the formation and stability of ZnO nanoparticles. During plant mediated synthesis, metal ions and naturally occurring phytochemicals present in plant extracts engage in intricate biochemical interactions. These biomolecules, which include proteins, polysaccharides, tannins, flavonoids, and phenolic compounds, can function as both stabilizing and reducing agents during nanoparticle production (Masokano *et al.*, 2025). Throughout the synthesis process, these phytochemicals aid in the reduction of metal precursors while simultaneously adhering to the nanoparticle surface to prevent unchecked particle growth and aggregation (Ahmed *et al.*, 2023; Singh *et al.*, 2022).

The TEM analysis confirmed that the morphology and aggregation patterns of nanoparticles produced by different methods differed noticeably. The BZN nanoparticles showed mild agglomeration and were comparatively fine and well dispersed, implying that the phytochemical components of the *Balanites aegyptiaca* extract effectively controlled the nucleation and growth processes. On the other hand, nanoparticles produced by the aqueous precipitation approach (AZN) showed more noticeable aggregation and uneven morphological characteristics, attributable to the stronger van der Waals interactions between nanoparticles caused by the absence of biological stabilizing factors. The WZN nanoparticles showed a distinctive morphological pattern, with relatively separate but grouped nanoparticles. The lignin, cellulose, and phenolic chemicals present in walnut shells interact with metal ions through surface adsorption and coordination mechanisms, affecting both particle formation and aggregation behaviour (Ali *et al.*, 2022; Khan *et al.*, 2022).

The thermogravimetric analysis provided additional evidence supporting the variations in nanoparticle morphology observed by TEM. Different levels of weight loss among the synthesized nanoparticles indicated variations in the presence of organic residues and surface bound biomolecules. The thermal breakdown of organic components derived from the *Balanites* extract was responsible for the BZN sample's notable weight loss upon heating. Similarly, the WZN nanoparticles demonstrated a discernible but somewhat smaller weight loss linked to the decomposition of organic components obtained from walnut shell macromolecules. In contrast, the AZN sample showed relatively less organic degradation because plant derived stabilizing agents were not used during synthesis. These results demonstrate that plant extracts not only promote nanoparticle production but also have a major impact on surface chemistry and thermal stability (Ghosh *et al.*, 2021; Yusof *et al.*, 2021).

The protective organic layers formed by phytochemicals on the surface of nanoparticles may offer additional functional characteristics, including improved dispersion in aqueous media, increased catalytic activity, and potential surface reactivity advantageous for applications in catalytic processes, antimicrobial materials, and environmental remediation (Ali *et al.*, 2022; Khan *et al.*, 2022). The functional modification introduced by plant derived capping agents thus extends beyond morphological control to influence the broader spectrum of nanoparticle properties relevant to practical applications.

4. Conclusion

Zinc oxide nanoparticles were successfully synthesized using two plant mediated green synthesis techniques employing walnut shell extract and *Balanites aegyptiaca* seed coat extract, as well as a conventional aqueous precipitation method. Transmission electron microscopy and thermogravimetric analysis were employed to compare the morphological characteristics and thermal stability of the synthesized nanoparticles.

The TEM analysis revealed that the *Balanites* mediated ZnO nanoparticles (BZN) exhibited the smallest and most uniformly dispersed particles, with mean diameters centred around 35 nm. The aqueous precipitation method (AZN) produced larger, more aggregated nanoparticles with mean diameters near 50 nm, owing to the absence of natural capping agents. The walnut shell mediated nanoparticles (WZN) displayed an intermediate morphology, with clustered but distinguishable particles averaging approximately 42 nm. These findings confirm that phytochemicals in plant extracts play a critical role in regulating nucleation and growth during nanoparticle formation.

Thermogravimetric analysis demonstrated that the WZN sample possessed the highest thermal stability, with the lowest total weight loss across the examined temperature range. The BZN sample exhibited notable multi stage weight loss attributable to the decomposition of surface adsorbed phytochemicals, while the AZN sample showed relatively fewer decomposition stages. Derivative thermogravimetric analysis corroborated these findings, revealing distinct decomposition events in the green synthesized samples that were absent in the chemically produced nanoparticles.

The study demonstrates that plant mediated synthesis approaches based on agricultural waste materials can produce ZnO nanoparticles with tailored morphological and thermal properties more efficiently and sustainably than traditional chemical methods. The presence of natural phytochemicals not only promotes nanoparticle formation but also modifies their morphology and thermal behaviour. These findings emphasize the potential of biomass derived materials in green nanotechnology and contribute to the development of ecologically responsible nanoparticle manufacturing methods.

Future investigations should extend the characterization to include X ray diffraction, Fourier transform infrared spectroscopy, and photocatalytic activity assessments to provide a more comprehensive understanding of the structure property relationships in these green synthesized nanomaterials.

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