

Research Article

Green Synthesis of Zinc Oxide Nanoparticles Using Yemeni Sidr Honey as a Bio-Templating Agent

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Abstract

Zinc oxide (ZnO) nanoparticles are versatile semiconductor materials with applications in photocatalysis, gas sensing, optoelectronics, and biomedical fields. Conventional chemical synthesis routes often involve toxic reagents and synthetic surfactants, creating demand for environmentally benign alternatives. This study reports a green sol-gel synthesis of ZnO nanoparticles using Yemeni Sidr honey (from *Ziziphus spina-christi*) as a natural bio-templating and capping agent, eliminating the need for added synthetic surfactants. In the synthesis, 12 g of Sidr honey was dissolved in 60 mL deionized water under stirring at 70 °C for 60 min. Then 10 g zinc nitrate hexahydrate was added, and the mixture was stirred on a sand bath at 70 °C for 8 h until a viscous gel formed. The gel was calcined in air at 450 °C for 3 h, yielding a white ZnO powder. The product was characterized by Fourier-transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), and field-emission scanning electron microscopy (FESEM). FTIR showed a strong Zn–O stretching band at 470 cm⁻¹, along with surface-adsorbed hydroxyl and carbonate species; bands associated with organic functional groups were not detected after calcination. XRD confirmed a single-phase hexagonal wurtzite structure (JCPDS 36-1451) with no detectable impurity phases, and the average crystallite size calculated using the Scherrer equation was 24.3 nm. FESEM images revealed predominantly spherical particles with hexagonal facets and noticeable agglomeration. The results indicate that Yemeni Sidr honey can serve as an effective natural template in a simple, low-cost, and non-toxic sol-gel route to ZnO nanoparticles. This approach may be extendable to the synthesis of other transition metal oxide nanostructures, although further optimization and property evaluation are needed.

Keywords

ZnO Nanoparticles, Green Synthesis, Yemeni Sidr Honey, *Ziziphus Spina-christi*, Sol-gel, Bio-templating, Hexagonal Wurtzite, XRD

1. Introduction

There is growing scientific interest in nanomaterial synthesis, driven by the distinctive physicochemical behavior these materials exhibit relative to their bulk counterparts. Particle shape and size are critical determinants of nanomaterial performance, directly governing the range of applications a given nanostructure can support [1-3]. Among the broad class of

nanostructured materials, metal oxide nanoparticles stand out for their wide-ranging utility in photocatalysis, sensing platforms, optoelectronic devices, and biomedical applications [4].

Zinc oxide (ZnO) ranks among the most versatile semiconductor materials, characterized by a wide band gap of 3.37 eV and a notably high exciton binding energy of 60 meV [5].

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These properties have enabled ZnO nanoparticles to be deployed across dye-sensitized solar cells, UV-blocking formulations, antimicrobial surface coatings, gas-sensing devices, and photocatalytic systems for breaking down organic contaminants [5, 6]. Given this broad utility, identifying synthesis routes that are both economical and environmentally sound has become an important research direction.

ZnO nanoparticle fabrication has been achieved through several chemical routes, among them coprecipitation, hydrothermal processing, and sol–gel chemistry. The sol–gel route in particular stands out for its comparatively mild processing temperatures, operational simplicity, affordability, and the fine control it affords over particle size and uniformity [7, 8]. That said, conventional synthesis protocols frequently rely on toxic reducing agents and surfactants, which has pushed researchers toward identifying greener substitutes.

Plant-derived extracts and natural biopolymers have gained traction as environmentally responsible platforms for nanoparticle fabrication. Sidr honey sourced from the nectar of *Ziziphus spina-christi* (Christ's thorn) represents a honey variety unique to Yemen and the broader Middle Eastern region [9, 10]. Its composition, rich in flavonoids, phenolic acids, sugars, and proteins, allows it to function as a natural capping and stabilizing medium, limiting particle aggregation without requiring any added surfactant [9, 10]. This honey variety has previously been employed in the fabrication of silver and gold nanoparticles [11–13]. Furthermore, other honey types, such as stingless bee honey, have been successfully utilized for the green synthesis of ZnO nanoparticles [14–16]. However, despite the demonstrated potential of both *Ziziphus spina-christi* plant extracts for ZnO synthesis and antimicrobial evaluation [17, 18] and various honey varieties for ZnO nanoparticle fabrication [14–16], the specific use of Yemeni Sidr honey for ZnO nanoparticle synthesis has not yet been investigated. To the best of our knowledge, no study has reported the synthesis of ZnO nanoparticles using Yemeni Sidr honey as a bio-templating and capping agent.

In the present study, we report, for the first time, the synthesis of zinc oxide nanoparticles using Yemeni Sidr honey as a natural template, zinc nitrate hexahydrate as the zinc precursor, and deionized water as the reaction medium, carried out through a straightforward sol–gel process free of any synthetic surfactant or chemical reducing agent. This route is non-toxic, adaptable, economical, and environmentally benign, with potential applicability to the synthesis of other metal oxide nanostructures. The resulting nanoparticles were characterized using FTIR, XRD, and FESEM. The findings confirm the formation of phase-pure hexagonal wurtzite ZnO with an average crystallite size of 24.3 nm and a predominantly spherical morphology with hexagonal facets, demonstrating the viability of Yemeni Sidr honey as a green bio-template. This work thus offers a simple, low-cost, and sustainable pathway for ZnO nanoparticle synthesis while valorizing a unique Yemeni natural resource.

2. Experimental

2.1. Materials

Yemeni Sidr honey was sourced from Bani Zaide, located in Haraz region of Yemen (harvest year: 2025). Zinc nitrate hexahydrate ($\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, $\geq 98\%$) was obtained from university lab and used as received. Deionized water was employed as the solvent in all experimental procedures.

2.2. Synthesis of ZnO Nanoparticles Using Sidr Honey

Sidr honey (12 g) was dissolved in deionized water (60 mL) under magnetic stirring at 500 rpm for 60 min at 70 °C to form a homogeneous honey solution. The pH of the solution was measured to be 8.3. $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (10 g) was then introduced, and the mixture was transferred to a thermostatted sand bath maintained at 70 °C with continuous stirring at 500 rpm for 8 h, during which a viscous yellowish gel formed. The gel was dried at 100 °C for 24 h and then calcined in air at 450 °C for 3 h (heating rate: 5 °C/min, natural cooling inside the furnace). This yielded a white ZnO powder (2.2 g, ~80% yield based on $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$). The synthesis was performed in triplicate to ensure reproducibility.

2.3. Instrumentation

FT-IR spectra were acquired on a Bruker Tensor II FT-IR spectrometer using the KBr pellet method. Spectra were recorded over the 4000–400 cm^{-1} range with a resolution of 4 cm^{-1} and 32 scans per sample. A background spectrum was collected and subtracted.

Crystal structure was examined via a Rigaku MiniFlex 600 X-ray diffractometer employing Cu K α radiation ($\lambda = 1.5406$ Å), operated at 40 kV and 15 mA. Diffraction patterns were collected at room temperature over a 2θ range of 20 °–80 ° with a step size of 0.02 ° and a scan speed of 2 °/min.

Surface morphology and particle size distribution were assessed using a JEOL JSM-7600F field emission scanning electron microscope (Japan) at an accelerating voltage of 15 kV and a working distance of 8.0 mm. Prior to imaging, samples were sputter-coated with a thin gold layer (approximately 10 nm) using a vacuum sputter coater to enhance surface conductivity. Particle size distribution was measured from FESEM images using ImageJ software by measuring the diameters of at least 100 individual particles.

2.4. XRD analysis

Crystallite dimensions of the synthesized ZnO nanoparticles were calculated from the XRD peak broadening data by applying the Scherrer equation (Eq. (1)):

$$D = \frac{0.9\lambda}{\beta \cos \theta} \quad (1)$$

where D is the average crystallite size (nm), K is the shape factor (0.9), λ is the Cu $K\alpha$ wavelength (0.154 nm), β is the full width at half maximum (FWHM) of the diffraction peak in radians, and θ is the Bragg diffraction angle [17]. The FWHM values were obtained for the (101) reflection, and the average crystallite size was calculated.

3. Results and Discussion

3.1. FTIR Analysis

Figure 1 presents the FTIR spectrum of the ZnO nanoparticles calcined at 450 °C for 3 h. The spectrum exhibits distinct absorption bands at 3420, 1620, 1380, 880, and 470 cm^{-1} . The

most prominent feature is the strong, sharp band at 470 cm^{-1} , which is assigned to the Zn–O stretching vibration of the hexagonal wurtzite lattice. This band position is consistent with previous reports on green-synthesized ZnO nanoparticles, where Zn–O stretching vibrations were observed in the 470–480 cm^{-1} range [18]. The presence of this band confirms the successful formation of the ZnO crystal lattice.

The strong, broad band centered at 3420 cm^{-1} is attributed to the O–H stretching vibration of hydroxyl groups, which may originate from both physically adsorbed water molecules and surface hydroxyl species. The medium-intensity band at 1620 cm^{-1} corresponds to the H–O–H bending mode of adsorbed water [19]. The detection of these bands indicates that the nanoparticle surface retains some moisture and hydroxyl functionality, which is typical for ZnO nanoparticles exposed to ambient conditions and can influence surface reactivity for applications such as photocatalysis and sensing.

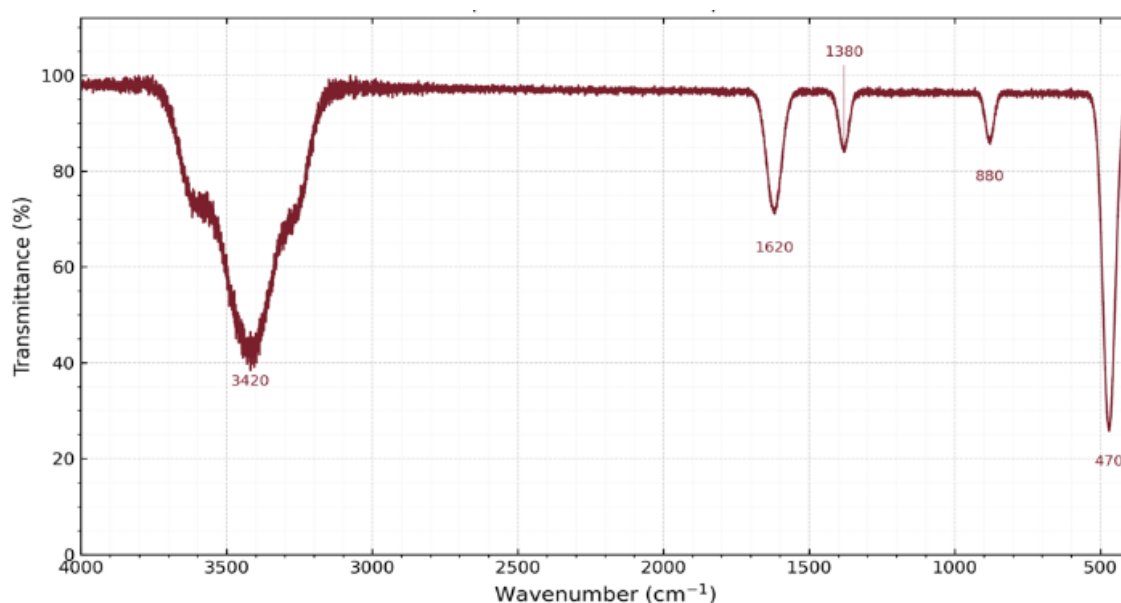


Figure 1. FTIR spectrum of the obtained ZnO nanoparticles.

The weak band at 1380 cm^{-1} is assigned to the asymmetric stretching mode of surface-bound carbonate species (CO_3^{2-}), likely formed through the adsorption of atmospheric CO_2 onto the ZnO surface. This assignment is supported by previous studies that reported similar carbonate bands in the 1380–1390 cm^{-1} region for ZnO-based materials [20]. The weak shoulder at 880 cm^{-1} is tentatively attributed to Zn–OH deformation vibrations associated with surface defect sites, consistent with reports of Zn–OH vibrations in this spectral region.

Notably, no absorption bands corresponding to organic

functional groups—such as carbonyl ($\text{C}=\text{O}$, ~1700 cm^{-1}) or aliphatic C–H stretching (2800–3000 cm^{-1})—were detected in the spectrum of the calcined sample. This observation suggests that the honey-derived organic template was substantially removed during calcination. However, it should be acknowledged that FTIR cannot definitively exclude the presence of trace carbon below its detection limit. Complementary techniques such as CHN elemental analysis or X-ray photoelectron spectroscopy would provide stronger evidence for complete organic removal. The observed FTIR bands and their assignments are summarized in Table 1.

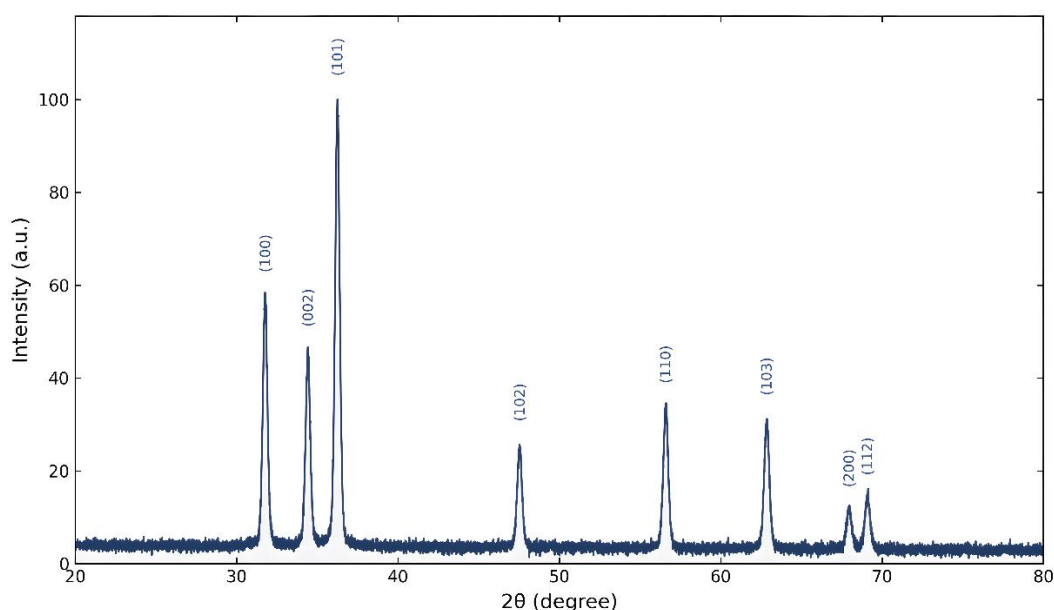
Table 1. FTIR absorption bands of the calcined ZnO nanoparticles and their assignments.

Wavenumber (cm ⁻¹)	Intensity	Assignment	Reference
470	Strong, sharp	Zn–O stretching (wurtzite lattice)	[18]
880	Weak	Zn–OH deformation (surface defects)	[20]
1380	Weak	ν_3 asymmetric stretching of CO ₃ ²⁻ (surface carbonate)	[20]
1620	Medium	H–O–H bending of adsorbed water	[19]
3420	Strong, broad	O–H stretching of adsorbed water/hydroxyl groups	[19]

3.2. XRD Analysis

The phase composition and crystallinity of the synthesized nanoparticles were examined by X-ray diffraction, with the resulting pattern shown in Figure 2. Eight well-defined diffraction peaks were observed at $2\theta = 31.77^\circ$; 34.42° ; 36.25° ; 47.54° ; 56.60° ; 62.86° ; 67.96° ; and 69.10° , corresponding respectively to the (100), (002), (101), (102), (110), (103), (200), and (112) crystallographic planes. These reflections match the standard hexagonal wurtzite ZnO structure (JCPDS Card No.

36-1451), with lattice parameters $a = b = 3.249 \text{ \AA}$ and $c = 5.206 \text{ \AA}$ [5, 18]. The c/a ratio was calculated to be 1.602, which is close to the ideal value of 1.633 for an ideal hexagonal close-packed structure, and the unit cell volume was determined to be $47.62 \text{ \AA}^3$ consistent with reported values for wurtzite ZnO [5]. The absence of any extraneous peaks confirms phase purity within the detection limit of the instrument, with no detectable secondary phases or unreacted precursor residues. The sharp and intense nature of the diffraction peaks further indicates a well-crystallized product.

**Figure 2.** XRD diffractogram of the biosynthesized ZnO nanoparticles calcined at 450°C for 3 h, indexed to the hexagonal wurtzite structure (JCPDS 36-1451).

The average crystallite size was calculated by applying the Scherrer equation to the full width at half maximum (FWHM) of the diffraction peaks:

$$D = \frac{0.9\lambda}{\beta \cos \theta} \quad (2)$$

where D is the crystallite size (nm), K is the shape factor (0.9), λ is the Cu $K\alpha$ wavelength (0.15406 nm), β is the FWHM in radians, and θ is the Bragg diffraction angle [17]. The crystallite size calculated from the dominant (101) reflection was 24.3 nm. This value is in good agreement with crystallite sizes reported for other honey-mediated ZnO nanoparticles. For instance, Deepika et al. reported a crystallite size of 25.31 nm

for ZnO nanoparticles synthesized using honey via a sol-gel route [21], while Sharmila et al. obtained honey-capped ZnO nanoparticles with comparable structural characteristics through an autocombustion method [22]. The slightly smaller crystallite size obtained in the present study may be attributed to the unique composition of Yemeni Sidr honey, which is rich in phenolic compounds and flavonoids that can act as efficient capping agents, limiting particle growth during calcination [9, 10]. Furthermore, the crystallite size is comparable to values reported for ZnO nanoparticles synthesized using other green routes, such as approximately 20 nm for ZnO synthesized using Siirt pistachio thin shell extract [23], and 23.7 ± 0.8 nm for eco-friendly synthesized ZnO nanoparticles [24].

The relatively small crystallite size is advantageous for applications requiring high surface area, such as photocatalysis and gas sensing. It should be noted that the Scherrer equation provides a lower-bound estimate of crystallite size, as it does not account for lattice strain contributions to peak broadening [17]. The use of complementary analytical methods such as

the Williamson-Hall analysis or the size-strain plot could provide a more comprehensive assessment of size and strain effects [25-28]. Grace et al. demonstrated that the Williamson-Hall method yields crystallite sizes that account for strain-induced broadening, providing values that may differ from those obtained by the Scherrer equation alone [25]. Khorsand Zak and Hashim further showed that advanced peak broadening analysis is essential for accurate crystallite size evaluation in doped ZnO systems [26]. Chérif et al. applied both Williamson-Hall and size-strain plot methods to ZnO nanocrystals, highlighting the importance of strain consideration [27]. Tamanna et al. compared multiple XRD analysis models, including the Monshi-Scherrer model, for ZnO characterization [28]. Advanced methods such as the size-strain plot have been applied to ZnO nanocrystals [29], and recent studies continue to refine these approaches [30]. This comprehensive size-strain analysis is recommended for future work. Table 2 summarizes the XRD data, including peak positions, hkl indices, and crystallite sizes.

Table 2. XRD data of the biosynthesized ZnO nanoparticles.

2θ (°)	Hkl	Crystallite size (nm)
31.77	(100)	---
34.42	(002)	---
36.25	(101)	24.3
47.54	(102)	---
56.60	(110)	---
62.86	(103)	---
67.96	(200)	---
69.10	(112)	---

3.3. SEM Analysis

The surface morphology and particle size distribution of the synthesized ZnO nanoparticles were investigated using field emission scanning electron microscopy (FESEM) at two magnification levels, as shown in Figure 3. The micrographs reveal that the nanoparticles exhibit a predominantly spherical morphology, accompanied by visible hexagonal facets consistent with the wurtzite crystal habit of ZnO. This mixed morphology is characteristic of green-synthesized ZnO nanoparticles, where the interplay between nucleation kinetics and the capping action of biomolecules can produce particles with both spherical and faceted geometries [31]. Similar spherical-to-hexagonal morphologies have been reported for ZnO nanoparticles synthesized using stingless bee honey, where

FESEM and TEM analysis confirmed spherical morphology with uniform distribution [16]. The presence of hexagonal facets in the present study provides direct morphological evidence of the wurtzite crystal structure, corroborating the XRD analysis.

A notable degree of particle agglomeration is evident in the micrographs, with individual nanoparticles clustering together rather than existing as fully isolated units. The occurrence of larger aggregated clusters is likely a consequence of the inherently high surface energy and elevated surface tension characteristic of ultrafine ZnO particles, which drives them toward agglomeration to minimize overall surface free energy [32]. This phenomenon is widely reported for green-synthesized metal oxide nanoparticles.

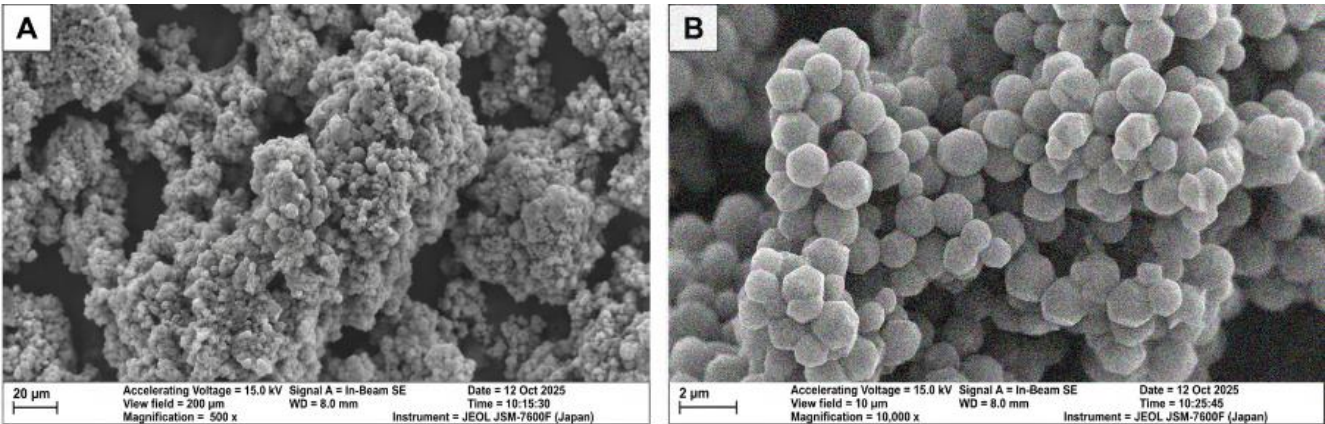


Figure 3. FESEM images of the biosynthesized ZnO nanoparticles calcined at 450 °C for 3 h at (A) 500× and (B) 10,000× magnification.

For instance, FESEM analysis of ZnO nanoparticles synthesized using *Codiaeum variegatum* extract revealed predominantly spherical to slightly irregular particles with appreciable agglomeration, attributed to similar surface energy effects [31]. Similarly, ZnO nanoparticles biosynthesized using *Ziziphus spina-christi* leaf extract exhibited a high density of spherical nanoparticles that aggregated to form nano-form morphologies with various irregular agglomerated shapes [33]. The agglomeration observed in the present study is therefore consistent with the behavior of ZnO nanoparticles synthesized via green routes, where the stabilizing action of biomolecules may be insufficient to fully overcome the intrinsic tendency of nanoparticles to aggregate.

The particle size distribution of the synthesized ZnO nanoparticles was estimated from the FESEM images. The nanoparticles exhibited a size range of approximately 30–50 nm, with agglomerated clusters reaching larger dimensions. This size range is comparable to values reported for other honey-mediated ZnO nanoparticles. For instance, FESEM analysis of honey-based ZnO nanopowders revealed spherical nanoparticles with a size of approximately 30 nm [34]. ZnO nanoparticles synthesized using stingless bee honey exhibited size

ranges between 15–30 nm, with a majority possessing spherical morphology [16]. ZnO nanoparticles biosynthesized using *Ziziphus spina-christi* leaf extract showed an average particle size of approximately 83 nm by SEM [33], while ZnO nanoparticles with bidara leaf biostabilizers exhibited sizes ranging from 42–87 nm [35]. Table 3 summarizes the comparative particle size data from various green synthesis routes.

It should be acknowledged that the FESEM analysis provides a qualitative assessment of morphology and an estimate of particle size, but it does not provide statistically robust particle size distributions. The agglomeration observed in the micrographs limits the accurate measurement of individual particle diameters, as many particles are clustered together. Transmission electron microscopy (TEM) would provide higher-resolution imaging capable of resolving individual crystallites and enabling more accurate particle size determination [36]. Additionally, energy-dispersive X-ray spectroscopy (EDS) analysis would confirm the elemental composition (Zn and O) and detect any residual carbon from the honey template, complementing the FTIR results [36]. These complementary techniques are recommended for future work to provide a more comprehensive understanding of the nanoparticle morphology and surface composition.

Table 3. Comparison of ZnO nanoparticle sizes from various green synthesis routes.

Synthesis Route	Morphology	Particle Size (nm)	Reference
Yemeni Sidr honey (present study)	Spherical with hexagonal facets	30–50 (agglomerated)	This work
Stingless bee honey	Spherical	15–30	[16]
Honey (autocombustion)	Spherical	~30	[34]
<i>Ziziphus spina-christi</i> leaf extract	Spherical, agglomerated	~83	[33]
<i>Ziziphus spina-christi</i> with bidara biostabilizer	Spherical	42–87	[35]
<i>Codiaeum variegatum</i> extract	Spherical to irregular	—	[31]

4. Conclusions

This study reports the first green synthesis of ZnO nanoparticles using Yemeni Sidr honey as a natural bio-templating agent via a simple sol–gel route. The method eliminates synthetic surfactants and reducing agents, yielding phase-pure hexagonal wurtzite ZnO after calcination at 450 °C for 3 h. FTIR confirmed Zn–O lattice formation (470 cm^{-1}) and substantial removal of the honey template. XRD revealed eight peaks indexed to JCPDS 36-1451, with an average crystallite size of 24.3 nm from the (101) reflection. FESEM showed spherical particles with hexagonal facets and typical agglomeration.

The synthesis is cost-effective, non-toxic, and environmentally benign, valorizing a unique Yemeni natural resource. The crystallite size is comparable to or smaller than those of other honey-mediated ZnO nanoparticles, suggesting that Sidr honey components act as efficient capping agents.

Key limitations include uncontrolled pH, Scherrer-based size underestimation due to strain, and qualitative FESEM sizing. Future work should optimize pH, honey concentration, and calcination temperature; employ TEM, EDS, and Williamson–Hall analysis; and evaluate photocatalytic, sensing, and biomedical performance. This honey-mediated strategy offers a scalable, sustainable pathway for other transition metal oxide nanostructures.

Abbreviations

EDS	Energy-Dispersive X-ray Spectroscopy
FESEM	Field Emission Scanning Electron Microscopy
FTIR	Fourier Transform Infrared Spectroscopy
FWHM	Full Width at Half Maximum

Appendix

JCPDS	Joint Committee on Powder Diffraction Standards
KBr	Potassium Bromide
SEM	Scanning Electron Microscopy
TEM	Transmission Electron Microscopy
UV	Ultraviolet
XRD	X-ray Diffraction
ZnO	Zinc Oxide

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Author Contributions

Amir Alqahm: Conceptualization, Data curation, Formal Analysis, Investigation, Methodology, Project administration, Resources, Software, Supervision, Validation, Visualization, Writing – original draft, Writing – review & editing

Data Availability Statement

The data supporting the outcome of this research work has been reported in this manuscript.

Conflicts of Interest

The author declare no conflicts of interest.

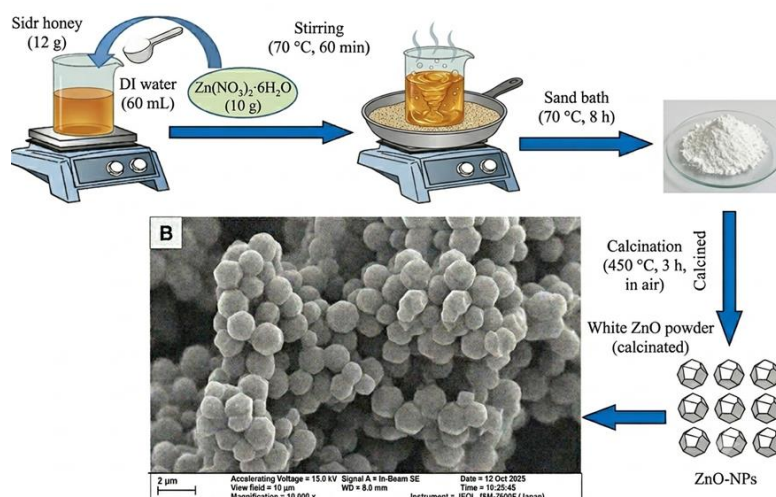


Figure 4. Graphical Abstract.

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Research Field

Amir Alqahm: Green synthesis of nanoparticles, Zinc oxide nanostructures, Sol-gel processing, Bio-templating agents, Natural honey utilization, Nanomaterial characterization, Sustainable chemistry, Metal oxide nanoparticles, Eco-friendly synthesis methods