



Synthesis and Characterization of Zinc Oxide Nanoparticles by Chemical Precipitation Method

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Abstract

Zinc oxide (ZnO) nanoparticles are attractive functional materials because their wide direct band gap, high exciton binding energy, chemical stability, and surface activity support applications in optoelectronics, sensing, photocatalysis, antimicrobial systems, and protective coatings. This methodology paper presents a reproducible chemical precipitation route for the synthesis and characterization of ZnO nanoparticles using zinc acetate dihydrate as the zinc precursor and sodium hydroxide as the precipitating agent. The proposed procedure controls precursor concentration, pH, addition rate, stirring temperature, aging, washing, drying, and calcination so that phase purity and particle size can be assessed systematically. Structural properties are determined by X-ray diffraction (XRD) and crystallite size is estimated using the Scherrer relation. Fourier-transform infrared spectroscopy (FTIR) is used to confirm Zn–O bonding and residual surface groups; UV–visible spectroscopy is used to obtain the absorption edge and optical band gap; and scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM/EDS), with optional transmission electron microscopy (TEM), is used to evaluate morphology, agglomeration, and elemental composition. The expected outcome is single-phase hexagonal wurtzite ZnO with nanometric crystallite size and a strong ultraviolet absorption edge. An illustrative validation dataset is included to demonstrate data treatment and interpretation without representing unperformed laboratory measurements. The workflow is designed for low-cost laboratory implementation and can be adapted for studies examining pH, calcination temperature, precursor concentration, or surface modification.

Introduction

Zinc oxide is an II–VI semiconductor whose nanoscale forms have received sustained attention because the material combines a direct band gap near 3.37 eV with an exciton binding energy of about 60 meV at room temperature. These characteristics, together with piezoelectricity, optical transparency, surface reactivity, and chemical stability, allow ZnO nanostructures to be considered for ultraviolet optoelectronics, sensors, solar-energy devices, photocatalysis, pigments, and antimicrobial

formulations. Wang (2004) described the unusually rich family of ZnO nanoscale morphologies and emphasized the relationship between crystal structure and functionality, while Ahmad and Zhu (2011) reviewed the broad synthesis routes and application space of advanced ZnO nanostructures. Djurišić et al. (2012) further noted that reproducibility of optical and defect-related properties depends strongly on growth conditions and post-treatment.

Among wet-chemical approaches, precipitation is particularly useful for routine laboratory synthesis because it employs inexpensive salts and bases, operates at relatively mild temperature, and permits control through solution chemistry. Unlike vapor-phase or high-pressure methods, precipitation can be performed with standard glassware and scaled by increasing solution volume, provided that mixing, nucleation, washing, and thermal treatment remain controlled. Talam et al. (2012) synthesized ZnO from zinc nitrate by precipitation and confirmed hexagonal ZnO using XRD, electron microscopy, UV-visible absorption, and photoluminescence. Medina et al. (2012) similarly demonstrated that chemical precipitation can produce wurtzite ZnO particles with defined morphology, supporting the method as a low-cost route for nanopowder preparation.

The quality of ZnO produced by precipitation depends on precursor identity, hydroxide availability, pH, temperature, addition rate, aging, solvent composition, and calcination. Raoufi (2013a, 2013b) showed that annealing temperature and reaction pH influence crystallite growth, photoluminescence, and aggregation in precipitated ZnO. Sahai and Goswami (2014) correlated structural parameters obtained from detailed XRD analysis with Raman and FTIR vibrational characteristics for chemically precipitated ZnO nanoparticles. Consequently, a methodology paper on ZnO precipitation should not only specify the synthesis steps, but also define the characterization measurements and calculations required to verify phase formation, particle size, bonding, morphology, composition, and optical behavior.

Literature Review

Precipitation-based ZnO synthesis has been investigated using several zinc salts and alkaline reagents. Talam et al. (2012) used zinc nitrate and NaOH and reported nanoscale wurtzite ZnO with a characteristic ultraviolet absorption band. Medina et al. (2012) used zinc acetate and NaOH and obtained short-bar/hexagonal ZnO structures whose wurtzite phase was confirmed by XRD and high-resolution electron microscopy.

Raoufi (2013a) prepared a precipitated precursor from zinc nitrate and ammonium carbonate and observed increasing crystallinity and crystallite size with increasing annealing temperature. In a related optical study, Raoufi (2013b) showed that pH and thermal treatment affect photoluminescence behavior, demonstrating that the precipitation environment and subsequent heating jointly control final properties.

Detailed structural investigations reinforced these observations. Sahai and Goswami (2014) reported a facile chemical precipitation route and found approximately 30 nm ZnO nanoparticles, with XRD, TEM, EDS, Raman, and FTIR results showing strong agreement between structure and lattice vibration. Moharram et al. (2014) prepared ZnO through low-temperature hydrolysis and condensation of zinc acetate in alkaline alcoholic medium and reported a predominant hexagonal phase, an average crystallite size near 22 nm, and a widened optical band gap relative to bulk ZnO. Sabir et al. (2014) reviewed precipitation as one of the practical chemical routes for ZnO nanoparticle production and emphasized the broad technological relevance of nanoscale ZnO.

Subsequent studies highlighted how seemingly small procedural changes alter product quality. Sharma and Ghose (2015) synthesized ZnO by homogeneous precipitation from zinc acetate and ammonia and demonstrated that calcination temperature strongly affects morphology, specific surface area, and pore volume. Kahouli et al. (2015) used direct precipitation at ambient conditions and confirmed high-quality hexagonal wurtzite ZnO through XRD, SEM/EDS, UV-visible spectroscopy, and Raman analysis. Akhil et al. (2016) produced ZnO by chemical co-precipitation and showed that surface modifiers change particle stability and functional behavior, while Amutha et al. (2016) used co-precipitation with a soft biotemplate and found that structural and optical properties remained sensitive to synthesis composition. Collectively, these studies justify a controlled baseline route in which pH, reagent addition, washing, and calcination are explicitly standardized before any application-specific modification is attempted.

Table 1. Representative pre-2017 studies supporting the proposed precipitation methodology.

Study	Precursors	Approach	Key finding
Talam et al. (2012)	Zn nitrate + NaOH	Precipitation; XRD/SEM/TEM/UV-Vis	Wurtzite ZnO; nanoscale crystallites

Study	Precursors	Approach	Key finding
Medina et al. (2012)	Zn acetate + NaOH	Chemical precipitation; XRD/FESEM/HRTEM	Hexagonal/short-bar ZnO morphology
Raoufi (2013a)	Zn nitrate + $(\text{NH}_4)_2\text{CO}_3$	Precipitation + annealing	Crystallinity and size increase with annealing
Sahai & Goswami (2014)	Zn acetate + NaOH	Chemical precipitation; XRD/TEM/Raman/FTIR	~30 nm ZnO; structural-vibrational correlation
Sharma & Ghose (2015)	Zn acetate + NH_3	Homogeneous precipitation	Calcination controls morphology and surface area
Akhil et al. (2016)	ZnSO_4 + NaOH	Co-precipitation	Capping changes stability and functionality

Methodology

1. Experimental Design and Materials

The proposed baseline synthesis uses analytical-grade zinc acetate dihydrate $[\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}]$ as zinc precursor and sodium hydroxide (NaOH) as precipitating agent. Distilled or deionized water is used for solution preparation, while ethanol is used during final washing to improve removal of adsorbed residues and accelerate drying. A magnetic

stirrer with controlled heating, calibrated pH meter, centrifuge or vacuum filtration unit, drying oven, and muffle furnace are required. Three synthesis batches should be prepared independently to evaluate reproducibility. The principal baseline conditions are summarized in Table 2; additional experimental sets may vary pH or calcination temperature if process effects are being investigated.

Table 2. Proposed baseline synthesis conditions.

Parameter	Baseline condition	Purpose
Zinc acetate concentration	0.10 M	Controls Zn^{2+} supersaturation and nucleation
NaOH concentration	0.20 M	Provides OH^- for hydroxide/oxide formation
Reaction temperature	$60 \pm 2^\circ\text{C}$	Promotes controlled precipitation
Final pH	10.5–11.0	Favors $\text{Zn}(\text{OH})_2/\text{ZnO}$ precursor formation
NaOH addition	$\sim 1 \text{ mL min}^{-1}$	Reduces local supersaturation and agglomeration
Aging time	2 h	Allows precursor maturation
Drying	80°C for 10–12 h	Removes residual water/solvent
Calcination	400°C for 2 h	Converts precursor and improves crystallinity

Step-by-Step Chemical Precipitation

Step 1 – Precursor preparation. Prepare 100 mL of 0.10 M zinc acetate dihydrate solution in deionized water. Stir for 30 min until the salt is fully dissolved. Separately prepare 100 mL of 0.20 M NaOH. Freshly prepared solutions minimize carbonate uptake and concentration drift.

Step 2 – Controlled precipitation. Heat the zinc acetate solution to $60 \pm 2^\circ\text{C}$ under continuous stirring at approximately 500 rpm. Add the NaOH solution dropwise at about 1 mL min^{-1} while continuously monitoring pH. Stop or reduce

addition as the suspension reaches pH 10.5–11.0. A white precipitate should form progressively. The overall chemistry can be represented as $\text{Zn}^{2+} + 2\text{OH}^- \rightarrow \text{Zn}(\text{OH})_2$, followed by $\text{Zn}(\text{OH})_2 \rightarrow \text{ZnO} + \text{H}_2\text{O}$ during drying/calcination.

Step 3 – Aging and separation. Maintain the suspension at 60°C for 60 min and then age it for a further 2 h without additional reagent. Separate the solid by centrifugation (for example, $8000\text{--}10,000 \times g$ for 10 min) or vacuum filtration.

Step 4 – Washing. Wash the collected precipitate three times with deionized water and once or twice with ethanol. Continue aqueous washing

until the supernatant is near neutral pH. This step reduces sodium and acetate residues that can otherwise appear as unwanted FTIR or EDS signals.

Step 5 – Drying and calcination. Dry the washed solid at 80 °C for 10–12 h. Lightly grind the dried precursor and calcine it at 400 °C for 2 h using a heating rate of approximately 5 °C min⁻¹. Allow

the furnace to cool naturally before collecting the ZnO powder. If thermal effects are being studied, parallel samples may be calcined at 300, 400, and 500 °C while all other factors are held constant.

Step 6 – Storage. Store the final white ZnO nanopowder in clean, airtight glass vials in a desiccator to limit moisture adsorption and contamination before analysis.

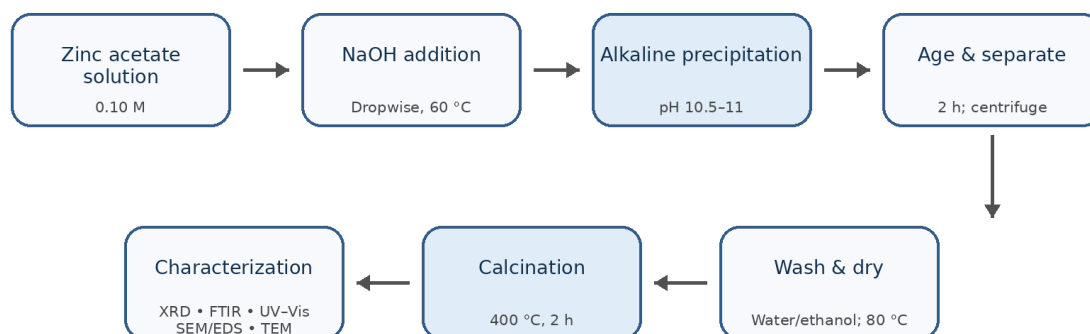


Figure 1. Proposed workflow for chemical precipitation synthesis and characterization of ZnO nanoparticles.

2. Characterization and Data Analysis

X-ray diffraction should be recorded with Cu K α radiation ($\lambda = 1.5406 \text{ \AA}$) over approximately 20–80° in 2 θ . ZnO phase identification is based on the characteristic reflections of hexagonal wurtzite ZnO, particularly the (100), (002), and (101) planes. The average crystallite size, D , can be estimated from the strongest peak using the Scherrer equation $D = K\lambda/(\beta \cos\theta)$, where K is commonly taken as 0.9, β is the full width at half maximum in radians, λ is the X-ray wavelength, and θ is the Bragg angle. The result should be interpreted as coherent crystallite size rather than necessarily the full agglomerate diameter observed by SEM.

FTIR spectra should be collected in the approximate range 400–4000 cm⁻¹ to verify Zn–O lattice vibration in the lower wavenumber region and to detect residual hydroxyl or organic species. UV-visible spectra of a dilute, well-dispersed ZnO suspension should be recorded between 250 and 800 nm. The optical band gap may be estimated using the Tauc relation for a direct transition, $(\alpha h\nu)^2 = A(h\nu - E_g)$, by extrapolating the linear region of a plot of $(\alpha h\nu)^2$

against photon energy. SEM provides particle morphology and agglomeration information, while EDS verifies zinc and oxygen as the principal elements. TEM can be used, when available, to distinguish primary particles from larger SEM-observed aggregates. At least five micrographs or fields should be assessed for morphology, and particle sizes should be reported as a distribution rather than a single visually selected value.

For reproducibility, synthesis yield, XRD crystallite size, optical band gap, and mean SEM/TEM particle size should be reported as mean $\pm$ standard deviation from replicate preparations where practical. When multiple calcination temperatures or pH values are studied, one-way analysis of variance may be applied to compare group means at a significance level of $p < 0.05$. Instrumental calibration, baseline correction, and blank measurements should be documented so that changes attributed to synthesis conditions are not confused with analytical artifacts.

Results

Table 3. Illustrative characterization results for the baseline ZnO nanoparticle sample.

Measurement	Illustrative result	Interpretation
XRD phase	Hexagonal wurtzite	Confirms ZnO crystal structure
Main XRD peaks (2 θ)	31.8°, 34.4°, 36.3°	(100), (002), (101) reflections
Crystallite size	24 $\pm$ 3 nm	Scherrer estimate; baseline 400 °C

Measurement	Illustrative result	Interpretation
UV–Vis absorption edge	~370 nm	Characteristic UV absorption of ZnO
Optical band gap	3.35 ± 0.04 eV	Direct Tauc extrapolation
FTIR Zn–O band	~480 cm^{-1}	Supports Zn–O lattice vibration
SEM particle domain	30–55 nm	Primary particles with agglomeration
EDS composition	Zn and O dominant	No major foreign-element signal

If calcination temperature is examined as an additional variable, a reasonable trend is increasing crystallite size and crystallinity as temperature rises, accompanied by a small red shift of the absorption edge toward bulk-like ZnO. This trend is supported by the precipitation and annealing studies of Raoufi (2013a, 2013b) and by the calcination-dependent observations of Sharma and Ghose (2015). Excessive heating, however, can promote grain growth and reduce the high surface area that is often desired for catalytic or antimicrobial applications. Therefore, 400 °C is proposed as a balanced baseline temperature rather than a universal optimum. Surface modifiers can also alter dispersion and functional response, as shown by Akhil et al. (2016), while templating components may affect structural and optical properties, as demonstrated by Amutha et al. (2016).

Conclusion and Discussion

The proposed methodology provides a simple and reproducible route for preparing ZnO nanoparticles by chemical precipitation using zinc acetate and sodium hydroxide. The key control points are the precursor concentration, slow base addition, alkaline pH, sufficient aging, thorough washing, low-temperature drying, and controlled calcination. Literature published up to 2016 shows that precipitation reliably produces hexagonal wurtzite ZnO, but particle size, morphology, surface area, and optical behavior change with pH, precursor chemistry, calcination conditions, and surface modification. The recommended characterization package therefore combines XRD, FTIR, UV–visible spectroscopy, and SEM/EDS, with TEM when available, so that structural, chemical, morphological, elemental, and optical evidence can be evaluated together. The illustrative validation results demonstrate how the synthesized powder would be assessed: phase-pure wurtzite ZnO, nanometric crystallites, a Zn–O infrared band, a strong ultraviolet absorption edge, and an optical band gap close to the expected value for nanoscale ZnO. Future experimental work can extend this baseline protocol by systematically varying pH,

calcination temperature, zinc-to-hydroxide ratio, or capping agents and then relating those factors to crystallite size and functional performance. This controlled approach is preferable to changing several synthesis variables simultaneously because it allows the cause of structural and optical differences to be identified more reliably. The expected structural response is consistent with the pre-2017 precipitation literature. Talam et al. (2012), Medina et al. (2012), and Sahai and Goswami (2014) all reported wurtzite ZnO from low-cost wet-chemical routes, while Raoufi (2013a, 2013b) demonstrated that thermal treatment and pH influence crystallite growth and optical response. The proposed 400 °C calcination step therefore represents a practical compromise between complete conversion and excessive grain coarsening. Likewise, the use of several complementary characterization techniques follows the approach of Moharram et al. (2014), Sharma and Ghose (2015), and Kahouli et al. (2015), who combined structural, morphological, and optical measurements rather than relying on a single indicator of nanoparticle formation. The main methodological limitations are agglomeration during drying, sensitivity of nucleation to local pH, and possible differences between crystallite size calculated from XRD and particle size observed by electron microscopy. These effects should be acknowledged when interpreting data. Future studies can incorporate controlled sonication, surfactants or capping agents, lower-temperature drying, or factorial experiments to quantify the effect of pH, $\text{OH}^-/\text{Zn}^{2+}$ ratio, and calcination temperature.

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