Effect of Amount of Leaf Extract and Precursor Salt on Green Synthesis of ZnO Nanoparticles: A Comparative Study (UV–Vis, FTIR, XRD)

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Abstract

The green synthesis of ZnO nanoparticles using Ocimum tenuiflorum leaf extract and zinc nitrate as a precursor was systematically investigated through UV-Vis spectroscopy, FTIR, and XRD, revealing the critical roles of precursor salt and leaf extract concentrations in determining nanoparticle quality, yield, and properties. Three samples (10 g leaf extract + 5 g zinc nitrate, 10 g leaf extract + 10 g zinc nitrate, and 20 g leaf extract + 5 g zinc nitrate) were prepared by chemical bath deposition technique. The absorbance peak, particle size distribution, and band gap were investigated by UV-Vis spectroscopy. Crystallinity and amount of organic residue were investigated using FTIR spectroscopy of all the three samples. Effect of variation in the amount of leaf extract on the defects in the samples was investigated using shifts in XRD peaks of the samples. Findings highlight that an optimal leaf extract-to-precursor ratio is essential for achieving high-purity, crystalline ZnO nanoparticles with uniform size distribution. Increasing the leaf extract enhances yield and crystal quality but introduces organic residues that alter surface chemistry, while excess precursor salt increases yield at the cost of structural defects and non-uniformity. This study underscores the potential of green synthesis for producing high-quality ZnO nanoparticles and provides a framework for future material optimization.

Keywords: ZnO nanoparticles, Leaf extract, Precursor concentration, Green synthesis, UV–Vis, FTIR, XRD.
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Introduction

ZnO is an important semiconductor material due to its direct wide band gap, low cost, and compatibility with optoelectronic and energy-based devices. Conventionally, ZnO nanoparticles have been synthesized by using different methods like chemical reduction, sol-gel technique, and hydrothermal techniques which often include hazardous reducing agents and processing at high temperature [1]. Green synthesis of ZnO from plant extracts provides an environment friendly alternative with low toxicity.

The phytochemicals in the leaf extracts act in dual roles: as reducing agents for the metal ions and as surface capping. However, the ultimate physicochemical properties of ZnO nanoparticles strongly depend on the balance in precursor salt-extract concentration [2,3]. Excess precursor results in larger-sized particles with structural defects, while excess extract may result in organic residues that are not removed.

Although there are many reports on plant-mediated synthesis, there is limited comparative work that considers how the amount of extract and precursor together affects optical and structural properties [4]. Therefore, three different compositions synthesized under identical conditions have been directly compared in this work.

Method

Materials

Fresh leaves of Ocimum tenuiflorum were collected locally. Zinc nitrate hexahydrate ($Zn(NO_3)_2 \cdot 6H_2O$) was used

without further purification. All solutions were prepared in distilled water [5, 6].

Preparation of Leaf Extract

Measured quantities (10 g and 20 g) of washed leaves were boiled in 100 mL water at 70 °C for 30 minutes, cooled, and filtered. The filtrate served as the reducing agent.

Synthesis of ZnO Nanoparticles

Three samples were prepared as shown in table 1. The extract was added drop wise into the precursor solution while stirring at 60 °C pH was adjusted close to 9.0 using NaOH. After 1h stirring and 2h ageing, precipitates were washed, dried at 80 °C, and calcined at 400 °C for 2 h.

Table 1: Three samples of Zn Nitrate Nanoparticles.

Sample	Leaf Extract	Zn Nitrate
S1	10 g	5g
S2	10 g	10g
S3	20 g	5g

Results and Discussion

UV–Vis Analysis

The UV-Vis absorption spectra of the three synthesized samples (designated as Sample 1, Sample 2, and Sample 3) were obtained and analysed as shown in figure 1. The following observations were made:

Absorbance Values

In each sample, the absorbance values were measured at different wavelengths. Sample 2 exhibited much higher absorbance values than Sample 1, while Sample 3 recorded an intermediate absorbance value. In sample 2, precursor salt was used in double amount compared to sample 1. Zinc Nitrate ($Zn(NO_3)_2$) acted as the source of zinc ions (Zn^{2+}) during the synthesis. Indeed, in the process, these zinc ions will take part in forming the ZnO nanoparticles. As a result, the zinc ions will undergo a series of chemical reactions with other components to realize the ZnO nanoparticles [7]. Thus, high amount of ZnO particles formed will have greater amount of absorbance with broadest absorption peak.

Peak Wavelengths

Sample 1 and Sample 3 have peak wavelengths of 325 nm and 328nm, respectively, while Sample 2 has the highest wavelength of about 332 nm. The difference between these values shows that there is some variation in the size and electronic transitions of the synthesized nanoparticles. High yield of ZnO nanoparticles is obtained for sample 2 but they are not of good quality i.e., nanoparticles in this sample may be non-uniform in size or shape and have structural defects compared to the samples 1 and 3. Using Tauc plots band gap of sample 1 is found to be 2.94eV, 2.61eV in sample 2 and 2.60 eV in sample 3 [8]. Smaller the size of nanoparticle, greater will be the quantum confinement effect and so will be the band gap. Higher band gap in sample 1 shows it has smaller particle size.

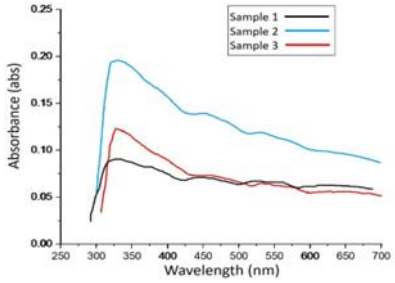


Figure 1: UV–Vis absorption spectra of ZnO nanoparticles synthesized using different extract-to-precursor ratios

FTIR Analysis

The FTIR spectra of all the three samples are as shown in figure 2. The results are as follows:

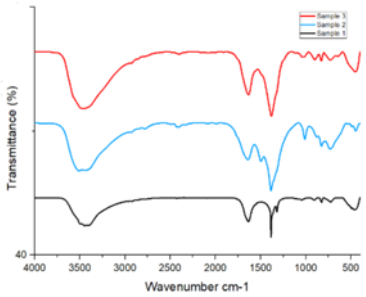


Figure 2: FTIR spectra of ZnO nanoparticles showing major functional groups and Zn–O vibration

Sample 1

It has the Sharpest peaks with minimal broadening or overlap, especially at 1400 cm^{-1}, 1300 cm^{-1}, and 500 cm^{-1} (Zn-O). The sharp peaks around 1300 - 1100 suggesting a controlled presence of organic capping. Functional groups from the extract are moderately present. The minimal additional peaks and noise reflect high purity, clean surface, and less organic residue [9]. It has the cleanest fingerprint, showing high purity crystalline ZnO.

Sample 2

In this sample, the peaks are broader and less defined, in particular in the 1000–1700 cm^{-1} region. Increased broadening and overlap may indicate the increased presence of organic residues with various vibrational modes that overlap. The 1400 cm^{-1} peak (C-H bending) is wider and less defined compared to Sample 1, with significant overlap into the 1300 cm^{-1} region. Peaks around 1200 cm^{-1} and 1100 cm^{-1} can be observed that may correspond to the C-O-C or C-OH vibrations, although lacking sharpness. The

fingerprint region for Sample 2 is present but significantly broader and not sharp compared to Sample 1, which can be related to the lower crystallinity and less uniform ZnO structure in Sample 2. More small peaks and noise appear between 1000 and 600 cm^{-1}, which may reflect more organic residues or surface defects contributing to the spectrum.

Sample 3

The results lie between 1 and 2, likely because of a better reducing agent ratio than Sample 2 but not as good as Sample 1. Peaks are intermediate in number and intensity, but less sharp than Sample 1. The 1400 cm^{-1} peak is slightly broader than that of Sample 1, though well defined, with some overlap into the 1300 cm^{-1} region. Also, the 1300 cm^{-1} peak is visible but slightly broader than Sample Peaks at approximately 1200 and 1100 cm^{-1} are present but a bit sharper and less noisy compared to Sample 2. Such peak characteristics indicate the moderate presence of organic residues, more than Sample 1 but less than Sample 2. The intensity is intermediate, reflecting a balanced presence of organic capping due to the higher leaf extract (20 g).

XRD Analysis

Elemental analysis of the samples was performed by X-ray diffractometers using Cu Kα as a radiation source of wavelength (λ = 1.5406 Å). d-spacing were calculated using Bragg's equation

$$2d\sin(\theta) = n\lambda$$

and size of the crystallites were calculated using Scherrer equation [10],

$$D = k\,\lambda\,/\beta\cos(\theta)$$

where β = Full wave Half Maximum, k = Scherrer constant = 0.9. The data analysis and result matching were done using Origin pro and X'pert Highscore software.

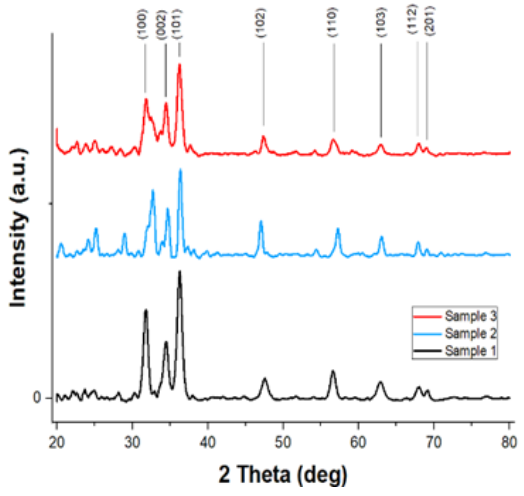


Figure 3: XRD pattern of ZnO nanoparticles confirming wurtzite crystal structure.

The XRD spectra for all three samples (figure 3) confirm the presence of hexagonal wurtzite ZnO structure known as Zincite, as evidenced by the presence of characteristic diffraction peaks indexed to planes (100), (002), (101), (102), (110), (103), (112), and (201), matching JCPDS card no. 36-1451. Following results are obtained:

Sample 1

It showed the most well-defined and well positioned peaks. It had a clean baseline with minimum amorphous hump. The intensities of the peaks were moderate. The average crystal size was 14.64 nm and crystallinity of the sample was 68%. The 2θ positions for the prominent ZnO peaks were 31.68, 34.55, 36.24, 47.57, 56.54, 62.99, 67.94, 69.10 with d-spacing 2.82, 2.59, 2.47, 1.90, 1.62, 1.47, 1.37, 1.35 respectively.

[Crystallinity = area of crystal peaks / area of (crystal + amorphous) peaks]

Sample 2

It has intense, thin and sharp peaks indicating high yield of crystal ZnO. However, these peaks were slightly shifted from their 2θ positions. Also, it had the highest number of secondary peaks which belong to Zn(OH)2 [Zinc Hydroxide]. The average crystal size was 19.56 nm and crystallinity was about 57%. The 2θ positions for the prominent ZnO peaks were 32.83, 34.64, 36.45, 47.09, 57.26, 63.19, 67.81, 68.99 with d-spacing 2.72, 2.58, 2.46, 1.92, 1.61, 1.47, 1.38, 1.35 respectively.

Sample 3

The peaks are is well positioned but less defined as sample 1. They are slightly broader, noisy and suppressed. It has the largest background amorphous hump. The average crystal size was 16.65 nm and crystallinity was about 47%. The 2θ positions for the prominent ZnO peaks were 31.78, 36.27, 47.32, 56.49, 63.05, 67.92, 69.05 with d-spacing 2.81, 2.61, 2.47, 1.91, 1.62, 1.47, 1.37, 1.35 respectively.

Conclusion

The work hereby affirms that the ratio of leaf extract to precursor is a decisive factor in the green synthesis of ZnO nanoparticles. The extract of leaves works as a reducing and stabilizing agent, reducing the precursor salt to metal-Oxide particles. The UV-Vis spectra of all the three samples show that with a slight increase in concentration, improvements in crystal quality as well as better yield with more homogeneous particle size and morphology can be obtained. Such improvement is due to increased availability of the phytochemicals such as polyphenols, flavonoids, and other biomolecules in the extract, and hence a greater number of active reducing sites that allow better control of the nucleation and growth processes involved in the

nanoparticle formation. The slight shifts in FTIR peaks suggest changes in bonding environments, likely due to residual organic matter or incomplete conversion. The increased intensity and broadening indicate that it contains more organic residues or unconverted precursors, consistent with excess precursor, limited reduction. Also, Intensity variation shows higher absorption but poorer resolution. The XRD spectra shows that the organic molecules scatter the X-rays diffusively, not constructively as crystals. Organic residue might have stuck on the surface of the nano particles which changed surface chemistry and led to overall increase in size.

A balanced composition, 10 g extract + 5 g precursor, resulted in the most crystalline and least defective nanoparticles with high bandgap energy and minimum organic residue. An excess of precursor increased yield while degrading quality in structure, and excess extract resulted in a higher organic content within the particles. Therefore, regulation of the two variables mentioned is absolutely necessary for reliable and sustainable nanoparticle synthesis.

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