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RESEARCH ARTICLE

Role of Piper betle extract in the Precipitation of ZnO Nanoparticles for improved Photocatalytic Efficiency

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Abstract

The present study delves into an eco-friendly and scalable method for creating zinc oxide (ZnO) nanoparticles using extract from Piper betle leaves. By harnessing the natural bio-reductive and capping abilities of the phytochemicals found in P. betle, the ZnO nanoparticles formed a hexagonal wurtzite structure, with an average crystallite size of about 20.3 nm, as confirmed by XRD analysis. The UV–Visible spectroscopy results showed a notable blue shift in absorption, leading to an estimated band gap of 4.10 eV, which is linked to quantum confinement effects. FTIR spectra confirmed the presence of biomolecules on the surface, while SEM imaging displayed quasi-spherical polycrystalline particles with some aggregation. To assess the photocatalytic potential, we evaluated the degradation of methylene blue (MB) dye under UV light, where the Piper betle–ZnO catalyst achieved over 90% degradation efficiency in just ˜80 minutes, with a pseudo-first-order rate constant of 0.0342 min^{-1}. These results highlight the collaborative role of P. betle's phytoconstituents in shaping the nucleation, growth, and surface chemistry of ZnO nanoparticles, which enhances their ability to absorb light and separate charge carriers. This green approach presents a promising strategy for developing effective photocatalysts aimed at cleaning up wastewater.

Keywords: ZnO nanoparticles, piper betle extract, precipitation, methylene blue dye degradation.

1 Introduction

In recent years, the unchecked release of synthetic dyes from industries such as textiles, paper, and plastics has led to significant pollution in our water ecosystems [6]. This not only threatens the environment but also poses serious health risks to humans due to the toxic nature of these dyes, their persistence in the environment, and their resistance to standard treatment methods [1]. Fortunately, advanced oxidation processes, especially semiconductor-mediated photocatalysis, have shown great promise for completely breaking down these pollutants when exposed to UV or visible light [23]. Among the most researched photocatalysts are zinc oxide (ZnO) nanoparticles, due to their wide band gap (3.2–3.3 eV), high exciton binding energy, chemical stability, and affordability [20]. However, traditional methods of synthesizing these nanoparticles, like

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sol-gel, hydrothermal, and chemical precipitation, often involve high temperatures, toxic solvents, or complicated procedures, which can hinder scalability and environmental friendliness [9]. Additionally, inherent defects in ZnO, such as zinc and oxygen vacancies, can negatively affect photocatalytic efficiency unless they are meticulously managed during the synthesis process [26]. Green synthesis methods address these challenges by utilizing plant-based phytochemicals, like flavonoids, terpenoids, and phenolics, as natural reducing and capping agents [14]. This eco-friendly approach not only removes the need for harmful chemicals but also often results in nanoparticles that boast improved surface functionalities and reactive sites [21]. So far, a range of botanical extracts from Cocos nucifera to Azadirachta indica have been effectively used to create ZnO nanoparticles that show impressive photocatalytic and antimicrobial properties [25]. Piper betle L. (betel vine) stands out for its abundance of phenolic compounds and essential oils, giving its extracts powerful reducing, stabilizing, and bioactive qualities [4]. Initial studies showcased its ability to synthesize noble-metal nanoparticles, and more recent research has expanded this to ZnO, revealing well-defined wurtzite structures with moderate crystallite sizes and a relatively wide band gap when using P. betle leaf extract [2]. These P. betle/ZnO hybrids also feature high surface areas (97 m^2/g) and porous structures that enhance pollutant adsorption and light absorption [24]. Additionally, petiolar extracts from P. betle have demonstrated the ability to produce ZnO nanoparticles with notable antioxidant and antibacterial properties [16], underscoring the versatile advantages of this green synthesis method. The role of P. betle phytochemicals in a controlled precipitation synthesis, where factors like extract concentration, pH, and reaction time are carefully adjusted, hasn't been fully explored yet. Traditional chemical precipitation methods that use zinc nitrate and NaOH often produce particles with a wide range of sizes and limited surface reactivity [8]. In contrast, a green-assisted precipitation approach could combine the straightforwardness of chemical methods with the beneficial properties of plant extracts [8].

In this study, ZnO nanoparticles were synthesized by a simple precipitation method using Piper betle leaf extract in a one-pot approach. We examined how P. betle phytochemicals affect nucleation, growth, and defect passivation. The resulting P. betle/ZnO photocatalysts show significantly enhanced degradation rates for methylene blue (with rate constants reaching about 0.0342 min^{-1} under UV light) and notable dye-removal efficiencies (over 90% in less than 80 minutes) compared to standard ZnO. By merging green chemistry with a scalable precipitation method, this study establishes a foundation for eco-friendly, high-performance ZnO photocatalysts designed for environmental remediation.

2 Materials and Methods

2.1 Materials

Zinc nitrate hexahydrate ($Zn(NO_3)_2{\cdot}6H_2O$, AR grade) and sodium hydroxide (NaOH, AR grade) were sourced from SRL (Sisco Research Laboratory, India). Fresh Piper betle leaves were picked up from local market in Kharar, Punjab, INDIA (30.7499°N, 76.6411°E), thoroughly washed with deionized water, and then air-dried in the shade for about 5 to 7 days. All solutions were made using triple-distilled (MilliQ) water.

2.2 Method

2.2.1 *Preparation of Piper betle Leaf Extract*

To prepare the extract, dried *P. betle* leaves were finely ground and were kept in airtight containers. 10 grams of this leaf powder was mixed with 100 ml of MilliQ water, then heated the mixture to 80 °C for 30 minutes while stirring continuously. Once it cooled down to room temperature, it was centrifuged at 4500 rpm for 10 minutes and filtered using Whatman No. 1 paper. The clear liquid that resulted was collected and stored at 4 °C for later use.

2.2.2 *Synthesis of ZnO nanoparticles using Piper betle leaves extract*

In a controlled environment, a 0.1 M solution of $Zn(NO_3)_2$ was stirred magnetically at room temperature. Gradually, 20 ml of P. betle extract was added drop by drop until a distinct color change, signaling the start of nucleation. The pH was then adjusted to 9.0 with 0.5 M NaOH, and the stirring continued for 2 hours. After that, we collected the resulting white precipitate through centrifugation at 8000 rpm for 15 minutes. It was washed three times with deionized water and once with ethanol, then dried at 70 °C for 10 hours. To finish, the powder was calcined at 400 °C for two hours to produce pure ZnO NPs.

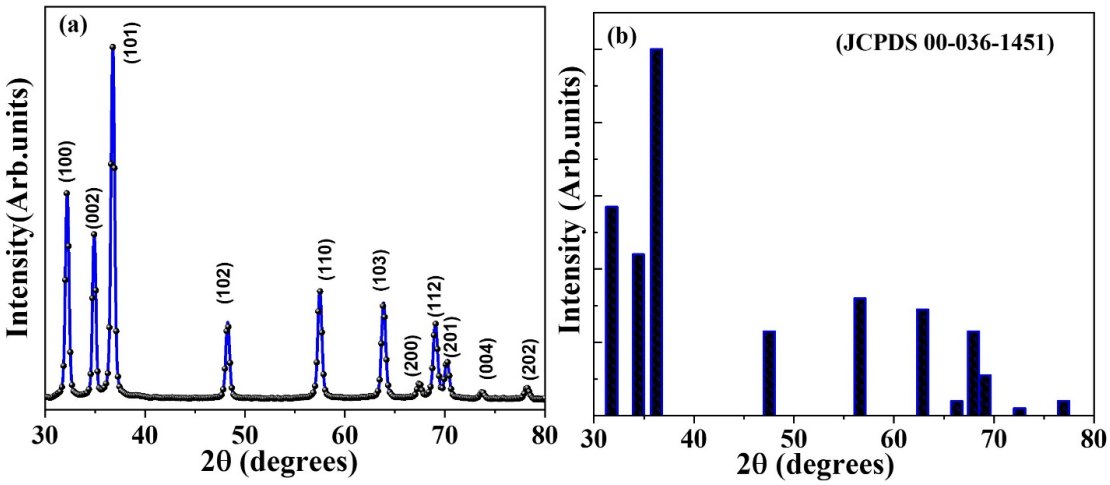

Figure 1. XRD analysis of Piper betle-mediated ZnO NPs (a) and JCPDS data (b).

2.3 Characterization techniques

The crystallographic structure and average crystallite size of ZnO NPs were analyzed using X-ray diffraction (XRD) with a Panalytical X'PERT PRO diffractometer (λ = Cu Kα), scanning from 2θ = 30–80°. To estimate the band-gap energy (Eg), the optical absorption spectrum was recorded on a Shimadzu UV-2600 spectrophotometer through Tauc analysis. The morphological features were observed with a Carl Zeiss SUPRA 55VP field-emission scanning electron microscope (FESEM). The elemental composition was analyzed via energy-dispersive X-ray (EDX) spectroscopy with the Oxford Instruments AZtec system. The functional groups were analyzed using Fourier-transform infrared (FTIR) spectroscopy on a Bruker Alpha II instrument (4000–500 cm^{-1}).

3 Results and discussion

3.1 X-ray diffraction analysis of *Piper betle*-mediated ZnO nanoparticles

The structural properties of the as-synthesized Piper betle–capped ZnO nanoparticles were investigated by X-ray diffraction (XRD). The resulting pattern (Figure 1 (a)) shows the distinct reflections of the hexagonal wurtzite phase at angles of $2\theta \approx 31.8°$(100), 34.4°(002), 36.3°(101), 47.5°(102), 56.6°(110), 62.8°(103), 66.4°(200), 67.9°(112), 69.1°(201), and 72.6°(202), which align perfectly with JCPDS card no. 36-1451 (Figure 1 (b)). There were no extra peaks observed, which confirms that it has phase purity and no secondary phases or excess organics.

By applying the Debye–Scherrer equation [10] (1) to the main (101) peak, we calculated an average crystallite size of 20.3 nm.

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

where K is the shape factor (0.9), λ is the X-ray wavelength (0.154 nm), β is the full width at half maximum (FWHM), and θ is the Bragg angle.

Additionally, the broadening of the peak hints at a bit of lattice strain, probably caused by interactions with Piper betle phytochemicals during the growth process. Overall, these XRD findings confirm that the green-assisted precipitation method successfully yields pure, nanocrystalline ZnO with a well-controlled particle size and impressive structural order.

3.2 UV-visible analysis of Piper betle mediated ZnO NPs

A closer examination of the optical properties of the Piper betle–capped ZnO NPs was conducted using UV–Vis spectroscopy (Figure 2 (a)). The resulting absorption spectrum displays a prominent excitonic peak around 274 nm, indicating the nanoparticles' effectiveness at capturing light in the UV range.

Using the absorbance data to construct a Tauc plot for a direct band-gap semiconductor [13] (Figure 2 (b)), the linear extrapolation of (αhv)2 against hv revealed an energy band gap (Eg) of approximately 4.10 eV. This noticeable blue shift when compared to bulk ZnO (which possesses a band gap of 3.3 eV) [17] is attributable to quantum confinement within the roughly 20 nm crystallites [5]. Additionally, the presence of surface-bound phytochemicals likely influences the local electronic structure, promoting higher-energy transitions.

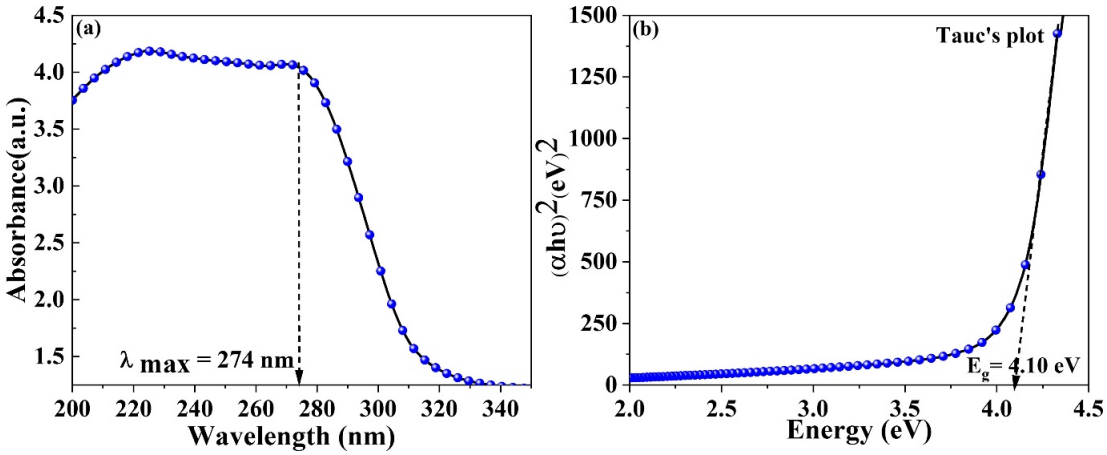

Figure 2. (a) UV-visible pattern of Piper betle mediated ZnO NPs, (b) Tauc's plot of Piper betle mediated ZnO NPs.

3.3 FTIR Analysis of Piper betle mediated ZnO NPs

The Fourier-transform infrared (FTIR) spectrum of the Piper betle/ZnO NPs (Figure 3) shows several bands that confirm the presence of both ZnO lattice vibrations and phytochemicals bound to the surface [7]. A broad band in the range of 3741–3833 cm^{-1} is linked to the O–H stretching of adsorbed water and phenolic –OH groups from the P. betle extract. There's a subtle feature around 3010 cm^{-1} that corresponds to aromatic C–H stretching, hinting at some leftover organic materials. A clear band at 2340 cm^{-1} is due to CO_2 adsorption that occurred during the pellet preparation. In the fingerprint region, the band at 1711 cm^{-1} is associated with C=O stretching from carboxylate or carbonyl groups, while the strong absorption at 1535 cm^{-1} is likely due to N–H bending or aromatic C=C vibrations from the extract's phytochemicals [12]. Lastly, the bands at 825 and 679 cm^{-1}, along with a strong feature around 500–600 cm^{-1}, are linked to ZnO lattice vibrations [22], confirming that the wurtzite ZnO structure has formed. All these spectral features together indicate that the biomolecules from Piper betle are effectively adsorbed onto the ZnO surface, which helps in capping and may also influence the nanoparticles' optical and photocatalytic properties.

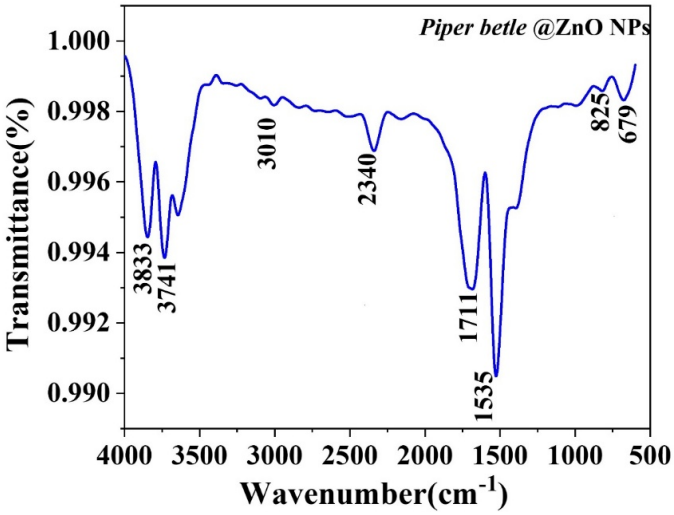

Figure 3. FTIR spectrum of Piper betle mediated ZnO NPs.

3.4 SEM analysis of *Piper betle* mediated ZnO NPs

The scanning electron microscopy (SEM) images of the Piper betle–capped ZnO NPs (Figure 4) reveal quasi-spherical particles that tend to clump together into larger formations. Each particle is about 30–60 nm in size and shows a notable amount of surface roughness; these qualities are tied to the capping effect of P. betle phytochemicals during their growth [3]. Also, energy-dispersive X-ray (EDX) spectroscopy performed in situ on the same areas confirms that only Zn and O are present, with weight percentages of 80.2% for Zn (K) and 19.8% for O (K). This translates to atomic fractions of 53.8% and 46.2%, respectively. The nearly stoichiometric Zn:O ratio underscores the phase purity of the nanocrystals and suggests that there's minimal incorporation of leftover organics or impurities from the precursors [18]. Additionally, the particle size is larger than the crystallite size (20.3 nm) calculated from XRD, indicating that the nanoparticles are polycrystalline in nature, made up of multiple smaller crystallites that have come together [11].

3.5 Photocatalytic degradation of Methylene Blue using *Piper betle*-mediated ZnO

The distinct absorption peak of MB dye at λ = 665 nm acted as a key indicator for tracking photodegradation [15]. When the Piper betle–ZnO photocatalyst was introduced and exposed to UV light, there was a steady decline in peak intensity over time intervals of 0, 15, 30, 45, 60, and 90 minutes (Figure 5), which indicated that the dye molecules

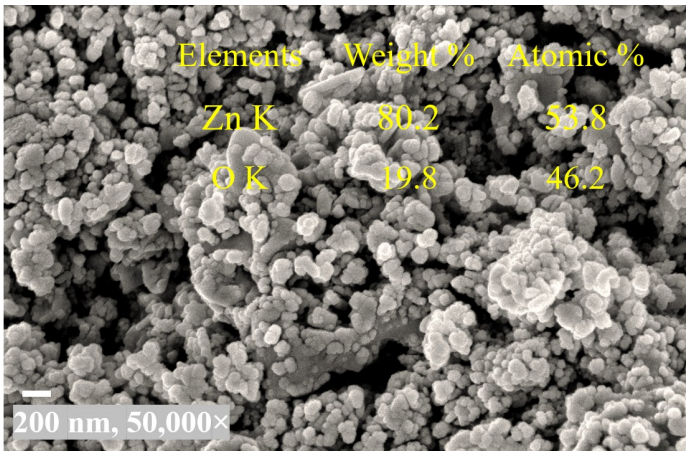

Figure 4. SEM image and corresponding EDX spectrum of Piper betle-capped ZnO NPs.

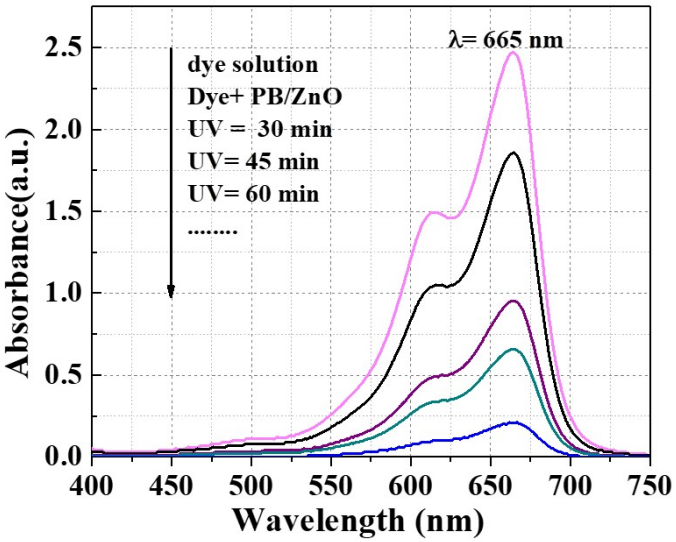

Figure 5. UV–Vis absorption spectra of methylene blue (MB) solution degraded by PB/ZnO photocatalyst under UV irradiation.

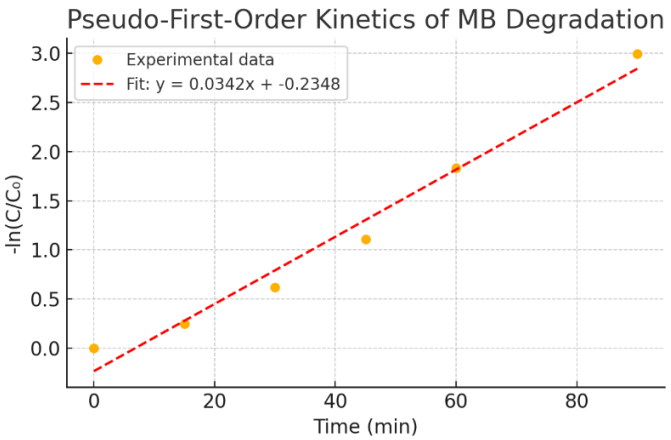

Figure 6. Pseudo-first-order kinetic plot for MB degradation by *Piper betle*-mediated ZnO under UV light.

were breaking down. Interestingly, the spectral profile didn't change in terms of peak position, which confirmed degradation rather than a transformation into intermediate dye species. The absorbance dropped sharply in the first 30 minutes and nearly reached zero by the 90-minute mark, showcasing the rapid and effective photocatalytic action of the green-synthesized ZnO. This impressive performance can be linked to the phytochemical compounds found in Piper betle extract, which act as natural stabilizers and capping agents during the formation of nanoparticles [19]. These biomolecules help reduce charge recombination, increase surface area, and facilitate efficient electron–hole separation during UV excitation. The arrow in the Figure 5 illustrates the gradual decrease in absorbance as irradiation time increases, highlighting the successful degradation of MB in the presence of P. betle–ZnO under UV light.

3.6 Photocatalytic Degradation Analysis

The photocatalytic performance of the synthesized Piper betle–ZnO NPs was evaluated by modeling the degradation kinetics of methylene blue (MB) under UV light. A pseudo-first-order rate equation was employed for this analysis:

$$-ln(\frac{C}{C_0}) = kt \tag{2}$$

where, C_0 denotes the initial concentration of the dye, C represents its concentration at time t, and k is the rate constant (min^{-1}).

A graph plotting $-ln(\frac{C}{C_0}) = kt$ against irradiation time (Figure 6), which spanned from 0 to 90 minutes, produced a straight line with a strong correlation coefficient ($R^2 = 0.976$). This finding strongly supports the use of the pseudo-first-order kinetics model for the degradation process. The calculated rate constant (k) for this system came out to be 0.0342 min^{-1}. This rate constant stands out when compared to many other ZnO-based systems, highlighting the remarkable photocatalytic activity of the Piper betle–ZnO. This enhanced performance is likely due to its bio-functional surface, reduced charge recombination, and better dye adsorption. Moreover, the observed rate constant is significantly higher than those reported for pristine ZnO and various other green-synthesized ZnO photocatalysts, indicating a notable boost in degradation efficiency. All photocatalytic experiments were conducted under UV irradiation (365 nm, 50 W), using an initial MB concentration of 10 mg/L and a catalyst dose of 20 mg per 100 ml.

4 Conclusion

The present study successfully synthesized zinc oxide (ZnO) nanoparticles using a green precipitation method that was facilitated by the extract of Piper betle leaves. This extract, rich in phytochemicals, served a dual purpose as both a reducing and stabilizing agent, allowing us to form nanocrystalline ZnO with a well-defined wurtzite structure, an average crystallite size of about 20.3 nm, and impressive surface functionalization. Various characterization techniques, including XRD, UV–Vis spectroscopy, FTIR, and SEM–EDX, were used to confirm the structural integrity, optical properties, and phytochemical surface capping of the nanoparticles. The resulting ZnO NPs showed a blue-shifted band gap of around 4.10 eV and displayed remarkable photocatalytic activity for breaking down methylene blue dye under UV light, achieving over 90% removal in just 80 minutes. Kinetic analysis indicated a pseudo-first-order reaction with a rate constant of 0.0342 min^{-1}, which outperformed many previously reported green and chemically synthesized ZnO systems. These results underscore the promise of using Piper betle in the synthesis process as a scalable and eco-friendly method for producing high-performance ZnO photocatalysts, ideal for environmental cleanup applications.

Data Availability Statement

Data will be made available on request.

Funding

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Conflicts of Interest

The authors declare no conflicts of interest.

Ethical Approval and Consent to Participate

Not applicable.

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