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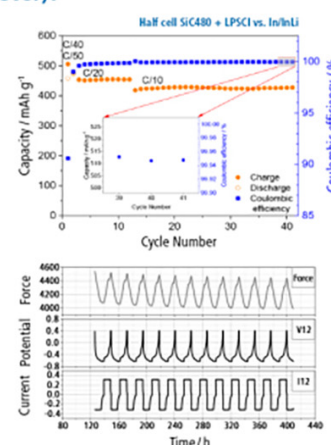
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Characterization and Photocatalytic Evaluation of ZnO Nanoparticles Synthesized with Terminalia mantaly Extract

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Abstract: This study outlines an environmentally friendly method for producing zinc oxide (ZnO) nanoparticles using Terminalia mantaly leaf extract. From SEM image, the synthesized ZnO nanoparticles exhibit irregular shapes and significant aggregation. XRD analysis confirms the nanoparticles crystallize in the hexagonal wurtzite phase, indicating high chemical purity. FTIR spectroscopy further supports this by identifying characteristic functional groups corresponding to ZnO, validating the formation of pure ZnO nanoparticles. Photocatalytic activity was evaluated through the degradation of methylene blue dye under UV-Vis irradiation, which followed pseudo-first-order kinetics with a degradation efficiency exceeding 99%. These results demonstrate the strong photocatalytic potential of the synthesized ZnO nanoparticles, supported by comprehensive morphological, structural, and chemical analyses. These findings highlight Terminalia mantaly leaf extract as a sustainable and eco-friendly reducing agent for ZnO nanoparticle synthesis with promising applications in environmental remediation.

Keywords: Terminalia mantaly, ZnO, XRD, eco-friendly synthesis, and methylene blue.

1. Introduction:

Currently, organic dye pollution from sectors such as printing, textiles, and painting, poses a significant threat to the environment because of their immense resistance to biodegradation. Methylene Blue (MB) is one of the dyes frequently used in industrial wastewater. This substance is widely used in the textile industry and is found in wastewater during the dyeing process. When this wastewater is discharged into water, it poses a risk to aquatic animals and plants. It can cause health issues such as eye discomfort, nausea, gastritis, central nervous system toxicity, and diarrhoea[1][2]. Traditional wastewater treatment methods include active carbon adsorption, oxidation, biodegradation, precipitation, and air floating. These processes are inefficient, emit secondary pollutants, and do not fully mineralize organic pigments[3].

Recently, there has been an increase in interest in developing nanosized metals and metal oxide particles owing to their distinctive properties, which make them suitable particles for a variety of applications, such as wastewater treatment, anti-microbial activities, and gas sensing. Among metal oxides, ZnO is considered one of the most promising semiconducting materials



because of its low toxicity, biocompatibility, photostability, and electrochemical coupling coefficient. It is a semiconductor with a band gap of approximately 3.36 eV, high exciton binding energy, and ultraviolet absorption. Owing to these features, ZnO has many applications in different fields when its size is reduced to the nanoscale, such as remarkable degradation of organic dyes through photocatalytic activity and demonstrating antimicrobial properties [4].

Physical, chemical, and biological (green) methods were employed to synthesise ZnO nanoparticles. Among these, the green method (plant extracts, bacteria, and fungi) is one of the most interesting methods used to synthesize ZnO nanoparticles, as the extracts are rich in phytochemicals, such as flavonoids, amino acids, tannins, and proteins, which act as reducing and stabilizing agents during the synthesis process. Different plant extracts like *Azadiracta indica*, *Artocarpus gomezianus*, *Aleo Veera*, *Moringa oleifera*, Maple leaf, tulsi, *Phyllan-tjusniruri*, methi [5][6][7] etc were used to synthesis ZnO nanoparticles. In the current study, *Terminalia mantaly* leaf extract was used to synthesize ZnO nanoparticles, which have properties such as being antitumour, antidiabetic, and antihypertensive. These nanoparticles were used to study wastewater treatment.

2. Materials and Methods

2.1 Materials:

Zinc nitrate hexahydrate was purchased from Sigma–Aldrich, and double-distilled water was obtained from the chemistry laboratory throughout the experiment. *Terminalia mantaly* leaves were collected from Anurag University, Venkatapur, Ghatkeser.

2.2 Preparation of the plant extract

First, *Terminalia mantaly* leaves were collected and washed under running water two to three times to remove dust particles. The leaves were dried and cut into small pieces. Next, 30 g of leaves were weighed and added to 200 mL of water in a 500 mL beaker. The mixture was stirred on magnetic stirrer for an hour and then leaf extract was allowed to cool and filtered in conical flask using Whatman filter paper. The resultant plant extract is stored at 4°C for future use.

2.3 Synthesis of zinc oxide nanoparticles

To synthesize ZnO nanoparticles, 3 grams of zinc nitrate is added to 30ml of plant extract in a beaker and heated at 60°C on a magnetic stirrer under constant stirring for 2 hours until a yellowish paste was formed. This paste was collected and washed for two times using ethanol to remove impurities present in the sample. The resulting sample was heated in a furnace at 400 °C for 2 h. The final product was collected in a sample holder and used for further characterization techniques and for the photocatalytic activity tests[2].

The procedure used to synthesize the nanoparticles is illustrated in Figure 1.



Figure 1. Procedure for the synthesis of ZnO nanoparticles

3. Characterizations:

The following characterization techniques were employed for the synthesized nanoparticles. X-ray diffraction (XRD) was used to analyze the phase and crystallite size, scanning electron microscope (SEM) was used to study the morphology of the nanoparticle and Fourier transform infrared spectroscopy (FTIR) was used to study the presence of functional groups within the sample.

4. Results and Discussion:

4.1 Scanning electron microscopy (SEM):

SEM was used to study the morphology of the sample. As shown in Figure 2(a), the SEM image consists of irregularly shaped particles with rough surfaces that are highly agglomerated. The particles have a wide range of sizes in the micrometer range. Strong interparticle attraction forces during synthesis and subsequent drying are often attributed to the high surface energy of small

particles, which causes this form of agglomeration. The absence of various rough grains corresponds to rapid nucleation and growth processes, which may have no influence on the formation of the particles. The current SEM analysis also revealed the presence of non-spherical ZnO nanoparticles. These zinc oxide nanoparticles have sizes ranging from 23 to 216 nm[8].

4.2 Energy Dispersive X-Ray

Figure 2(b) presents the EDS spectrum, revealing the elements present in the sample. The EDS report confirms that the sample consists of ZnO nanoparticles, indicated by the exclusive presence of Zn and O peaks. The absence of any other impurity peaks within the detection range of the instrument indicates that the produced substance has a high level of chemical purity. Surface oxygen adsorption, measurement error, or the intrinsic limitations of EDS in precisely quantifying elements could be the cause of the slight deviation from the ideal atomic ratio. The atomic percentages of oxygen and zinc atoms are given by 47.71% and 52.30 %. The weight percentages of oxygen and zinc atoms are given by 18.25% and 81.75%.

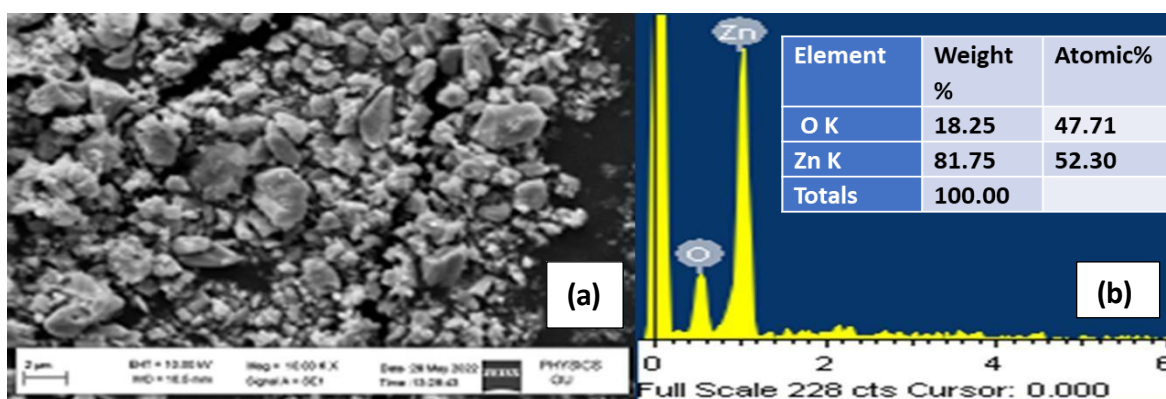


Figure 2. (a) FE-SEM image shows the surface morphology and particle agglomeration of synthesized ZnO nanoparticles. (b) Corresponding EDS spectrum confirming the elemental composition

4.3. X Ray Diffraction:

The X-ray diffraction (XRD) patterns of the synthesized ZnO nanoparticles (NPs) matched those reported in the literature. The XRD patterns of ZnO NPs synthesized using TM leaf extract are shown in Figure 3. Different crystallographic planes are represented by separate diffraction peaks in the XRD pattern of the sample. Interestingly, the (100) plane is connected to the peak at 2θ at an angle of 31.51° . Other peaks at 34.20° , 36.01° , 47.34° , 56.38° , 62.68° , 66.06° , 67.69° , 68.93° , 72.43° , and 76.79° correspond to the (002), (101), (102), (110), (103), (200), (112), (004), and (202) [9] planes, respectively. According to the Scherrer equation, the crystallite size is in the nanometre range, indicating the formation of a nanocrystalline material. The Debye Scherrer equation is used to determine the particle size and it is given by the equation $D = \frac{k\lambda}{\beta \cos\theta}$

[10]

where D is the particle size, K is the Debye–Scherrer constant, λ is the wavelength of X-rays, θ is the diffraction angle, and β is the full width at half maximum. The calculated crystallite sizes ranged from approximately 10 to 24 nm, with an average size of 18.17 nm.

The lattice parameters of the ZnO nanoparticles were calculated using the (100) and (002) diffraction planes. The lattice constants values are calculated using lattice relation

$$\frac{1}{d^2} = \frac{4(h^2 + hl + k^2)}{3a^2} + \frac{l^2}{c^2} \quad [9]$$

and the obtained values are $a = 3.28 \text{ \AA}$ and $c = 5.24 \text{ \AA}$.

The corresponding c/a ratio is 1.60, which is consistent with the characteristic value of hexagonal wurtzite ZnO, confirming the structural stability. The volume of the unit cell was calculated using the formula $0.866a^2c$ and, which was approximately $48.8 \text{ \AA}^3$, further confirming the formation of a stable hexagonal ZnO structure. Additionally, the dislocation density, which provides a measure of crystal defects, was calculated using the inverse square of the crystallite size and was $0.055[11]$.

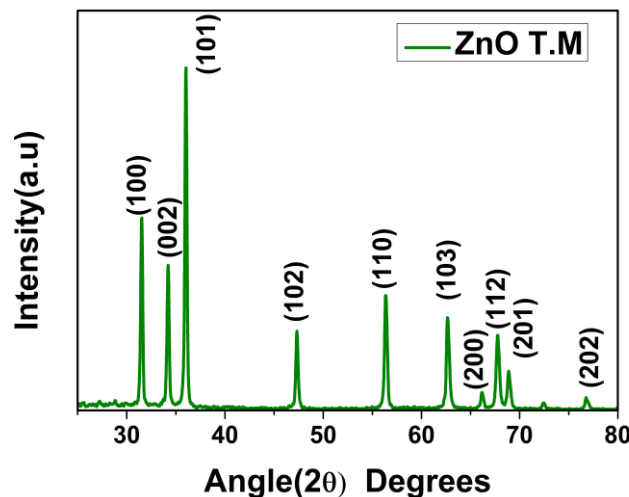


Figure 3: XRD patterns of synthesised ZnO nanoparticles prepared using TM leaf extract.

4.3.1. Williamson–Hall plot

Peak broadening in X-ray diffraction patterns arises from the combined effects of finite crystallite size and lattice strain. The Williamson–Hall (W–H) method was employed to separate these contributions using a uniform deformation model (UDM). The fundamental equation used in W–H method is given by $\beta \cos\theta = \frac{k\lambda}{D} + 4\epsilon \sin\theta$ [12].

To separate the contributions of crystallite size and lattice strain to peak broadening, a Williamson–Hall analysis was performed by plotting $\beta \cos\theta$ versus $4\sin\theta$. The linear nature of the plot confirms uniform lattice deformation in the ZnO nanoparticles, as shown in Figure 4 (a). The crystallite size and micro strain were estimated to be approximately 20.4 nm and 4.1×10^{-3} , respectively, from the intercept and slope of the Williamson–Hall plot. The crystallite size obtained using the Williamson–Hall method is slightly larger than that obtained using the Scherrer equation, indicating that peak broadening is influenced by both size and strain effects.

This observation confirms the nanocrystalline and defect-rich nature of the synthesised ZnO nanoparticles.

4.3.2. Size-strain plot (SSP)

The size-strain plot (SSP) method is an advanced X-ray diffraction (XRD) line broadening analysis that assumes that strain broadening follows a Gaussian distribution, whereas size broadening follows a Lorentzian distribution. Compared with the Williamson–Hall method, the SSP method provides more weight to the low-angle diffraction peaks, thereby improving the accuracy of crystallite size estimation.

The SSP relationship is expressed as follows:

$$(d_{hkl} \beta \cos \theta)^2 = \frac{k\lambda}{D} (d_{hkl}^2 \beta \cos \theta) + \left(\frac{\varepsilon}{2}\right)^2$$

[12] Where β is the FWHM in radians, θ is the Bragg angle in radians, d_{hkl} is the interplanar spacing, k is the shape factor (0.9), and λ is the wavelength (0.15406 nm).

The SSP plot of $(d_{hkl} \beta \cos \theta)^2$ by taking on X axis and $d_{hkl}^2 \beta \cos \theta$ on Y axis as shown in figure 4(b). From the slope and intercept of the fitted line, the crystallite size and micro strain were estimated to be 21.6 nm and 3.9×10^{-3} , respectively. The slightly larger crystallite size obtained from the SSP method compared to the Scherrer and Williamson–Hall methods confirm the reliability of the SSP approach for nanocrystalline ZnO.

Table 1 compares the particle size and strain.

Table1:

Method	Crystallite Size (nm)	Micro strain
Scherrer	18.17	—
Williamson–Hall	20.4	4.1×10^{-3}
Size–Strain Plot	21.6	3.9×10^{-3}

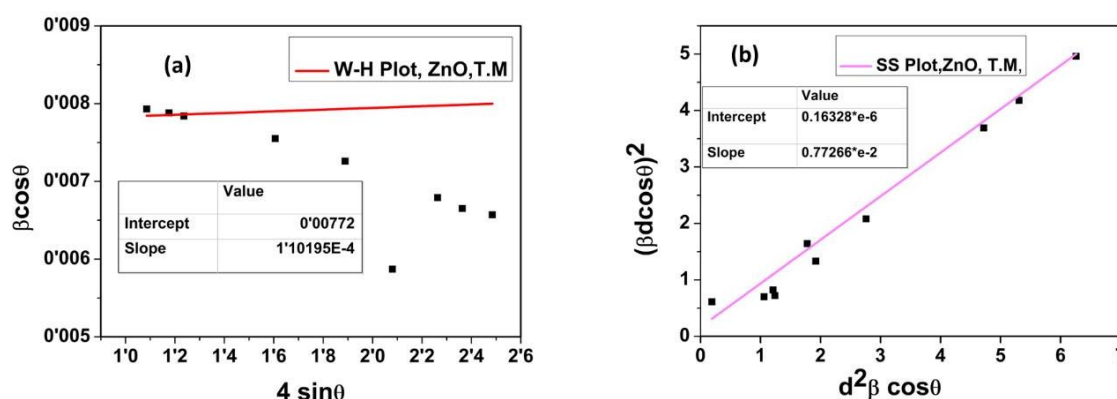


Figure 4: (a) W-H plot of synthesized nanoparticles using TM leaf extract. (b) Size-strain plot of ZnO nanoparticles synthesized using TM leaf extract.

4.4 Fourier transform infrared spectroscopy (FTIR):

Fourier-transform infrared (FTIR) spectroscopy was used to identify the functional groups in the sample. The FTIR spectrum of the synthesized ZnO nanoparticles was captured in the range of $173\text{--}4075\text{ cm}^{-1}$, and is depicted in Figure 5. The observed peaks were attributed to various bond modes by comparison with the existing literature.

The peaks formed in between $400\text{--}600\text{ cm}^{-1}$ indicate the presence of metal oxides. In this FTIR spectrum, the peaks formed at 470 and 436 cm^{-1} [11] confirm the presence of ZnO nanoparticles. The FTIR spectrum shows several characteristic peaks corresponding to different functional groups present in the sample. The sharp absorption peak observed at 3614 cm^{-1} is attributed to free O–H stretching vibrations, indicating the presence of alcohol and phenolic groups [13]. The peaks at 2889 and 2426 cm^{-1} correspond to aliphatic C–H stretching vibrations, indicating the presence of alkane groups. The peak observed at 1384 cm^{-1} corresponds to symmetric N–O stretching, indicating the presence of nitro compounds.

Furthermore, the absorption bands at 1044 cm^{-1} and 1112 cm^{-1} are attributed to the C–N stretching vibrations of aliphatic amines. The peak observed at 826 cm^{-1} is assigned to the C–Cl stretching vibration, confirming the presence of alkyl halides. The low-frequency band at 436 cm^{-1} corresponds to the C–halogen stretching vibration, indicating the presence of halogen-containing compounds in the sample.

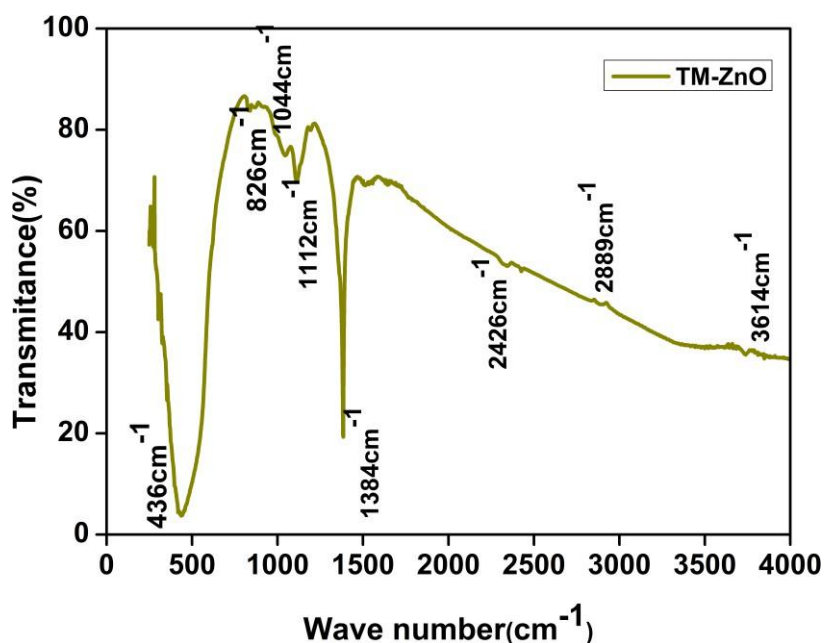


Figure 5 FTIR spectrum of ZnO nanoparticles.

5. Photocatalytic activity

Figure 6 shows the temporal UV–Vis absorption spectra of methylene blue in the presence of a ZnO photocatalyst under light irradiation. The characteristic absorption band of MB at

approximately 550–560 nm gradually decreases with increasing irradiation time, indicating effective photocatalytic degradation.

In this absorption study, 0.05 g of synthesized ZnO nanoparticles was added to 50 mL of a methylene blue solution with a concentration of 50 ppm. This mixture was stirred under a UV-Vis light source, with samples collected every 30 min. Prior to activating the UV light, the solution was stirred in the dark for 1 h to reach the adsorption–desorption equilibrium. The activation of the UV light was considered to be at $T=0$ min. Every 30 min, a 4–5 mL sample of the suspension was collected and immediately centrifuged to separate the catalyst. The resulting clear solution was then analysed using UV-Vis spectroscopy, which indicated a reduction in the characteristic absorption peak of methylene blue at $\lambda_{\text{max}} = 560$ nm. The degradation percentage is calculated using given equation, $\text{Degradation (\%)} = \frac{A_0 - A_t}{A_0} \times 100$ where A_0 is the initial concentration and A_t final concentration.[14]

The degradation efficiency reached approximately 99% after the final irradiation period, demonstrating the excellent photocatalytic activity of the ZnO nanoparticles. The degradation kinetics followed a pseudo-first-order reaction model, as confirmed by the linear relationship between $\ln(A_0/A_t)$ and irradiation time [15]. The calculated rate constant was $k \approx 0.032 \text{ min}^{-1}$, suggesting rapid charge carrier generation and efficient reactive species formation during the photocatalytic process.

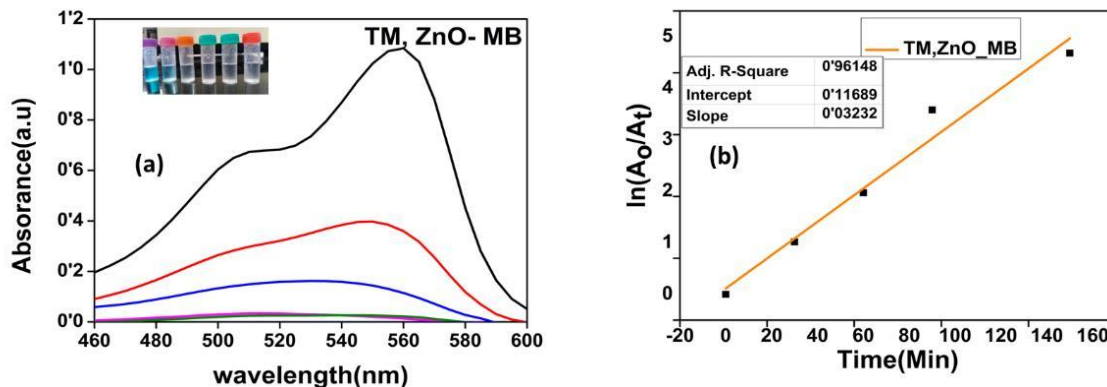


Figure 6: (a) Degradation of MB of synthesized nanoparticles using TM leaf extract. (b) Kinetics of MB dye degradation, which is a first-order linear fit of synthesized ZnO nanoparticles using TM leaf extract.

5.1 Mechanism:

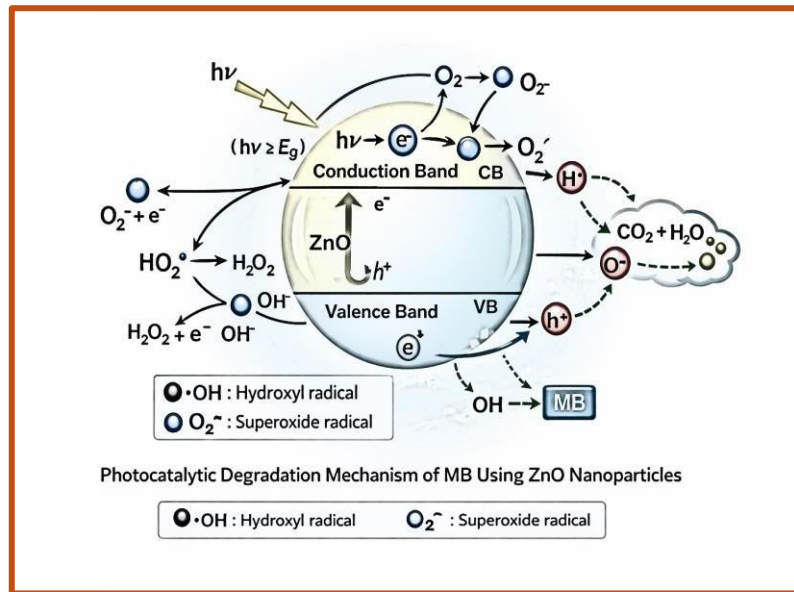
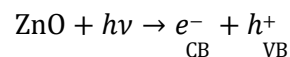
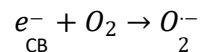


Figure 7. Possible mechanism for the photocatalytic degradation of methylene blue on ZnO nanoparticles under light irradiation

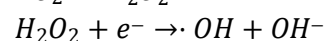
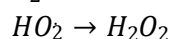
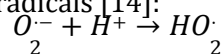
Figure 7 illustrates the photocatalytic degradation mechanism of methylene blue (MB) using ZnO nanoparticles under light irradiation. When ZnO is exposed to photons with energy equal to or greater than its band gap ($h\nu \geq E_g$), electrons in the valence band (VB) are excited to the conduction band (CB), generating electron-hole pairs as expressed by:



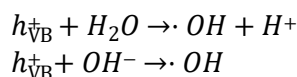
The photogenerated electrons in the conduction band migrate to the surface of the ZnO nanoparticles and react with dissolved molecular oxygen (O_2) to form superoxide radical anions (O_2^-):



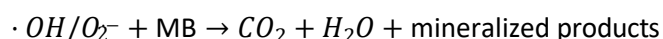
These superoxide radicals undergo further protonation and reduction reactions, producing hydroperoxyl radicals (HO_2) and hydrogen peroxide (H_2O_2), which subsequently decompose to generate highly reactive hydroxyl radicals [14]:



Simultaneously, photogenerated holes in the valence band act as strong oxidizing agents and react with surface-adsorbed water molecules or hydroxide ions to directly produce hydroxyl radicals.



The generated reactive oxygen species, particularly hydroxyl radicals and superoxide radicals, are highly oxidative and non-selective. These species attack the chromophoric structure of methylene blue molecules adsorbed on the ZnO surface, leading to the cleavage of aromatic rings, de-methylation, and successive oxidation reactions. Ultimately, MB is mineralized into environmentally benign end products such as carbon dioxide (CO₂), water (H₂O), and inorganic ions:



Efficient charge carrier separation and continuous generation of reactive radicals significantly suppress electron-hole recombination, thereby enhancing the photocatalytic degradation efficiency of methylene blue.

6. Conclusions:

Terminalia mantaly leaf extract was successfully used in a green technique to produce ZnO nanoparticles, evidence of the plant's effectiveness as a biogenic reducing and stabilizing agent. Structural analysis revealed that the synthesized ZnO nanoparticles exhibited a stable hexagonal wurtzite phase with nanoscale crystallite size and defect-rich properties. Owing to their effective charge carrier generation and reactive oxygen species generation, the resulting ZnO nanoparticles demonstrated good photocatalytic degradation of methylene blue dye. This study demonstrates an innovative application of Terminalia mantaly in the synthesis of ZnO nanoparticles and encourages further research into biogenic methods for the synthesis of ecologically safe nanomaterials with potential applications in wastewater treatment and catalysis.

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