

RESEARCH PAPER

ZnO Nanoparticles for Photocatalytic Detoxification of Methyl Violet-Contaminated Water: Optimization and Environmental Health Perspectives

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ARTICLE INFO

Article History:

Received 19 May 2026

Accepted 05 September 2026

Published 01 October 2026

Keywords:

Health Perspectives

Methyl Violet

Nanoparticle

Photocatalytic Detoxification

ZnO

ABSTRACT

The present study explored the photocatalytic degradation of the basic dye methyl violet (MV) from aqueous solution using an efficient, porous zinc oxide (ZnO) nanoparticle catalyst via an advanced oxidation process (AOP). The hydrothermally prepared zinc oxide nanoparticles were characterized for their structural and morphological properties by using scanning electron microscopy (SEM), transmission electron microscopy (TEM), Fourier transform infrared spectroscopy (FTIR), and X-ray diffraction (XRD). This analysis showed a clear indication for the formation of nanoparticles with high surface area, morphology, and chemical pureness, which are favorable to photocatalytic activity. Photolysis experiments were performed under controlled conditions to assess the effects of zinc oxide weight, dye concentration, and light intensity. For the determination of the best weight of zinc oxide, the values were set in the range of between 0.05 and 0.3 g, with a constant time of irradiation of 90 minutes, as well, from the experiments, it appears that for irradiation time of 90 minutes and using 0.20 g of zinc oxide (ZnO) the dye concentrations were from 50 to 150 mg/L. All suspensions were neutralized in the dark for 10 min prior to light exposure to investigate adsorption. A spectrophotometer measured the residual dye concentrations in the supernatant at the maximum absorption wavelength ($\lambda_{\text{max}} = 570 \text{ nm}$). The photocatalytic efficiency was markedly reduced (similar to that at 0.1 g) at 0.05 g, a weight that provides insufficient adsorption sites, whereas increasing the zinc oxide weight increases the active sites.

How to cite this article

Abdul-Reda Hussein U., Sabeeh Jabur H., Falah Hassan F., et al. ZnO Nanoparticles for Photocatalytic Detoxification of Methyl Violet-Contaminated Water: Optimization and Environmental Health Perspectives. J Nanostruct, 2026; 16(4):5320-5331. DOI: 10.22052/JNS.2026.04.068

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INTRODUCTION

Dyestuff, textile, paper, leather, and pharmaceutical industries are well known as major sources of pollutant discharges, which cause environmental pollution. Such polluted waters – usually from dyeing – contain an excessive amount of chemicals, exhibit high color strength, chemical stability and low biodegradability. Releasing these wastewater effluents into natural water sources carries significant risks to public health and environment. Such pollutants block the path for the light entering into the water body and obstruct photosynthesis and the aquatic ecosystem, in addition, most of these dyes and their degradation products are recognized, carcinogen, toxic, and mutagenic [1-3].

Synthetic dyes are chemically classified into several major categories which include reactive dyes, cationic dyes, anionic dyes, azo and triphenylmethane dyes, which are toxic and hazardous. The dyes, which are resistant to conventional chemical and biological degradation, possess stable aromatic structures. Hence, preliminary treatments to treat such pollutants using methods of flocculation, coagulation, biological processes, membrane filtration and adsorption have high operational costs. Driven by the need for better alternative options for dye treatment, advanced oxidation process (AOPs) based on the generation of very strong oxidants that transform very persistent toxic organic pollutants into innocuous end products like carbon dioxide and water have now been well established

as highly effective options for the treatment of dye wastewater [4-6]. Amongst AOPs, heterogeneous photocatalysis utilising semiconductor materials has attracted considerable attention due to its simplicity, sustainability, and high degradation efficiency. Several photo-catalytic surfaces and nano-materials have been investigated, including ZnO, TiO₂, Fe₂O₃, WO₃, and composite metal oxides [7-9]. Among the most important materials is zinc oxide (ZnO) nanoparticles, which are considered among the most promising photocatalysts due to their high oxidation capacity, high photosensitivity, high stability, non-toxicity, low cost, and ease of preparation. Zinc oxide also possesses a large energy band gap (3.2 eV), a large surface area, and high electron mobility, which facilitate efficient charge separation and enhance photocatalysis [10-12]. The photocatalysis of zinc oxide (ZnO) begins when it is illuminated with radiation having energy greater than or equal to the band gap. As a result, it produces e^-/h^+ pairs. These generated charges commonly interact with molecular O₂ and H₂O, yielding highly reactive species (e.g., superoxide radical species ($\bullet O_2^-$) and hydroxyl radical species ($\bullet OH$)), which degrade harmful dyes [13-17].

Methyl violet (MV) is a toxic cationic dye widely used in dyeing, textiles, printing, and other industrial applications. Methyl violet is comparatively stable in the environment owing to its stability with regard to biological degradation. But it can be a deadly poison to reproductive life. Removing it from wastewater is one of the most important environmental demands because it can

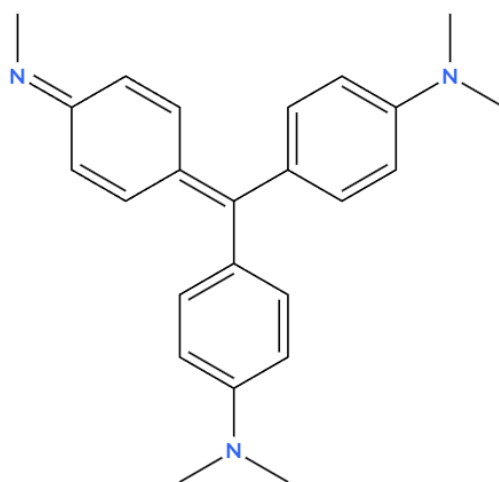


Fig. 1. Chemical structure of Methyl Violet (MV) dye.

be a potential mutagen and carcinogen. Methyl violet is a triphenylmethane derivative having a molecular formula of $C_{24}H_{28}N_3Cl$. Structurally, three fused aromatic rings attached to the carbon centre make it highly colour-stable and highly absorbent in the light, with a λ_{max} at 570 nm. Fig. 1 illustrates the molecular structure of methyl violet. Due to its use across various industrial sectors, the treatment of coloured toxic pollutants has become a pressing challenge. The current study aims to evaluate the photocatalytic efficiency of a zinc oxide nanocatalyst for methylphenol decomposition under optimal operating conditions [18-20]. The dependence of the photocatalytic process on various kinetic factors such as zinc oxide nanoparticles weight (0.05–0.30 g) and the initial dye concentration was investigated for light penetration and degradation kinetics. This study demonstrates how the use of zinc oxide nanoparticle catalysis with identification of optimal adsorption and photo-oxidation conditions, can be an environmentally benign agent for the green and sustainable conversion of toxic pollutants.

MATERIALS AND METHODS

Methyl violet (MV), a basic triphenylmethane dye, is used to remove toxic contaminants. This basic dye is characterized by its water solubility and chemical stability, making it suitable for use in catalytic photolysis. Methyl violet was obtained

from Fluka (purity > 98.0%). Standard solutions (1000 mg/L) of the MV dye were prepared by dissolving 1g of the MV dye powder in 1000 mL distilled water. To adjust the pH, a base and an acid were prepared using sodium hydroxide and hydrochloric acid (purchased from Fluka) to achieve the required pH for the experiment. The organic solvents, methanol and ethanol (purity $\geq$ 99%), were purchased from Merck and used as is for analysis and cleaning. All chemicals used in this research were used without any further purification and are of high purity.

Preparation of ZnO Nanoparticles

ZnO nanoparticles were prepared using a hydrothermal treatment method. Oxalic acid dihydrate ($H_2C_2O_4 \cdot 2H_2O$) (the precipitating/complexing agent) and zinc acetate dihydrate ($Zn(CH_3COO)_2 \cdot 2H_2O$) (the source material). 10 gm of zinc acetate dihydrate and 5 gm of oxalic acid dihydrate. The resultant mixture was dissolved in 100 mL of distilled water and the mixture magnetically stirred about 30 min at room temperature until completely dissolved forming a clear, homogenous solution. The final solution was transferred into a stainless steel autoclave with a Teflon liner and was subjected to hydrothermal synthesis at 160 °C for 24 h in order to initiate crystallization and control nanocrystal growth. Upon cooling to room temperature, a white precipitate appeared in the solution. The

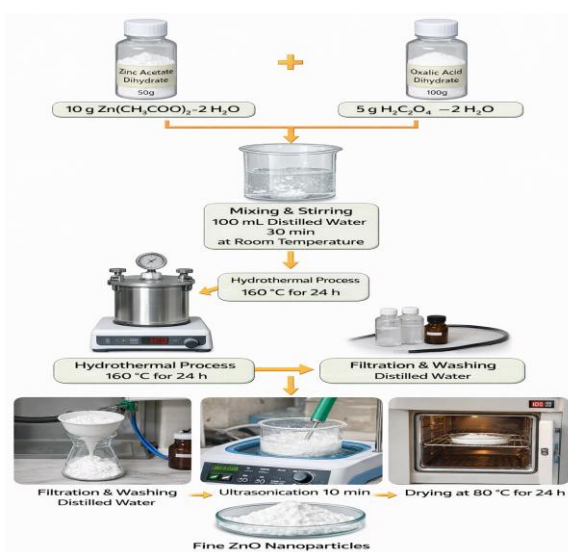


Fig. 2. Preparation of ZnO Nanoparticles.

obtained precipitate was centrifuged and washed extensively with distilled water for purification to remove any unreacted material and excess ions. Ultrasonic dispersion (10 min) for improved dispersion between the particles and reducing the clusters was then performed on the suspension. The final step in producing the highly porous crystalline zinc oxide nanopowder was drying the sample in an oven at 80 °C for 24 h. (Fig. 2).

Photocatalytic Degradation

In vitro photocatalytic reactor based on UVA light, methyl violet dye photocatalytic performance of different zinc oxide nanoparticles (ZnO NPs). To study the influence of dye concentration on photocatalytic efficiency, a series of dye solutions was prepared with concentrations ranging from 50 to 150 mg/L. A volume of 200 mL of dye solution was placed into the reactor for each experiment,

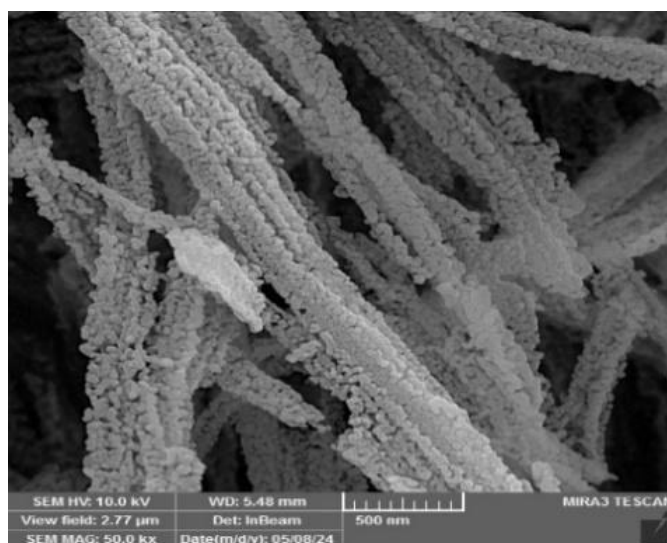


Fig. 3. FE-SEM micrographs of ZnO NPs recorded at different magnifications.

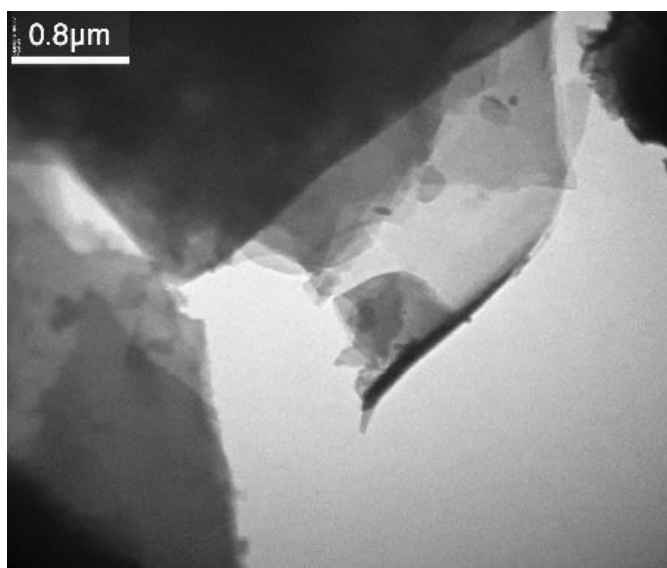


Fig. 4. TEM micrographs of ZnO NPs recorded at different magnifications at 0.8 μm.

and catalytic amounts of zinc oxide nanoparticles of 0.05–0.3 g were utilized. Adsorption equilibrium was established by stirring the solution at 300 rpm for 10 min in the dark. At around 10 minutes, an initial sample was extracted and designated $t=0$. Subsequently, UVA light irradiation with a 300 W xenon lamp (with a $\lambda > 350\text{--}370\text{ nm}$) was employed to initiate the photocatalytic decomposition process without ultraviolet radiation. The distance of the light source from the reactor was kept at 50 cm. The influence of radiation intensity on catalyst activity was examined by varying the energy density of the incident radiation. Over 90 minutes, 5 mL samples were collected under irradiation. Centrifugation at 2500 rpm for 10 minutes was employed to pellet the catalyst molecules from these samples. The clear liquid layer formed was subjected to UV-Vis analysis to quantify the residual dye concentration. Using the following Equation (1), the decomposition efficiency (%) was calculated.

$$\text{Degredation effectincy \%} = \frac{C_0 - C_t}{C_0} \times 100 \quad (1)$$

RESULTS DISCUSSION

Characterization of ZnO nanoparticles

FE-SEM images (Fig. 3) show ZnO nanoparticles with a nanostructured appearance. The images reveal that the particles are crystalline in shape

and size, uniformly distributed across the surface, with agglomeration. The microscopic images also show that the zinc oxide nanoparticles are distributed in a porous, interconnected, lattice-like or layered structure. This helps to improve the structural properties.

In Fig. 4, three-dimensional units and shapes for structural composition are shown, and from the transmission electron microscopy (TEM) images, it can be seen that the ZnO surface is a homogenous particle with an irregular three-dimensional shape. The amalgamation and homogeneous distribution of zinc oxide nanoparticles resulted in a rough, highly porous surface. The formed porous structure increases the specific surface area and the number of active sites available for photocatalytic reactions, respectively, the surface area was found to be 32 g.m^{-2} and this is an essential feature of this system to enhance adsorption and photocatalytic performance. In general, FE-SEM and TEM results suggest the synthesized zinc oxide nanoparticles show uniform distribution, large surface area and porosity beneficial for photocatalytic application [21–23].

Fig. 5 shows the XRD pattern of the zinc oxide nanoparticles, indicating high crystallinity with narrow, well-defined diffraction peaks over a wide $2\theta^\circ$ range ($26\text{--}76^\circ$). The peak locations at $\theta=31.7^\circ$, $\theta=34.4^\circ$, and $\theta=36.2^\circ$ correspond to the (100), (002), and (101) crystal planes of the hexagonal

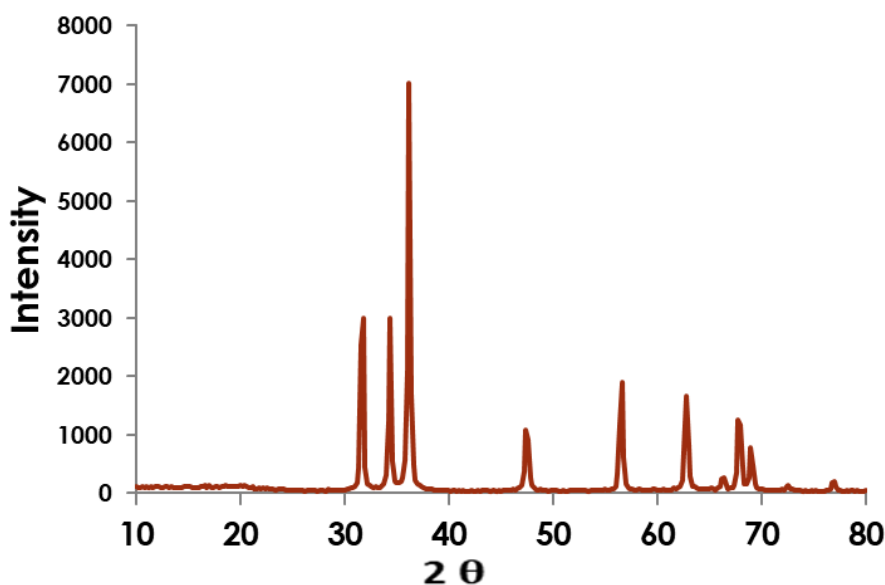


Fig. 5. X-ray diffraction of ZnO nanoparticles.

Wurtzite-type structure associated with zinc oxide, which testify that crystalline ZnO nanomaterial was successfully prepared. It is clear from the absence of other peaks that the prepared material is pure, with no impurities or secondary phases (e.g., mixed oxides), which reflects the effectiveness of the hydrothermal nanomaterial preparation method. The intensity of the peaks also reflects the material's crystallinity, while the relative peak amplitudes suggest that the nanocrystals are small, which correlates with a high specific surface area and more active sites [24, 25].

The Fourier transform infrared (FTIR) spectra of ZnO nanoparticles before and after dye photocatalytic provide information on surface functional groups and the chemical changes during the photolytic reaction (Fig. 6). A strong absorption band in the 3200–3500 cm^{-1} region is assigned to O–H stretching vibrations of surface hydroxyl groups and physically adsorbed water, which are responsible for moisture detection and active sites for hydrogen bonding. Weak bands seen at 2850–2950 cm^{-1} are due to aliphatic C–H stretching vibrations and may be contributed from some remnant organic species. New peaks around 1600–1650 cm^{-1} were assigned to H–O–H bending vibrations or carbonyl/ imine ($\text{C}=\text{O}/\text{C}=\text{N}$) group and “bands about 1380–1450 cm^{-1} and

1000–1200 cm^{-1} ” as ascribed to C–N, C–C, and C–O/C–O–C, confirming the adsorption of MV dye molecule or its degradation intermediates on the surface of ZnO after treatment confirmatively [26, 27]. Confirming the formation and integrity of the crystalline ZnO, the characteristic, sharp absorption band within the range of 400–600 cm^{-1} corresponds to the Zn–O lattice vibrations. This band intensity and organic signals derived from photolysis suggest a surface interaction between ZnO NPs and the dye or its photolysis products. In general, the FTIR results indicate that there are both passive hydroxyl groups and stable Zn–O bonds available, which would enable easy adsorption and promote enhanced photocatalytic activity by providing reactive surface sites for pollutant degradation [28, 29].

Effect of Initial Dye Concentration

The influence of initial MV dye concentration on photocatalytic degradation behavior of ZnO nanoparticles was systematically investigated under identical experimental conditions. Other variables, i.e., catalyst dosage, solution volume, light intensity and irradiation time, were constant while dye concentration was varied in the range of (50 to 150) mg L^{-1} . The degradation efficiencies associated with them are shown in Fig. 7.

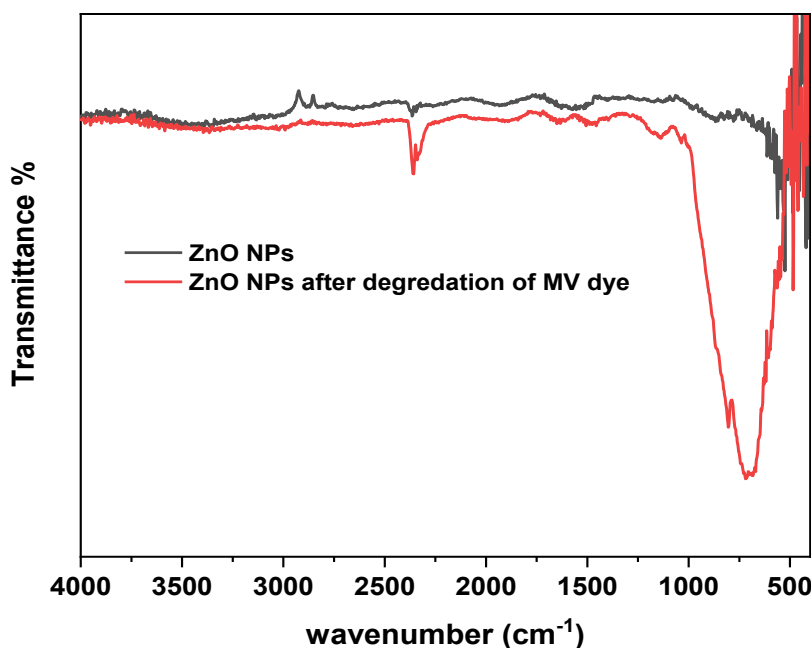


Fig. 6. FTIR spectra of zinc oxide nanoparticles before and after the MV dye photolysis.

The findings highlighted that the initial actual contaminant concentration is influential in efficient photocatalytic mineralization. At dye concentration below the lowest (100 mg L⁻¹) the best removal efficiency was observed with the fastest degradation occurring within the shortest irradiation time. In small amounts, the solution appears clear, meaning that photons within the liquid will penetrate further into the solution and be more effectively absorbed on the ZnO catalyst surface. As a result of this, the formation of electron-hole pairs are increased, which helps in forming reactive oxygen species (ROS) such as hydroxyl radical ($\bullet\text{OH}$) and superoxide radical anion ($\text{O}_2\bullet^-$) [5], leading predominantly to the oxidative degradation of dye molecules [30]. However, both degradation efficiency and reaction rate rose slightly before declining as the initial dye concentration increased. I did have a few reasons for this behavior though. Optical density of the dye-containing solutions increases with higher dye concentration, allowing more light to be attenuated, while few photons penetrate to the catalyst surface membrane (the inner filter effect) [31, 32].

Moreover, dye molecules at a high concentration tend to compete for active adsorption sites on the surface of ZnO, which reduces the amount of catalytic active sites to generate reactive species.

Third, both intermediate products generated during degradation may remain and block surface vacant sites, leading to low photocatalytic activity. Thus, while the absolute amount of dye removed increases with increased concentration, the percentage removal efficiency declines at higher initial concentrations. These results demonstrate that significantly lower pollutant concentrations are advantageous for high photocatalytic efficiency, owing to effective light harvesting and surface reaction kinetics. A maximum photocatalytic degradation efficiency of methyl violet (MV) dye (91.45%) was obtained at an initial concentration of 100 mg L⁻¹. depicts significant deviation of PDE% in photocatalytic degradation (PDE) by varying the MV dye concentration, demonstrating that the degradation performance always depends on the pollutant loading [33, 34].

Effect of ZnO nanoparticle on Photocatalytic Degradation of MV Dye

The effect of nanoparticle weight on the removal of the toxic dye methyl violet (MV) was systematically investigated under optimal laboratory conditions, including pH, temperature, dye decomposition time, dye concentration of 100 mg/L, and airflow rate of 10 mL/min. The nanoparticle weight was varied while all optimal conditions were held constant. Fig. 8 illustrates

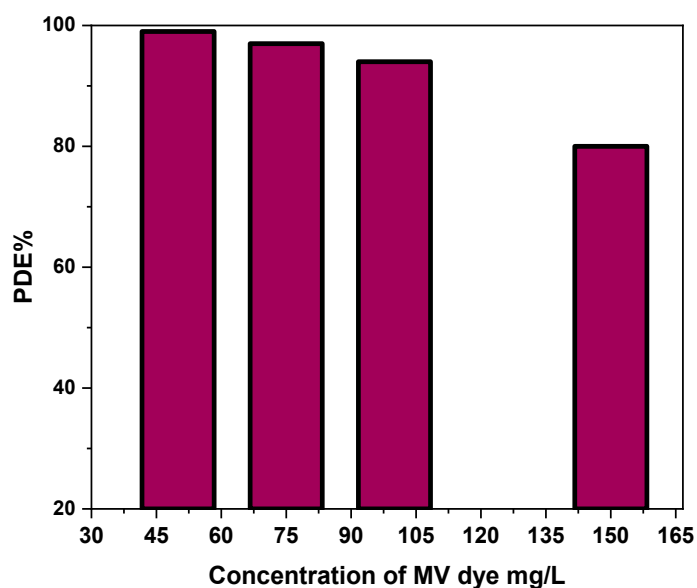


Fig. 7. Effect of Initial Dye Concentration on efficiency of photo-catalytic degradation using pH 7, 0.2g/200 mL ZnO NPs.

the dye decomposition efficiencies. The results indicate that increasing the nanoparticle weight initially improves photodegradation efficiency, reaching a maximum at 0.2 g per 200 mL. This improvement is attributed to an increase in the number of active surface sites, thereby enhancing photon and oxygen absorption and facilitating

electron-hole pair generation. Consequently, the interaction between the dye molecules and the surface becomes more efficient, thus promoting and accelerating the photodegradation process [35, 36].

In some studies, increasing the amount of catalyst did not improve photocatalytic

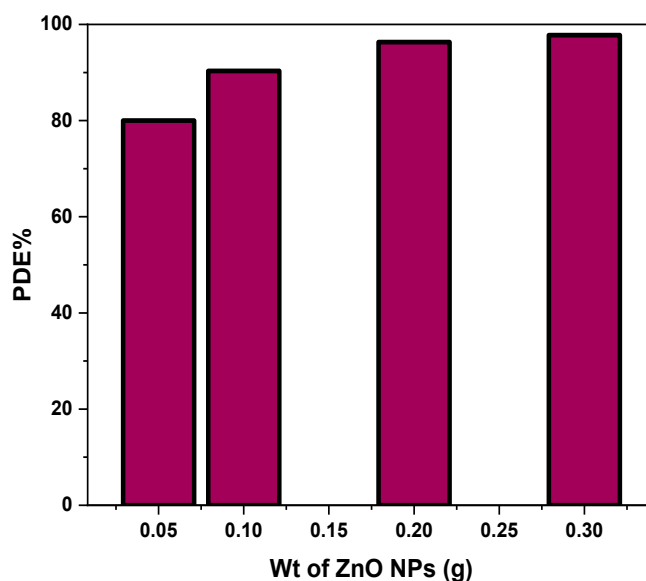


Fig. 8. Effect of the weight of ZnO NPs on the efficiency of photo-catalytic degradation using pH 7, 100 mg/L of MV dye, 0.2g/200 mL ZnO NPs.

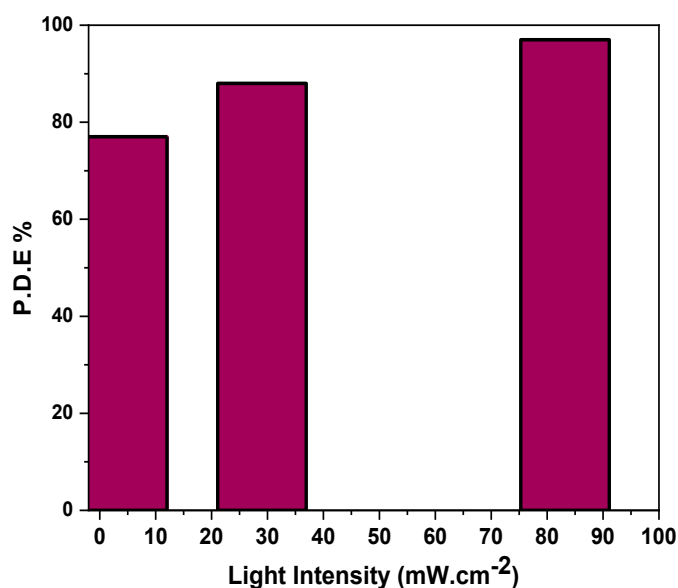


Fig. 9. Effect of Light Intensity onto efficiency of photo-catalytic degradation using pH 7, 100 mg/L of MV dye, 0.2g/200 mL ZnO NPs.

decomposition efficiency; rather, it decreased it. This behavior is attributed to the blocking effect resulting from the increased catalyst particles in the solution, as well as light scattering. Turbidity in the solution also limits light penetration and reduces photocatalytic efficiency, as photons do not reach the catalyst's inner layers. Conversely, when the amount of nanomaterial is low, a significant decrease in decomposition efficiency occurs because the number of surface-active sites is limited, thereby reducing the generation of reactive species. Therefore, an optimal nanomaterial weight of 0.2 g/200 mL was chosen to achieve maximum photocatalytic decomposition of MV dye [37, 38].

Effect of Light Intensity

The influence of light intensity on the photocatalytic degradation of methyl violet (MV) using the ZnO nanocomposite was systematically investigated by varying the irradiation intensity from 83.1, 29, and 4.1 mW cm⁻² while maintaining all other operational parameters constant, including an initial dye concentration of 100 mg L⁻¹, a catalyst loading of 0.2 g per 200 mL, an air flow rate of 10 mL min⁻¹, and ambient temperature. The corresponding degradation efficiencies are

presented in Fig. 9.

The results reveal a direct positive correlation between light intensity and photocatalytic degradation performance. As the light intensity increased, the degradation rate improved significantly. This enhancement is primarily due to the increased photon flux reaching the catalyst surface, which increases electron excitation from the valence band (VB) to the conduction band (CB) of ZnO. Consequently, a higher number of electron-hole pairs are generated, leading to the formation of more reactive oxygen species (ROS), such as hydroxyl radicals ($\cdot\text{OH}$) and superoxide radicals ($\text{O}_2\cdot^-$). These highly oxidative species accelerate the breakdown and mineralization of dye molecules [39]. At the highest tested intensity (83.1 mW cm⁻²), the system exhibited the highest photocatalytic activity, achieving a degradation efficiency of 94.89%. Overall, increasing light intensity enhances photocatalytic efficiency by promoting charge carrier generation and improving surface redox reactions, thereby facilitating faster pollutant degradation.

Proposed Mechanism of Photocatalytic Degradation

Based on the semiconductor properties of

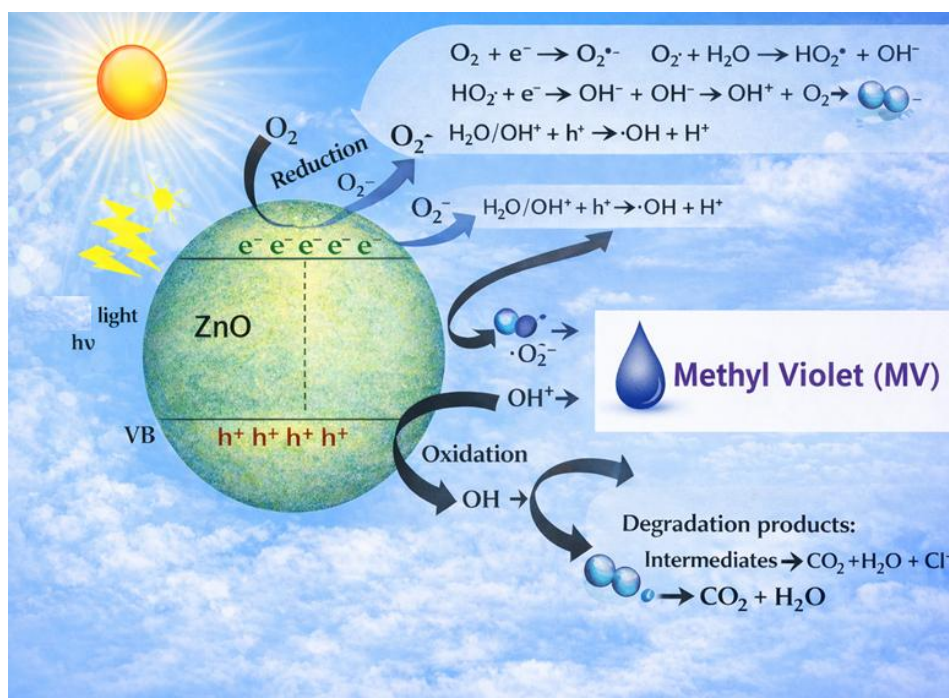


Fig. 10. Suggestion Mechanism of Photocatalytic Degradation of MV dye onto ZnO NPs.

ZnO and the respective photoinduced charge-transfer processes occurred at catalyst surface, photocatalytic degradation of MV dye over ZnO nanoparticles are proposed. ZnO is a wide band-gap semiconductor ($E_g \sim 3.2$ eV) and, upon irradiation with photons of energies above the band-gap, electron is excited from the valence band (VB) to the conduction band (CB) resulting in the formation of electron-hole pairs. These charge carriers migrate to the surface and then drive redox reactions with adsorbed species [40, 41].

In this process, conduction band electrons react with dissolved molecular oxygen to form superoxide radicals ($O_2^{\bullet-}$), whilst valence band holes oxidize hydroxyl groups or water molecules on the surface, yielding highly reactive hydroxyl radicals ($\bullet OH$) [36]. These reactive species are undoubtedly the main oxidizing species that attack and eventually decompose adsorbed MV molecules on the ZnO surface. The adsorption of MV increases interfacial contact, facilitating rapid carrier transfer and increasing the likelihood of oxidation. MV molecules might also behave as photosensitizers under UVA irradiation, in which case the excited dye molecules provide electrons into the CB of ZnO, thereby promoting ROS generation and accelerating degradation.[42]. The chromophoric structure of the dye is cleaved which is followed by progressive breakdown to smaller intermediates and ultimately mineralisation to CO_2 and H_2O via the synergistic effects of photon absorption, efficient charge separation, surface adsorption of the substrate and subsequent radical-mediated oxidation overall the photocatalytic process is translationally controlled by the generation of a photocharge, surface electron transfer and the radical reactions that constitute a photocatalytic-driven pathway that highlights the indispensability of ZnO nanoparticles to photo-assisted effective degradation of organic dye pollutants [43, 44]. The suggestion mechanism shown in Fig. 10.

CONCLUSION

This work describes the synthesis of a low-cost, eco-friendly and hydrothermally synthesised zinc oxide (ZnO) nanoparticles used as a novel, inexpensive and eco-friendly nanomaterial under UV light to efficiently photocatalyze the degradation of toxic organic pollutants such as methyl violet dye. Characterization of the photocatalyst. FTIR, SEM, TEM, and XRD were performed to characterise

the morphological, structural, and compositional properties of the photocatalyst. The ceramic purity, surface area, porosity, and crystallinity of the nanomaterial were characterised using the techniques described above. We also showed that 90 min of irradiation was enough to photolyze the methyl violet dye effectively using photocatalytic experiments. Such good performance is attributed to the porosity of the prepared surface and its higher light-absorption efficiency, which promotes the generation of more reactive oxygen species to oxidize the contaminants. The degradation behaviour was significantly affected by the operating parameters, with an increase in the dye concentration resulting in decreased photolysis performance due to light-penetration limitations, whereas at lower concentrations the dye degradation process was favoured by improved light penetration. The efficiency of degradation increased with the nanomaterial's weight due to an increase in active surface sites, whereas high light intensity enhanced dye degradation by increasing the photon flux and generating more electron-hole pairs. Overall, the results demonstrate that the hydrothermally prepared nanomaterial yields an inexpensive, readily available, and highly effective treatment for hazardous toxic organic pollutants in aquatic systems.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

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