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MORALITY BEFORE KNOWLEDGE

Utilization of water hyacinth as a reducing agent in microwave-assisted synthesis of zinc oxide nanoparticles for photocatalytic applications

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ABSTRACT

Background: The textile industry is the second largest contributor to industrial pollution worldwide, accounting for about 10% of carbon emissions and 20% of wastewater, mainly from textile dyeing. One of the most widely used dyes is methylene blue, a thiazine-based cationic aromatic dye that threatens aquatic ecosystems and human health. Conventional wastewater treatments are less effective in Indonesia due to high operational costs. Photodegradation using semiconductor photocatalysts offers a simple and affordable alternative. Zinc oxide nanoparticles provide advantages over titanium dioxide, including lower cost and excellent electrical, mechanical, and optical properties. This study aims to synthesize zinc oxide nanoparticles using water hyacinth leaf extract as a natural reducing agent, combined with microwave irradiation to enhance eco-friendly synthesis. **Methods**: The extract was applied at concentrations of 4%, 8%, and 12%. Characterization using X-ray diffraction, Fourier-transform infrared spectroscopy, and scanning electron microscopy confirmed the formation of zinc oxide nanoparticles. **Findings**: The nanoparticles displayed average particle sizes of 134.42 nm, 120.54 nm, and 102.64 nm, respectively. Photodegradation performance analyzed by ultraviolet–visible spectrophotometry showed that methylene blue, with maximum absorbance at 664 nm, exhibited significant absorbance reduction after exposure to the nanoparticles, confirming their photocatalytic activity. **Conclusion**: Water hyacinth-assisted microwave synthesis produces zinc oxide nanoparticles efficiently, offering a low-cost and environmentally friendly solution with strong potential for textile dye wastewater treatment. **Novelty/Originality**: The originality of this research lies in integrating water hyacinth extract and microwave irradiation, which accelerates nanoparticle synthesis while promoting sustainability, making it particularly suitable for application in developing countries.

KEYWORDS: methylene blue; photocatalysis; titanium dioxide; zinc oxide.

1. Introduction

The textile industry is recognized as the second-largest contributor to global industrial pollution, accounting for approximately 10% of carbon emissions and nearly 20% of

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worldwide industrial wastewater discharge, particularly from textile dyeing processes (Rahman et al., 2022). The major source of this pollution originates from synthetic dyes, which are extensively used to impart color and durability to textile products. Among these dyes, methylene blue (MB) is one of the most widely applied due to its high chemical stability, strong coloration efficiency, and relatively low production cost (Badriyah & Putri, 2017).

Methylene blue is a cationic aromatic heterocyclic dye with the molecular formula $C_{16}H_{18}ClN_3S$ and a maximum absorption wavelength (λ max) at 663 nm. It exhibits a high molar absorption coefficient of approximately 8.4×10^4 L·mol^{-1}·cm^{-1}, making it an effective coloring agent in industrial applications. However, these same physicochemical properties contribute to its persistence in aquatic environments. The discharge of MB-containing wastewater can significantly reduce light penetration and dissolved oxygen levels, disrupt aquatic biodiversity, and inhibit photosynthetic activity, thereby deteriorating water quality. In addition to its environmental impacts, MB exposure poses serious risks to human health, including respiratory, digestive, and neurological disorders, as well as skin irritation upon direct contact (Khan et al., 2022).

To mitigate dye-related pollution, various physical, chemical, and biological treatment methods have been developed, including adsorption, reverse osmosis, ultrafiltration, microbial degradation, and enzymatic treatment. Although these methods can effectively remove dyes from wastewater, they often require high operational and maintenance costs, sophisticated infrastructure, and substantial energy input. As a result, their large-scale application remains challenging, particularly in developing countries such as Indonesia (Nursalam et al., 2023). In this context, photocatalytic degradation has emerged as a promising alternative, as it utilizes light energy and semiconductor-based catalysts to decompose organic pollutants into less harmful or mineralized products.

Titanium dioxide (TiO_2) has traditionally been the most extensively studied photocatalyst due to its chemical stability and strong oxidative capability. However, in recent years, zinc oxide nanoparticles (ZnO-NPs) have attracted considerable attention owing to their superior or comparable photocatalytic performance. ZnO is a wide-bandgap semiconductor with a bandgap energy of 3.37 eV and a high exciton binding energy of 60 meV, which facilitates efficient generation of electron-hole pairs under light irradiation. Moreover, ZnO exhibits excellent optical and electrical properties, high chemical stability, and strong photocatalytic activity. Importantly, the production cost of ZnO is reported to be approximately 75% lower than that of TiO_2 and aluminum oxide (Al_2O_3), making ZnO-NPs an economically attractive photocatalyst for wastewater treatment applications (Ong et al., 2018; Soto-Robles et al., 2021; Mawarni et al., 2021).

Despite these advantages, conventional synthesis routes for ZnO nanoparticles generally rely on chemical methods that involve toxic solvents, harsh reaction conditions, and the generation of hazardous byproducts, raising concerns regarding environmental sustainability (Drummer et al., 2021). In response to these challenges, green synthesis approaches based on plant extracts have been increasingly explored. In such approaches, plant-derived phytochemicals, including polyphenols, flavonoids, terpenoids, and alkaloids, serve as both reducing and stabilizing agents. This dual function is consistent with previous studies reporting that natural biopolymers and plant-derived compounds can simultaneously act as reducing and stabilizing agents during nanoparticle synthesis, thereby minimizing particle aggregation and improving nanoparticle stability (Foliatini, 2015). Green synthesis also offers advantages such as low toxicity, cost-effectiveness, environmental compatibility, and improved nanoparticle stability compared with conventional chemical synthesis (Chan et al., 2021; Izadiyan et al., 2020). Several studies have successfully synthesized ZnO-NPs using plant extracts, such as *Prosopis juliflora* leaves, demonstrating the feasibility and environmental benefits of eco-friendly synthesis routes (Ramadanti & Maharani, 2022). Nevertheless, the identification of new and abundant plant sources remains essential to further enhancing synthesis efficiency and broadening practical applicability.

Water hyacinth (*Eichhornia crassipes*), a highly abundant and invasive aquatic plant, represents a promising yet underutilized biomass resource for green nanoparticle synthesis. Previous studies have reported that water hyacinth leaf extract contains various bioactive compounds, including phenols, flavonoids, terpenoids, and alkaloids. Kasim et al. (2020) successfully employed this extract as a reducing agent for the synthesis of silver nanoparticles (Ag-NPs), highlighting its strong reducing capability. However, to date, the potential application of water hyacinth leaf extract as a bioreductant in the synthesis of ZnO nanoparticles has not been thoroughly investigated, indicating a clear research gap.

To further enhance the efficiency of green synthesis processes, microwave-assisted methods have been proposed as energy-saving and cost-effective alternatives. Microwave irradiation enables rapid and uniform heating, promotes homogeneous nucleation, and facilitates the formation of nanoparticles with controlled morphology and crystallinity, while significantly reducing reaction time and energy consumption. Previous studies have also demonstrated that microwave-assisted synthesis produces ZnO nanoparticles with improved crystallinity, enhanced photocatalytic performance, and shorter processing times compared to conventional heating methods (Ahammed et al., 2020; Sari et al., 2019; Nursalam et al., 2023). Despite growing interest in microwave-assisted synthesis, the integration of microwave irradiation with water hyacinth extract as a bioreductant for ZnO-NPs remains unexplored.

Therefore, this study aims to address this gap by developing a novel microwave-assisted green synthesis approach for ZnO nanoparticles using water hyacinth leaf extract as the reducing agent. The synthesized ZnO-NPs are systematically characterized, and their photocatalytic performance in degrading methylene blue is evaluated. Based on the phytochemical composition of water hyacinth and the advantages of microwave-assisted synthesis, the resulting ZnO nanoparticles exhibit enhanced photocatalytic activity, offering a sustainable and efficient alternative to conventional synthesis methods for treating textile wastewater.

2. Methods

2.1 Synthesis of ZnO nanoparticles using water hyacinth leaf extract

This study was carried out in the laboratories of the Department of Physics and Chemistry at Institut Teknologi Sepuluh Nopember (ITS), Surabaya, Indonesia. Water hyacinth (Eichhornia crassipes) leaves were collected from local water bodies in Surabaya, an area where the plant is an invasive species. After thorough washing to remove dirt and contaminants, the leaves were shade-dried for two weeks and ground into a fine powder. The powdered leaves were then boiled in distilled water to extract bioactive compounds, which served as the reducing agent for the synthesis of zinc oxide nanoparticles (ZnO-NPs). The extract was prepared at three concentrations: 4%, 8%, and 12% (w/v) by dissolving the powder in water and filtering the solution.

The synthesis of ZnO nanoparticles was carried out by dissolving zinc acetate dihydrate (0.2 M) in deionized water, followed by the addition of the prepared water hyacinth leaf extract. The solution was mixed and its pH adjusted to 9 using a 1 M sodium hydroxide (NaOH) solution to promote the precipitation of zinc oxide. The mixture was then subjected to microwave irradiation at 800 W for 5 minutes, using a microwave oven. The microwave-assisted method accelerates nanoparticle formation by providing rapid and uniform heating, enhancing nucleation and control over particle size and morphology. After microwave irradiation, the mixture was cooled to room temperature, and the precipitates were collected by centrifugation. The collected particles were washed several times with deionized water to remove any residual impurities, and then dried in an oven at 60°C overnight to obtain ZnO nanoparticles.

2.2 Characterization and photocatalytic activity evaluation of ZnO nanoparticles

The synthesized ZnO-NPs were characterized using various techniques to confirm their structure and properties. X-ray diffraction (XRD) was employed to determine the crystalline phase and structural integrity of the nanoparticles. The XRD patterns were analyzed to confirm the formation of a pure ZnO phase, with no secondary or impurity phases present. Fourier-transform infrared spectroscopy (FTIR) was used to identify the functional groups associated with the surface chemistry of the nanoparticles. Scanning Electron Microscopy (SEM) provided detailed images of the nanoparticle morphology, particle size distribution, and agglomeration behavior, enabling an in-depth understanding of how the water hyacinth extract concentration influenced the particle formation process.

To evaluate the photocatalytic activity of the synthesized ZnO-NPs, a degradation study of methylene blue (MB) dye was conducted under UV light. A 15 ppm MB solution was prepared, and 0.1 g of ZnO-NPs was added to 50 mL of the dye solution. The photocatalytic degradation experiment was conducted under UV irradiation, and the degradation progress was monitored by measuring the absorbance of methylene blue at 664 nm using a UV–Vis spectrophotometer, and the degradation progress was monitored over a period of 60 minutes. Samples were collected at 15-minute intervals, and the solution absorbance was measured using UV-Vis spectrophotometry. The decrease in absorbance was used to calculate the degradation efficiency of MB, with a higher degradation efficiency indicating better photocatalytic performance. The study was conducted with each of the three extract concentrations (4%, 8%, and 12%) to assess the impact of the extract concentration on the photocatalytic activity of the ZnO nanoparticles.

3. Results and Discussion

3.1 Mechanistic role of EDEG as a bioreductor and capping agent in ZnO-NPs Synthesis

Water hyacinth (Eichhornia crassipes) leaf extract (EDEG) functions as an effective bioreductor in zinc oxide nanoparticle synthesis through the electron-donation capabilities of its diverse polyphenolic compounds, enabling the conversion of Zn^{2+} ions to ZnO under mild conditions. The plant extract contains abundant secondary metabolites with strong reducing power, including phenolic compounds (9.73% of extract), flavonoids (10.49%), tannins, terpenoids, and alkaloids. Among these, polyphenolic compounds with ortho- (1,2-) or para- (1,4-) dihydroxyl substituents (catechol, quercetin, and caffeic acid), function as reversible electron shuttles, undergoing proton-coupled electron transfer (PCET) that enables the sequential donation of electrons to Zn^{2+} ions. The reaction path based on the experiment done by Garino et al. (2019) is based on the hydrolysis of the zinc precursor due to the presence of the hydroxide, as shown in the following reaction scheme:

$$NaOH \rightarrow Na^{+}\,(aq) + OH^{-}\,(aq) \quad \text{(Eq. 1)}$$

$$Zn(Ac)_2 \rightarrow Zn^{2+}\,(aq) + 2Ac^{-}\,(aq) \quad \text{(Eq. 2)}$$

$$Zn^{2+}\,(aq) + 2OH^{-}\,(aq) \rightarrow Zn(OH)_2\,(aq) \quad \text{(Eq. 3)}$$

$$Zn(OH)_2\,(aq) + 2H_2O \rightarrow Zn(OH)_4{}^{2-} + 2H^{+}\,(aq) \quad \text{(Eq. 4)}$$

$$Zn(OH)_4{}^{2-} + 2H^{+}\,(aq) \rightarrow ZnO\,(s) + 3H_2O \quad \text{(Eq. 5)}$$

Critically, compounds containing ortho- or para-dihydroxyl groups exhibit reversible redox cycling through resonance stabilization of semiquinone-like radical intermediates, whereas meta-positioned hydroxyl groups lack this capacity and function only as non-renewable antioxidants. The antioxidant activity of water hyacinth extracts with DPPH IC_{50} values as low as 35.83 ppm directly correlates with their reducing power and Zn^{2+} reduction

capability, demonstrating the strong electron-donating capacity of the extract's polyphenolic composition. The efficiency of bioreduction is highly pH-dependent, with alkaline conditions (pH ≥ 8-10) dramatically enhancing the electron-shuttle capability of phenolic compounds through deprotonation-coupled electron transfer, which explains why synthesis protocols raise pH immediately after zinc precursor addition to ensure sustained electron donation rather than irreversible antioxidant consumption (Ahmad et al., 2024; Kalaivani & Ravi, 2023; Zelekew et al., 2021).

The synthesis mechanism reveals that water hyacinth extract simultaneously governs both the chemical reduction pathway and the morphological control of synthesized ZnO-NPs. In typical green synthesis protocols, aqueous extraction of water hyacinth tissue releases phytochemicals into solution, which are then mixed with zinc acetate dihydrate precursor with stirring. Upon pH adjustment to approximately 10 using NaOH, the alkaline environment triggers simultaneous Zn^{2+} reduction by phenolic electron shuttles and ZnO nucleation through the reaction of OH^- with reduced zinc species. The remaining phenolic compounds and organic species act as stabilizing capping agents, preventing uncontrolled nanoparticle agglomeration and controlling crystal growth kinetics to yield well-defined morphologies. The capping layer also improves colloidal stability and limits excessive particle growth during synthesis, which is a common mechanism observed in plant-mediated nanoparticle synthesis (Chugh et al., 2021). Following synthesis, calcination at 800 W for 5 minutes in a microwave and drying in an oven at 60°C overnight to remove most organic residue while preserving the ZnO crystal structure (Chutrakulwong et al., 2024).

The mechanism of ZnO-NPs formation using water hyacinth extract is fundamentally different from the reduction mechanism involved in Ag-NPs synthesis done by Kasim et al. (2020). In the synthesis of Ag-NPs, the primary process is a true redox reaction, in which Ag^+ ions undergo a change in oxidation state from +1 to 0 through direct electron transfer from polyphenolic compounds. Phenolic groups bearing ortho- or para-dihydroxyl substitutions (e.g., catechol, quercetin, and caffeic acid) act as active reducing agents, becoming oxidized to quinone or semiquinone forms that are stabilized by resonance. This process proceeds via proton-coupled electron transfer (PCET), producing Ag^0 atoms that subsequently nucleate and grow into stable silver nanoparticles. In contrast, the transformation of Zn^{2+} into $Zn(OH)_2$ and subsequently into ZnO does not involve a change in the oxidation state of zinc and therefore cannot be classified as a reduction reaction. Instead, the process is governed by pH-driven hydrolysis and precipitation.

3.2 ZNO-NPs structural and morphological analysis

The synthesized ZnO nanoparticles (ZnO-NPs) were systematically characterized using Scanning Electron Microscopy (SEM), X-ray Diffraction (XRD), and Fourier Transform Infrared Spectroscopy (FTIR) to elucidate their morphological features, crystal structure, and surface chemical composition. Nanoparticles possess unique physicochemical properties, including high specific surface area and size-dependent optical and catalytic behavior, making structural characterization essential for understanding their functional performance (Khan et al., 2019). Comprehensive analysis of these parameters is essential, as the structural and morphological properties of ZnO-NPs strongly influence their photocatalytic behavior.

The SEM analysis, as depicted in Figure 1b, reveals that the synthesized ZnO nanoparticles (ZnO-NPs) exhibit an irregular surface morphology characterized by a combination of prismatic and amorphous-like structures. Such morphological diversity is typical of ZnO-NPs synthesized via green synthesis methods, where the presence of organic compounds from plant extracts influences the growth and aggregation of the nanoparticles. The organic compounds in plant extracts, rich in functional groups like hydroxyl and carboxyl, interact with the ZnO particles during synthesis. These interactions can either promote or hinder particle growth, resulting in a variety of shapes and structures. This irregular morphology can be advantageous for photocatalytic applications, as it can enhance

the surface area and provide more active sites for reactions. Furthermore, the presence of prismatic features suggests preferential growth along specific crystallographic directions of the wurtzite ZnO lattice, while the amorphous-like regions may be associated with incomplete crystallization or surface disorder induced by organic capping agents. Such surface heterogeneity can contribute to localized electronic states, which may act as charge trapping sites and influence photocatalytic reactivity. The combination of ordered crystalline domains and disordered surface regions is therefore considered beneficial for balancing charge transport and surface reaction kinetics.

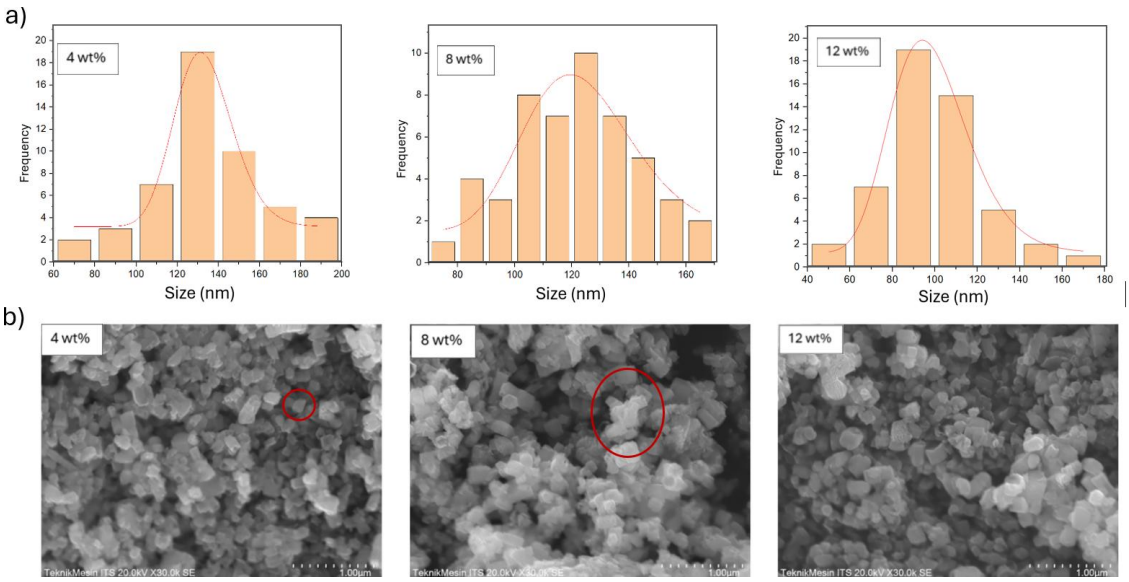

Fig. 1. (a) Particle size distribution; (b) & SEM micrographs of synthesized ZnO-NPs 4 wt%, 8 wt%, and 12 wt%

The particle size distribution shown in Figure 1a indicates that the synthesized ZnO-NPs have sizes ranging from approximately 60 nm to 200 nm. The average particle diameters for ZnO-NPs synthesized with precursor concentrations of 4 wt%, 8 wt%, and 12 wt% were determined to be 134.42 nm, 120.54 nm, and 102.64 nm, respectively. The reduction in particle size with increasing precursor concentration suggests that higher precursor concentrations lead to an enhanced nucleation rate under the present synthesis conditions. Faster nucleation results in the formation of smaller particles, as the material is distributed more rapidly into the growing nanoparticles, limiting their subsequent growth. This phenomenon indicates improved control over the nucleation and growth processes, which is particularly beneficial for photocatalytic applications, where smaller particles typically exhibit higher surface areas and more reactive sites. In photocatalytic systems, reduced particle size is directly associated with shorter charge carrier diffusion lengths, which lowers the probability of bulk electron–hole recombination before charges reach the surface (Ong et al., 2018). Consequently, the smaller average particle size observed for the 12 wt% ZnO-NPs is expected to facilitate more efficient charge separation and enhance surface redox reactions. In contrast, the larger particles formed at lower precursor concentrations may suffer from increased recombination losses due to longer migration distances within the bulk material.

Despite the overall successful control over particle size, the SEM micrographs also reveal some degree of particle agglomeration. The large specific surface area combined with the high surface energy of nanoparticles promotes aggregation, resulting in the development of agglomerated structures exhibiting fractal characteristics (Zhang et al., 2021). This can be a result of non-optimal synthesis conditions, including improper irradiation duration or pH conditions during synthesis. These factors may cause insufficient stabilization of the nanoparticles, leading to their aggregation into clusters. While agglomeration reduces the effective surface area available for photocatalytic reactions, the fact that the particles remain within the nanoscale range suggests that they still have

sufficient surface area for effective photocatalytic activity (Li et al., 2010). However, such agglomeration could hinder the particles' performance by limiting the availability of active sites, which is a well-known challenge in nanoparticle synthesis.

It is important to note that the extent of agglomeration appears less pronounced for samples synthesized at higher precursor concentrations, which may be attributed to faster nucleation and more uniform particle formation under microwave irradiation (Kunze et al., 2019). This behavior further supports the superior photocatalytic performance observed for the 12 wt% ZnO-NPs, as reduced agglomeration helps preserve accessible surface area and active reaction sites. The agglomeration observed in this study is consistent with previous reports in the literature, where ZnO-NPs synthesized via green methods tend to form aggregates under certain conditions. Insufficient stabilization or prolonged synthesis times are known to promote particle clustering, thus reducing the effectiveness of the nanoparticles in catalytic applications (Li et al., 2010). To mitigate this issue, researchers have used various strategies, such as adding surfactants or optimizing irradiation and pH conditions, to improve the dispersion of the particles and prevent agglomeration. In the context of this study, while agglomeration may slightly reduce the effectiveness of the ZnO-NPs, their overall size distribution and the presence of nanoscale features suggest that they remain viable candidates for photocatalytic applications. Moreover, partial agglomeration may still allow inter-particle charge transfer pathways, which can contribute positively to photocatalytic activity when aggregation does not fully block active surface sites.

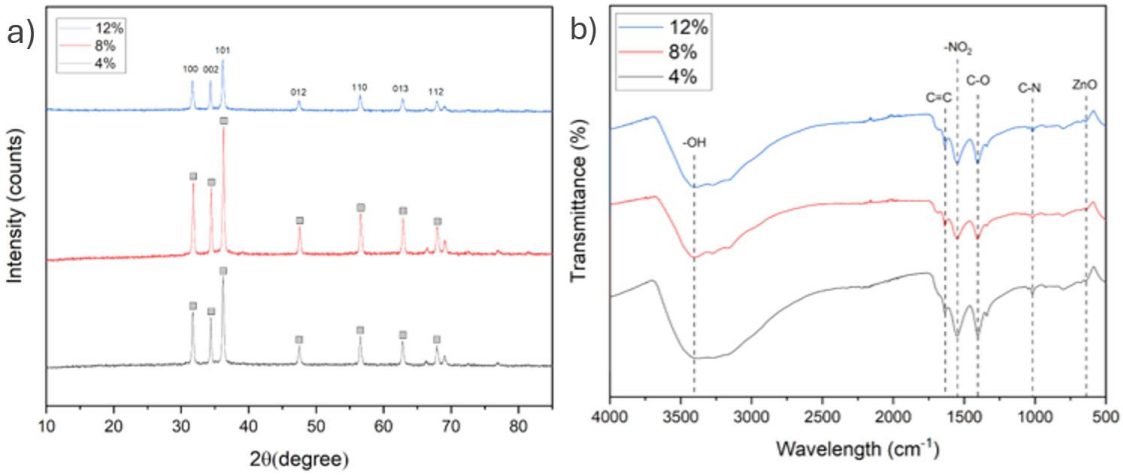

Fig. 2 (a) XRD pattern; (b) FTIR spectrum of synthesized ZnO-NPs

Structural characterization using XRD (Figure 2a) confirms that the synthesized nanoparticles possess a crystalline hexagonal wurtzite ZnO structure. Phase identification performed using MATCH! and HighScore Plus software shows excellent agreement with the standard reference pattern (JCPDS No. 36-1451). The absence of secondary diffraction peaks or impurity phases indicates high phase purity and demonstrates the selectivity of the synthesis route. The sharp and well-defined diffraction peaks observed in the XRD pattern suggest a high degree of crystallinity, which is essential for efficient charge transport and reduced recombination of photogenerated electron–hole pairs (Xie et al., 2019). Chemical composition analysis further reveals approximately 80.3% Zn and 19.7% O, closely corresponding to stoichiometric ZnO. These findings confirm that the synthesis process effectively promotes crystalline ZnO formation without introducing undesirable secondary phases.

The hexagonal wurtzite structure of ZnO is highly favorable for photocatalytic applications due to its anisotropic crystal nature and the presence of intrinsic polar surfaces, particularly along the (002) plane (Paul et al., 2025). These polar facets generate internal electric fields that promote the effective separation of photogenerated electron–hole pairs, thereby suppressing charge recombination and enhancing photocatalytic efficiency. In addition, variations in precursor concentration may influence crystallite size

and lattice distortion, which can alter defect characteristics and electronic properties, ultimately affecting the photocatalytic performance of ZnO.

Further structural verification was conducted using FTIR spectroscopy (Figure 2b). FTIR spectroscopy is widely employed to identify chemical bonds and functional groups associated with nanomaterials by detecting characteristic vibrational modes, making it an effective technique for confirming Zn–O bond formation and residual organic compounds originating from plant extracts (Terpugov, 2020). A distinct absorption band observed near 636 cm^{-1} is attributed to Zn–O stretching vibrations, providing direct evidence of ZnO formation. A broad absorption band in the range of 3380–3404 cm^{-1} corresponds to O–H stretching vibrations, which are likely associated with adsorbed moisture or surface hydroxyl groups formed during synthesis. Additional absorption bands at 1629 cm^{-1}, 1539–1551 cm^{-1}, 1403 cm^{-1}, and 1016 cm^{-1} are assigned to C=C, NO_2, C–O, and C–N functional groups, respectively. These functional groups are likely derived from residual organic compounds originating from the reducing or stabilizing agents present in the water hyacinth leaf extract. The presence of such surface functional groups may enhance surface hydrophilicity and facilitate dye adsorption during photocatalytic reactions.

In addition to surface chemistry effects, residual organic species and lattice imperfections can contribute to the formation of intrinsic defects such as oxygen vacancies. These defects are known to act as electron trapping sites, prolonging charge carrier lifetimes and promoting the formation of reactive oxygen species during photocatalytic reactions. Consequently, the combined influence of optimized particle size, favorable crystal structure, and controlled defect density provides a rational explanation for the observed photocatalytic performance trend, where ZnO-NPs synthesized at 12 wt% exhibit superior activity compared to those prepared at 8 wt% and 4 wt%. The enhanced performance of the 12 wt% sample can therefore be attributed to synergistic effects arising from smaller particle size, improved charge separation, and defect-assisted surface reactivity (Dwiyanti et al., 2025).

3.3 Photocatalytic performance of ZNO-NPs

The photocatalytic performance of ZnO nanoparticles (ZnO-NPs) synthesized with different EDEG concentrations was evaluated through the degradation of methylene blue (MB) under ultraviolet (UV) irradiation. An aqueous MB solution with an initial concentration (C_0) of 15 ppm was mixed with ZnO-NPs and exposed to UV light for 0–120 minutes. Aliquots were withdrawn every 15 minutes, centrifuged to remove the photocatalyst, and the absorbance of the clear supernatant was measured using a UV–Vis spectrophotometer at the characteristic maximum absorption wavelength of MB ($\lambda_{max} \approx$ 664 nm). The experimental results are presented in Fig. 3.

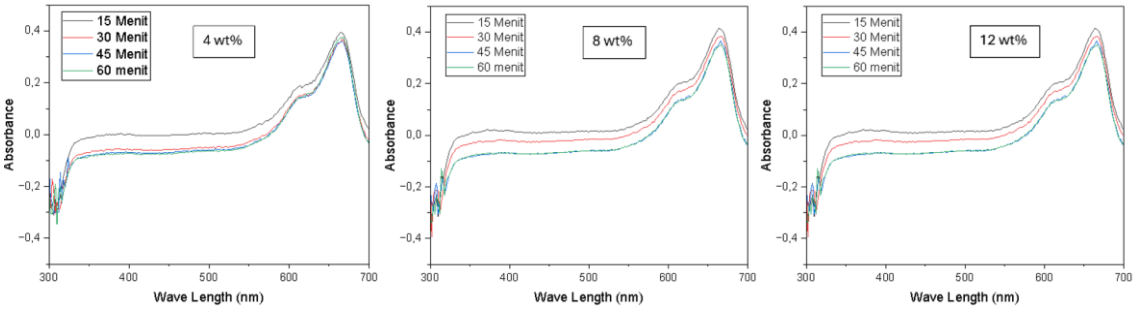

Fig. 3. Absorption spectra of MB in the presence of 4 wt%, 8 wt%, and 12 wt% EDEG

Fig. 3 shows the absorption spectra of MB in the presence of ZnO-NPs synthesized using 4 wt%, 8 wt%, and 12 wt% EDEG. A gradual decrease in absorbance intensity at 664 nm with increasing irradiation time is observed for all samples, indicating the progressive degradation of MB. No significant shift in the maximum absorption wavelength was detected, suggesting that the photocatalytic process proceeds predominantly through

molecular degradation rather than simple adsorption or structural transformation of the dye. The MB concentration at each irradiation time (C_t) was calculated from the measured absorbance using the Beer–Lambert law:

$$A = \varepsilon \cdot l \cdot C \quad \text{(Eq. 6)}$$

where A represents the measured absorbance of the methylene blue solution, ε is the molar absorptivity of methylene blue, l is the optical path length of the cuvette (1 cm), and C is the concentration of MB. The photocatalytic degradation efficiency after 120 minutes of UV irradiation was calculated using the following equation:

$$Degradation\ efficiency\ (\%) = \frac{C_0 - C_t}{C_0} \times 100 \quad \text{(Eq. 7)}$$

where C_0 is the initial methylene blue concentration before UV irradiation (15 ppm), which represents the starting concentration of the dye solution, and C_t is the methylene blue concentration after t = 120 minutes of irradiation, which reflects the remaining dye concentration following the photocatalytic process.

The degradation of methylene blue (MB) using ZnO nanoparticles (ZnO-NPs) synthesized using different EDEG concentrations (4, 8, and 12 wt%) shows relatively promising results, although these are still lower than the performance of commercial ZnO, which achieves a degradation efficiency of 56%. After two hours of UV irradiation, ZnO-NPs with 4, 8, and 12 wt% EDEG resulted in degradation percentages of 32%, 44%, and 53%, respectively. This trend indicates that increasing the EDEG concentration enhances the photocatalytic activity of the ZnO-NPs, with the degradation efficiency improving progressively with higher EDEG concentrations. The enhancement in photocatalytic activity can be attributed to several factors, such as improved dispersion of ZnO-NPs in the solution, increased surface area available for interactions with MB molecules, and the potential reduction in the aggregation of nanoparticles. All these factors contribute to a more effective photocatalytic reaction, providing more active sites for the degradation of MB. Despite these improvements, the degradation efficiency achieved by ZnO-NPs with EDEG modification still does not reach the level of commercial ZnO, indicating that ZnO-NPs with EDEG modification, while effective, have yet to match or exceed the performance of the well-established commercial photocatalysts. This also highlights the need for further exploration into optimizing ZnO-NPs for enhanced photocatalytic performance.

The addition of EDEG plays a significant role in synthesizing the physical and chemical properties of the ZnO nanoparticles, thereby enhancing their photocatalytic efficiency. Of the concentrations tested, the 12 wt% EDEG concentration yielded the highest degradation efficiency at 53%, which can be attributed to several key factors. First, at this concentration, EDEG likely improves the dispersion of ZnO-NPs, preventing agglomeration and maintaining a high surface area, which is critical for photocatalytic reactions. Additionally, EDEG may help stabilize the ZnO nanoparticles, improving their structural integrity under UV irradiation. Despite these advantages, the performance of ZnO-NPs synthesized with EDEG still falls short of that of commercial ZnO, which indicates that there is an optimal range of EDEG concentration beyond which no further improvement is observed. It is possible that higher concentrations of EDEG could lead to aggregation of ZnO-NPs or excessive blocking of active sites, thereby reducing their overall photocatalytic performance.

ZnO functions as an n-type semiconductor photocatalyst with a band gap energy of 3.3 eV, making it highly responsive to ultraviolet (UV) light. Its photocatalytic properties are largely attributed to this band gap, which allows ZnO to absorb UV light and drive redox reactions. The process of photocatalytic degradation of methylene blue (MB) involves a complex multi-step mechanism, starting with light absorption, followed by the generation and separation of charge carriers, and culminating in the formation of reactive oxygen

species (ROS) that contribute to the degradation of the MB molecule (Ghamarpoor et al., 2024). When ZnO is exposed to UV light with a wavelength of 380 nm or less, the energy of the incoming photons (Ephoton ≥ 3.3 eV) is sufficient to excite electrons from the valence band (VB) to the conduction band (CB), creating photoexcited electron-hole pairs. This phenomenon can be expressed as $ZnO + h\nu \rightarrow e^{-}CB + h^{+}VB$, where the energy provided by UV light excites the electron to a higher energy state, initiating the photocatalytic reaction.

The enhanced photocatalytic performance of the synthesized ZnO nanoparticles can be attributed to their reduced particle size and unique structural features induced by microwave-assisted green synthesis. The decrease in average particle size from 134.42 nm to 102.64 nm with increasing EDEG concentration shortens the migration distance of photogenerated charge carriers, thereby suppressing bulk recombination of electron–hole pairs. Smaller particles also provide a higher surface-to-volume ratio, which increases the availability of active sites for dye adsorption and surface redox reactions. Furthermore, microwave irradiation promotes rapid and uniform nucleation, leading to improved crystallinity and defect formation that are beneficial for photocatalytic processes.

However, ZnO's large band gap also presents limitations, particularly in its ability to utilize visible light. Since the material is highly responsive only to UV light, its performance is inherently constrained by the limited availability of UV light compared to the broader spectrum of visible light. As a result, while ZnO is effective for UV-driven photocatalysis, it is less efficient under natural sunlight or in environments where UV light is scarce. This limitation becomes particularly relevant when trying to enhance the photocatalytic efficiency of ZnO for environmental applications, where visible light utilization is crucial for broader and more sustainable use.

The photoexcited electrons and holes generated in the conduction and valence bands, respectively, can either recombine quickly, releasing energy in the form of heat and reducing the efficiency of the photocatalytic reaction, or they can migrate to the surface of ZnO, where they participate in redox reactions. On the surface, electrons accumulate in the conduction band, while the holes remain in the valence band, creating an electric field that encourages the spatial separation of charges. This charge separation is essential for the efficient generation of reactive oxygen species (ROS), such as superoxide anion (O_2^{-}) and hydroxyl radicals (OH·), which are the key agents responsible for breaking down MB molecules. The efficient migration and separation of these charge carriers are critical for the success of the photocatalytic degradation process, as they ensure that the energy from UV light is effectively used to degrade organic pollutants. However, without efficient charge separation, the electrons and holes may recombine too quickly, leading to a loss of photocatalytic efficiency (Ghamarpoor et al., 2024).

The separated charge carriers in ZnO-based photocatalysis initiate two parallel redox processes on the surface of ZnO. In the first pathway, photoexcited electrons from the conduction band (e^{-}_{CB}) reduce dissolved oxygen molecules (O_2) to generate superoxide radical anions ($O_2\bullet^{-}$), as described by the reaction $e^{-}_{CB} + O_2 \rightarrow O_2\bullet^{-}$. The superoxide radicals ($O_2\bullet^{-}$) formed can then undergo protonation, leading to the formation of hydroperoxyl radicals ($O_2\bullet^{-} + H^{+} \rightarrow O_2H\bullet^{-}$), which are also reactive intermediates in the photocatalytic process. The second parallel pathway involves the oxidation of water molecules or hydroxide ions (OH^{-}) adsorbed on the ZnO surface by photogenerated holes ($h^{+}VB$) in the valence band. These reactions produce hydroxyl radicals (•OH), which are critical in the photocatalytic degradation of pollutants. Two possible reactions take place depending on the species involved: $h^{+}_{VB} + H_2O \rightarrow \bullet OH + H^{+}$ or $h^{+}_{VB} + OH^{-} \rightarrow \bullet OH$. The hydroxyl radical (•OH) is the primary oxidizing species in ZnO photocatalysis, playing a dominant role in aqueous environments, especially in the degradation of organic pollutants like methylene blue (MB) (Ghamarpoor et al., 2024).

The generated reactive oxygen species (ROS), including superoxide radical anions ($O_2\bullet^{-}$) and hydroxyl radicals (•OH), attack the methylene blue molecules through oxidative degradation. Methylene blue (MB) can exist in solution as monomers or aggregated dimers, and the degradation mechanism depends on the form of MB present. For monomeric methylene blue, ROS primarily attack the conjugated aromatic system and the N-S bonds of

the dye. The main sites of attack are the n→π* transitions, involving the nitrogen and sulfur atoms in the MB structure, and the π→π* transitions in the aromatic benzene rings. The reaction follows MB + •OH→ Degradation Intermediates. For dimeric methylene blue, the aggregated dimers are more resistant to direct degradation. However, initial UV irradiation can break the hydrogen bonds between the MB molecules, dissociating the dimers into reactive monomers. These monomers are then readily degraded by ROS through the same oxidative mechanisms, as described above (Atta et al., 2024).

In addition to particle size effects, the presence of surface defects and oxygen vacancies plays a critical role in enhancing the photocatalytic activity of ZnO-NPs synthesized via microwave-assisted routes. Oxygen vacancies act as shallow electron traps that inhibit rapid electron–hole recombination while simultaneously enhancing oxygen adsorption on the ZnO surface. Adsorbed oxygen molecules at vacancy sites are more readily reduced by photogenerated electrons to form superoxide radicals ($O_2•^-$), which subsequently participate in the generation of hydroxyl radicals (•OH). These reactive oxygen species are primarily responsible for the oxidative degradation of methylene blue molecules adsorbed on the ZnO surface (Zhang et al., 2018).

The continued oxidation of degradation intermediates leads to complete mineralization of methylene blue, ultimately yielding carbon dioxide (CO_2), water (H_2O), and various mineral products. This mineralization process follows pseudo-first-order kinetics, meaning the rate of degradation is proportional to the concentration of the dye. The degradation efficiency is influenced by several factors, including catalyst mass, light intensity, and pH (Prabakaran & Pillay, 2019).

Several factors significantly influence the photodegradation process of methylene blue. Dissolved oxygen acts as an essential electron acceptor in the process. Higher oxygen concentrations enhance the formation of superoxide radicals, which in turn increases the overall degradation rate. At neutral to slightly basic pH, hydroxide ions (OH^-) are more readily available, promoting the formation of hydroxyl radicals through hole oxidation. Additionally, ZnO nanoparticles with a higher specific surface area provide more active sites for the adsorption of methylene blue molecules and the subsequent generation of ROS, leading to more efficient degradation. UV light intensity is another critical factor that affects the photoexcitation rate. In advanced systems, UV laser irradiation at 305 nm has been shown to enhance the degradation process by breaking up MB aggregates, significantly reducing the degradation time from 24 hours to approximately 3 hours (Zhang et al., 2018).

4. Conclusions

The synthesis of zinc oxide nanoparticles (ZnO-NPs) using water hyacinth leaf extract combined with microwave-assisted synthesis demonstrated a cost-effective and environmentally friendly approach. The resulting ZnO-NPs showed high purity with no secondary phases, confirming the effectiveness of the synthesis method. Photocatalytic degradation of methylene blue (MB) dye revealed promising results, with degradation efficiencies of 32%, 44%, and 53% for extract concentrations of 4%, 8%, and 12%, respectively. The highest degradation efficiency was achieved at the 12% extract concentration, indicating the importance of extract concentration in enhancing the photocatalytic activity of the nanoparticles. This suggests that water hyacinth extract can serve as an effective green reducing agent for ZnO-NP synthesis with significant potential in wastewater treatment.

The microwave-assisted method enhanced the efficiency of nanoparticle synthesis by promoting rapid and uniform heating, which reduced energy consumption and synthesis time. The use of water hyacinth, an invasive aquatic plant, further adds environmental value by utilizing a readily available biomass resource while mitigating the plant's environmental impact. Although the photocatalytic efficiency of the synthesized ZnO-NPs was slightly lower than commercial ZnO, the results indicate that this green synthesis approach is a viable, scalable alternative for the production of ZnO-NPs, especially for applications in the degradation of textile dyes like methylene blue. Further optimization of the synthesis

parameters could improve the catalytic performance, making this method even more effective for large-scale environmental applications.

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Author Contribution
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The data that support the findings of this study are available from the corresponding author upon reasonable request.

Conflicts of Interest
The authors declare no conflict of interest. The funder had no role in the design of the study; in the collection, analyses, or interpretation of data; in the writing of the manuscript; or in the decision to publish the results.

Declaration of Generative AI Use
During the preparation of this work, the authors used ChatGPT (OpenAI) to assist in improving the English language and refining the structure of the manuscript. After using this tool, the authors reviewed and edited the content as needed and take full responsibility for the content of the publication.

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