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# Photocatalytic Degradation of Organic Dye Pollutants Using ZnO Nanoparticles Synthesized from *Plumeria alba* L. Leaf Extract

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## ABSTRACT

**Background**: Dye pollutants are hazardous to environment and community health. ZnO nanoparticles synthesized by leaf extract of *Plumeria alba* L. were tested to degrade pollutants through photocatalysis. The pollutants used in this study are organic dyes-Methylene Blue (MB), Bromophenol Blue (BB), Malachite Green (MG) and Crystal Violet (CV). These dye pollutants are commonly used in various industries, washed away, and mixed up in the water bodies.

**Methods**: The present work presents a green production technique for ZnO nanoparticles (ZnO NPs) that is easy, cost-effective, eco-friendly, and uses *P. alba* L. aqueous leaf extract and 0.5 M dehydrated zinc acetate. Fourier transform infrared spectroscopy (FT-IR) and UV-visible spectroscopy (absorption band at 355 nm) were used to characterize the synthesized ZnO NPs.

**Results**: FT-IR spectra revealed that polyols, terpenoids, and proteins with functional groups of amines, alcohols, ketones, and carboxylic acids were involved in the steps leading to the production of ZnO NP. TEM results confirmed the average size of NPs is 19.4 nm. Spectrophotometric results showed that MB, BB, MG and CV dyes (with absorption maxima at 656 nm, 590 nm, 616nm and 582 nm, respectively) were degraded gradually with increased irradiation time.

**Conclusion**: It was thus demonstrated that green synthesized ZnO NPs are a potent photocatalyst that may be effectively used to degrade hazardous and toxic contaminants in aquatic environments.

**Key-words:** *Plumeria alba* L., Photocatalytic Degradation, Green synthesis, ZnO Nanoparticles, ZnO NPs

## INTRODUCTION

Water pollution is mainly caused by industrial effluents, particularly in cities where the number of industries for fabric dying are higher. Industries involved in the coloring release dangerous waste materials into natural streams and rivers, posing serious environmental and health risks that require attention.

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Sanganer, Jaipur (Rajasthan) is the hub of fabric dying and is globally recognized for hand-blocked prints. Dyes are colored chemicals that have a strong bond with fiber and give color to different types of materials in an easy way. According to their definition, dyes are colored, ionizing, and aromatic organic chemicals that exhibit an affinity for the substrate to which they are applied [1]. Whether a chromophore absorbs photons in the visible or UV spectrum determines whether or not it may give a complex color. Due to their increased absorption of some light wavelengths over others, dyes look colored [2]. When dyes are subjected to water, water bodies impart color, reducing sunlight penetration and photosynthetic activity and degrading water quality. Dyes also increase

 

BOD and reduce the amount of dissolved oxygen needed to support aquatic life, which results in toxicity, pollution, and eutrophication. Pollutants and by-products are often poisonous, mutagenic, and carcinogenic [3]. Traditional wastewater treatment methods include adsorption, coagulation/flocculation, and precipitation. These methods can take a long time to complete and produce expensive secondary sludge to dispose of Muhd *et al.* [4].

Sustainable and eco-friendly technologies are always preferable to deal with environmental hazardous pollutants. Another significant area that is utilized to break down organic dyes is nanotechnology. Due to their tiny size, aligned surface, and distinctive chemical and physical characteristics, nanoparticles are unique and pave the way for sustainable solutions to pollutions. Physical, chemical, or biological methods can be used to synthesize nanoparticles. Green synthesis of nanostructures is inexpensive, straightforward, and environmentally benign.

Only a few studies on ZnO NPs' synthesis using *Plumeria* plant extract have been carried out till date. *P. obtuse* leaf [5] and *P. obtuse* flower [6] extracts were used for synthesizing Zinc oxide nanoparticles and their antibacterial and antifungal properties were tested. *P. alba* flower extract was used to synthesize gold nanoparticles [7]. Rhodamine B was degraded by Rajendrachari *et al.* [8] utilizing ZnO NPs (green synthesized using *Alchemilla vulgaris*) while illuminated by an AM 1.5 solar simulator. For the first time, a straightforward, ecologically friendly process for creating zinc oxide nanoparticles (ZnO NPs) using an aqueous leaf extract of *P. alba* L. is shown here, along with its application in the photocatalytic degradation of organic colours.

Elumalai *et al.* [9] also described that ZnO nanoparticles synthesized using plant extract are likely to get agglomerated due to their small size. In place of simple ZnO nanoparticles, nanocomposites and heterojunctions are more efficient in photocatalytic performances [10-12]. Similar to the above report, tea extracts and its photocatalytic activity under visible light were used in a low-cost Ag-ZnO NC biosynthesis method developed to break down common organic pollutants [13]. $Cr_2O_3$/ZnO composite prepared by *Eichhornia crassipes* extract as capping agent degraded MB dye with 85% degradation efficiency [14].

## MATERIALS AND METHODS

**Preparation of leaf extract-** Fresh leaves of *P. alba* L. were collected and cleaned to remove any dust or impurities using running tap water. We let extra water air dry at ambient temperature. After weighing 5g of leaves and roughly crushing them using a pestle and mortar, 100ml of distilled water was added to a conical flask. After an hour of heating the solution to 50°C, Whatman No. 1 filter paper was used for filtering.

**Green synthesis of Zinc Oxide Nanoparticles-** 0.5 M Zinc acetate dihydrated [$Zn(O_2CCH_3)_2(H_2O)_2$; mol. wt. 219.46g/mol] was dissolved in 50 ml distilled water and kept in stirrer for 1 h. Similarly, 2 M of Sodium hydroxide (NaOH; mol. wt. 40 g/mol) was dissolved in 50 ml distilled water and kept on stirrer for approx. One h. Zinc acetate solution was added to NaOH solution drop by drop and mixed again for 1 hour. After adding plant extract, the previously mentioned mixture was agitated for another two hours. After two hours of incubation, a yellow-colored precipitate emerged, indicating the creation of ZnO NPs. Pellet was obtained after the precipitate was separated from the reaction solution by centrifugation at 10,000 rpm for ten minutes at six degrees Celsius. In order to eliminate contaminants, the pellet was centrifuged three times using purified water. Finally, the pellet was dried in a vacuum oven at 80°C for two hours. For future research, white-colored powder of zinc oxide nanoparticles was stored in airtight centrifuge tube (Fig. 1).

**Characterization of ZnO nanoparticles-** The synthesized NPs were characterized using UV-Vis spectroscopy, FTIR and HRTEM. UV-visible absorption spectroscopy is a method that is frequently used to analyze the optical properties of nano-sized particles. To confirm the formation of the zinc oxide nanoparticles, the UV-visible absorption spectra were recorded using a Thermo Scientific Multiskan GO UV-Vis spectrophoto meter. By employing the KBr pellet technique and an FTIR spectrophotometer (Shimadzu IR Affinity-1) with a scan range of 400 to 4000 $cm^{-1}$ and a resolution of $cm^{-1}$, ZnO NPs' FTIR spectra were captured. HR TEM was performed at Sophisticated Instrument Facility at AIIMS, New Delhi. Ultrasonicated nanoparticles were subjected to TECNAI G20 HR-TEM machine for high-resolution images. Size of different NPs were recorded and photographed.

 

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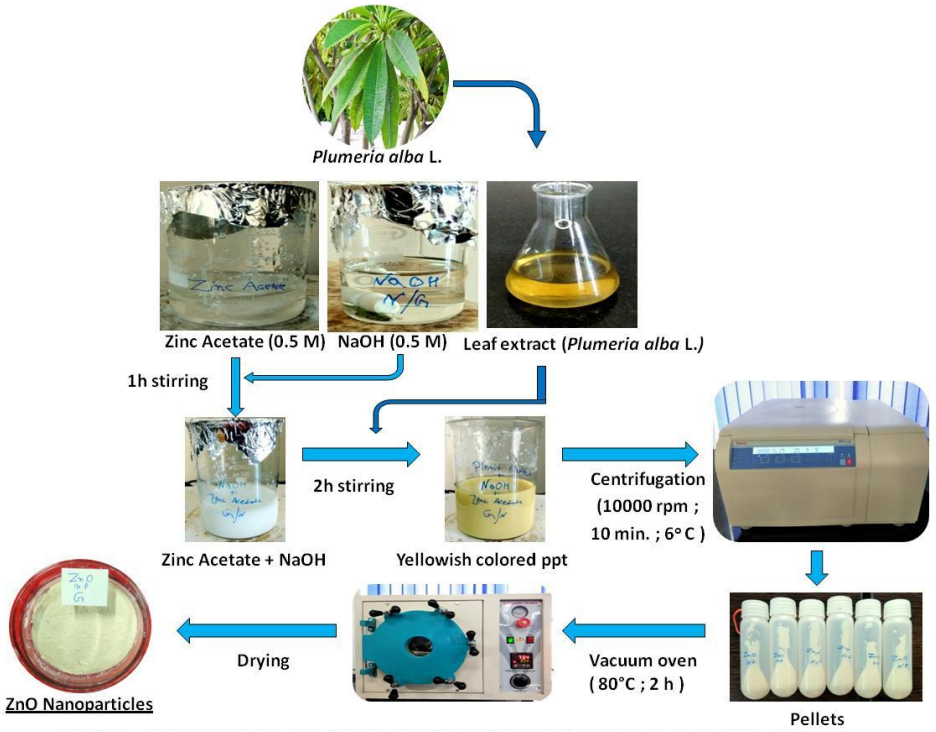


**Fig. 1:** Steps involved in green synthesis of ZnO NPs using *Plumeria alba* L. leaf extract.

**Photocatalytic Degradation of Organic Pollutants-** To assess how well biologically produced ZnO NPs degraded four distinct organic dyes—Methylene Blue (MB), Bromophenol Blue (BB), Malachite Green (MG), and Crystal Violet (CV)—as water pollutants, dyes were treated with NPs. The experiment was conducted in September in the Centre for Converging Technologies lab on the University of Rajasthan campus. It was a sunny day with an outdoor temperature of 30-30 degrees Celsius. Different dye pollutants were added into a beaker containing 50 ml of distilled water to pollute the water. We filled the beakers with 100 mg of ZnO NPs. The photocatalytic degradation procedure involved exposing them to direct sunshine.

Two control sets were maintained simultaneously: (i) Polluted water without nanoparticles but exposed to sunlight; (ii) Polluted water with nanoparticles but not exposed to the sunlight and kept in dark.

Following predetermined time intervals, the pollutants' degradation with zinc oxide was visually monitored, and the quantity of leftover dye was calculated by measuring the pollutants' absorbance at respective wavelength. The step-wise experimental procedure adopted to evaluate the photocatalytic degradation of organic pollutants is shown in Fig. 2.

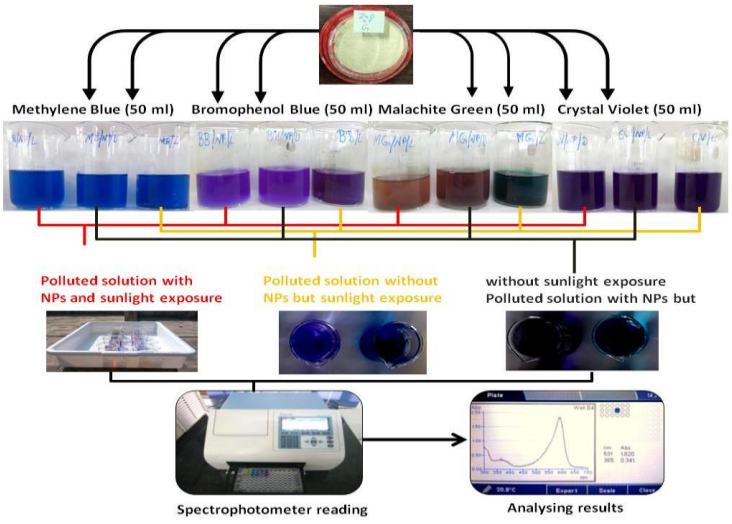


**Fig. 2:** Phases in the photocatalytic breakdown of organic dye pollutants

 

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## RESULTS

Zinc oxide nanoparticles synthesized applying a green method and leaf extract from *P. alba* L. During the synthesis process yellow colored precipitate was obtained which represents the synthesis of ZnO NPs. The appearance of Yellow-colored precipitate was also previously reported [15] using *Passiflora caerulea* L. (Passifloraceae) leaf extract and 1mM zinc acetate.

### Characterization of green synthesized ZnO Nanoparticles

**UV-Visible absorption spectroscopy-** UV-Visible absorption spectroscopy for the synthesized Zinc oxide nanoparticles exhibits a strong absorption band at about 355 nm. The UV-Vis absorption spectra of the biosynthesized ZnO NPs at room temperature shows a prominent absorption band at about 355 nm (Fig. 3).

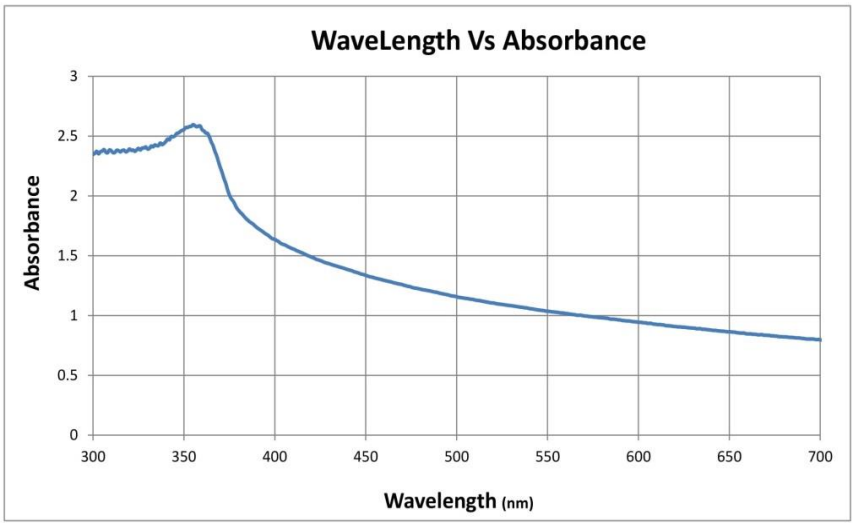


**Fig. 3:** Absorbance of green-synthesized zinc oxide nanoparticles (ZnO NPs) prepared using *P. alba* leaf extract by UV-Vis spectrophotometer

**FTIR of Zinc Oxide nanoparticle-** FT-IR was used to identify potential functional groups of biomolecules that could be involved in synthesizing ZnO NPs. Fig. 4(a) and (b) shows the FTIR spectra for ZnO NPs synthesized using *P. alba* L. leaf extract and for *P. alba* L. leaf extract respectively. The O-H and C-H stretching of alcohol and alkyl groups, respectively, are represented by the absorption bands at 3201 $cm^{-1}$ and 2866 $cm^{-1}$ in the FT-IR spectra of the leaf extract of *P. alba* L. (Fig. 4b). The conjugated alkenes' C=C stretching is shown by the absorption peak, which is situated at around 1602 $cm^{-1}$. The O-H stretching of carboxylic acid is correlated with stretching vibrations at 2655 $cm^{-1}$. C=O stretching in the ester complex is the cause of the stretching vibrations at 1745 $cm^{-1}$. Bands at 1390 $cm^{-1}$ showed phenol stretching vibrations. The C-N stretching of the amines is shown by the band at 1055 $cm^{-1}$.

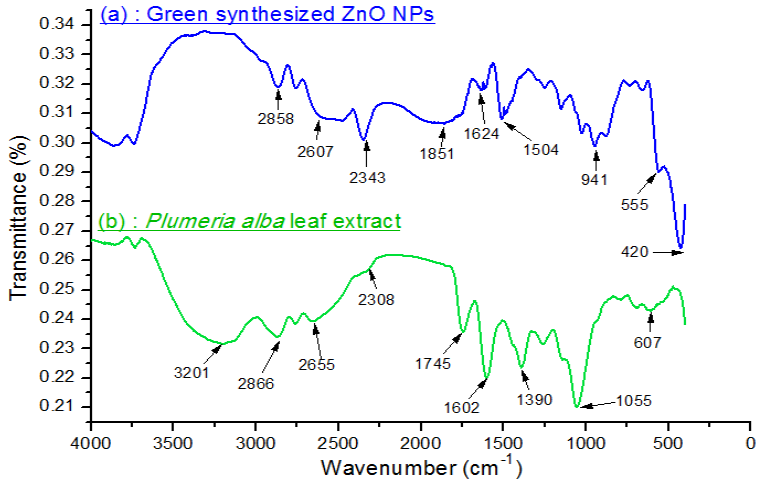


**Fig. 4:** FT-IR spectra (a) Green synthesized ZnO NPs (b) *P. alba* L. leaf extract

 

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**SEM and HRTEM-** The scanning electron microscopic (SEM) analysis, as depicted in Fig. 5, illustrates that ZnO nanoparticles exhibit an agglomerated structural morphology. The compact nature of these agglomerated nanoparticles can be attributed to the application of heat in the electric furnace, which contributes to their crystalline nature. This process also aids in the reorganization of the biomolecules responsible for capping and stabilizing the metal NPs synthesized from the leaf extract of *P. alba*.

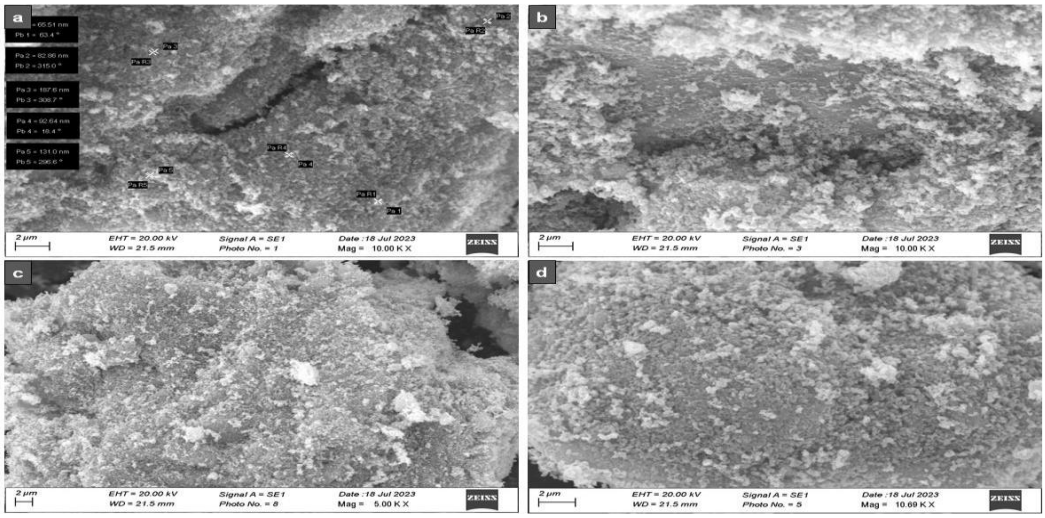


**Fig. 5:** Scanning Electron Microscopy of ZnO Nanoparticles

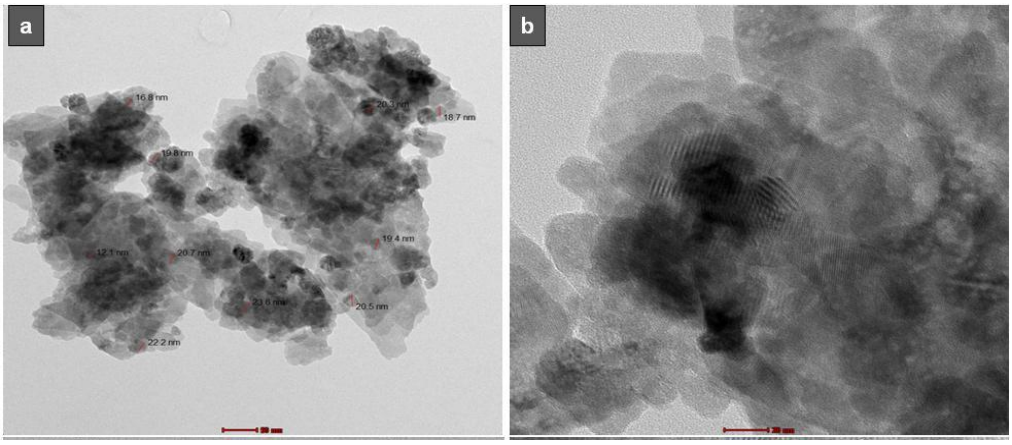


**Fig. 6:** HRTEM of ZnO nanoparticles

**Photocatalytic Degradation of Organic Pollutant-** At specific intervals, the degradation of dye pollutants with zinc oxide NPs was visually monitored, and the spectroscopic absorbance of the dye pollutants at their respective wavelengths was utilized to quantify the remaining dye concentration using a multiplate UV-Vis spectrophotometer.

In presence of ZnO NPs and under sunlight exposure, a gradual change in the color of polluted water was observed from colored to colorless water. Complete clean water was observed after 8 h, 12 h of solar irradiation for Methylene Blue and Bromophenol Blue polluted water, respectively and after 20 h for Malachite Green and Crystal Violet polluted water, indicating the significant degradation of dye pollutants by photocatalytic process.

The results obtained from spectrophotometry show that the dyes MB, BB, MG, and CV—which show prominent absorption peaks at 656 nm, 590 nm, 616 nm, and 582 nm, respectively—became invisible after 8, 12, 20, and 20 hours of exposure.

In the absence of ZnO NPs but under sunlight exposure, only a minor alteration in color and a slight reduction in the absorption spectrum of the polluted water were noted. This was likely due to the photolysis of dye pollutants by solar radiation. There was a little shift in the absorption spectra and colour in the presence of ZnO NPs but not in light. This was likely due to the adsorption of dye pollutants on the nanoparticle surface.

 

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Fig. 7 and 8 show color change observed for MB, BB, MG, CV polluted water under different experimental conditions. Additionally, Fig. 7 displays the absorbance spectrum vs. wavelength for various time intervals for Methylene blue, Bromophenol blue, Malachite green, and Crystal violet. All four dyes were examined for photocatalytic degradation using ZnO NPs. These NPs have the potential to effectively decompose all four dyes.

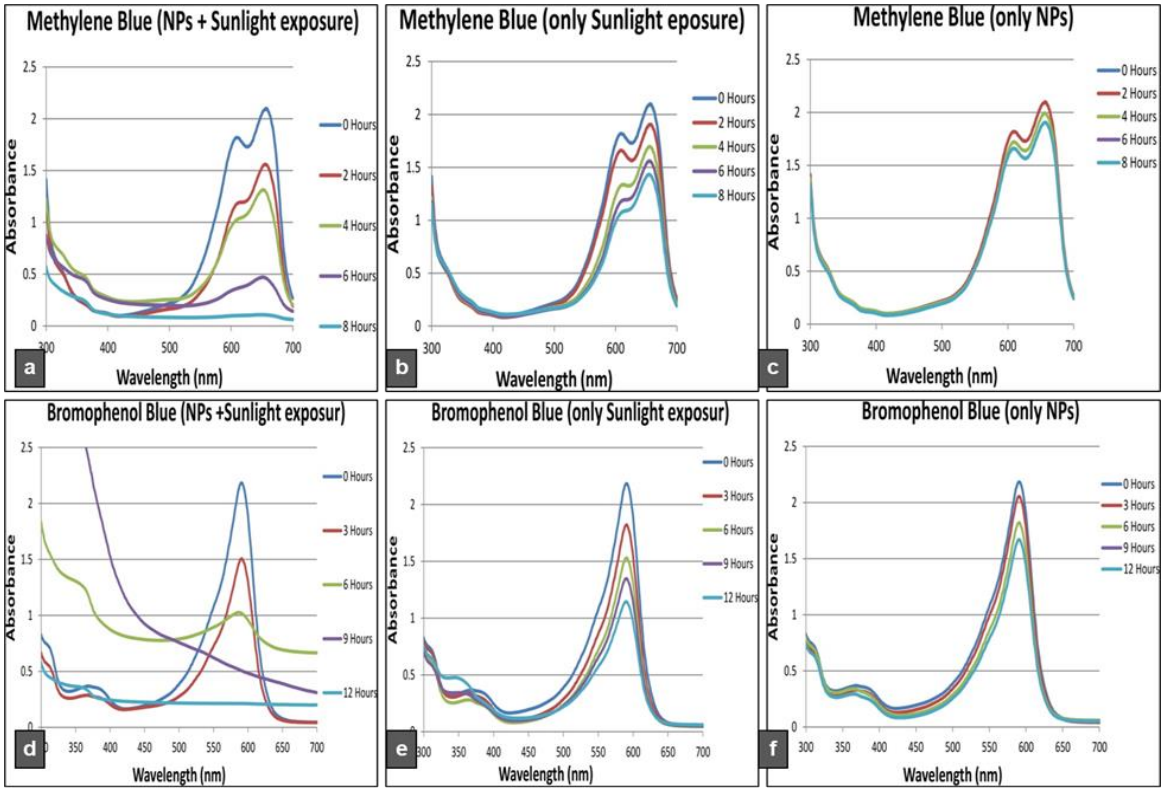


**Fig. 7:** Photocatalytic degradation of Methylene Blue- a: with Nanoparticles after sunlight exposure; b: without Nanoparticles after sunlight exposure; c: Nanoparticles without sunlight, and Bromophenol Blue-d: Nanoparticles and sunlight exposure; e: only sunlight exposure without Nanoparticles; f: only Nanoparticles without sunlight exposure.

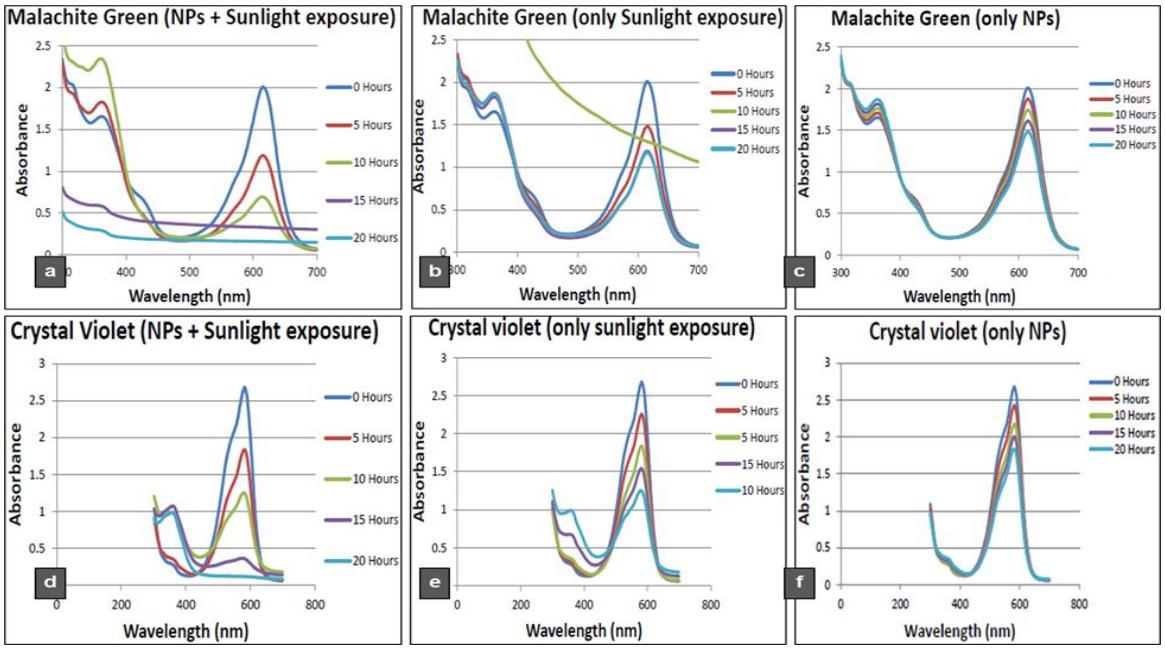


**Fig. 8:** Photocatalytic degradation of Melachite Green- a: with Nanoparticles after sunlight exposure; b: without Nanoparticles after sunlight exposure; c: Nanoparticles without sunlight, and Crystal Violet-d: Nanoparticles and sunlight exposure; e: only sunlight exposure without Nanoparticles; f: only Nanoparticles without sunlight exposure.

 

## DISCUSSION

The current study presents an efficient, secure, and cost-effective process for producing ZnO NPs in an ecologically friendly manner without the use of toxic or dangerous materials, utilising leaf extract from *P. alba* L. Proteins and phytochemicals containing carboxylic acid, ester, alcohol, or amine functional groups are involved in biosynthetic processes, according to the identification of the nanoparticles using UV and FTIR spectroscopy.

As the size of zinc oxide (ZnO) decreases from bulk to the nanometer scale, quantum confinement becomes increasingly significant. This results in a shift in the absorption spectrum of ZnO towards the blue side of the electromagnetic radiation spectrum. The band gap energy increases as wavelength decreases because of the quantum confinement. Therefore, the transition will be more energetic (blue-shifted). ZnO NP's band gap energy was estimated using the following equation:

$E_g = hc/\lambda$ [17]

h denotes Planck's constant (6.626× 10−34 Js), c signifies the speed of light (2.99× 108 m s−1), and λ represents the peak wavelength (355 nm). Additionally, 1.6×10-19 Joules is equivalent to 1 electron Volt (eV).

According to the abovementioned equation, the synthesized ZnO NPs employing *P. alba* have a band gap energy of 3.49 eV, suggesting the particles have undergone a blueshift relative to bulk ZnO (3.37 eV).

FTIR analysis of ZnO NPs synthesized from *Azadirachta indica* (L.) leaf extract [9] revealed shifts in the following bands: "3352 - 3429 $cm^{-1}$, 2391 - 2931 $cm^{-1}$, 1647 -1637 $cm^{-1}$, 1452 -1452 $cm^{-1}$, 1070 -1056 $cm^{-1}$, 771 -875 $cm^{-1}$, and 522 -457 $cm^{-1}$". This study confirms the involvement of polyols, terpenoids, and proteins containing functional groups such as amines, alcohols, ketones, and carboxylic acids in the bio-reduction processes [9]. As in the present study, the alteration observed in the FT-IR spectra of ZnO NPs after biosynthesis, particularly in the bands ranging from 2866-2858 $cm^{-1}$, 2655-2607 $cm^{-1}$, 2308-2343 $cm^{-1}$, 1745-1851 $cm^{-1}$, 1602-1624 $cm^{-1}$, 1390-1504 $cm^{-1}$, 1055-941 $cm^{-1}$, and 607-555 $cm^{-1}$, suggests the participation of proteins and phytochemicals containing functional groups such as carboxylic acid, ester, alcohols, and amines in the biosynthesis process. Fig. 4 shows the ZnO NPs' FTIR spectra produced using sustainable approaches. It has absorption bands at 2858 $cm^{-1}$, 2607 $cm^{-1}$, 2343 $cm^{-1}$, 1851 $cm^{-1}$, 1624 $cm^{-1}$, 1504 $cm^{-1}$, 941 $cm^{-1}$, 555 $cm^{-1}$, and 420 $cm^{-1}$, among other wavelengths. The band at 420 $cm^{-1}$ confirms that zinc oxide nanoparticles can stretch vibrations [18,19].

The first absorbance spectrum was recorded at zero time, and its peak value is the highest. The absorbance spectrum was recorded after illumination began, revealing that the peak value decreased. This demonstrates how the experiment's absorbance value decreases as time passes. The solution's blue, purple, brown, and violet hues—respectively for methylene blue, bromophenol blue, malachite green, and crystal violet—finally turn colourless after 8 hours, 12 hours, 20 hours, and 20 hours, when the absorbance peak becomes almost smooth. Methylene blue, a water contaminant, has been demonstrated to be photo-catalytically degraded by $TiO_2$ nanoparticles [20]. This study demonstrated photocatalytic degradation of bromophenol blue in an aqueous medium using three different chitosan conjugated magnetic nanoparticles (CCMN) of Co, Ni, and Fe synthesized by co-precipitation approach. According to their findings, all of the produced CCMN demonstrated good to exceptional photocatalytic properties, with Fe-CCMN (94.5%) showing the maximum degradation and Co-CCMN (85.1%) and Ni-CCMN (83.0%) showing the lowest degradation. Four batches of catalysts with excellent photocatalytic activity were produced by recycling and reusing them [21].

Aminuzzaman *et al.* [22] also discovered that all four dyes exhibited a little change in colour and absorption spectrum when exposed to sunlight without ZnO NPs and when ZnO NPs were present but not exposed to solar radiation. Malachite Green dye only experiences a 6.25% colour deterioration due to photolysis when exposed to natural sunshine "in the absence of biosynthesized ZnO NPs". MG's adsorption on ZnO NPs resulted in a degradation efficiency of just 13.72% for the identical MG dye solution with biosynthesized ZnO NPs "in the dark for 180 minutes". However, the MG dye solution's successful oxidation utilizing hydroxyl radicals (•OH) produced by the photocatalytic process resulted in a degradation efficiency of over 99% after 180 minutes of sun exposure.

## CONCLUSIONS

The study investigated using zinc oxide nanoparticles (ZnO NPs) to degrade organic dyes, including Methylene blue, Bromophenol blue, Malachite green, and Crystal violet, in water. The outcomes demonstrated the

potential photocatalytic capabilities of the synthesized ZnO NPs by displaying how well they degraded the dye contaminants when exposed to sunlight. This suggests that biosynthesized ZnO NPs can be an effective photocatalyst for degrading hazardous and toxic contaminants in aquatic environments.

The combination of nanotechnology and biotechnology, along with the use of catalysts and the unique properties of nanomaterials, has the potential to impact pollution reduction and water treatment significantly. In underdeveloped nations, access to clean drinking water has become increasingly challenging. Shortly, this affordable and safe technology holds out hope for the recovery of contaminated waterways and the supply of drinkable water.

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**Data collection-** Shubham Mathur, Vasudev Gautam
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**Literature search-** Preeti Mishra
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**Critical review-** Preeti Mishra
**Article editing-** Preeti Mishra
**Final approval-** Preeti Mishra

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