Synthesis of photocatalytic zinc oxide nanoflowers using *Peltophorum pterocarpum* pod extract and their characterization

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Abstract

Zinc oxide nanoflowers (ZnONFs) were prepared by employing the pod extract of *Peltophorum pterocarpum* as a green resource and characterized by various methods. UV–vis spectrum displayed a peak at 361 nm which confirmed the formation of ZnO nanoparticles. The optical band gap was calculated as 3.43 eV. FE-SEM images exposed the flower-like morphology and EDX portrayed strong signals for Zn and O. XRD studies substantiated signature peaks for the wurtzite phase of ZnONFs and the lattice parameters matched well with the literature. Mesoporous nature was confirmed by BET analysis which yielded a high specific surface area of 19.61 m²/g. FTIR bands at 420.48 and 462.92 cm⁻¹ affirmed the Zn and O bonding vibrations. The photocatalytic potential of the ZnONFs was successfully examined for the removal of methylene blue dye under natural solar light. The experimental data were fitted to Langmuir–Hinshelwood's first-order equation and the kinetic constant was calculated as 0.0114 min⁻¹.

Keywords Zinc oxide nanoflowers · *Peltophorum pterocarpum* · Methylene Blue dye · Photodegradation · Green synthesis

Introduction

Nanotechnology is emerging as a forerunner in many fields in recent times. Due to the unique physicochemical properties as compared to their bulk materials, the nanomaterials are extensively explored in various domains such as food, textile, cosmetics, electronics, medical sciences, energy, construction, and environmental remediation (Varadavenkatesan et al. 2020a, b; Pandey et al. 2020a). Zinc oxide nanoparticles (ZONPs) are one such valuable metal oxide nanoparticles which have reaped attention at present due to its unique characteristics like wide bandgap, catalytic efficiency, and nontoxic nature. ZONPs are extensively used as adsorbents, photocatalysts, anti-microbial agents,

drug-delivery agents, self-cleansing agents, and semiconductors (Mirzaei and Darroudi 2017).

Several conventional approaches such as chemical, physical, and electrochemical methods are used in the preparation of ZONPs. However, these popular strategies are not cost-effective and face difficulties during purification due to the formation of hazardous by-products. Moreover, the researchers also faced additional important problems such as the achievement of monodispersity and control over the morphology during the preparation of nanoparticles. To eliminate these problems, green resources such as plant extracts and microbial extracts were explored and proven as outstanding substitutes for the preparation of ZONPs (Vinayagam et al. 2020a). The synthesis processes involving these green resources are very rapid, cheap, and easily scalable. The various biomolecules present in these extracts reduce and stabilize the nanoparticles (Groiss et al. 2017). Few examples of green resources used for the preparation of ZONPs include *Linum usitatissimum* (Alkasir et al. 2020), *Moringa oleifera* (Letsholathebe et al. 2020), *Salvadora persica* (Alharthi et al. 2020), *Ilex paraguariensis* (Bandeira et al. 2020), *Sambucus spp.* (Alamdari et al. 2020) *Abelmoschus esculentus* (Mirgane et al. 2020), *Peltophorum pterocarpum* (Pai et al. 2019a), *Cyanometra ramiflora*

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(Varadavenkatesan et al. 2019) and *Calliandra haematocephala* (Vinayagam et al. 2020b).

Wastewaters loaded with industrial dye effluents are a major environmental concern (Pandey 2017). An example of a cationic dye in methylene blue that has far-reaching applications in diverse areas such as textile, paper and pharmaceutical sectors. It is imperative that the dye is sufficiently removed prior to the waters entering the aquatic bodies as the dye poses critical ecological and health concerns (Pandey et al. 2020b). The usual methods employed for dye elimination (e.g. adsorption) suffer from a lot of disadvantages including cost and pace of the dye transfer. Our research group has already explored the usage of the *Peltophorum pterocarpum* pod extract to prepare silver (Raja et al. 2015a) and iron oxide nanoparticles (Dash et al. 2019). As demonstrated in these reports, the biomolecules of the extract were responsible for the reduction and stabilization of the nanoparticles.

Therefore, in the current study, we focused on the (1) fabrication of ZONPs by utilizing the *P. pterocarpum* pod extract, (2) characterization, and (3) photocatalytic ability to decolorize a model pollutant—methylene blue (MB) dye in the presence of solar irradiation.

Materials and methods

Materials

Analytical grade zinc acetate dihydrate, NaOH, and MB dye were purchased from Merck, India, and used without any further purification. Distilled water was used for all the experiments. Fresh pods of *P. pterocarpum* tree were collected from Manipal, India.

Peltophorum pterocarpum pod extract preparation

P. pterocarpum pod extract (PPPE) was prepared by referring to our earlier reports (Dash et al. 2019; Raja et al. 2015b). Briefly, the pods of *P. pterocarpum* were first washed with distilled water to remove the surface dirt and then air-dried. The cleaned pods were boiled with water (1:10) to yield a light-yellow extract indicating the release of the polyphenols from the plant material. After cooling and filtering, the extract was collected and preserved in the refrigerator for future use.

Production of zinc oxide nanoflowers (PP-ZnONFs)

Nanoparticles were synthesized by mixing 50 mL of zinc acetate dihydrate (50 mM) and 10 mL of PPPE and subsequent addition of 10 mL NaOH (2 M) to form colloidal suspension on heating. Settling of suspension was observed

in a course of time resulting in precipitated ZnONFs. After multiple water-washes, the particles were centrifuged and oven-dried (at 95 °C for 7 h) to obtain pure PP-ZnONFs. The dried powder was pulverized using a mortar and pestle and stored in a plastic vial.

Photocatalytic activity of PP-ZnONFs to degrade MB dye

The photocatalytic potential of the PP-ZnONFs was studied by the degradation of MB dye under natural solar light. The photocatalytic experimentations were performed in a glass beaker. Initially, 40 mg of PP-ZnONFs were mixed with 100 mL of MB dye (25 ppm) and stirred uniformly for 1 h in dark to finish adsorption–desorption process. Afterward, the contents were placed under solar irradiation on a sunny day between 10:30 AM and 3:00 PM with constant stirring. At fixed time intervals, samples were collected and the MB dye concentrations were analyzed by recording the absorption intensity ($\lambda_{\text{max}} = 663 \text{ nm}$) using UV–vis spectroscopy.

Characterization

The synthesized PP-ZnONFs were characterized using many sophisticated methods. The synthesis of PP-ZnONFs was monitored by the absorption spectrum of colloidal suspension using a UV–visible spectrophotometer (SHIMADZU—UV1700). Field Emission Scanning Electron Microscope (FE-SEM) (Carl ZEISS, Sigma, Oxford Instruments) was used to examine the individual elements and morphology of the PP-ZnONFs. Pore volume and specific surface area (SSA) were determined using the Brunauer–Emmett and Teller (BET) apparatus (Smart Instruments, Mumbai). X-ray diffraction (Rigaku Miniflex 600) spectrum was used to analyze the crystalline type. Fourier Transform Infrared Spectroscopy (FTIR-SHIMADZU-8400S) was used to investigate the specific functional moieties of the PP-ZnONFs.

Results and discussions

Visual examination and UV–vis analysis

The confirmatory spot check for the development of PP-ZnONFs was the color change of the reaction mixture when the pale yellow PPPE (Fig. 1A) was mixed with the colorless zinc acetate solution (Fig. 1B). This leads to the formation of a colloidal suspension (Fig. 1C) upon heating. The subsequent addition of NaOH leads to a white precipitate (Fig. 1D) in due course. These observations were in concordance with our earlier reports for the fabrication of ZnONPs using various green resources (Varadavenkatesan et al. 2019; Vinayagam et al. 2020b; Pai et al. 2019).

Fig. 1 Formation of PP-ZnONFs **(A)** *P. pterocarpum* pod extract. **B** Zinc acetate solution. **C** Colloidal suspension of ZnONFs. **D** Precipitated ZnONFs

As mentioned earlier, the PPPE contains various types of biomolecules that combine with the metallic salt, zinc acetate to form an intermediate complex through hydrogen bonding (Suresh et al. 2018). Under the basic environment, this forms metal hydroxide which yields zinc oxide nanoparticles while heating. The reduction of Zn was possible and spontaneous because of the fact that the standard reduction potential of biomolecules of plant materials is between 0.3 and 0.8 V whereas for Zn it is just -0.76 V (Haynes 2014). Apart from the reduction, the biomolecules help in the stabilization of nanoparticles by self-assembling and lead to the formation of flower-like nanostructures. A similar mechanism was described for the ZnONFs synthesized using *Bacillus licheniformis* extract and zinc acetate precursor (Tripathi et al. 2014).

Following the visual observation, the PP-ZnONFs development was confirmed by making a PP-ZnONFs dispersion in water and conducting UV–vis spectral examination of the same. As evinced from Fig. 2, a distinctive absorption peak was observed at 361 nm due to the surface plasmon

resonance (SPR) of zinc oxide nanoparticles (Varadavenkatesan et al. 2019). The absence of other peaks substantiated the purity of the ZnONFs. The surface plasmon resonance (SPR) of the ZnONPs alters the particle morphology and dielectric constant of the response channel (Chakraborty et al. 2020). The optical bandgap of PP-ZnONFs was calculated using the Planck-Einstein relationship and found out as 3.43 eV. The value obtained was concordant with the previously reported literature. For example, bandgap values of 3.4 eV and 3.44 eV were obtained in studies using *Jatropha gossypifolia* leaf extract and *Cyanometra ramiflora* leaf extracts (Varadavenkatesan et al. 2019; Krishnan et al. 2020).

SEM and EDX studies

The FESEM image of PP-ZnONFs with the magnification of 20 kX and 80 kX are shown in Fig. 3A and B. Aggregated flower-shaped ZnO-NFs were observed. Clusters of particles were observed due to crystal growth. The

Fig. 2 UV–visible spectroscopic analysis of PP-ZnONFs

formation of aggregations was possibly owing to the divergence, electrostatic attraction, and high surface energy PP-ZnONFs (Darvishi et al. 2019). The presence of organic compounds in the extract may also cause agglomeration (Vinayagam et al. 2020b). The average thickness of the nanoflower petals was calculated as 59 nm using ImageJ software. The observation of the nanoflower-shaped zinc oxide nanoparticles is common for the green synthesis using the extracts of *P. pterocarpum* (Pai et al. 2019), *E. sanguinea* (Ekennia et al. 2020) and *W. coagulans* (Hasan et al. 2021).

The EDX spectra (Fig. 3C) showed the presence of zinc, oxygen, and carbon atoms. To be specific, Zn indicated strong signals at 8.6 keV and 1 keV. Similarly, O exhibited a strong signal at 0.5 keV. Therefore, the existence of these signals corroborated the establishment of ZnO nanoparticles. The C signal might be because of the participation of phytochemicals during the synthesis and capping of ZnONFs (Pai et al. 2019).

The higher weight percentage of Zn (66.59%) and O (24.29%) witnessed the presence of ZnO nanoparticles. Also, the atomic compositions (%) of the individual elements in the sample were observed as, Zn: 30.91, O: 46.06, and C: 23.03. Similar kinds of atomic compositions (%), Zn: 36, O: 44.85 and C: 15.68 were reported for the synthesis of ZnONPs by utilizing *Justicia spicigera* leaf extract (Soto-Robles et al. 2021). The purity of the ZnO nanoparticles was ascertained by the non-appearance of other peaks.

XRD studies

The XRD image of the sample with the Miller indices of peaks is depicted in Fig. 4. The diffraction signals at the 2θ values of 31.31° , 34.06° , 35.84° , 47.20° , 56.23° , 62.48° , 67.62° , 68.74° and 76.74° corresponded to (1 0 0), (0 0 2), (1 0 1), (1 0 2), (1 1 0), (1 0 3), (1 1 2), (2 0 1) and (2 0 2) Miller indices which were precisely indexed to the pure hexagonal wurtzite phase of zinc oxide nanoparticles (JCPDS Card No.: 36–1451) (Ekennia et al. 2020). The lattice parameters ‘a’ and ‘c’ were computed as 3.269 Å and 5.2092 Å, respectively, which matched with the literature (Pudukudy and Yaakob 2015). The ratio of the lattice parameters ‘c/a’ was equal to 1.594 which matched well with the ZnONPs synthesized using the flower extract of *Aspalathus linearis* (Diallo et al. 2015).

The sharp and distinct diffraction signals indicated the high crystalline nature of the nanoparticles. No other signals were observed. The full width at half maximum (FWHM), ‘ β ’ of these peaks yielded a mean crystallite size of 17.79 nm (Table 1) calculated by the Debye–Scherrer equation which matched well with previous studies (Alamdari et al. 2020; Vinayagam et al. 2020b).

BET studies

It is well-known that the flower-like ZnONPs have more SSA as compared to other morphological forms (Luo et al. 2014). The SSA and pore volume were analyzed by BET

Fig. 3 **A** FESEM image of PP-ZnONFs at 20 kX magnification. **B** FESEM image of PP-ZnONFs at 80 kX magnification and **C** EDX spectrum of PP-ZnONFs

method. As expected, the PP-ZnONFs showed a high SSA of $19.61 \text{ m}^2/\text{g}$ which was almost 6 times higher than that of commercial ZnO nanoparticles ($3.23 \text{ m}^2/\text{g}$) (Chakrabarti and Dutta 2004). It is apparent from Table 2 that the SSA obtained in the present work is much higher than the other green synthesized ZnO nanoparticles. Besides, the pore volume was $0.0826 \text{ cm}^3/\text{g}$ which confirmed the mesoporous structure (16.85 nm) of pores. Therefore, the high surface area and mesoporous structure of PP-ZnONFs make them vital representatives for many applications.

FTIR studies

The composition of the PP-ZnONFs, with respect to the functional moieties, was deduced from the FTIR spectrum (Fig. 5). The broadband in the range of $3400\text{--}3600 \text{ cm}^{-1}$ was mainly due to the stretching vibration of the phenolic hydroxyl group (Darvishi et al. 2019; Soto-Robles et al. 2021; Lu et al. 2019). The band at 2359 cm^{-1} was due to the C–H stretching

vibrations of aromatic aldehyde (Akbarian et al. 2020). A weak band at 1541 cm^{-1} corresponded to C=C stretching bonds of aromatic compounds in the extract (Vinayagam et al. 2020b; Hu et al. 2019). The band at 1175 cm^{-1} belonged to C–O stretching (Darvishi et al. 2019; Chen et al. 2019).

The bands at 637, 718, and 781 cm^{-1} in the fingerprint region corresponded to aromatic C–H groups in the extract (Darvishi et al. 2019). The bands at 421 and 463 cm^{-1} may be attributed to characteristic Zn and O bonding vibrations (Suresh et al. 2018; Li et al. 2019; Gopalakrishnan et al. 2019). Therefore, the signals for various functional moieties ascertained the contribution of the phytochemicals present in the PPPE for the formation of ZnONFs.

Photocatalytic activity of PP-ZnONFs to degrade MB dye

The UV–vis spectral images of photodegradation of MB dye by PP-ZnONFs were shown in Fig. 6A along with the

Fig. 4 XRD spectrum of PP-ZnONFs **Table 1** Computation of average crystallite size of PP-ZnONFs

S.no	2θ (°)	"h k l" plane	β (°)	Crystallite size (nm)
1	31.31	1 0 0	0.4742	19.33
2	34.06	0 0 2	0.3691	25.01
3	35.84	1 0 1	0.5169	17.95
4	47.20	1 0 2	0.5246	18.36
5	56.23	1 1 0	0.7004	14.288
6	62.48	1 0 3	0.6108	16.90
7	67.62	1 1 2	0.6374	16.67
8	68.74	2 0 1	0.7737	13.82
9	76.74	2 0 2	0.6329	17.79
				Average = 17.79

discoloration of MB dye as a function of time. It can be witnessed from the images that the MB dye solution became colorless after 180 min of solar irradiation. The decrement in the absorbance of MB dye at $\lambda_{\max} = 663$ nm substantiated the degradation process. From Fig. 6B, it can be inferred that 88.39% of the MB dye was photodegraded in 180 min solar irradiation.

It can be inferred from Table 3 that the degradation efficiency of MB dye by the PP-ZnONFs is much higher than the recent reports on the green synthesized ZnO nanoparticles. This enhanced photocatalytic activity of the PP-ZnONFs may be due to the small particle size, mesoporosity, and high specific surface area.

The kinetic data of the photodegradation was fitted to the Langmuir–Hinshelwood first-order equation as given by,

Table 2 BET surface area of various green synthesised ZnO nanoparticles

Source	SSA (m ² /g)	Ref
<i>Calliandra haematocephala</i> leaf	9.18	Vinayagam et al. (2020b)
<i>Musa balbisiana</i> peel	11.402	Tamuly et al. (2015)
<i>Peltophorum pterocarpum</i> leaf	13.56	Pai et al. (2019)
<i>Cyanometra ramiflora</i> leaf	16.27	Varadavenkatesan et al. (2019)
<i>Peltophorum pterocarpum</i> pod	19.61	This study

Fig. 5 FTIR spectrum of PP-ZnONFs

$$\ln \left[\frac{A_f}{A_0} \right] = -k_{\text{phd}} t$$

where “ t ” is the solar irradiation time in min, A_0 and A_f are the initial and final concentration of MB dye and k_{phd} is the degradation constant. Figure 6C shows the relationship between $\ln \left[\frac{A_f}{A_0} \right]$ and t with linear relationship and the value of k_{phd} was determined as 0.0114 min^{-1} .

The incident sunlight has more energy than the bandgap energy of PP-ZnONFs. Therefore, as depicted in Fig. 7 the photocatalytic mechanism involves the creation of electrons (e^-) in the conduction band (CB) and holes (h^+) in the valance band (VB) when sunlight irradiates on the surface of PP-ZnONFs. These generated e^- converts dissolved O_2 to form superoxide free radicals (O_2^-) and h^+ produce $\cdot\text{OH}$ free radicals. These intermediates are highly active which act as oxidizing and reducing agents and thus are responsible for the photodegradation of MB dye as explained by many

researchers (Tabrizi Hafez Moghaddas et al. 2019; Ganesh et al. 2019; Pandimurugan and Thambidurai 2016).

Conclusion

Green synthesized Zinc oxide nanoflowers (ZnONFs) have been synthesized by utilizing the pod extract of *Peltophorum pterocarpum* for the first time. The formation of ZnO nanoparticles was confirmed by the characteristic signals shown in UV–vis spectrum, EDX image, XRD pattern, and FTIR spectrum. FE-SEM image confirmed the flower-like nanostructures with few aggregations and BET analysis ascertained the mesoporosity with a relatively higher specific surface area than commercial ZnO nanoparticles. 88.39% of MB dye was photodegraded under sunlight within 180 min in the presence of ZnONFs. Therefore, the green-synthesized ZnONFs obtained in the present study can play a significant role in the removal of harmful dyes present in industrial wastewater.

Fig. 6 Photocatalytic degradation of MB dye by PP-ZnONFs. **A** UV-vis spectra and color change of MB dye under different time intervals. **B** Plot of % degradation of MB dye vs time. **C** Plot of $\ln(A_t/A_0)$ vs time

Table 3 Recent reports on the photocatalytic potential of various green synthesised ZnO nanoparticles to degrade MB dye

S.no	Green resource	Irradiation source	Time of degradation (min)	Degradation (%)	Ref
1	<i>Cassia fistula</i>	Sunlight	120	50	Suresh et al. (2015)
2	<i>Suaeda japonica</i> Makino	UV light	60	54	Shim et al. (2019)
3	<i>Linum usitatissimum</i>	UV light	120	80	Tabrizi Hafez Moghaddas et al. (2019)
4	<i>Bacillus licheniformis</i>	UV light	60	83	Tripathi et al. (2014)
5	<i>Camellia sinensis</i>	UV light	120	84	Nava et al. (2017)
6	<i>P. pterocarpum</i> pods	Sunlight	180	88.39%	This study

Fig. 7 Mechanism of photocatalytic degradation of MB dye by PP-ZnONFs

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Data availability The raw/processed data required to reproduce these findings cannot be shared at this time as the data also forms part of an ongoing study.

Declarations

Conflict of interest The authors declare that they have no conflict of interest.

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