Characterization and photocatalytic activity of ZnO nanoflowers synthesized using *Bridelia retusa* leaf extract

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Abstract

In the current work, the leaf extract of *Bridelia retusa* was used for the first time to synthesize zinc oxide nanoparticles (ZnONPs). A zinc nanoparticle-specific 364-nm peak was discerned via UV–Vis studies with a typical bandgap energy of 3.41 eV. FE-SEM micrographs revealed flower-shaped structure of the ZnONPs. EDS analysis corroborated the presence of zinc and oxygen. XRD spectrum established the wurtzite structure, sized at 11.06 nm. The mesoporous texture (4.89 nm) of the nanoparticles was deduced from BET analysis, proving a higher specific surface area than commercial ZnONPs. FTIR spectroscopy resulted in absorption bands typical for ZnONPs. Within a span of 165 min, under solar irradiation, the ZnONPs facilitated the photocatalytic degradation of Rhodamine B dye upto 94.74%. Exhibiting pseudo-first-order kinetics, the process had a degradation constant of 0.0109 min⁻¹. It was concluded that numerous factors led to the high degradation efficiency. High values of bandgap energy and specific surface area, along with the mesoporous and crystalline nature of the ZnONPs led to the observed effect. The ZnONPs were also stabilized by the phytochemicals in the *B. retusa* leaves. The study is thus able to successfully demonstrate the huge potential in the field of environmental nanoremediation. The viability of using ZnONPs as solar photocatalysts for treating dye-laden industrial wastewater was thus attested.

Keywords Zinc oxide nanoflowers · *Bridelia retusa* · Rhodamine B dye · Photodegradation · Green synthesis

Introduction

Nanomaterials have shown magnificent applications ranging from technology to biological science, to produce useful products. The nanoparticles have a large surface-to-volume ratio which makes them suitable for various sectors such as energy, medical, cosmetics, electronics, catalysis,

wastewater treatment, food, and agriculture. One of the important metal oxide nanoparticles is zinc oxide nanoparticles (ZnONPs) which have distinct features such as sensing, UV blocking, thermal and chemical stability, and biocompatibility. Moreover, it has high bandgap energy with enormous binding energy and hence, utilized in different fields, including cosmetics, medicine, and environmental remediation (Kadam et al. 2020). Recently, green synthesis has grown to be a key motivating force for nanoparticle synthesis (Hasan 2021). The release of massive quantities of colorants in the water bodies is a major environmental crisis (Varadavenkatesan et al. 2020a, b). Different types of dyes are discharged from various industries which make undesirable impacts on living beings (Chakraborty et al. 2020). It is essential to remove these dyes from wastewater before entry into water bodies (Sharifpour et al. 2018). Among various dye-removal technologies, a popular technique is advanced oxidation process (AOP) that relies on highly reactive oxidants to destroy organic pollutants till their mineralization produces CO₂ and H₂O as end products.

The synthesis of ZnONPs is mostly achieved by expensive chemical and physical routes. Moreover, the chemical

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methods utilize toxic chemicals and the release of these chemicals in effluents is an environmental concern (Bandeira et al. 2020). To avoid such problems, there is a necessity to explore cheap and eco-friendly methods for the synthesis of nanoparticles. Plant extracts are more efficient due to their accessibility and the possession of several metabolites in them, helping to reduce Zn^{2+} to ZnONPs (Akbarian et al. (2020)). Moreover, the plant extracts are enriched with wide ranges of phytochemicals like polyphenols and flavonoids, deemed to be bio-reducing agents with no negative effects on the environment (Shayegan Mehr et al. 2018). Their use as reducing and capping agents has led to recognition as biologically benign materials for the formulation of eco-friendly and low-cost nanoparticles (Manjari et al. 2019; Krishnan et al. 2020). Rhodamine B (RhB) is a water-soluble dye, used as a coloring agent in many industries. The release of RhB-contaminated wastewaters may cause reproductive and nervous systems disorders and hence, it is essential to remove RhB dye from wastewater (Sonker et al. 2020). Photocatalysis is one AOP process, comprising the effective use of light energy to generate hydroxyl radicals for the degradation of dyes (Bishnoi et al. 2018; Sirdeshpande et al. 2018). Being cheap, non-toxic, and able to absorb a larger fraction of solar irradiation, the ZnONPs are preferred over TiO_2 and Al_2O_3 —the conventional photocatalysts (Ong et al. 2018). Besides, the ZnONPs have the same bandgap energy similar to TiO_2 .

Few recent reports on the synthesis of ZnONPs include the use of various plant extracts such as *Azadirachta indica* (Haque et al. 2020), *Ilex paraguariensis* (Bandeira 2020), *Camellia sinensis* L (Akbarian et al. 2020), *Acacia caesia* (Manjari et al. 2019), *Jatropha gossypifolia* (Krishnan et al. 2020), *Lippia adoensis* (Demissie et al. 2020), *Justicia spicigera* (Soto-Robles 1225), and *Averrhoa carambola* (Chakraborty et al. 2020). In addition, our research group also has published leaf extract-mediated ZnONPs using *Calliandra haematocephala* (Vinayagam et al. 2019), *Cynometra ramiflora* (Varadavenkatesan et al. 2019), and *Peltophorum pterocarpum* (Pai et al. 2019a). Already our research group has reported the potential of *Bridelia retusa* leaf extract to act as a reducing and capping agent for the synthesis of silver nanoparticles (Ramesh et al. 2017). There are reports available in the literature to utilize various plant extract-mediated ZnONPs as photocatalysts to degrade harmful dyes such as methylene blue (MB) (Haque et al. 2020; Vinayagam et al. 2019; Pai et al. 2019a), RhB (Varadavenkatesan et al. 2019), Basic Red 51 (Yashni et al. 2020), Congo red (Chakraborty et al. 2020), and other pollutants like p-nitrophenol (Kadam et al. 2020).

Taking into cognizance the current state of work, in the present study, we report the synthesis of ZnONPs using the *B. retusa* leaf extract. Post synthesis, we characterize the zinc oxide nanoparticles by various analytical methods.

Moreover, we demonstrate the photocatalytic degradation of RhB dye under sunlight in the presence of the synthesized ZnONPs.

Materials and methods

Materials

Zinc acetate dihydrate (Merck, India) was used as the zinc precursor. RhB dye and sodium hydroxide (analytical-grade) were procured from Merck, India. Distilled water was used for all the experiments.

Preparation of *Bridelia retusa* leaf extract

Healthy leaves of *B. retusa* were collected from Manipal, India, and rinsed with tap water initially, followed by distilled water-wash. 10 g of leaves were added to 200 mL of water and heated at 80 °C for 15 min to produce a pale-brown broth, labelled as BRLE.

Preparation of *Bridelia retusa*–facilitated ZnONPs

0.05 M zinc acetate, BRLE, and 1 N NaOH were mixed at a volume ratio of 2.5:1:1 yielding a creamy suspension. The resulting contents were heated for 1 h at 80 °C, forming an ivory precipitate and thus indicating the formation of ZnONPs. Later, the precipitate was removed by centrifugation with multiple water-washes and dried at 90 °C overnight, which resulted in a white powder, termed “BR–ZnONFs”.

Photodegradation of RhB dye under sunlight

The photodegradation ability of the BR–ZnONFs was examined by the degradation of RhB dye in the presence of sunlight. The experiments were performed by taking 100 mL of RhB dye (13 μM) in a conical flask with 40 mg BR–ZnONFs and magnetically mixed in dark for 1 h to obtain a homogeneous suspension. Later, the flask was exposed to natural sunlight irradiation with constant stirring on a sunny day during October 2020. The absorbance spectra of the samples withdrawn at fixed time intervals were recorded and the concentrations were calculated using the absorbance data ($\lambda_{\text{max}} = 553 \text{ nm}$).

Characterization of BR–ZnONFs

The BR–ZnONFs were characterized by various techniques. The formation of ZnONPs and the concentration of RhB dye at regular intervals were examined by a UV–vis spectrometer (Shimadzu, UV-1800). The morphological

characteristics and elemental compositions were analysed using a Field Emission Scanning Electron Microscope (FE-SEM) (Carl ZEISS, Sigma, Oxford Instruments) coupled with Energy Dispersive X-ray Spectroscopy (EDS). For the phase analysis and crystallinity, an X-ray diffractometer (Rigaku, Miniflex 600) was used. Fourier Transform Infrared (FTIR) spectrophotometer (Shimadzu, 8400S) was used for the analysis of functional groups present on the surface of BR–ZnONFs. The porous nature and the specific surface area of the BR–ZnONFs were examined by the Brunauer–Emmett–Teller (BET) apparatus (Smart Instruments, Mumbai).

Results and discussions

Visual monitoring and UV–Vis studies

The formation of an ivory-colloidal suspension (Fig. 1c) was the indication of the incipient formation of ZnONPs, when the colourless 0.5 M zinc acetate solution (Fig. 1a), 1 M NaOH, and pale-brown BRLE (Fig. 1b) were mixed. Heating the contents at 80 °C for 1 h precipitated the nanoparticles (Fig. 1d) which were further purified and dried at 90 °C overnight. The white-powder (Fig. 1e) obtained was designated as BR–ZnONFs.

The UV–Vis spectra of the diluted BRLE and 0.1 wt% suspension of BR–ZnONFs are shown in Fig. 2. The BRLE

(Fig. 2a) showed a broad peak at 276 nm which may be assigned to the phytocompounds present in the leaf extract (Groiss et al. 2017). This peak was not present in the spectrum of BR–ZnONFs (Fig. 2b) ascertaining the interaction between the phytocompounds and zinc acetate to form nanoparticles. The interaction was due to the reduction potential values of zinc and phytocompounds as explained in the literature (Vinayagam et al. 2019; Varadavenkatesan et al. 2019). Besides, Fig. 2b shows a peak at 364 nm—a characteristic feature of ZnONPs which may be due to the surface plasmon resonance or the transfer of electrons from the valence to the conduction band (Rambabu et al. 2020). The peak obtained in this study was in concordance with our earlier study for the green synthesis of ZnONPs using *P. pterocarpum* leaf extract wherein a peak at 365 nm was observed. Planck–Einstein equation was used to determine the bandgap energy and was calculated as 3.41 eV, similar to the *Jatropha* gum-mediated ZnONPs (Geetha et al. Sep. 2016).

FE-SEM–EDS analysis

FE-SEM images (Fig. 3a, b) revealed ‘flower-shaped’ zinc oxide nanoparticles. These asteroidal uniform nanoparticles homogeneously exuded out from the center, being powerfully held to the spherical pedicle and providing an almost precise 3D alignment (Hasan 2021). The alkaline conditions used during the synthesis process formed flower-like ZnONPs. Flower-like structures are very

Fig. 1 Synthesis process of BR–ZnONFs

Fig. 2 UV–Vis profile of the *Bridelia retusa* leaf extract and BR–ZnONFs

common for ZnONPs synthesized using plant extracts as reported by many researchers such as the extracts of *Carissa edulis* (Fowsiya et al. 2016), *Codonopsis lanceolate* (Lu May 2019), *Laurus nobilis* L. (Fakhari et al. 2019), *Juglans regia* L. (Darvishi et al. 2019), *Calliandra haematocephala* (Vinayagam et al. 2019), *Cyanometra ramiflora* (Varadavenkatesan et al. 2019), and *Peltophorum pterocarpum* (Pai et al. 2019a). The sheets had massive structural and surface defects making BR–ZnONFs porous materials. These defects play a significant role in the photocatalysis by acting as extra sites during photocatalytic degradation (Din et al. 2020). The agglomerations noticed in the images may be due to the interactions between the BR–ZnONFs and of the phytocompounds present in the leaf extract (Vinayagam et al. 2020).

The EDS image (Fig. 3c) showed strong signals for Zn (at 1 & 8.63 keV) and O (at 0.53 keV), confirming the existence of ZnO in the sample. As depicted in Fig. 3c, the weight % composition of Zn and O are 63.57% and 26.49%, respectively, which substantiated the presence of ZnO. The composition of the BR–ZnONFs matched well with the ZnONPs synthesized using the extracts of *J. spicigera* (Soto-Robles 1225). Also, a peak for C (at 0.27 keV) was witnessed which may be due to the contribution of phytocompounds during the formation of ZnONPs. Similar results for the EDS of green synthesized ZnONPs have been described in earlier work (Rambabu et al. 2020).

XRD studies

The XRD pattern of the BR–ZnONFs is exemplified in Fig. 4 which confirmed the crystalline nature and orientation of zinc oxide nanoflowers. The sharp peak indicated the smaller size of the formed BR–ZnONFs (Praveen et al. 2019). The peaks at 2θ magnitudes of 31.53° , 34.13° , 35.97° , 47.21° , 56.28° , 62.62° , 67.56° , and 76.82° were indexed to (100), (002), (101), (102), (110), (103), (112), and (202) planes which matched with JCPDS-36–1451 and confirmed the formation of a crystalline hexagonal assembly (Awwad et al. 2020). No additional signals were noticed signifying the purity of the BR–ZnONFs. The average crystallite size was calculated using Scherrer formula (Vinayagam et al. 2017) and was estimated as 11.06 nm, concordant with our previous study (Pai et al. 2019b). The lattice parameters were calculated conferring to the Bragg's equation and reflecting the hexagonal structure of ZnONPs. The parameters 'a' and 'c' were estimated at 3.27 Å and 5.25 Å for samples, corroborating with the literature (Bandeira 2020).

BET studies

The BET specific surface area (SSA) of the BR–ZnONFs was estimated as 6.71 m²/g, almost two times higher than the commercial ZnONPs (3.23 m²/g) (Chakrabarti and Dutta 2004). Moreover, the SSA obtained in this study was comparatively higher than the ZnONPs synthesized by chemical method (5.178 m²/g) (Duo et al. 2016). Besides, the pore

Fig. 3 FE-SEM image with 40kX (A), 100kX magnifications (B), and EDS image of BR–ZnONFs (C)

Fig. 4 XRD pattern of the BR–ZnONFs

diameter was determined as 4.89 nm which confirmed the mesoscopic nature of the nanoparticles. Hence, the high surface area and mesoscopic nature of the BR–ZnONFs may play a pivotal part in various applications such as catalysis, photocatalysis, and wastewater treatment.

FTIR studies

FTIR analysis at room temperature was performed for the BR–ZnONFs to assess the functional groups present in it. The biomolecules present in the extract play an imperative role in reducing and stabilizing the nanoparticles which can be recognized by FTIR analysis (Anand et al. 2019). The FTIR spectrum (Fig. 5) shows strong bands (cm^{-1}) at 3369.64, 2358.94, 1649.14, 1548.84, 1419.61, 1041.56, 723.31, 634.58, and 422.41. The bands between 400 and 800 cm^{-1} were designated to Zn and O bonding vibrations

confirming the formation of ZnONPs (Bhuyan et al. 2015). The remaining bands endorsed the presence of various functional groups such as alcoholic, ketonic, alkanes, amines, and other carbonyl groups which belonged to the phyto-compounds of BRLE. Table 1 summarizes the various FTIR bands and the functional groups to which they correspond. The presence of these functional groups corroborated the participation of the phytochemicals of BRLE to form BR–ZnONFs from zinc acetate.

Photodegradation of RhB dye under sunlight

The photocatalytic degradation of RhB dye under sunlight irradiation was examined in the presence of BR–ZnONFs. The time-dependent absorption spectra of RhB dye is shown in Fig. 6. The fading-away of the RhB dye color can be witnessed from Fig. 6A confirming the dye degradation

Fig. 5 FTIR spectrum of BR–ZnONFs

Table 1 FTIR bands and their corresponding groups

Wavenumber (cm^{-1})	Band assignments	Reference
3369.64	O–H stretching band may be due to polyphenols or adsorbed water	Ekennia, et al. 2020)
2358.94	Symmetric and asymmetric valency bands of C–H	Kaliraj et al. 2019)
1649.14	C=O stretching in polyphenols	Ullah et al. 2019)
1548.84	Bending vibrations of N–H secondary amines	Padalia and Chanda 2017)
1419.61	C–C vibrations	Praveen et al. 2019)
1041.56	C–O bonds of alkaloids and flavones	Sharma 2016)
723.31, 634.58	ZnO vibrational stretching	Ekennia, et al. 2020; Sharma 2016)
422.41	Crystalline ZnO	Padalia and Chanda 2017)

Fig. 6 Photocatalytic degradation of Rhodamine B dye under sunlight irradiation in the presence of BR-ZnONFs

progress. Moreover, it can be visualized from the spectra that the intensity of the absorbance at 553 nm started declining as the sunlight exposure time increased. A degradation efficiency of 94.74% was observed within 165 min of sunlight exposure. This value was significantly higher than the values reported for the degradation of RhB dye by ZnONPs. For instance, only 38% and 71% degradation efficiencies for the RhB dye degradation under sunlight radiation were reported for the ZnONPs synthesized using algae (Vijai Anand et al. 2019) and chemical methods (Al-Bedairy and Alshamsi 2018) respectively. The enhanced degradation could be ascribed to the crystalline and mesoporous nature, high surface area, and good electron gaining characteristics of the BR-ZnONFs. Moreover, the improved photocatalytic activity may be due to the stabilization effect of the phyto-compounds present in the BRLE. Similar conclusions were demonstrated for the solar light degradation of MB dye by neem leaf extract-mediated ZnONPs (Haque et al. 2020).

The kinetics of the photocatalytic degradation of RhB dye can be explained by pseudo-first-order kinetics: $\ln \frac{C_0}{C_t} = kt$

where C_0 and C_t are the concentrations of the RhB dye at the initial and final time, 't', and k is the degradation constant. A linear plot with a regression coefficient (R^2) of 0.9631 was obtained by plotting $\ln (C_0/C_t)$ vs 't' which yielded a degradation constant (k) of 0.0109 min⁻¹.

The fundamental mechanism (Fig. 7) of the photocatalytic degradation of RhB by the BR-ZnONFs under solar radiation is due to the generation of electron-hole pairs when the solar light energy illuminates the surface of the

BR-ZnONFs (Prasad et al. 2018). These electron-hole pairs move spontaneously to the BR-ZnONFs surface and begin redox reactions with molecular oxygen and hydroxide ions. Oxidation of water to reactive hydroxyl ($\cdot\text{OH}$) and H^+ occurs in the valence band due to the existence of holes. Electrons in the conduction band reduce surface oxygen molecules to form superoxide radicals ($\text{O}_2^{\cdot-}$). The extra electrons generated stimulate the effects to yield hydroxyl radicals (Rana et al. 2016). The monodispersive nature fostered the high-level exciton splitting to create immense amounts of $\cdot\text{OH}$ and $\text{O}_2^{\cdot-}$ which readily degraded the RhB dye molecules to colorless products, resulting in high dye degradation efficiency of BR-ZnONFs (Rambabu et al. 2020).

Conclusions

ZnONPs were synthesized using the aqueous extract of *B. retusa* leaves and used as solar photocatalysts to efficiently degrade RhB dye. Initially, the formation of BR-ZnONFs was visually confirmed by the color changes of the reaction mixture. Later, the samples were characterized by various techniques to examine the elemental composition, surface morphology, crystalline nature, surface functional groups, surface area, and nature of pores. High degradation efficiency (94.74%) of RhB dye was observed under solar light in the presence of BR-ZnONFs. The degradation process was modeled according to pseudo-first-order kinetics. Hence, this study suggests that the BR-ZnONFs can be used as effective photocatalysts to remove dyes from wastewater.

Fig. 7 Degradation mechanism of Rhodamine B dye by BR-ZnONFs under sunlight irradiation

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Declarations

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

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