



IMPROVED ANTIFUNGAL ACTIVITY OF ZNO NANOPARTICLES BIOSYNTHESIZED USING BLACK CARDAMOM

Ashwani Kumar Singh¹, Pallavi Singh² and Amit Srivastava^{3*}

¹School of Physical Sciences, Jawaharlal Nehru University, New Delhi-221005, India

²Department of Botony (MMV), Banaras Hindu University, Varanasi, U.P. 221005, India

³Department of Physics, TDPG College, Jaunpur, U.P. 222001, India

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ABSTRACT

This paper demonstrates a one step, simple, and green method to synthesize Zinc oxide nanoparticles using black cardamom extract. Black cardamom extract is used as reducing agent for ZnO nanoparticles. Use of black cardamom can be justified under the light of the fact that it contains strong reducing properties. Number of characterization techniques for the analysis of synthesized material has been carried out to confirm the formation of ZnO nanoparticles. XRD pattern, UV-Vis and PL spectra explicitly confirm the formation of ZnO nanoparticles. A careful TEM investigation gives provides information about the shape and size distribution of nanoparticles. Further, antifungal activity test of these synthesized ZnO nanoparticles has been done which shows a better inhibition response for fungus.

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INTRODUCTION

Zinc oxide (ZnO) is a promising material for a variety of useful applications, like piezoelectric transducers, optical waveguides, surface acoustic wave devices, varistors, phosphors, transparent conductive oxides, chemical and gas sensors, spin functional devices, and UV-light emitters (1, 2). These properties have stimulated the search for new methodologies of synthesis of well-controlled ZnO nanostructures (3-14). ZnO is a wide-gap semiconductor with a direct gap around 3.4 eV, i.e. in the near-UV and crystallizes preferentially in the hexagonal wurtzite-type structure. Its wide bandgap makes ZnO a promising material for photonic applications in the UV or blue spectral range, while the high exciton-binding energy (60 meV)(1) allows efficient excitonic emission even at room temperature. In addition, ZnO doped with transition metals shows great promise for spintronic applications [15]. It has also been suggested that ZnO exhibits sensitivity to various gas species, which makes it suitable for sensing applications. Moreover, its piezoelectric property (originating from its non-centrosymmetric structure) makes it suitable for electromechanical sensor or actuator applications. Also, ZnO is biocompatible which makes it suitable for biomedical applications. Last but not least, ZnO is a chemically stable and environmentally friendly material. Consequently, there is considerable interest in studying ZnO in the form of powders, single crystals, thin films, or

nanostructures. The fabrication methods for ZnO nanostructures can be divided into two groups: spontaneous growth and template based synthesis (for example, using an alumina template). Fabrication without a template can occur either by using metal catalysts or may be self-catalyzed. The use of metal catalysts, such as Au, can be an advantage for achieving aligned and selective area growth [16]. Aligned nanorods can also be obtained by a hydrothermal method without any metal catalyst. The degree of alignment and the achieved aspect ratio was dependent on the seed layer used and the fabrication conditions. The influence of these factors on ZnO morphology was studied in detail recently (17). A variety of ZnO nanostructure morphologies, such as Nanowires, nanorods, tetrapods, and nanoribbons/belts, have been reported (18-35). ZnO nanostructures have been fabricated by various methods, such as thermal evaporation, (18-20) metal-organic vapor phase epitaxy (MOVPE), [26] laser ablation, [27] hydrothermal synthesis, [21] and template-based synthesis. In this article, I have shown a green route to synthesize ZnO nanoparticles using black cardamom as reducing agent. Black cardamom is widely used extensively in India, in foods, beverages, mouth fresheners, and native medicine. Black cardamom has been used as reducing agent in our synthesis protocols. Further it has never been used as reductant for ZnO nanoparticles before.

Experimental details

MATERIALS AND METHODS

Zinc nitrate hexahydrate ($Zn(NO_3)_2 \cdot 6H_2O$) and NaOH was purchased from sigma, and were used without any further

*Corresponding author: Amit Srivastava

Department of Physics, TDPG College, Jaunpur, U.P. 222001, India

purification. Dried black cardamom easily available in commercial market. These materials have been adopted as starting materials for the synthesis of ZnO nanoparticles.

Preparation of Black Cardamom Extract

In the synthesis protocol adopted here, black cardamom has been used as a reducing agent, prepared by simply dipping 5 gm of it for 24 h into 100 mL of double-distilled water. The solid content was filtered out, leaving the residual extract of dark brownish color. This extract was further used in consecutive steps for the synthesis of ZnO nanoparticles.

Synthesis of ZnO nanoparticles

Solution of required chemical $Zn(NO_3)_2 \cdot 6H_2O$ was prepared by dissolving 5.588 gm $Zn(NO_3)_2 \cdot 6H_2O$ into 200ml double distilled to make the final concentration of the solution 0.1M. 100 ml black cardamom extract was further added under vigorous stirring at 300rpm slowly. Mixture was kept under stirring for next 5 minutes and 100 ml (1M) NaOH solution was further added under stirring at room temperature. Solution was further kept under stirring for next 2 hours. After 2 hours, solution was removed from stirring plate and left overnight. Particles get settled at bottom, carefully filtered and centrifuged at 10000 rpm for 15 minutes thrice. Particles thus obtained were collected and dried at room temperature.

Characterizations of ZnO nanoparticles

Confirmation of the synthesis of ZnO nanoparticles was done by using various techniques like XRD, TEM, UV-Vis, and PL. samples for XRD were prepared by placing a drop of synthesized material on a glass slide and allowed it to dry. XRD measurement was performed at Regaku Miniflex 600. XRD patterns were recorded by operating X-ray tube at 45 kV and 35 mA. The radiation used was Cu- $K\alpha$ ($k = 1.5406 \text{ \AA}$). Sample for TEM was prepared by placing 10 μ l diluted reaction mixture on a carbon coated copper grid. TEM investigations were performed by using JEOL 2100F microscope. The details of formation and shape of Au nanoparticle were also analyzed employing Perkin Elmer Lambda 750S UV-Visible spectrometer. Samples for UV-Vis and PL were prepared by mixing 0.1ml reaction mixture into 2 ml double distilled water. UV- Spectra was recorded on 750S UV-Visible spectrometer while PL spectra was recorded by using Edinburgh FL920 spectrometer.

Antifungal test

Pathogenic fungi, *B. cinerea* was obtained from the culture collection of the gastroenterology laboratory at the IMS, BHU. MacConkey agar was used for culturing *B. cinerea* on plates at 25 °C in the dark. The component of Luria-Bertani (LB) medium was used in growing and maintaining this culture. All the media used for culture, was supplied from HiMedia laboratories, India. Antifungal tests were performed by the agar dilution method (Fraternale et al. 2003) with some modification. The autoclaved MacConkey agar media with ZnO NPs at concentrations of 0, 3, and 6 mmol/L were poured into the Petri dishes (9 cm diameter). The fungi were inoculated after the media solidified. A disc (1.4 cm) of mycelial material taken from the edge of 7-day-old fungal cultures was placed in the center of each Petri dish. The Petri dish with the inoculum was then incubated at 25 °C. The efficacy of ZnO NP treatment was evaluated at the time intervals of one day for 10 days by measuring the diameter of

fungal colonies. All tests were performed in triplicate and the values were expressed in centimeters.

RESULTS AND DISCUSSION

Structural analysis

The as synthesized products were subjected to XRD characterization for the identification of the gross structure. Xray diffraction (XRD) confirmed that all nanostructures have hexagonal (wurtzite) ZnO structure (JCPDS file no. 36-1451) and the corresponding pattern is shown in Fig.1.

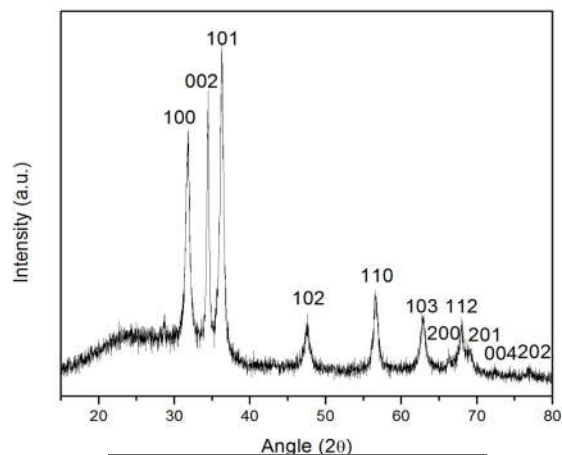


Fig.1 XRD pattern of as synthesized nanomaterial

These XRD peaks matches well with the standard 2 theta and hkl values of ZnO nano crystal. This confirms the formation of ZnO nanoparticles.

Optical characterization

UV-visible reflectance spectra of as synthesized pure ZnO has been illustrated in Fig. 2. The reflectance spectrum was studied without taking into account the transmission losses. For pure ZnO NPs sharp peaks close to 390, 382, 378, 375 and 370 nm were observed, which was believe to arise from the near band edge free excitons.

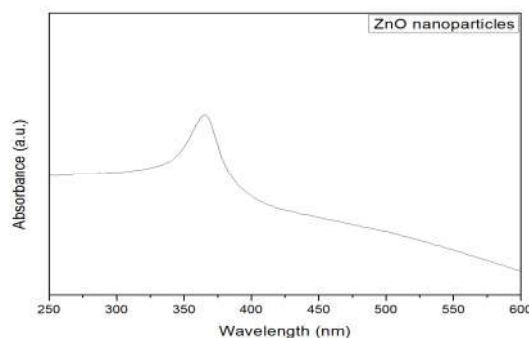


Fig.2 UV-Vis spectra of as synthesized nanomaterial

Fig.3 shows the PL spectra of ZnO nanoparticles at excitation wavelength 310 nm. The PL spectrum exhibits a peak located at 420 nm. The 420 nm, peak is well understood and due to the exciton emission. Although, there seems to be no general agreement on the type and nature of such defects the possible variety of defects could be zinc and oxygen interstitials, oxygen vacancies, donor-acceptor complexes, surface states, etc.

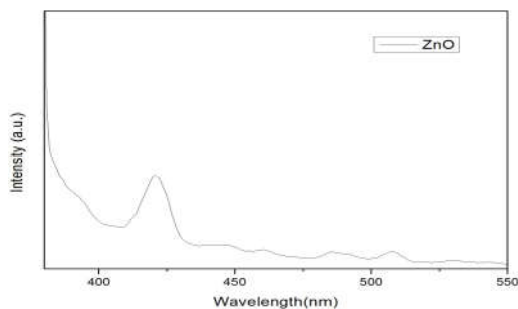


Fig.3 PL spectra of as synthesized nanomaterial

Microstructural characterizations

TEM investigations were done to find out the microstructural properties and particle size distribution of synthesized nanomaterials. Fig.4 shows the TEM micrographs of as synthesized nanoparticles

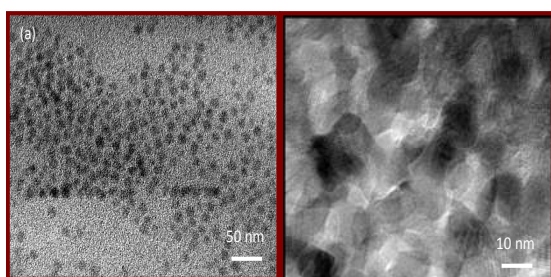


Fig.4 TEM micrographs of as synthesized nanomaterial

Fig 4a is a low magnification image of ZnO nanoparticles. This image gives the information about average shape and size of the synthesized nanoparticles. Particles are spherical in shape and average size of the particles is approximately 20-25 nm. Further fig.4b shows the magnified image of nanoparticles.

Antifungal effect of ZnO NPs

Figs. 5 show the effect of ZnO NPs on the growth of *B. cinerea* that was cultivated on MacConkey agar containing different concentrations of ZnO NPs (0, 3, and 6 mmol/L) and incubated at 25 °C for 10 days. It is observed that, the use of ZnO NP suspension was effective in inhibiting fungal growth. The average growth of *B. cinerea* was inhibited by from 63% to 80% in terms of colony growth diameters after 10 days of incubation as the concentration of ZnO NPs increased from 0 to 6 mmol/L. These results indicate that ZnO NPs at concentration greater than 3 mmol/L can significantly inhibit the growth of *B. cinerea*

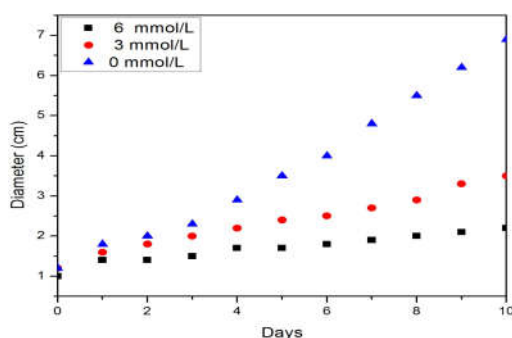


Fig.5 Antifungal activities of ZnO NPs against *Botrytis cinerea* on media in the presence of different concentrations of ZnO NPs.

CONCLUSION

A new and novel route of the synthesis of ZnO nanoparticles using black cardamom as natural precursor has been demonstrated. XRD pattern matches well with the known standard diffraction peaks of ZnO nanocrystals. TEM, UV-Vis and PL studies also confirms the formation of ZnO nanoparticles. Further, the results on the inhibition of fungus show an effective impact on its growth. It has also been observed that this inhibition becomes more effective as the concentration of ZnO nanoparticles in media is increased further.

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