



ISSN: 0976-3031

Available Online at <http://www.recentscientific.com>

CODEN: IJRSFP (USA)

International Journal of Recent Scientific Research
Vol. 8, Issue, 8, pp. 19224-19227, August, 2017

International Journal of
Recent Scientific
Research

DOI: 10.24327/IJRSR

Research Article

FACILE GREEN SYNTHESIS OF ZnO NANOPARTICLES USING LEAF EXTRACT

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DOI: <http://dx.doi.org/10.24327/ijrsr.2017.0808.0651>

ARTICLE INFO

Article History:

Received 15th May, 2017
Received in revised form 25th
June, 2017
Accepted 23rd July, 2017
Published online 28th August, 2017

Key Words:

Zinc Oxide nanoparticles, Optical
properties, Morphological analyses.

ABSTRACT

We describe a simple combination reaction between zinc nitrate and leaf extract of *Solanum nigrum* to prepare ZnO nanoparticles. The synthesized NPs subjected to structural, optical, and morphological analyses. The structural analysis demonstrates that the prepared ZnO NPs are crystalline and exhibit hexagonal wurtzite. The optical properties of the products were studied by ultraviolet-visible and room temperature photoluminescence measurements. The morphology of the products was analyzed by field emission scanning electron microscopy analysis.

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INTRODUCTION

Zinc oxide, a II-VI semiconductor with a wide direct band gap of 3.37 eV exhibits excellent optical and electrical properties and hence it can be widely used in solar cells, field emitters, surface acoustic wave devices and transparent conducting materials [1]. Previously reported that, metal NPs have various functions that are not observed in bulk phase [2,3] and have been studied extensively because of their exclusive catalytic, optical, electronic, magnetic and antimicrobial [4,5] wound healing and anti-inflammatory properties [6]. The different types of NPs are used in different applications, among them; ZnO NPs have a wide range of applications like cosmetics and sunscreen lotions because of their efficient UV-A and UV-B efficient absorption properties without scattering visible light [7]. ZnO NPs have also used as the antimicrobial properties [8,9] and agriculture and anticancer therapy [10,11]. The production of ZnO NPs increased day by day and also increased the accidental exposure to humans and animals. Biological methods for NPs synthesis using microorganisms, enzymes, and plants or plant extracts have been suggested as possible eco-friendly alternatives to chemical and physical methods. Thus, there is a need for a new simple and eco-friendly green synthesis processes, those avoids the use of toxic chemicals and high energy inputs. Recently, a variety of research work has been carried out to investigate the

bioreduction of various metal ions (e.g. Ag, Au, Pd, and Pt) into metal NPs [12-15].

Solanum nigrum is a medicinal plant belonging to the family Solanaceae. Its common names are Makoil and blacknight shade. *S. nigrum* has been used traditionally to treat various ailments such as pain, inflammation fever and enteric diseases. It possesses many activities like anti-tumorigenic, anti-oxidant, anti-inflammatory, diuretic, anti-pyretic agent, and anti-bacterial respectively. It is also used against sexually transmitted diseases. *S. nigrum* possesses numerous compounds that are responsible for the bioreduction. Its active components are glycoalkaloids, glycoproteins, and polysaccharides, polyphenolic compounds such as gallic acid, catechin, protocatechuic acid (PCA), caffeic acid, epicatechin, and naringenin [16]. Hence, the present study was aimed to rapidly green synthesis of ZnO NPs using aqueous leaves extract of *Solanum nigrum*, to investigate the biomolecules responsible for synthesis of ZnO NPs and finally to evaluate its antibacterial activities.

MATERIALS AND METHODS

Preparation of the leaf extract

The plant leaves are collected from Polur, Thiruvannamalai District (12°15' N, 79°07' E), Tamil Nadu, and India. The collected leaves subjected to washing several times with

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distilled water to remove the dust particles. In the preparation of leaves extract, 20 g of fine cut leaves in 250 mL glass beaker mixed with 100 mL of distilled water. Boiling of the mixture for 20 minutes changed the color of the aqueous solution from watery to light yellow. After allowing the extract to cool to room temperature, using a Whatman filter paper broth filtration took place.

Preparation of zinc oxide NPs

For the synthesis of ZnO nanoparticles, 30, 40 and 50 mL of leaves extract allowed to boil using a stirrer–heater. Then, 5 gm of zinc nitrate added to the above solution as the temperature reached 60 °C. This mixture further boiled until its color changed into a dark yellow. The obtained paste further transformed to the ceramic crucible and annealed at 400 °C for 2 hours. The finally arrived light white colored powder consumed for different characterizations.

RESULTS AND DISCUSSION

XRD analysis

Structure and phase purity of ZnO NPs are shown in Fig.1. From the diffractogram of XRD are very well matched with the hexagonal phase (wurtzite structure) by comparison with the data from JCPDS card No.89-1397 and no indication of a secondary phase or impurity peaks were obtained. The strong and narrow diffraction peaks indicate that the product has good crystalline structure. The sharp intense diffraction peaks appearing at about 2θ of 31.29°, 33.95°, 36.04°, 47.05°, 56.09°, 62.38°, 65.90°, 67.45°, 68.60°, 72.03°, and 76.48° corresponding with those from (100), (002), (101), (102), (110), (103), (200), (112), (201), (004) and (202) orientations, respectively.

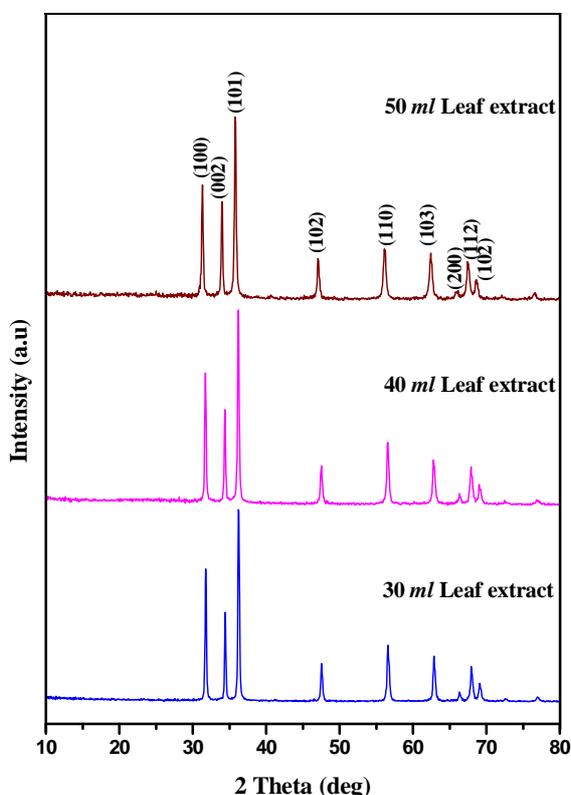


Fig 1 XRD spectrum of ZnO NPs synthesized using *Solanum nigrum* leaf extract

The average grain size of the sample was calculated using the Scherer’s equation.

$$D = \frac{K\lambda}{\beta \cos\theta} \text{ \AA} \quad \dots\dots\dots (1)$$

Where D is the average crystallite size in Å, K is the shape factor (0.9), λ is the wavelength of X-ray (1.5406 Å) CuKα radiation, θ is the Bragg angle, and β is the corrected line broadening of the NPs. The obtained sizes are 39.62, 34.05 and 29.79 nm, respectively for 30, 40, and 50 ml of extract addition.

Optical studies

Figure 3.3a shows the optical absorption spectrum of ZnO NPs synthesized by using *Solanum nigrum* extract. The sample has a clear and strongly observed absorption peak below at 400 nm.

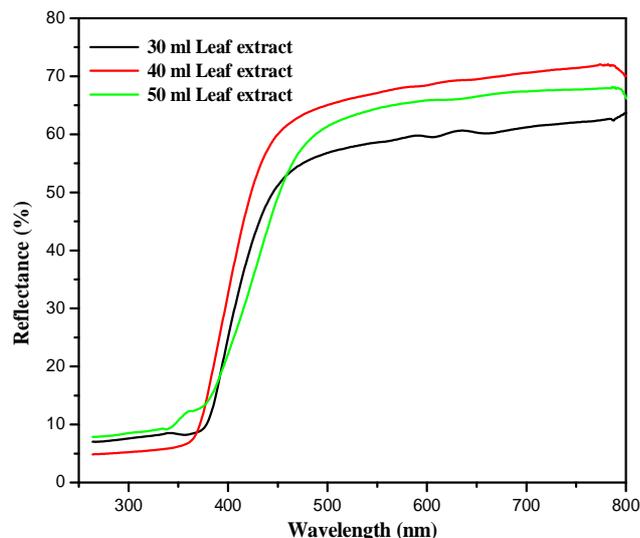


Fig 2a UV-DRS spectrum of ZnO NPs synthesized using *Solanum nigrum* leaf extract

The band gap energy (E_g) of ZnO was obtained from the wavelength value corresponding to the intersection point of the vertical and horizontal part of the spectrum, using the equations:

$$E_g = \frac{hc}{\lambda} \text{ eV} ; E_g = \frac{1240}{\lambda} \text{ eV} \quad \dots\dots\dots (2)$$

The band gap energy corresponds to the absorption limit and can be roughly evaluated by the above relation. Where, E_g is the band gap energy (eV), h is the Planck’s constant (6.626×10^{-34} J s), C is the light velocity (3×10^8 m/s) and λ is the wavelength (nm). From Fig.3.3a, the absorption edges are positioned at 366,361 and 358 nm, respectively for 30, 40 and 50 mL of extract addition. The corresponding band gap value of 3.38, 3.43 and 3.46 eV which indicates red shift in the UV region

Indirect band gap energy (Kubelka–Munk plot)

The reflectance spectra were analyzed using the Kubelka-Munk relation (equation 3). To convert the reflectance data into a Kubelka-Munk function (equivalent to the absorption coefficient) F (R), the following relation was used.

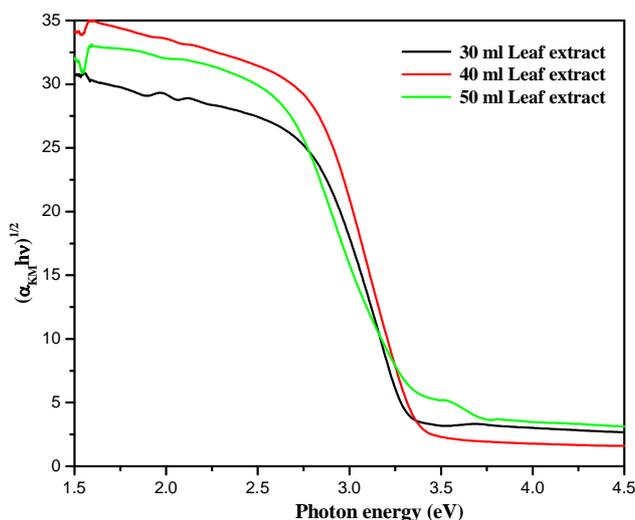


Fig 2 b Plot of indirect band gap energy for ZnO NPs

$$F(R) = \frac{(1-R)^2}{2R} \dots\dots\dots (3)$$

Where, R is the reflectance value. Band gap energy of the samples was estimated from the variation of the Kubelka-Munk function with photon energy. Fig.2b shows the Kubelka-Munk plots for the ZnO NPs. It is used to determine their band gap energy associated with their indirect transitions. The ZnO exhibits indirect E_g of 3.35, 3.41 and 3.38 eV.

Photoluminescence (PL) analysis

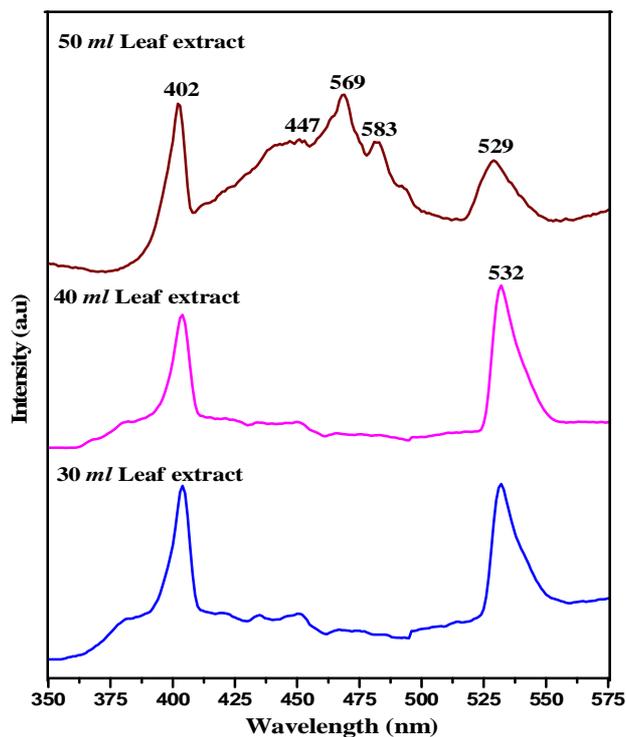


Fig 3 Photoluminescence spectrum of ZnO NPs synthesized using *Solanum nigrum* leaf extract

Photoluminescence (PL) studies were performed to emphasize its emission properties as shown in Fig. 3. The photoluminescence of ZnO sample suggested five emission bands, including three blue bands at 402, 447, 469 and at 483 nm, probable green band at 529 nm have been observed from the prepared ZnO NPs sample. The blue band at 417, 440 and

462 nm may be in correlation with the defect structures in ZnO crystal. The green bands at 520 nm and shoulder red band at 675 nm may be correlated to a transition between the oxygen vacancy and interstitial oxygen [17].

FT-IR analysis

Possible biomolecules responsible for the reduction of ZnO and capping agent of bioreduced ZnO NPs through particular bond vibration peaks identified from defined wave numbers in FT-IR technique. The FT-IR spectra of control leaf extract (before reaction without Zn (NO₃)₂ and synthesized ZnO (after reaction with Zn (NO₃)₂ are shown in Fig. 4 The absorption bands at 3429, 2926, 2349, 1635, 1404, 1261, 1031, 605, 540, and 470 cm⁻¹ in *Solanum nigrum* leaf extract would be shifted into 3425, 2924, 1627, 1382, 1126, 1030, 833, and 445 cm⁻¹ in synthesized ZnO NPs sample. The peak found at around 1450-1500 cm⁻¹ showed the stretching of N-H vibration. Whereas the stretching of ZnO NPs were found around 400-800 cm⁻¹ [18]. The FT-IR spectra showed the presence of bonds due to O-H stretching (around 3429 cm⁻¹) and aldehydic C-H stretching (around 2924 cm⁻¹) [19]. The peak at around 1240-1261 cm⁻¹ present in both the cases signified amide III band of the random coil of protein [20]. Peak at 1404 cm⁻¹ may be assigned to the symmetric stretching of the carboxyl side groups in the amino acid residues of the protein molecules [21]. The band at 1029 cm⁻¹ corresponds to C-N stretching vibration of amine [22]. The bands incidence at 1126, 1030 and 605 cm⁻¹ illustrates the chemical bonding, crystal structure and relative intensities of the IR bands of the carbonate [23,24].

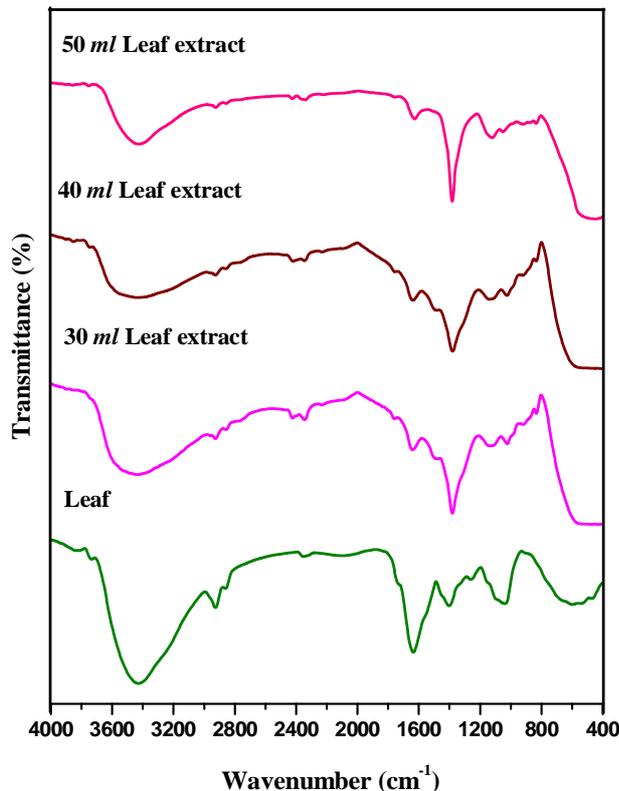


Fig 4 FT-IR spectra of leaf extract and ZnO NPs synthesized using *Solanum nigrum* leaf extract

Therefore the synthesized ZnO NPs were surrounded by proteins and metabolites such as terpenoids having functional groups. From the analysis of FTIR studies, we confirmed that the carbonyl groups from the amino acid residues and proteins

has the stronger ability to bind metal ions, indicating that the proteins could possibly from the metal NPs (i.e.; capping of silver NPs) to prevent agglomeration and thereby stabilize the medium. This suggests that the biological molecules could possibly perform dual functions of formation and stabilization of ZnO NPs in the aqueous medium.

FE-SEM analysis

The FE-SEM image of prepared ZnO NPs was presented in Fig. 5. The diameter of the cluster ZnO NPs found to be in the range of 20–30 nm and is nearly spherical in shape with rough surface. EDX spectrum shows the high values of zinc (62%) and oxygen (29%) respectively. These results confirmed the present of zinc as a majority label compared to oxygen in precursor material.

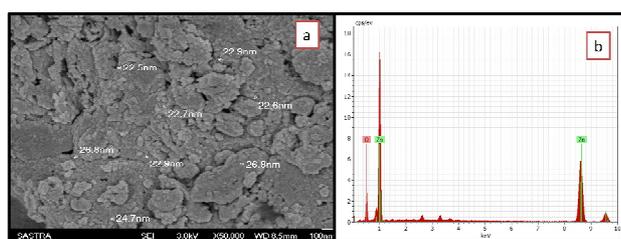


Fig 5 FE-SEM image and EDX spectrum of ZnO NPs synthesized using 50 ml of *Solanum nigrum* leaf extract

The EDX analysis displays the optical absorption peaks of ZnO NPs and these absorption peaks were due to the surface plasmon resonance of Zinc oxide NPs. The origin of these elements lies in the biological components; mostly align along with ZnO NPs [25].

CONCLUSION

ZnO NPs synthesized were successfully using a *Solanum nigrum* leaf extract mediated bio-reduction process. The structure, morphology and size (dimension) of prepared ZnO NPs were examined by XRD, FT-IR, and FE-SEM analysis. UV-Vis-DRS studies confirmed the indirect band gap 3.38, 3.41, 3.35 eV and photoluminescence was found the blue band at 402, 447, 469 and 483 nm. The average grain size lies between 20-30 nm were obtained from XRD study as well as FT-IR spectra revealed the functional groups of stretching bands for ZnO NPs were found around $800\text{--}400\text{ cm}^{-1}$. Finally, the present study is so helpful and useful to the scientific community for using the ZnO NPs as the potent applications to the semiconducting, pyroelectric, piezoelectric, catalysis, optoelectronics, antidiabetic and antihyperlipidemic activities. Besides, it is inexpensive, stable and nontoxic, safe and eco-friendly ZnO nanoparticles from the *Solanum nigrum* leaf extract.

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How to cite this article:

Anbuvarannan M.2017, Facile Green Synthesis of ZnO Nanoparticles Using Leaf Extract. *Int J Recent Sci Res.* 8(8), pp. 19224-19227. DOI: <http://dx.doi.org/10.24327/ijrsr.2017.0808.0651>
