Research Article

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Synthesis and Characterization of Pure and Capped Zinc Oxide Nanoparticles using *Pesidium gujava* Leaf Extract

M. Kokilavani, K. Surya, V. Kalaiselvi*, N. Vidhya, V. Ramya

Department of Physics, Navarasam Arts & Science College for Women, Erode, TN, India

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*nk.arthi.kalai@gmail.com

ABSTRACT

In this work, Zinc oxide nanoparticles, which are mostly applied in the fields of medical science and anti-bacterial activities, were synthesized with and without *Pesidium guajava* leaf extract by using Microwave irradiation method. The zinc oxide nanoparticles were characterized by various techniques like XRD, EDS, FTIR and SEM. X-Ray diffraction spectra revealed the crystalline size of the sample. The qualitative and quantitative components of the ZnO nanoparticles were analyzed using Energy-dispersive X-Ray microscopy. The functional groups of ZnO nanoparticles were investigated through Fourier Transform Infrared Spectroscopy. The surface morphological structure of zinc oxide nanoparticles was determined using Scanning Electron Microscopy (SEM).

Keywords: Zinc oxide nanoparticles; Elemental composition; Antibacterial.

1. INTRODUCTION

Nanotechnology deals with materials measured in a billionth of a meter. A nanometer is 1/80,000-th of the diameter of a human hair or approximately ten hydrogen atoms wide. Nanotechnology is a multi-disciplinary science; it includes knowledge from biology, chemistry, physics and other disciplines. Zinc oxide is an inorganic compound with the formula ZnO. It usually appears as a white powder, nearly insoluble in water (Divya *et al.* 2018; Birustani *et al.* 2018; Bala *et al.* 2015). Zinc oxide is a multi-functional substance with its special physical and chemical properties, including high chemical stability, high electro-chemical binding coefficient, wide spectrum of radiation absorption and high photostability (Manjunatha *et al.* 2019; Iqbal *et al.* 2020).

Pesidium guajava (*P. guajava*) or the common guava is an evergreen shrub or small tree belonging to the family Myrtaceae. *P. guajava*; it is issued as a popular medicine against diarrhea and is also used for wound dressing, ulcers, rheumatic pain (Sutradar *et al.* 2010; Santhosh Kumar *et al.* 2017). *P. guajava* leaves are also being reported to have antibacterial, anti-inflammatory and anti-cancer properties (Ezealisiji *et al.* 2019; Ganasangeetha *et al.* 2013; Patil *et al.* 2016).

P. guajava is selected for this study due to its high content of polyphones (garlic acid, protocatechuic acid, caffeic acid, ferulic acid, chlorogenic acid, ellagic acid, guavin), flavanoids (quercetin, leucocyanidin, kaempferol, quercetin3-l-arabinofuranoside), carotenoids (carotene, lutein, lycopene, cryptoxanthin, rubixanthin, criptoflavin, neochrome, phytofluene), triterpenes (oleanolic acid, ursolic acid, sitosterol, uvaol) present in the crude water extract of leaves (Kalaiselvi *et al.* 2018a; 2018b; Vijajakumar *et al.* 2016a; 2016b; Chaudhuri *et al.* 2017). The afore-mentioned biomolecules have functional groups that can coordinate Zn (II) and will help in stabilization during the formation of ZnO nanoparticle (Saha *et al.* 2018; Ramesh *et al.* 2015; Dobrucka *et al.* 2016).

2. MATERIALS AND METHODOLOGY

Zinc oxide (ZnO) nanoparticles were prepared by using zinc acetate dehydrate ($C_4H_{10}O_6Zn$) and leaf extract of *Pesidium guajava* (Guava leaves) as a capping agent. The chemical reagents like NaOH solution, acetone and deionized water were used.

2.1 Synthesis of ZnO Nanoparticles

10.975 g of zinc acetate dehydrate was dissolved with 100 ml of deionized water, and the solution was stirred for half an hour. 10 ml of NaOH solution was added drop-wise to the above mixture to maintain the pH level at 12. The final mixture was stirred for half an hour. The gelatinous precipitate was obtained.

This obtained precipitate was aged for 24 hours and double-washed with deionized water. Then the precipitate was kept in microwave oven for 30 minutes at 75 ºC; then dried at 400 °C for four hours. The fine pure ZnO nanoparticles were collected by grinding the dried cake using mortar.

2.2 Preparation of Plant Extract

Fresh and good quality leaves were collected and washed using distilled water. These leaves were cut into small pieces using a knife. 30 g of chopped leaves were weighed and taken into a beaker with 100 ml of distilled water and then boiled for 30 minutes at 75 ºC. By this time aqueous part turns green. The extract was filtered using Whatman No. 1 filter paper to get a clear solution.

2.3 Synthesis of ZnO Nanoparticles using Capping agent (*Pesidium guajava)*

10.975 g of zinc acetate dehydrate was dissolved with 100 ml of deionized water and, the solution was stirred for half an hour for the reduction of zinc ions. 10 ml of *Pesidium guajava* leaf extract was added into the 100 ml of zinc acetate dehydrate solution. Then the mixture was stirred for half an hour using a magnetic stirrer. 10 ml of NaOH solution was added drop-wise to the above mixture to maintain the pH level at 12. The final mixture was stirred for half an hour. The gelatinous precipitate was obtained. This obtained precipitate was aged for 24 hours and double-washed with deionized water. Then the precipitate was kept in the microwave oven for 30 minutes. *The* fine *Pesidium guajava* capped ZnO nanoparticles were collected by grinding the dried cake using mortar.

3. CHARACTERIZATION TECHNIQUES

The prepared samples were characterized by using various techniques like Fourier transform infrared spectroscopy (FTIR), ,X-Ray diffraction (XRD), Scanning electron microscopy (SEM), Energy dispersive X-Ray spectroscopy (EDAX) and Anti-bacterial activity.

3.1 FTIR

FTIR Spectroscopy is an analytical technique used to identify organic, polymeric, and in some cases, inorganic materials. The FTIR analysis method uses infrared light to scan test samples and observe chemical properties.

3.2 XRD

X-ray diffraction (XRD) is a technique used in materials science for determining the atomic and molecular structure of a material. This is done by irradiating a sample of the material with incident X-rays and then measuring the intensities and scattering angles of the X-rays that are scattered by the material.

3.2.1 Grain Size

The crystalline size was calculated by using Debye-Scherrer formula,

$$D = k\lambda/\beta \cos \theta$$

where, k - Wavelength of XRD, λ - Full width half maximum, θ - Bragg's angle and k - a constant

3.2.2 Lattice constants and Unit cell volume:

The lattice parameters were calculated by the equation of

$$1/d^2 = (4(h2+hk+k^2)/3a^2) + (l^2/c^2)$$

where, d - plane spacing a, c - the lattice parameters which confirm the hexagonal structure.

The unit cell volume (V) of the sample is given by the below equation:

$$V = (\surd 3/2) + a^2+c^0 \ (5.3)$$

3.3 SEM

SEM analysis is a powerful investigative tool that uses a focused beam of electrons to produce complex, high magnification images of a sample's surface topography.

3.4 EDAX

Energy Dispersive X-Ray Analysis (EDX), is an X-ray technique used to identify the elemental composition of materials. Applications include materials and product research, trouble-shooting, deformulation and environmental pollutant analysis.

4. RESULTS

4.1 FTIR Analysis

Fourier Transform Infrared Spectroscopy (FTIR) identifies chemical bonds in a molecule by producing an infrared absorption spectrum. The FTIR spectrum of the prepared pure and capped ZnO samples were plotted between the wavelength ranges about 4000-400 cm^{-1} as shown in Fig. 1. The absorption peaks at 3848.11 cm^{-1} and 3854.96 cm^{-1} corresponded to the O=H stretching banded (alcohol). The peaks at 3441.73 cm^{-1} and 3637.40 cm^{-1} indicated the N=H bond (Amine). The absorbance at 2800 cm^{-1} and 2793.15 cm^{-1} has represented the C=H bond (Alkynes). The absorption peaks at 1657.46 cm^{-1} and 16.77 cm^{-1} indicated the presence of C=HO stretching (Hydrogen). And then N=O stretching (Nitro) absorption was represented by the peaks at 1413.91 cm^{-1} and 1441.27 cm^{-1}, respectively. The results indicated that the synthesized ZnO nanoparticles were stabilized by biomolecular constituents present in the leaf extract of *Pesidium guajava* as shown in Table 1.

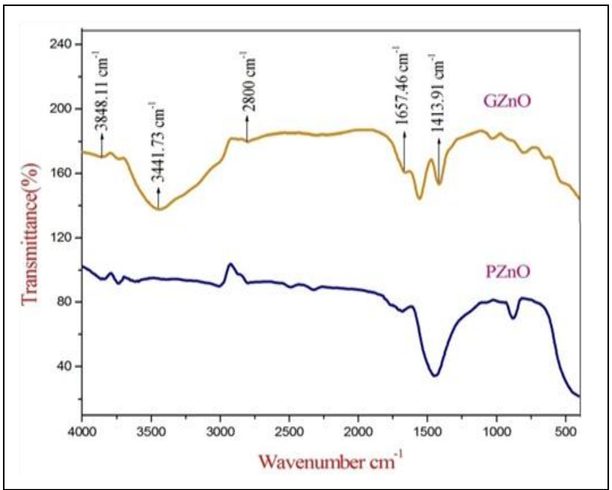

Fig. 1: FTIR spectrum of pure and capped ZnO nanoparticles.

4.2 XRD Analysis

XRD is primarily used for phase identification of a crystalline material and can provide information on unit cell dimensions. The analyzed material was finely ground and homogenized and the average bulk composition was determined. The XRD pattern of prepared PZnO and GZnO were shown in Fig 2. The prepared sample confirms the presence of hexagonal structure. The diffraction peaks of the prepared PZnO at 2θ = 36.44, 63.03, 68.1 and 69.29 and then the diffraction peaks of GZnO at 2θ = 36.647, 63.056, 67.192and 69.222are identified. Both are corresponding to the hkl planes are (101), (103), (112), and (201). The average crystalline size (D) of PZnO and GZnO was 18.67 and 14.26nm. Thus the average crystalline size of PZnO is higher than the GZnO due to the capping of leaf extract. The unit cell volume (V), lattice parameters a and c decreased due to an increase in crystalline size (Table 2).

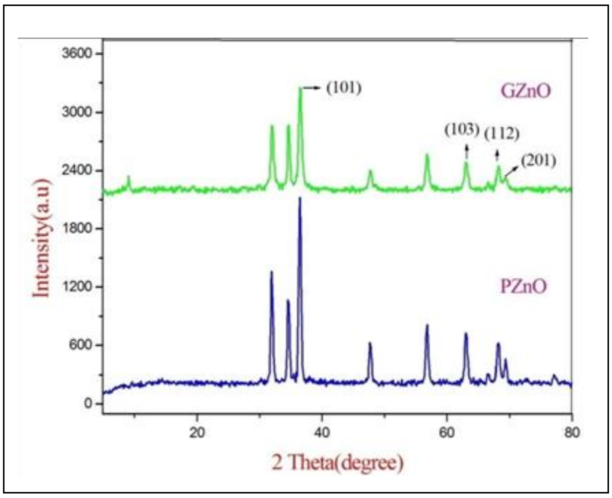

Fig. 2: XRD Patterns of pure and Capped ZnO nanoparticles.

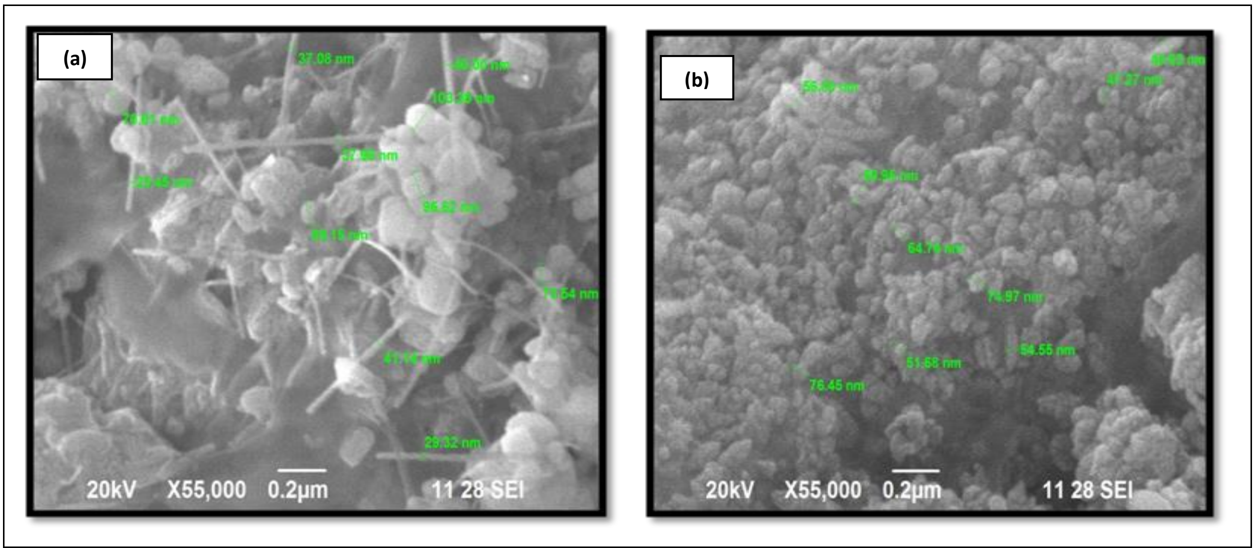

Fig. 3: SEM analysis for pure and Capped ZnO NPs: (a) Needle structure (Pure ZnO) and (b) Spherical structure (Capped ZnO).

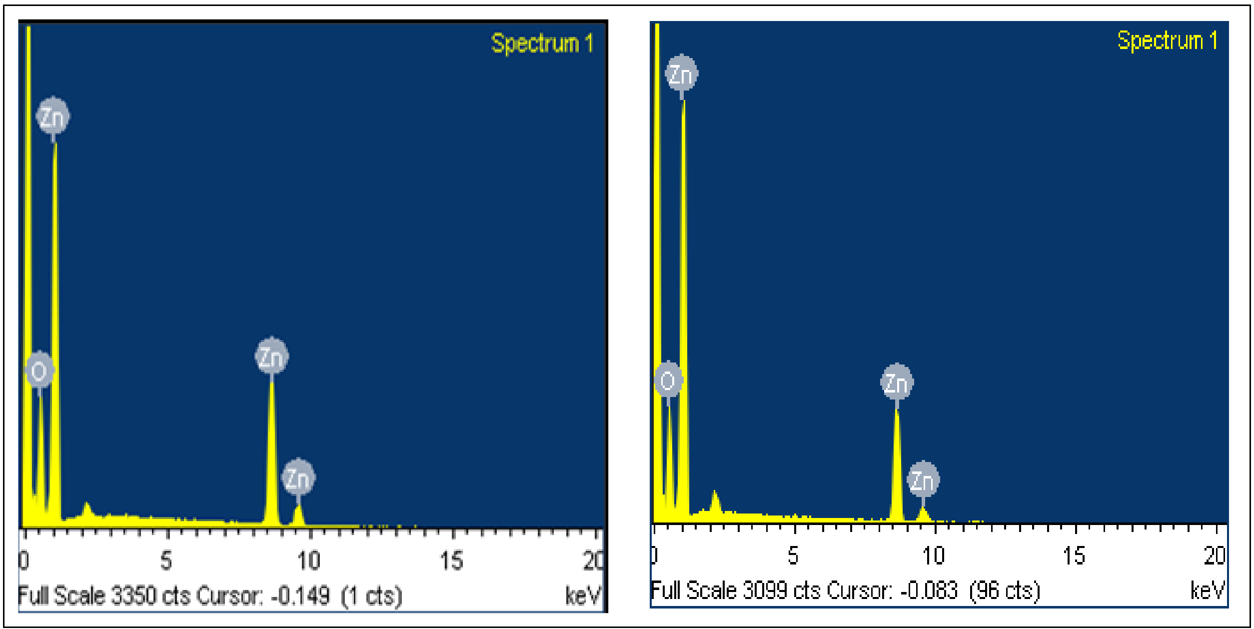

Fig. 4: EDAX analysis for pure and capped ZnO.

4.3 SEM Analysis

The morphological analyses of the synthesized materials were done by scanning electron microscope (SEM). The two-dimensional SEM images of pure and capped zinc oxide nanoparticles are shown in Fig. 3. The pure ZnO nanoparticles have shown needle-like structure, whereas the capped ZnO nanoparticles have shown the spherical structure. The particle size of pure ZnO NPs were 25.45 to 103.36 nm and that of capped ZnO NPs were 47.27 to 76.45 nm.

4.4 EDAX Analysis

Energy-dispersive X-ray spectroscopy (EDX) provides a qualitative and quantitative analysis of the prepared samples. The functional groups of Zn and O were present in the sample. The atomic weight for Zn in pure ZnO was 40.54% and O was 59.46%; whereas, the atomic weight of Zn in capped ZnO was 35.51 and O was 64.49. The EDX analyses of pure ZnO and capped ZnO were shown in Fig. 4.

6. CONCLUSION

In this research work, the zinc oxide nanoparticles were synthesized with and without the capping agent of *Pesidium guajava* leaf extract. The samples were analyzed by FTIR, XRD, SEM and EDX.

- The FTIR analyses has revealed the different functional groups present in pure and capped ZnO.
- XRD pattern has shown the presence of hexagonal structure, crystalline structure and unit cell dimensions of the prepared samples.
- SEM has confirmed the morphological structure of the prepared sample - needle-like shapes for pure ZnO and spherical structure for the capped ZnO, revealing that the capping agent can change some morphological structure of the given samples.
- EDX has confirmed the presence of foreign elements in the prepared samples. Both the samples were having zinc (Zn) and oxide (O).

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CONFLICTS OF INTEREST

The authors declare that there is no conflict of interest.

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