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

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Received: 14.02.2020 Accepted: 13.03.2020
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ABSTRACT

Nanotechnology is a promising concept in the field of science and technology. In this present work, the ZnO nanoparticles were synthesized with and without *Pesidium guajava* leaf extract by using the Microwave irradiation method. The formation of zinc oxide nano particles has been characterized by various techniques like XRD, EDS, FTIR, and SEM. X-Ray diffraction (XRD) spectra reveal the crystalline size of the sample. The qualitative and quantitative components of the ZnO-nanoparticles analyzed using Energy-dispersive X-Ray microscopy (EDS). The functional groups of the ZnO nanoparticles were investigated through Fourier Transform Infrared Spectroscopy (FTIR). The surface morphological structure of zinc oxide nanoparticles were obtained in Scanning Electron Microscopy (SEM). The Zinc oxide nanoparticles are mostly found and applied in field of medical science and anti-bacterial activities.

Keywords: Zinc Oxide nanoparticles; Elemental composition; Antibacterial.

1. INTRODUCTION

Nano means one-billionth. Thus nanotechnology deals with materials measured in a billionth of a meter. A nanometer is 1/80,000 the diameter of a human hair or approximately ten hydrogen atoms wide. Nanotechnology is the science of very small things. But nanotechnology is not just involved with small things. 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 multifunctional substance with its special physical and chemical properties, including high chemical stability, high electrochemical 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* is issued as a popular medicine against diarrhoea and is also used for wound dressing, ulcers, rheumatic pain (Sutratar et al. 2010; Santhosh Kumar et al. 2017). *P. guajava* leaves are also being reported to show antibacterial, anti-inflammatory, and anticancer properties (Ezealisi Ji 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), flavonoids (quercetin, leucocyanidin, kaempferol, quercetin-3-arabinofuranoside), carotenoids (carotene, lutein, lycopene, cryptoxanthin, rubixanthin, criptoflavin, neochrome, phytofluene), triterpenes (oleanolicacid, ursolicacid, 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; Dobruca et al. 2016).

2. MATERIALS & METHODOLOGY

Zinc oxide (ZnO) nanoparticles were prepared by using zinc acetate dehydrate (C₂H₄O₂Zn) 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

10.975g of zinc acetate dehydrate was dissolved with 100ml of deionized water, and the solution was stirred for half an hour. The 10 ml of NaOH solution was stirred for half an hour. The 10 ml of NaOH solution was
added drop-wise to the above mixture to maintain the pH level 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 °C. And again furnace was also used to dry the material at 400 °C for four hours. The fine pure ZnO nanoparticles were collected by grained the dried cake using mortar.

2.2 Preparation of Plant Extract

Fresh and good quality leaves are collected, and it was washed in running tap water and then again washed using distilled water. These leaves were cut into small pieces using a knife. The 30g of chopped leaves were weighed and taken into a beaker with 100 ml of distilled water. Moreover, it was boiled for 30 minutes at 75 °C. 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)

10.975g of zinc acetate dehydrate was dissolved with 100ml of deionized water and, the solution was stirred for half an hour for the reduction of zinc ions. 10ml of Pesidium guajava leaf extract was added into the 100ml of zinc acetate dehydrate solution. Then the mixture was stirred for half an hour using a magnetic stirrer. The10 ml of NaOH solution was added drop-wise for the above mixture to maintain the pH level 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 grained 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 calculated by using the Debye scherrer formula is given by,

\[ D = \frac{k\lambda}{\beta \cos \theta} \]

Where, k - Wavelength of XRD, \( \lambda \) - Full width half maximum, \( \theta \) - Bragg’s angle, k - Constant

3.2.2 Lattice constants and unit cell volume:

The lattice parameter calculated by the equation of

\[ \frac{1}{d^2} = \frac{4(h^2 +hk +k^2)}{3a^2} + \frac{(l^2)}{c^2} \]

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:

\[ V = (\sqrt{3}/2) + a^2c \] (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, troubleshooting, deformation, Environmental pollutant analysis and more.

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$^{-1}$ is shown in fig 1. The verity of peaks is absorbed in the different wavenumbers. A large number of absorption peaks is 3848.11 cm$^{-1}$ and 3854.96 cm$^{-1}$ it corresponds to the O=H stretching banded (alcohol). The peak at 3441.73 cm$^{-1}$ and 3637.40 cm$^{-1}$ indicates the N=H bond (Amine). The absorbance at 2800 cm$^{-1}$ and 2793.15 cm$^{-1}$ ant it has represented the C=H bond (Alkynes). The absorption peaks at 1657.46 cm$^{-1}$ and 16.77 cm$^{-1}$ it indicates the presence of C=HO stretching (Hydrogen). And then N=O stretching (Nitro) absorption represents the peak at 1413.91 cm$^{-1}$ and 1441.27 cm$^{-1}$, respectively. Those results are indicating that the synthesized ZnO nanoparticles are stabilized by biomolecular constituents present in the leaf extract of Pesidium guajava is shown in Table 1.

### 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 is finely ground, homogenized, and the average bulk composition is 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 then the diffraction peaks of GZnO at 2θ=36.647, 63.056, 67.192 and 69.222 are 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 14.26 nm. 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, and it is shown in table 2.
4.3 SEM Analysis

The morphological analyses of the synthesized materials are analyzed 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 (a) were given the needle structure. Moreover, a capped ZnO nanoparticle (b) shows the spherical structure. The particle size of pure ZnO was 25.45 to 103.36 nm, and capped ZnO nanoparticles are 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 sample. Moreover, it's also used to calculating the chemical composition of the elements. The functional groups of Zn and O are present in the sample. The atomic weight for Zn in PZnO was 40.54%, and O was 59.46%. And the atomic weight of Zn in GZnO was 35.51 and O is 64.49, respectively. The EDX analysis of PZnO and GZnO is shown in fig 4.

6. CONCLUSION

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

- The FTIR analyses reveal the different functional groups are present in the given pure ZnO and capped ZnO.
- XRD pattern shows the presence of hexagonal structure, and it is used to determine the crystalline structure of the sample and unit cell dimensions of the prepared sample PZnO and GZnO.
- SEM confirms the morphological structure of the prepared sample. Moreover, it gives the Needle shapes for the pure ZnO and spherical structure for the Capped ZnO, so the SEM analysis reveals that the capping agent can change some morphological structure of the given samples.
- EDX confirms that the foreign elements are present in the prepared samples. The PZnO and GZnO nanoparticles are having zinc (Zn) and oxide (O).

REFERENCES

Alaghemand, A., Khaghani, S., Bihamta, M. R., Gomarian, M. and Ghorbanpour Mansour., Green synthesis of zinc oxide nanoparticles using Nigella sativa L. extract: The effect on the height and number of branches, J. Nanostruct., 8(1), 82-88 (2018). https://dx.doi.org/10.22052/JNS.2018.01.010
Bala, N., Saha, S., Chakraborty, M., Maiti, M., Das, S., Basu, R. and Nandy, P., Green synthesis of zinc oxide nanoparticles using *Hibiscus subdariffa* leaf extract: Effect of temperature on synthesis, anti-bacterial activity and anti-diabetic activity, *RSC Advances*, 5(7), 4993-5003 (2015).
https://dx.doi.org/10.1039/C4RA12784F

Birusanti, A. B., Mallavarapu, U., Nayakanti Devamma and Espenti, C. S., Plant-Mediated ZnO nanoparticles using *Ficus Racemosa* leaf extract and their characterization, anti-bacterial activity, *Asian J. Pharm. Clin. Res.*, 11(9), 463-467 (2018).
https://dx.doi.org/10.22159/ajpcr.2018.v11i9.28084

Chaudhuri, S. K. and Lalit Malodia, Biosynthesis of zinc oxide nanoparticles using leaf extract of *Calotropis gigantea*: Characterization and its evaluation on tree seedling growth in nursery stage, *Appl. Nanosci.*, 7(8), 501-512 (2017).
https://dx.doi.org/10.1007/s13204-017-0586-7

Divya, M., Vaseeharan, B., Abinaya, M., Vijayaumar, S., Govindarajan, M., Naiyf S. Alharbi., Kadaikunnan, S., Jamal M. Khaled and Benelli, G., Biopolymer gelatin-coated zinc oxide nanoparticles showed high anti-bacterial, antibiofilm and anti-angiogenic activity, *J. Photochem. Photobiol. B.*, 178, 211-218 (2018).
https://dx.doi.org/10.1016/j.jphotobiol.2017.11.008

Dobrucka, R. and Jolanta Długaszewska, Biosynthesis and anti-bacterial activity of ZnO nanoparticles using *Trifolium pratense* flower extract, *Saudi J. Biol. Sci.*, 23(4), 517-523 (2016).
https://dx.doi.org/10.1016/j.sjbs.2015.05.016

Ezealisiji, K. M., Xavier Siwe – Noundou, Blessing Maduelosi, Nkemakolam Nwachukwu and Rui Werner Macedo Krause, Green synthesis of zinc oxide nanoparticles using *Solanum torvum* (L.) leaf extract and evaluation of the toxicological profile of the ZnO nanoparticles—hydrogel composite in Wistar albino rats, *Int. Nano Lett.*, 9, 99-107 (2019).
https://dx.doi.org/10.1007/s40089-018-0263-1

Gnanasangeetha, D. and SaralaThambavani, D., One pot synthesis of zinc oxide nanoparticles via chemical and green method, *Res. J. Mater. Sci.*, 1(7), 01-08 (2013).

Iqbal, J., Abbasi, B. A., Ahmad, R., Shahbaz, Zahra, S. A., Kanwal, S., Munir, A., Rabbani, A. and Mahmood, T., Biogenic synthesis of green and cost effective iron nanoparticles and evaluation of their potential biomedical properties, *J. Mol. Struct.*, 1199, 126979 (2020).
https://dx.doi.org/10.1016/j.molstruc.2019.126979

Kalaiselvi, V. and Mathammal, Rm., Synthesis and characterization of pure and triethanolamine capped hydroxyapatite nanoparticles and its antimicrobial and cytotoxic activities, *Asian J. Chem.*, 30(8), 1696-1700 (2018).
https://dx.doi.org/10.14233/ajchem.2018.21214

Kalaiselvi, V., Mathammal, R., Vijayakumar, S. and Vaseeharan, B., Microwave assisted green synthesis of Hydroxyapatite nanorods using *Moringa oleifera* flower extract and its antimicrobial applications, *Int. J. Vet. Sci. Med.*, 6(2), 286-295 (2018).
https://dx.doi.org/10.1007/s13204-017-0586-7

Manjunatha, R. L., Usharani, K. V. and Naik, D., Synthesis and characterization of ZnO nanoparticles: A review, *J. Pharmacogn Phytotrop.,* 8(3), 1095-1101 (2019).

Patil, B. N. and TariKere, C. T., *Limonia acidissima* L. leaf mediated synthesis of zinc oxide nanoparticles: A potent tool against *Mycobacterium tuberculosis*, *Int. J. Mycobacteriol.*, 5(2), 197-204 (2016).
https://dx.doi.org/10.1016/j.ijmyco.2016.03.004

Ramesh, M., AnbuVannan, M. and Viruthagiri, G., Green synthesis of ZnO nanoparticles using *Solanum nigrum* leaf extract and their anti-bacterial activity, *Spectrochim. Acta A: Mol. Biomol. Spectrosc.*, 136, 864-870 (2015).
https://dx.doi.org/10.1016/j.saa.2014.09.105

Saha, R., Kartik Subramani, PechiMuthu Raju Subbiah Arunachala Kumar, Rangaraj Suriyaprabha, Rajendran Venkatachalam, *Psidium guajava* leaf extract-mediated synthesis of ZnO nanoparticles under different processing parameters for hydrophobic and anti-bacterial finishing over cotton fabrics, *Prog. Org. Coat.*, 124, 80-91 (2018).
https://dx.doi.org/10.1016/j.porgcoat.2018.08.004

Santhoshkumar, J., Venkat Kumar, S. and Rajeshkumar, S., Synthesis of zinc oxide nanoparticles using plant leaf extract against urinary tract infection pathogen, *Resource - Efficient Technologies*, 3(4), 459-465 (2017).
https://dx.doi.org/10.1016/j.refit.2017.05.001

Sutraddhar, P. and Mitali Saha, Green synthesis of zinc oxide nanoparticles using tomato (*Lycopersicon esculentum*) extract and its photovoltaic application, *J. Exp. Nanosci.*, 11(5), 314-327 (2010).
https://dx.doi.org/10.1080/17458080.2015.1059504

Vijayakumar, S. and Vaseeharan, B., Antibiofilm, anti-cancer and ecotoxicity properties of collagen based ZnO nanoparticles, *Advanced Powder Technology*, 29(10), 2331-2345 (2018).
https://dx.doi.org/10.1016/j.apt.2018.06.013
Vijayakumar, S., Vaseeharan, B., Malaikozhundan, B. and Shobiya, Malaikkarasu, Laurus nobilis leaf extract mediated green synthesis of ZnO nanoparticles: characterization and biomedical applications, *Biomed. Pharmacother.*, 84, 1213-1222(2016).
https://dx.doi.org/10.1016/j.biopha.2016.10.038

Vijayakumar, S., Malaikozhundan, B., Gobi, N., Vaseeharan, B. and Murthy, C., Protective effects of chitosan against the hazardous effects of zinc oxide nanoparticle in freshwater crustaceans *Ceriodaphnia cornuta* and *Moinamcrura.* "*Limnologica,* 61(2), 44-51(2016).
https://dx.doi.org/10.1016/j.limno.2016.09.007