WORLD JOURNAL OF PHARMACEUTICAL RESEARCH
SJIF Impact Factor 7.523
Volume 6, Issue 10, 818-826. Research Article ISSN 2277– 7105

BIOSYNTHESIS AND CHARACTERIZATION OF *ZINC OXIDE* NANOPARTICLE USING *FICUS RELIGIOSA* LEAVES EXTRACT

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Article Received on
07 July 2017,

Revised on 28 July 2017,
Accepted on 17 Aug. 2017

DOI: 10.20959/wjpr201710-9365

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ABSTRACT

The objective of this study is to synthesize zinc oxide nanostructures with the most practical ways by using *Ficus Religiosa* leaves extract and characterize the nanostructures. ZnO nanoparticles were synthesized using Zinc Nitrate ($Zn(NO_3)_2$) as a precursor and *Ficus Religiosa* leaves extract solvent and distilled water were used as medium. ZnO nanoparticles were characterized by using XRD, UV-Visible spectroscopy, EDX and SEM, FTIR. Result of EDX characterization shows that the ZnO nanoparticles has good purity with (Zinc content of- 72.48% and; Oxygen content of- 27.52%). XRD result spectrum displays mainly oxygen and zinc peaks, which indicate the crystallinity in nature as exhibited. SEM micrographs shows that synthesized ZnO have a cubical structure. The obtained ZnO nanoparticles are homogenous and consistent in size which corresponds to the XRD result that exhibit good crystallinity.

KEYWORDS: Biosynthesis, XRD, EDX, SEM and FTIR.

INTRODUCTION

Zinc oxide plays an important role in current industry due to its special characteristics such as anti-corrosion, anti-bacteria, has low electrons conductivity and excellent heat resistance. The ZnO nanoparticles are of significant interest as they provide many practical applications worldwide. The most important application of ZnO nanoparticles would be as antibacterial agents. The increases surface area and smaller size of these particles make them an ideal antimicrobial agent. Lot of attention has been diverted to the green synthesis of metal nanoparticles using biological material as the reducing and stabilizing agents and due to the

usage of ecofriendly, non-toxic and safe reagents during the biosynthesis process, green synthesis has been considered in the field of toxic chemical and physical methods.[1-3] In the biological method, plant extracts are used for controlled and precise synthesis of several metallic nanoparticles.[2] High surface and a large fraction of surface atoms are responsible for the nanoparticles' atom-like behavior.[4,5] Despite the fact that conventional methods use less time for synthesizing nanoparticles, they contribute to environmental toxicity because they require toxic chemicals as capping agents. Green nanotechnology is an eco-friendly alternative and is cost effective and utilizes proteins as natural capping agents. Syntheses of metal nanoparticles by plants utilize various secondary metabolites, enzymes, proteins and or other reducing agents.

In the recent years, resistance of fungal infections has emerged as major health problem.[6] *Candida* spp. represents one of the most common pathogens which are responsible for causing hospital acquired sepsis with an associated mortality rate upto 40%.[7] During the past three decades, research in developing green methods has been increased considerably for the search of new and effective medicines of natural origin. Hence, in the present study, we report the synthesis of ZnO NPs, reducing the Zinc ions present in Zinc nitrate by the aqueous extract of *Ficus Religiosa* leaves. Furthermore, these biologically synthesized nanoparticles were found to produce a high fungicidal activity.

EXPERIMENTAL

Preparation of extract

Ficus Religiosa leaves were collected from Mumbai region. They were thoroughly washed thrice with deionized water and dried in oven for 48 hours at 40–50^{o}C. Then, they were crushed into fine powder using grinder. An intense green colored powder of *Ficus Religiosa* was obtained which was used in the present study. 20 g of finely cut *Ficus Religiosa* leaves were boiled in 100 ml water for 10 min and filtered to obtain *Ficus Religiosa* leaves extract. Identification of active phytoconstituents (Table No.1) was done by the methods mentioned in.[8,20]

Table.1 Phytochemical constituents of *Ficus Religiosa* aqueous leaf extract

Phyto-constituents	Reagents	Results
Carbohydrates	Molisch's, Benedict's	+, +
Flavonoids	Alkaline Reagent, Shinoda's	+, +
Alkaloids	Mayer's, Wagner's	+, +
Tannin	Lead acetate, Ferry chloride, Wagner's	+, +, +
Steroids	Liebermann Burchard's	+
Terpenoids	Thionyl chloride	+
Saponin	Gelatin	+
Glycosides	Legal's, Borntrager's	+, +
Reducing Sugar	Fehling solutions	+

+ Presence; – Absence

*all tests have been performed in triplicate.

It is suggested that the aldehyde groups are responsible for reduction of zinc oxide to zinc oxide nanoparticles and also stabilize the nanoparticles.[8,20]

Preparation of zinc oxide nanoparticles

For the ZnO nanoparticles synthesis, 50 ml of *Ficus Religiosa* leaf extract was boiled to 60-80^{o}C using magnetic stirrer and heater. Then 0.02 moles of Zinc Nitrate were added to the leaf extract of *Ficus Religiosa* plant when temperature reaches 60^{o}C. Then boil the solution till it is reduced to deep yellow paste. This paste is dried in oven at temperature 100- 130^{o}C for 40-45 mins. This obtained yellow powder was then collected in a quartz crucible and sintered in a horizontal furnace at 900^{o}C for 3 hours. A white colored powder was obtained and it was carefully collected and packed for further characterization. The material was mashed in a mortar-pestle so as to get a fine nature of particles for characterization[20]

Materials and Characterization

Synthesizing Zinc Oxide nanostructure in this research includes the use of several materials such as Zinc Nitrate ($Zn(NO_3)_2$) ≥99% purity (Sigma aldrich) and *Ficus Religiosa* leaves extract. Zinc Nitrate was used as precursor and plant leaves extract was used as a reagent.

Structural and optical properties of the ZnO nanoparticles were determined by using EDX and Scanning Electron Microscopy (SEM) (Hitachi: H-7500; Resolution: 2 Å), X-ray Diffraction (XRD) (Rikagu Mini-2 using CuKα1, λ = 0.15406 nm radiations), Differential Scaning Colorimetery (DSC) (TA Instruments DSC Q10) in the range 50-600 °C, Fourier Transform Infra-Red spectroscopy (FTIR) (Perkin Elmer, FTIR-380) in the wavelength range of 400 - 4000 cm^{-1} and UV-Visible spectroscopy (Systronic-2203).

RESULTS AND DISCUSSIONS

Zinc Oxide Nanostructures

Based on the experimental work that has been done, there are series of chemical reaction that takes place. The complete hydrolysis of zinc nitrate with the aid of *Ficus Religiosa aqueous* leaves extract solution should result in the formation of a ZnO nanoparticles. The final product was obtained as a result of the equilibrium between the hydrolysis and condensation reaction. Due to the heating, Zinc nitrate within the solution undergoes hydrolysis forming nitrate ions and zinc ions. The abundance of electrons in the oxygen atoms makes the hydroxyl groups (-OH) of leaves extract molecules bond with the zinc ions.[10] The overall chemical reaction to form ZnO nano-powder when *Ficus Religiosa aqueous* leaves extract was used as solvent as well as reagent stated as follow in Eq. (1):

$$Zn(NO_3)_2 + \textit{Ficus Religiosa aqueous}\ \text{leaves extract} \xrightarrow[\text{Stirring}]{\triangle} Zn\text{-}(OH)_2\text{-}2NO_3$$

$$Zn\text{-}(OH)_2\text{-}2NO_3 \xrightarrow{\text{Sintering at } 900^{o}C} ZnO + 2NO_3\uparrow + H_2O\uparrow$$

Zinc hydroxide nitrate is an intermediate product of the hydrolysis reaction, formed in the presence of H_2O and OH^- ions. It can be easily transformed into ZnO at higher temperature and with prolonged refluxing. Nitrate is water soluble and could therefore be removed from the end product. High purity ZnO nano-powder could therefore be obtained successfully by this green technique.[11]

UV-Visible spectroscopy

For analytical study of the prepared sample, the amount of absorption within wave length of 200– 500 nm was observed by UV-Vis spectroscopy. It is known that an absorption band at about 320 nm due to surface plasmon resonance in ZnO nanoparticles. Figure.1 shows the UV-Vis spectra of ZnO nanoparticles recorded between 200 and 500 nm. As illustrated the SPR band cantered 320 nm confirms the formation of ZnO nanoparticles. The increment in the values of optical band gap arises due to improvement in the crystallinity during annealing treatment.[12,13] The energy band gap value is reflected in the inset of Figure1. which comes out to be 3.87 eV, that is, more than pure ZnO (3.37 eV).

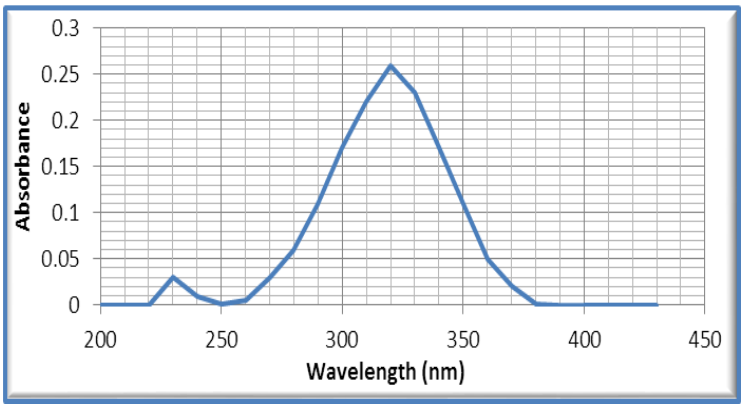

Figure 1: UV-Visible spectrum of Zinc oxide nanopowder.

FTIR spectroscopy

Figure2. shows FTIR spectra of ZnO nanoparticles. Infrared studies were carried out in order to ascertain the purity and nature of the metal nanoparticles. Metal oxides generally give absorption bands in fingerprint region i.e. below 1000 cm^{-1} arising from inter-atomic vibrations. The peak observed at 3452.30 and 1119.15 cm^{-1} are may be due to O-H stretching and deformation, respectively assigned to the water adsorption on the metal surface. The peaks at 1634.00, 620.93 cm^{-1} are corresponding to Zn-O stretching and deformation vibration, respectively. The metal-oxygen frequencies observed for the respective metal oxides are in accordance with literature values.[14,15]

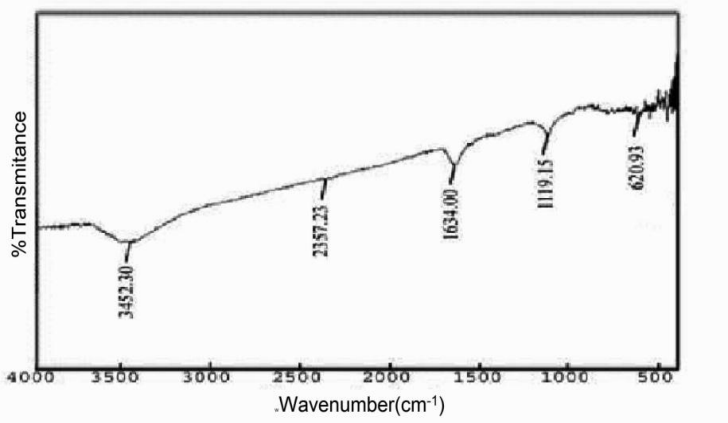

Figure 2: FTIR spectra of ZnO nanoparticles.

X-Ray Diffraction (XRD)

Figure 3 represents the Xray diffraction pattern of ZnO nanopowder. A definite line broadening of the XRD peaks indicates that the prepared material consist of particles in nanoscale range. From this XRD patterns analysis, we determined peak intensity, position and width, full-width at half-maximum (FWHM) data. The diffraction peaks located at

31.84◦, 34.52◦, 36.33◦, 47.63◦, 56.71◦, 62.96◦, 68.13◦, and 69.18◦ have been keenly indexed as hexagonal wurtzite phase of ZnO[16,17] with lattice constants $a = b = 0.324$nm and $c = 0.521$nm (JPCDS card number: 36-1451)[18], and further it also confirms the synthesized nanopowder was free of impurities as it does not contain any characteristics XRD peaks other than ZnO peaks. The synthesized ZnO nanoparticle diameter was calculated using Debye-Scherrer formula[19]

$$d = 0.89\lambda / \beta \cos \theta$$

Where 0.89 is Scherrer's constant, λ is the wavelength of X-rays, θ is the Bragg diffraction angle, and β is the full width at half-maximum (FWHM) of the diffraction peak corresponding to plane (101). The average particle size of the sample was found to be 76.21nm which is derived from the FWHM of more intense peak corresponding to (101) plane located at 36.33◦ using Scherrer's formula.

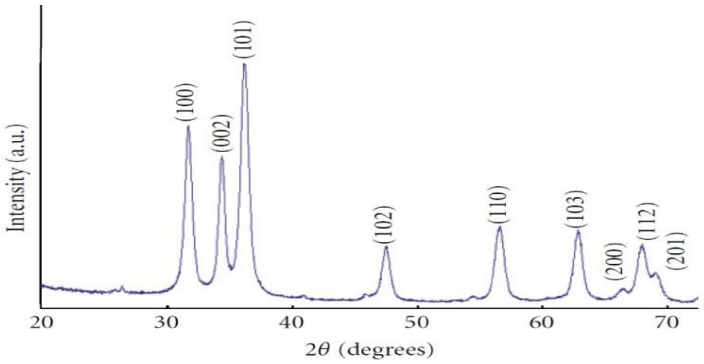

Figure 3: XRD pattern of prepared ZnO nanoparticles.

SEM and EDX analysis

Figure. 4 represents the SEM pictures of ZnO nanoparticles at different magnifications. These pictures confirm the formation of ZnO nanoparticles. These pictures substantiate the approximate cubical shape to the nanoparticles, and most of the particles exhibit some faceting. From the pictures, it also can be seen that the size of the nanoparticle is less than 80 nm which was in good agreement with the particle sizes (80 nm) calculated from the Debye-Scherrer formula.

EDX characterization suggested that the ZnO powder has good purity (Zinc content – 78.74%; Oxygen content – 21.26%) as shown in Figure. 5 and Table 2, in which very little impurities can be seen. Theoretically, expected stoichiometric mass per-cent of Zn and O are 80.3% and 19.7%.[7] The composition of zinc element is higher than in synthesized ZnO nano-powder.

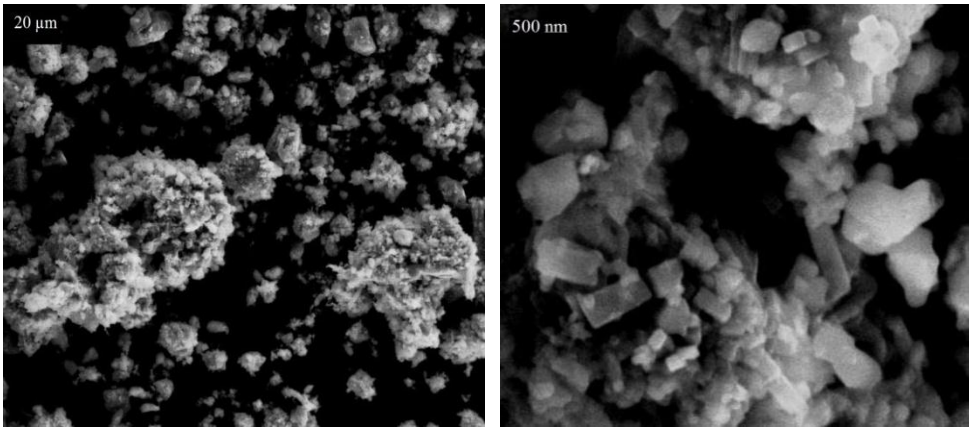

Figure 4: SEM pictures of ZnO nanoparticles at different magnifications.

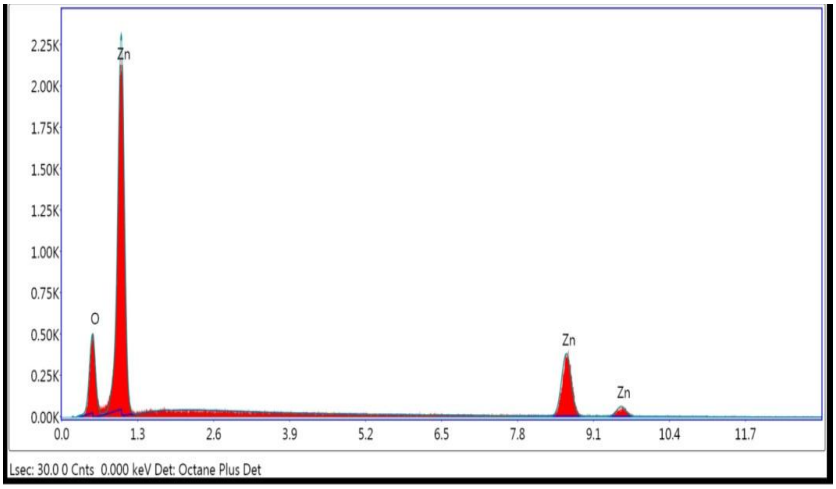

Figure 5: EDX graph of ZnO nanoparticles.

Table 2. EDX specifications of ZnO nanoparticle.

Element	Weight %	Atomic %	Net Int.	Error %	Kratio	Z	R	A	F
O K	27.52	60.80	352.73	9.05	0.11	1.2	0.88	0.34	1
ZnK	72.48	39.20	495.30	3.14	0.66	0.91	1.03	1	1

CONCLUSION

ZnO nanoparticles of hexagonal wurtzite structure are synthesized by *Ficus Religiosa* leaves extract. From SEM study, it is found that ZnO nanoparticles are of cube shape with average size of 70-80 nm. The FTIR spectral analysis reveals the characterstics peaks for Zn-O stretching. The absorption of water molecules on the ZnO nanoparticles is confirmed by FTIR spectra. The UV-Visible study shows blue shift absorption at ~320 nm. Allowed direct band gap energy of ZnO nanoparticles are found to be higher as compared to their bulk counterpart.

ACKNOWLEDGEMENT

We wish to express our sincere gratitude to Mithibai College and Department of Chemistry, for giving us the chance to do research, Head of the Chemistry Department, Mrs. Rajeshwari Mirji, for providing all facilities to work in Laboratory. Earth Science Department, IIT Bombay, for XRD analysis and ICON Analytical Laboratory, Mumbai for SEM and EDS analysis.

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