Research Article

Journal of Environmental Nanotechnology

Green Synthesis and Characterization of ZnO Nanoparticles using *Hibiscus rosa-sinensis* Leaf Extract

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Received: 10.03.2020 Accepted: 22.04.2020 Published: 30-09-2020

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ABSTRACT

The present study was aimed at the synthesis of ZnO nanoparticles using *Hibiscus rosa-sinensis* leaf extract. The synthesized nanoparticles were characterized by using various techniques such as XRD, SEM, EDAX, FTIR and Antibacterial Activities. The synthesized nanoparticles were formed by using zinc nitrate doped with *Hibiscus rosa-sinensis* leaf extract.

Keywords: *Hibiscus rosa-sinensis*; Scanning Electron Microscopy; Zinc oxide.

1. INTRODUCTION

Green synthesis is an emerging area in bio-nanotechnology and provides economic and environmental benefits; it is an alternative to chemical and physical methods. In this method, non-toxic, safe reagents which are eco-friendly and safe are used. Various natural resources available in nature, such as plant extracts, cyclodextrin and chitosan have been studied to synthesize metal oxide nanoparticles.

Zinc (Zn)**,** a chemical element, and a low-melting metal of Group 12 (II b or zinc group) of the Periodic table, is essential to life and is one of the most widely used metals. Zinc is of considerable commercial importance. Zinc is an important trace element in the human body. It is found in high concentrations in the red blood cells as an essential part of the enzyme carbonic anhydrase, which promotes many reactions relating to carbon dioxide metabolism. The zinc present in the pancreas aids in the storage of insulin. Zinc is a component of some enzymes that digest protein in the gastrointestinal tract. Zinc deficiency in nut-bearing and fruit trees causes such diseases as pecan rosette, little leaf and mottle leaf. Zinc in the hemosycotypsin of snails' blood transports oxygen in a way analogous to iron in the human blood (Hong *et al.* 2006; Diallo *et al.* 2015; Chaudhary *et al.* 2019).

Zinc oxide is a white, powdery mineral with a long history of use as sun protection. It is also used to create other products, such as diaper rash ointments and makeup. ZnO nanoparticles easily dissolve in soil and are up-taken by plants. They are employed in a wide range of applications in agriculture due to their unique properties. Results suggest that the application of ZnO NPs could enhance plant growth and development (Hong *et al.* 2009).

Zinc nitrate does not have a broad scale use but is used for the synthesis of coordinating polymers on a laboratory scale. The controlled decomposition of zinc oxide can also be used for the generation of various structures, including nanowires. It can also be used as a dyeing mordant. $Zn(NO_3)_2$ is an inorganic chemical compound with the chemical name Zinc nitrate. It is also called Zinc dinitrate or Celloxan or Zinc Nitrate Hexahydrate. It is widely used as a catalyst to manufacture medicine, dyes and various other chemicals. Inhaling dust causes irritation in the throat and nose. Swallowing Zinc dinitrate can lead to corrosion of the alimentary tract. Contact with the skin results in irritation and can cause rashes (Sirelkhatim *et al.* 2015; Pal *et al.* 2019).

A natural dye has been tried with the *Hibiscus rosa-sinensis* flowers and leaf extract on cotton fabric. The flowers and leaves of the plant are used to produce a lovely reddish Hibiscus dye. Methods such as Ultrasonic Automiser and Padding Mangle were used for dying. Treated sample has moderate to fair color fastness properties. The physical and mechanical properties of the treated samples seemed to be good (Sridar *et al.* 2018). In the present study, the ability of *Hibiscus rosa-sinensis* leaf extract (HLE) to act as a natural coagulant for the water treatment was tested. An insignificant effect of alkalinity on the performance of HLE was observed. The

addition of NaCl increased the dissolution of active coagulation species and enhanced the efficiency of HLE significantly. But the optimal dosages of HLE were lesser than that of alum. Thus, HLE can be used as a coagulant aid for the effective treatment of water.

2. MATERIALS AND METHODS

2.1 Material

All the chemical such as Zinc nitrate, distilled water and other sodium hydroxide ingredients utilized in this work were purchased from Erode, Tamilnadu, India. The leaves of *Hibiscus rosa-sinensis* were collected from in and around Arachalur, Tamilnadu, India.

2.2 Preparation of *Hibiscus rosa-sinensis* Leaf Extract

The plant extract was prepared by taking 20 g of *Hibiscus rosa-sinensis* leaves. The leaves were washed several times using running tap water and then again washed using double distilled water to remove dust particles; then they were dried and boiled for 25 minutes in 100 ml of distilled water. When the color of the solution changed to light green, the extract was filtered and stored at room temperature.

2.3 Green Synthesis of ZnO and Leaf-capped Nanoparticles

Hibiscus rosa-sinensis leaves (Fig. 1) were collected and washed with double distilled water to remove dust particles and then dried to remove residual moisture. The plant extract was prepared by taking 10 g of *Hibiscus rosa-sinensis* leaves and boiled for 25 minutes in 100 ml of distilled water. Then the extract was filtered using Whatman filter paper to get a clear solution. In this method, 10 g of Zinc nitrate was dissolved with 100 ml of distilled water and stirred for about 30 minutes. The leaf extract (10 ml) was added drop-wise into the above solution, which changed the color of the solution to light green; then sodium hydroxide solution was added drop-wise to the mixture to maintain pH level at 12. The synthesized sample was aged for 24 hours. Thus, the residue was kept in a microwave oven at 350 W for 30 minutes. The dried product was grained in a mortar, and then the fine ZnO nanoparticles were obtained.

Fig. 1: *Hibiscus rosa-sinensis*Flower and leaves.

2.3 Characterization Techniques

2.3.1 XRD Analysis

The prepared samples were analyzed using XRD technique. The XRD pattern predicted the lattice parameter (a and c), unit cell volume and crystalline size of the sample. The XRD pattern of prepared samples was well-matched with JCPDS File. The lattice parameter of the sample was calculated using the equation:

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

where, d is the spacing between the planes and a and c are the lattice parameters. The unit cell volume (V) of the sample was found using the equation:

$$V = (\sqrt{3}/2) + a^2 + c^2$$

The average crystalline size of the sample was determined by using Scherrer's formula,

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

where, D denotes the average crystalline size of the sample, K represents the broadening constant, λ denotes the wavelength of CuKα radiation source (1.54 Å), β represents the full width at half-maximum and θ represents the angle of diffraction.

2.3.2 FTIR Analysis

Fourier transform spectroscopy analysis (FTIR) is an analytical methodology used in industry and academic laboratories to understand the structure of individual molecules and the composition of molecular mixtures.

2.3.3 SEM & EDAX Analysis

Scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM/EDX) is the best known and most widely used surface analytical technique. High-resolution images of surface topography with excellent depth of field are produced using a highly focused scanning (primary) electron beam.

3. RESULTS & DISCUSSION

3.1 XRD Analysis

XRD analysis is used to determine the crystalline size and phase identification of the nanoparticles. The XRD pattern of ZnO nanoparticles has been shown in Fig. 2. The XRD pattern indicates that the ZnO sample is of hexagonal structure, and it well-matched with JCPDS files. For ZnO, the average crystalline size was given in Table 1. The diffraction peaks of ZnO nanoparticles occurred at 2θ = 36.44, 47.71 and 69.29 in planes of 101, 102 and 201, respectively.

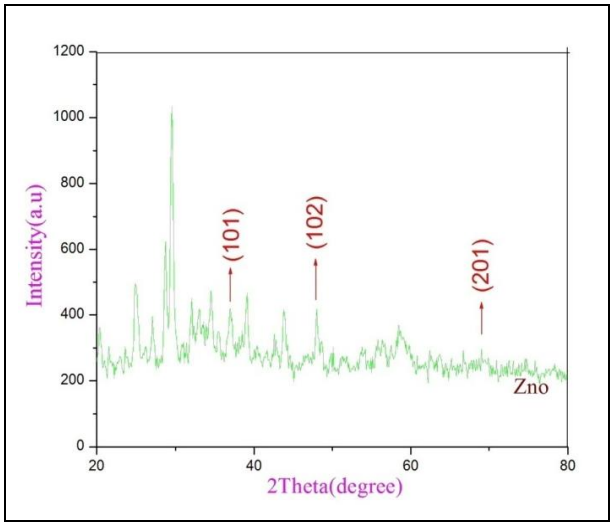

Fig. 2: Synthesized sample with XRD pattern for ZnO.

3.2 FTIR Analysis

FTIR spectra of the prepared ZnO samples were recognized using a wavelength range of 400 – 4000 cm^{-1} (Table 2). The observed peak resulted from the green synthesis method is from 3861.49 to 879.54 cm^{-1}. The vibrations of a variety of groups were present at different wavenumbers of IR radiation. The broad peak was absorbed at 3861.49 cm^{-1} and 3960.65 cm^{-1} (Alcohol), which revealed O-H stretching band. C-H stretching was confirmed from the absorption peak of 2800.64 and 2809.44 cm^{-1} (Alkynes). N=O stretching from the absorption peaks at 1450.47 cm^{-1} and 1450 cm^{-1} (Nitro) were confirmed. The peaks at 3589.53 cm^{-1} and 3446.81 cm^{-1} revealed the stretching vibrations of N-H (Amine) bond. Introducing a capping agent has created a minor change in the functional group analysis of the samples. The spectrum (Fig. 3) reveals the FTIR graph of ZnO.

Table 1. Lattice constant, crystalline size, and unit cell volume of the synthesized samples.

Sample	2θ (deg)	FWHM (deg)	D (Å)	Intensity (Counts)	Crystalline Size (nm)	Average Crystalline Size (nm)	hkl	Lattice Constant		Unit Cell Volume (Å^3)
								a=b	c	
	36.44	0.47	2.46	119	17.79		101			45.60
ZnO	47.71	0.49	1.90	283	17.72	18.47	102	3.1	5.2	45.82
	69.29	0.48	1.35	163	19.92		201			47.81

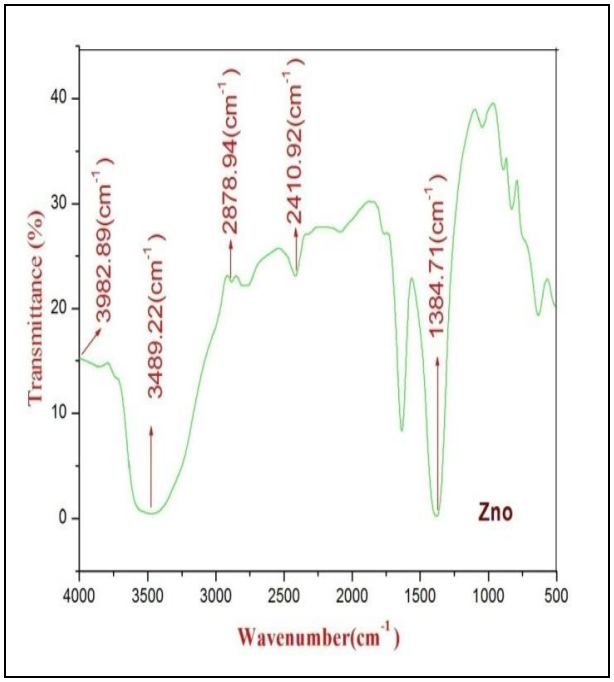

Fig. 3: FTIR spectrum of ZnO.

3.3 SEM and EDAX

The Scanning electron microscope (SEM) analysis determines the shape and morphology of ZnO nanoparticles. Fig. 4 has illustrated the morphological description and elemental composition of ZnO, showing spherical-shaped structure for ZnO. The capping agent can create a minor change in the morphology of the sample.

The Energy Dispersive X-Ray Spectroscopy is used to investigate the elemental composition and chemical analysis of ZnO. The EDAX analyses consist of spectra showing peaks corresponding to the elements making up the accurate composition of the sample.

The analysis observed Zn (Zinc) and O (Oxygen) for ZnO, confirming the purity of the sample. In EDAX, the presence of Zn and O revealed the absence of impurities in the sample. The EDAX analysis has been shown in Fig. 5.

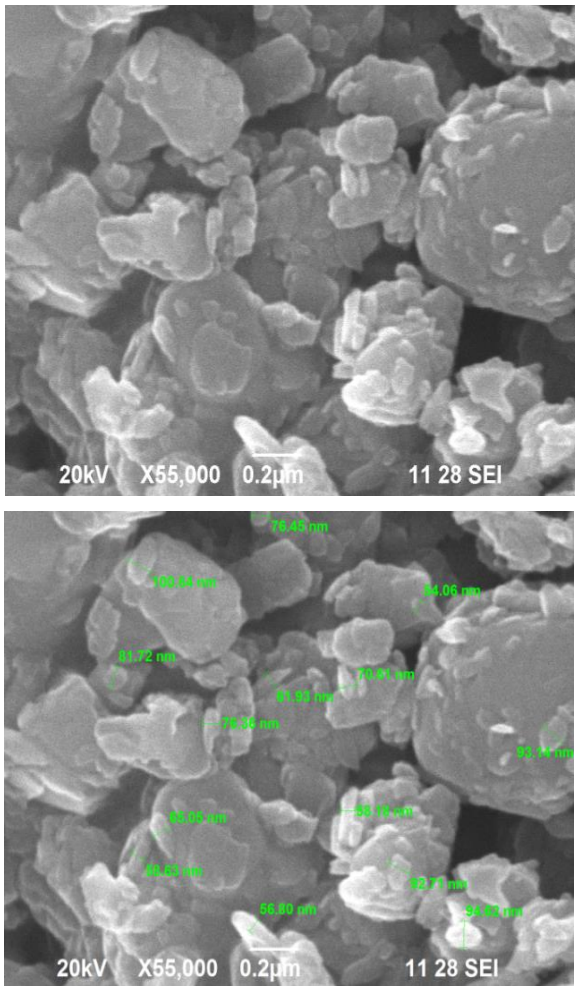

Fig. 4: SEM analysis of ZnO.

Table 2. Functional group of ZnO

Sample	Wave Number (cm^{-1})				
	O-H Stretching vibration (free)	O-H Stretching vibration (banded)	C-H Stretching vibration	N=O Stretching vibration	N-H Stretching vibration
ZnO	2410.92	3982.89	2878.94	1384.71	3489.22

Table 3. EDAX spectra of pure ZnO.

Sample	Element	App Conc.	Intensity Corrn.	Weight%	Weight % Sigma	Atomic %
ZnO	O	43.77	1.3608	47.92	0.69	78.99
	Zn	30.47	0.8718	52.08	0.69	21.01
Total						100.00

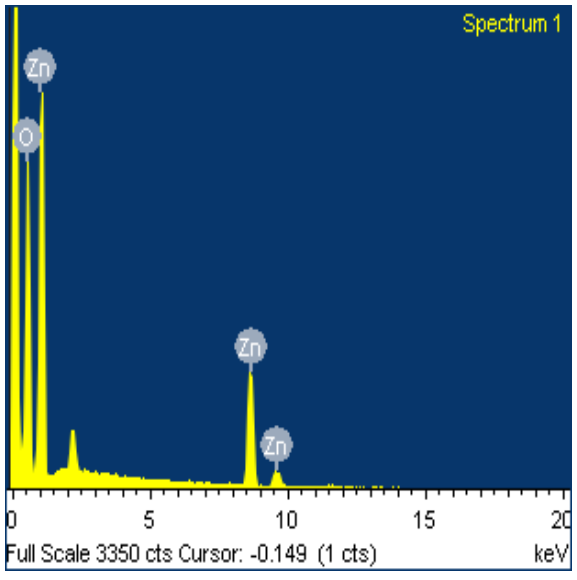

Fig. 5: EDAX analysis of ZnO.

4. CONCLUSION

In this present study, the synthesis of Zinc oxide nanoparticles by the green synthesis method was reported. XRD analysis predicted the crystalline size, lattice parameters and unit cell volume of the sample. The average crystalline size is 18.47 nm. FTIR study revealed the functional groups present in the sample. SEM analysis revealed the morphological structure; it has shown a spherical-shaped morphology. EDAX analysis determined the sample's elemental composition such as Zn (Zinc) and O (Oxygen) for ZnO. It represents the purity of the product.

FUNDING

This research received no specific grant from any funding agency in the public, commercial, or not-for-profit sectors.

CONFLICTS OF INTEREST

The authors declare that there is no conflict of interest.

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