



Solanum Nigrum Leaf Extract capped Synthesis of ZnO Nanoparticles

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ABSTRACT

The present work deals with the green synthesis of Zinc oxide nanoparticles using *Solanum nigrum* leaf extract. The synthesized nanoparticles were characterized by using XRD, SEM, EDAX, and FT-IR. The results of the X-ray diffraction (XRD) pattern shows that the crystal size of the sample and has a hexagonal wurtzite structure. The functional groups present in the sample were analyzed using FT-IR spectroscopy. The morphological surface of the prepared sample was identified using Scanning Electron Microscope (SEM). Then the purity and elemental composition of the sample were determined by Energy Dispersion X-ray Diffraction (EDAX) analysis.

Keywords: FT-IR; Scanning Electron Microscopy; *Solanum nigrum*; Zinc oxide.

1. INTRODUCTION

Many new products and devices have been developed by Nanotechnology with a wide range of applications such as nanomedicine, nanoelectronics and biomaterial energy production and Environmental friendly consumer products. (Hari Priya *et al.* 2014; Manjunatha *et al.* 2014). Definition of nanotechnology was established by the National Nanotechnology Initiative, which defines nanotechnology as the manipulation of matter with at least one dimension sized from 1 to 100 nanometres (Ramesh *et al.* 2015). Dr. Richard Feynman, words "There is a plenty of rooms at the bottom" gave the basic identity for Nanotechnology (Saha *et al.* 2018).

Zinc oxide is a white colour powder insoluble in water. It is applied in products like cosmetics, food supplements, rubbers, glass, cement, plastics, ceramics, ointments, foods, batteries, ferrites, lubricants, paints, sealants, pigments, fire retardants, and first-aid tapes. Most applications use the reactivity of oxide as a precursor to other zinc compounds (Kalaiselvi *et al.* 2018)). The Green synthesis of Nanoparticles is the advanced technique due to its simplicity, inexpensive, and non-toxic in more stable materials (Raut *et al.* 2015). Green chemistry emerged from a variety of existing information and research efforts in the framework of increasing attention to problems of chemical pollution and resource depletion (Santhoshkumar *et al.* 2017). Biological approaches have been developed as green synthesis processes using agents extracted from plants over chemical substances. The advantages of green synthesis in chemical and physical approaches are low cost, low energy and simple technique (Yasotha *et al.* 2020).

Solanum Nigrum is a plant widely used in the medical field. The leaves of this plant are used to treat mouth ulcer. It is known as *Manathakkali keerai* in Tamil Nadu. It is a home remedy for mouth ulcers and used for cooking like spinach. This is considered to be antioxidant, anti-inflammatory, diuretic, and antipyretic. Some researchers suggest that this plant inhibits the growth of cervical carcinoma in mice. The active constituent of the plant, solanines, inhibit the explosion of dissimilar cancer cells in vitro, such as breast cancer and pancreatic cancer. Its anti-tumour mechanism is mainly through the induction of different cell and molecular pathways, leading to apoptosis and autophagy of cells and molecules and inhibiting tumour metastasis (Sutradhar *et al.* 2016; Vijayakumar *et al.* 2020).

2. MATERIALS AND METHODS

2.1 Materials

All chemical such as Zinc nitrate, distilled water, sodium hydroxide used in work were purchased from Erode, Tamilnadu. The leaves of *Solanum nigrum* were collected from in and around Kundadam, Tamilnadu, India (Fig. 1).

2.2 Preparation of *Solanum Nigrum* Leaves Extract

The plant extract was prepared by taking 20g of *Solanum nigrum* leaves. The leaves were washed several time using distilled water and boiled 25 minutes in 100ml of distilled water. Once the solution was changed light green colour, the extract was filtered and stored at room temperature for further study.

2.3 Synthesis of ZnO Leaf Capped Nanoparticles

Take 10ml of leaf extract and 5g of zinc nitrate in separate beakers with 50ml of distilled water, and it was stirred for 25 minutes by using a magnetic stirrer. The leaf extract was added dropwise into the zinc nitrate solution, and this mixture was stirred 25 minutes by using a magnetic stirrer. The colour of the solution was changed to light green and sodium hydroxide solution was added dropwise to the mixture to maintain pH of 12. The synthesized sample was ageing at 24 hours. Thus the settled precipitate was kept in a microwave oven at 350w for 30 minutes. The dried sample was grained using a mortar, and then the fine powder of *Solanum nigrum* doped ZnO nanoparticles was obtained.



Fig. 1: *Solanum nigrum*

3. CHARACTERIZATION TECHNIQUES

3.1 XRD

The prepared samples were analysed using XRD (X-ray diffraction) technique. This XRD pattern predicts 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 card no: 36-1451 (which is corresponding to the hexagonal Wurtzite phase). The lattice parameter of the sample is where d is the spacing between the planes, a and c are the lattice parameter. The unit cell calculated using the following equation

$$1/d_2 = (4(h_2+hk+k_2)/3a^2) + (l_2/c_2)$$

Volume (V) of the sample was described using the given equation:

$$V = (\sqrt{3}/2)a_2+c_2$$

The average crystalline size of the sample was determined by using the 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 Cu K α radiation source(1.54Å^o), β represents the full width at half maximum, angle of diffraction is denoted by θ (Sangeetha *et al.* 2011; Atena Alaghemand *et al.* 2018).

3.2 SEM

A scanning electron microscope (SEM) is a type of electron microscope that produces images of a sample by scanning the surface with a focused beam of electrons. The electrons interact with atoms in the sample, producing various signals that contain information about the surface topography and composition of the sample. SEM can achieve resolution better than 1 nm (Vijayakumar *et al.* 2016).

3.3 FT-IR

Fourier Transform Infrared Spectroscopy (FT-IR) analysis, or FTIR Spectroscopy, is an analytical technique used to identify organic, polymeric, and, in some cases, inorganic materials. The FT-IR spectroscopy uses infrared light to scan test samples and observe chemical properties (Kalaiselvi *et al.* 2014).

3.4 EDX

The intensity or area of a peak in an EDX spectrum is proportional to the concentration of the corresponding element in the specimen. For determining elemental content, the electron-beam current is assumed to be uniform throughout the specimen, and electron channelling is avoided by avoiding strong diffraction conditions. (Ramya *et al.* 2020).

4. RESULTS AND DISCUSSION

4.1 XRD

XRD Patten reveals the grain size of the sample (Fig. 2). The average crystalline size (D) of the Zinc Oxide is 8.07nm. The crystalline size of the nanoparticle was determined by peak values. The diffraction peak of ZnO nanoparticle at $2\Theta=34.68^\circ, 36.49^\circ, 47.60^\circ$ and hkl planes(002), (101), (102) are observed (table 1). The crystalline size is calculated by the Debye formula can be written as,

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

Where,

K \longrightarrow is the constant of 0.9.

λ \longrightarrow is the X-ray wavelength.

β \longrightarrow is the width half maximum [FWHM] of the peak in XRD patten.

Θ \longrightarrow is Bragg's angle.

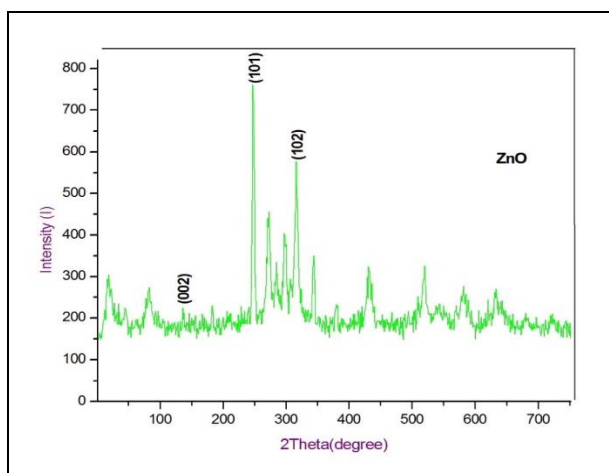


Fig. 2: XRD patten of ZnO

4.2 FT-IR Analysis

FT-IR spectrum of the prepared ZnO samples was recognized using at a wavelength range of 400 – 4000 cm^{-1} is shown in Fig. 3. The vibrations of a variety of groups are present in the sample at different wavenumbers of IR radiation. The broad peak absorbed at 3952.14cm^{-1} - 3479.12cm^{-1} . This can be in contact with

O- H stretching bonded. C-H stretching confirms the absorption peak at 2885.51 cm^{-1} (Alkynes). C- N stretching is confirmed from the absorption peaks at 1384.89 cm^{-1} (Nitro). The peak observed at 3469.94 cm^{-1} was due to stretching vibrations of N-H (Amine) bond (table 2). Introducing a capping agent has created a minor change in the functional group analysis of the samples. Fig 3 reveals the FTIR graph of and ZnO.

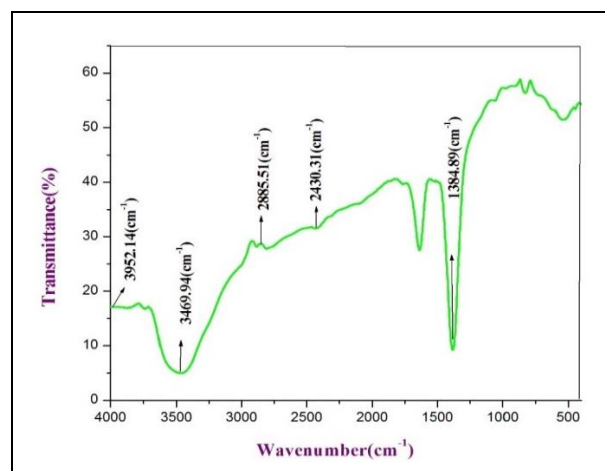


Fig. 3: FTIR spectrum of ZnO

Table 1: XRD pattern of prepared sample

Sample name	2θ (deg)	Crystalline size (nm)	Average Crystallize Size (nm)	hkl value	Lattice Constant (Å)		Unit cell volume (Å)
					a=b	C	
ZnO	34.68	8.20	8.07	002	3.2	5.1	46.22
	36.49	8.16		101			46.99
	47.60	7.86		102			47.44

Table .2: FTIR analysis of ZnO nanoparticles

Sample name	Wave Number(cm^{-1})				
	O-H Stretching vibration	C-H Stretching vibration	C-C Stretching vibration	C-C Stretching vibration(Bond)	C-N Stretching vibration
ZnO	3479.12	2885.52	1636.60	1553.42	1386.27

4.3 Scanning Elector Microscope Analysis

A scanning electron microscope is a useful technique to determine the morphology and particle size of the sample. The SEM analysis shows the cluster shape particle aggregated surface (Fig. 4).

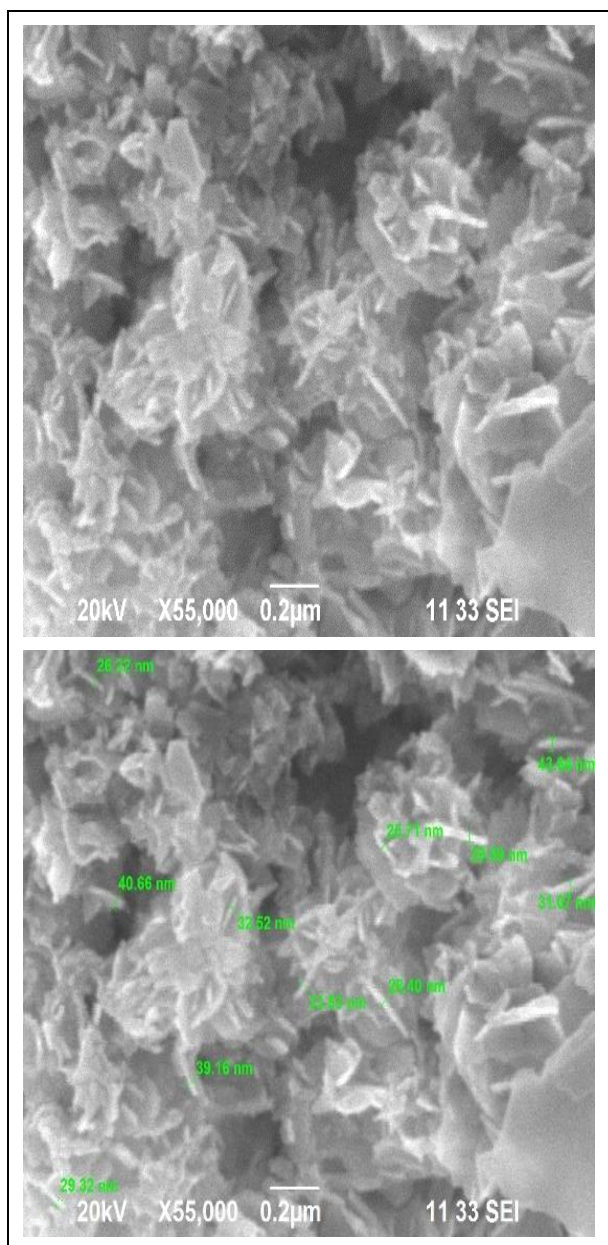


Fig. 4: SEM image of ZnO

4.4 Energy Dispersive X-Ray Diffraction Analysis

The dispersive energy X-ray (EDX) spectroscopy used to investigate the chemical composition of the material. The EDX results confirm the presence of the element. This is composed of Zn (Zinc) and O (Oxygen), as shown in Fig (5). It demonstrates the purity of the sample.

Table 3.3: EDAX spectra of capped ZnO

Element	App conc	Intensity corn	Weight	Weight sigma	Atomic%
O	36.05	1.2696	40.63	0.75	73.66
Zn	36.91	0.8895	59.37	0.75	26.34
Totals			100.00		

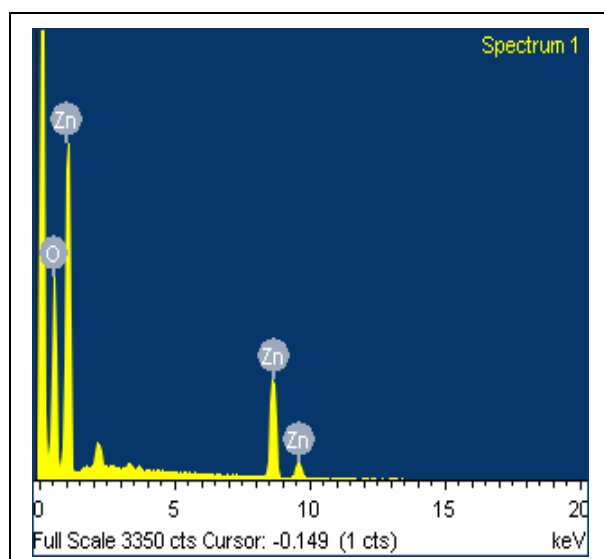


Fig. 5: EDX spectra of capped ZnO

5. CONCLUSION

The present study of ZnO Nanoparticles with and without capping agent of solanum nigrum leaf extract was synthesized by sol gel assisted microwave irradiation method. The synthesized sample was characterized using XRD, FTIR, SEM, EDAX. The XRD pattern indicates the crystalline size of the sample, and the average crystalline size is 8. 07nm. Then SEM analysis reveals the cluster shape particle morphology in the surface. The FTIR spectrum confirms the function group of the sample. EDX analysis predicts the presence of chemical composition in the sample. It confirms the ZnO nanoparticles.

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CONFLICTS OF INTEREST

The authors declare that there is no conflict of interest.

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