Characterization of zinc oxide nano particles synthesized via chemical and green method

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In recent years, the development of efficient green chemistry methods for synthesis of metal oxide nanoparticles has become a major focus of researchers. They have investigated in order to find an eco friendly technique for production of metal oxide nanoparticles. In this work our aim to synthesize of zinc oxide nano particles via chemical and green method. The zinc oxide nano particles were synthesized by mixing zinc sulphate ($ZnSO_4$) solanum procumbens extract and KOH. The synthesized zinc oxide nano-particles were characterized by XRD, FT-IR and UV-vis spectroscopy and Photoluminescence studies. Further, the synthesized zinc oxide nano particles were tested for antibacterial activity by stand art disc diffusion method.

(Received June 8, 2023; Accepted September 5, 2023)

Keywords: Green synthesis, ZnO nano particles, XRD, FTIR, UV, PL

1. Introduction

Nanotechnology is the device for intend, invention, characterization and applications of nanostructure materials. It generally deals with the structures sized between 1-100 nanometers in at least one dimension. It has been emerged as an increasing and quickly changing field and presents possible opportunities to build better materials and products [1-5]. Nanostructured materials are a strictly substantial object that possesses optical and electrical properties that depend remarkably on the dimension and shape of the nanoparticles. This is due to internment of the charge carriers in the thin space of the nanocrystal. The properties of nanoparitcles powerfully depend on their size. Their high specific surface area results in high chemical reactivity. The decrease of their size also leads to an increase of the band-gap energy that is known as quantum size effect.

ZnO nanoparticles, due to their unique physical and chemical properties and low cost preparation, have been of great interest recently. ZnO nanoparticles have varied applications as heat transfer systems [6], as super strong materials [7], as sensors [8] and as catalysts [9]. Their other properties like antimicrobial activity, disinfecting property and stability as matrix spring particles can be further exploited for use in wall paints and plasters to coat hospital equipment [10].

The synthesis of ZnO nanoparticles is an active area of academic and, more importantly, application research in nanotechnology. A variety of chemical and physical procedures such as chemical reduction [11], electrochemical reduction [12], chemical vapor deposition [13], thermal decomposition [14] and solvo thermal reduction [15] have been reported for synthesis of metallic nanoparticles. However, these methods have many problems including use of toxic solvents, generation of hazardous byproducts, high energy consumption and are non eco friendly. Taking this aspect into consideration there is an essential need to develop clean, reliable, biocompatible, cost-effective, environmentally friendly and sustainable procedures for synthesis of nanoparticles [16].

Considering the vast potentiality of plants as sources for the green synthesis of different nanoparticles, and especially ZnO nanoparticles researchers worked with plant extracts, some specific plant parts or whole plant for the green synthesis [17]. Many of them reported that extracts from plants like Moringa oleifera [18], Calolropis procera leaves [19], Pongamia pinnata & Cassia

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https://doi.org/10.15251/JOR.2023.195.505

fistula [20,21], Ixora coccinea leaf [22] efficiently yield ZnO nanoparticles on green synthesis and so were used for the same. The present study also concentrated on green synthesis of ZnO nanoparticles using solanum procumbens.

2. Materials and methods

Solanum trilobetum (thoothuvalai) had been collected from our college campus (sadakathullah appa college).it has washed with normal water after washed by distilled water twice. Using a grinder extraction has been prepared up to the level of 50 ml. in the midst of the time the precursor material 4.095gm of zinc sulphate ($ZnSo_4$) salt has taken into a beaker and added with distilled water of 100ml to dissolved it. The setup was kept on magnetic stirrer for one hour. After one hour the leaf extract has been added slowly using a pipette in to the salt water at a temperature of 95^0C.During this process the precipitating agent KOH was added to change the PH level. The process has been run about minimum 3 hours. Ending of this process the sample has filter with Waltman filter paper. Then the precipitate has been taken out and swapped on the Petri dish for put in an oven at 100^0C. The sample has been taken out after 48 hours for calcination. Before Calcination the sample weight has 3 grams. After Calcination under 375^0 at one hour the yield of the sample was 2.632 grams respectively.

3. Characterization of ZnO nanoparticles

The optical properties were investigated using a UV-Vis-DRS were recorded in air at room temperature in the wave length range of 200-500 nm using Shimadzu UV - 2450 spectrophotometer. Surface structure was characterized by a Fourier-transform infra red (FT-IR) spectrophotometer (JASCO FT-IR 460 plus). The crystalline structure of the nanoparticles was studied by an X-ray diffractometer (XRD; XPERT PRO X-RAY) with Cu Kα radiation at 25 ºC and the structural assignments were made with reference to the JCPDS powder diffraction files. The photoluminescence (PL) emission and excitation spectra were recorded at room temperature by use of a Shimadzu RF-5301 PC spectro fluoro photometer and also the synthesized zinc oxide nano particles were tested for antibacterial activity by standard disc diffusion method.

4. Results and discussions

4.1. XRD studies

The XRD measures were carried out at room temperature using panalytical Xpert-Pro software. Identification of phases was carried out by comparing the diffraction pattern obtained from XRD with standard JCPDS database. The lattice parameters and cell volume were calculated using UNIT CELL software. Figure .1 shows that XRD pattern of ZnO nano particles and Figure .2 shows that Comparison between observed XRD and JCPDS No:8913-97. A good match of peaks is absorbed when the data is compared with JCPDS no:8913-97. The peaks at 31.83, 34.55, 36.41 ,47.60, 56.64,66.37 and 72.56 is indexed as (1 0 0), (0 0 2), (1 0 1) (1 0 2) , (1 1 0), (2 0 0), (0 0 4) and it is found that hexagonal wurtzite structure . The lattice parameters are (a) 3.253Å, and (c) 5.231Å. Figure1. XRD pattern of ZnO nanoparticles, Figure 2. Comparison between observed XRD and JCPDS No: 8913-97.

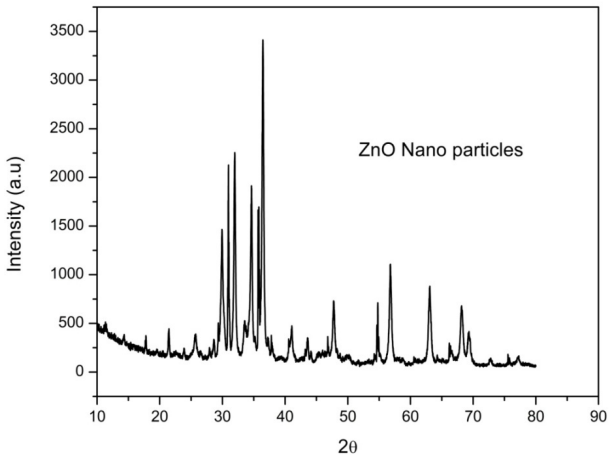

Fig. 1. XRD pattern of ZnO nanoparticles.

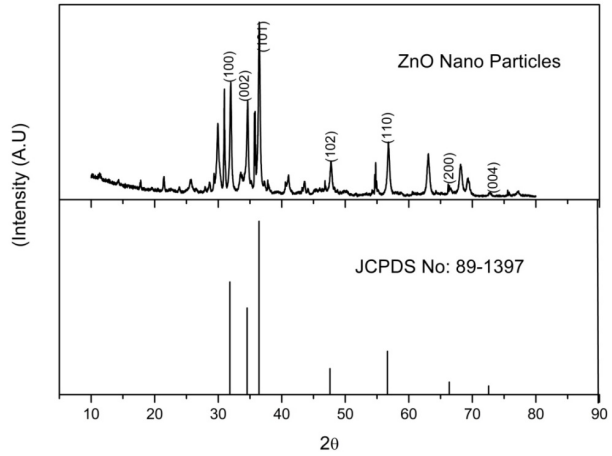

Fig. 2. Comparison between observed XRD and JCPDS No: 8913-97.

The average crystallite size (D) was calculated from the full-width at half-maximum (FWHM) of the most intense peak of the (101) plane of ZnO nanoparticles using the Debye Scherrer formula for spherical particles [Eq. (1)]. $D = 0.89\lambda/ (\beta \cos \theta)$ (1) Where λ is the wavelength (Cu Kα), β is the full width at the half-maximum of the ZnO nanoparticles and θ is the diffraction angle. From this equation the average particle size was estimated to be 51.20 nm.

Williamson and Hall proposed a method for calculating size and strain broadening by looking at the peak width as a function of 2θ. W-H plot is shown in Figure.3. It is plotted with sin θ on the x- axis and $\beta\cos\theta$ on the y-axis (in radians). A linear fit is got for the data and from it; particle size (52 nm) and strain (0.00472) are extracted from y intercept and slope respectively.

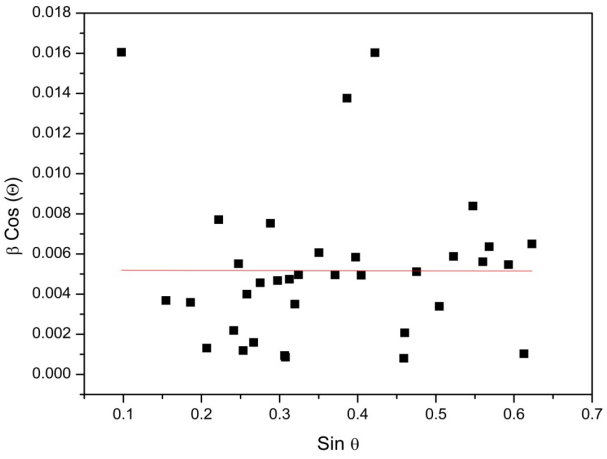

Fig. 3. WH flot for ZnO nanoparticle.

4.2 FTIR analysis

Figure 4 below shows that 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 3450 and 1120 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 1682.00, 620 cm^{-1} are correspond to Zn-O stretching and deformation vibration, respectively. The metal-oxygen frequencies observed for the respective metal oxides are in accordance with literature values [23] reported similar FTIR spectra observed of zinc oxide nanoparticles in their investigation.

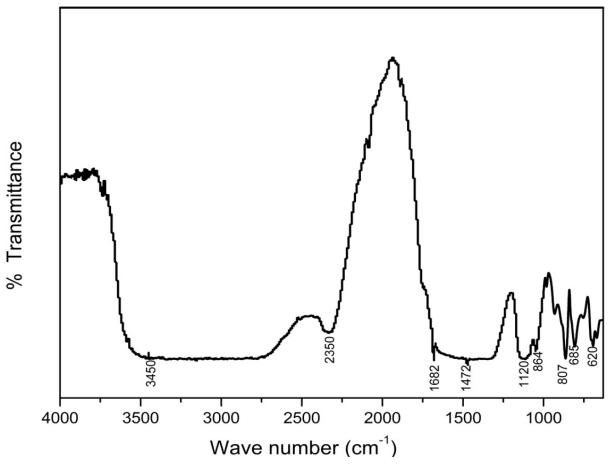

Fig. 4. FTIR Spectra of ZnO nano particles.

4.3. UV spectral studies

Optical absorption properties of the ZnO nanoparticles were investigated at room temperature by UV- Vis Spectroscopy. Figure 5 show the absorbance spectrum of the ZnO sample with absorption band in 360 nm wavelength range.

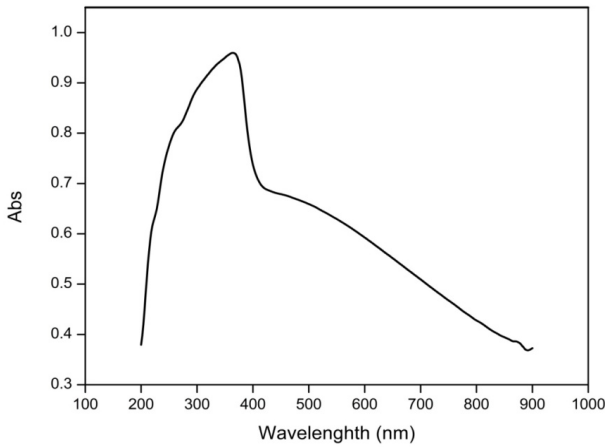

Fig. 5. The absorbance spectrum of ZnO nano particles.

4.4. Tauc's plot

The optical band-gap energy was calculated using Tauc's equation

$$\alpha h\upsilon = k(h\upsilon - Eg)^n$$

where k is a constant, hυ is the photon energy, Eg is the allowed energy gap, n=½ for allowed direct transition, and n=2 for allowed indirect transition. Tauc plot (αhν versus hν) is shown in Figure 6 and the band-gap energy was calculated as 2.82 eV for ZnO NPs synthesized using solanum procumbens leaf extract which was in good agreement with the result [24].

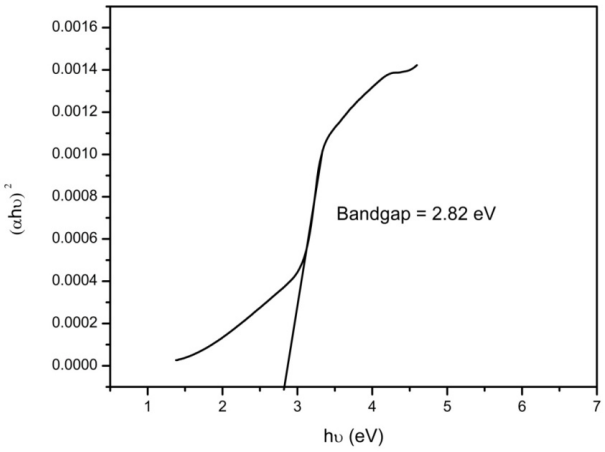

Fig. 6. Tauc Plot for ZnO nano particles.

4.5. Antibacterial activity

Streptomycin is an antibiotic used to treat a number of bacterial infections. This includes tuberculosis, Mycobacterium avium complex, endocarditis, brucellosis, Burkholderia infection, plague, tularemia, and rat bite fever. For active tuberculosis it is often given together with isoniazid, rifampicin, and pyrazinamide. It is given by injection into a vein or muscle.

The antimicrobial activity of bio synthesized ZnO nanoparticles was analyzed against Streptomycin, by disc diffusion method for different concentration. It was observed that microbial growth, decrease with the increase in concentration of biosynthesized ZnO nanoparticles. The ZnO nanoparticles synthesized from solanum procumbens leaf extract are nontoxic to multidrug

resistant microorganisms. From this study it showed that they have great potential in biomedical applications, Also, because of the biological reducing and capping agents these ZnO nanopartices are also environment friendly.

The zone of inhibition in the gram negative bacteria is tabulated in the table 1. It is observed that 50 µl samples showed more inhibition zone.

Table 1. Antibacterial Activity data for the ZnO nanoparticles.

Sample	**E.coli**			
Solanum Procumbens	**10µl**	**30µl**	**50µl**	**Streptomycin**
ethanol(mm)	0.6	1.3	1.5	1.8

4.6. PL spectra

Photoluminescence spectroscopy is used for the essential interpretation of chemical compositions, structure, impurities, energy transfer, photoelectric activity and electronic structure of nano particles. Figure 7 shows that the photoluminescence spectra of ZnO is recorded at room temperature. The spectrum exhibits two emission peaks one is located at 390 nm corresponding to near band gap excitonic emission and the other located at 520 nm due to the presence of singly ionized oxygen vacancies .The emission is caused by the radiative recombination of a photogenerated hole with an electron occupying the oxygen vacancies .Further the spectrum also reveals the narrow size distribution of nano particles in the powder as the luminescence peak full width half-maximum is only in few nanometers[25].

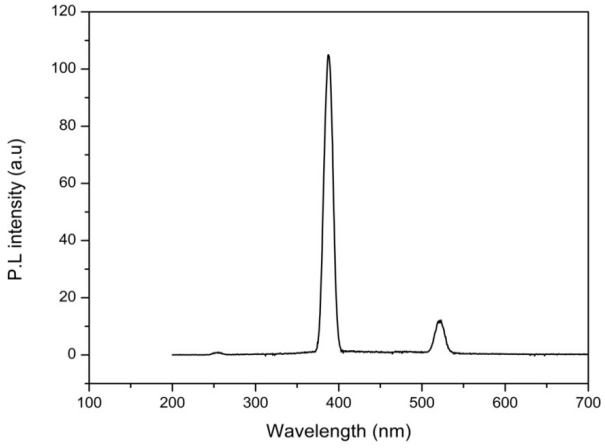

Fig. 7. The PL spectra of ZnO nanoparticles.

5. Conclusion

We synthesized zinc oxide nanoparticles by Chemical and green method using zinc sulfate and KOH in distilled water. XRD study gives pure hexagonal wurtzite structure of ZnO with size of 51.20 nm. The vast potentiality of plants as sources for the green synthesis of different nanoparticles and especially ZnO nanoparticles with plant extracts for the green synthesis. The present study on green synthesis of ZnO nanoparticles using solanum procumbens were more reliable, variety of application including health care, antibacterial activities and industry application.

Acknowledgements

The authors are thankful to the Sadakathullah Appa College, Tirunelveli for rendering the support for the research.

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