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Synthesis and Characterisation of ZnO Nanoparticals by XRD, EDX, SEM, FTIR and UV-DRS

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ABSTRACT

Nano sized materials have been an important subject in basic and applied science. ZnO nano particle were synthesized using a simple co-precipitation method with zinc nitrate and poly ethylene glycol as a starting material. The sample where characterized by X-ray diffraction (XRD) Scanning electron, microscopy (SEM), Energy dispersive spectroscopy(EDX), SEM images show various morphological changes of ZnO obtained by the above method. The average crystallite sizes of the samples were calculated from the full width at half maximum of XRD peaks by using Debye-Scherer's formula. EDS shows that the above route produced highly pure ZnO nanostructures. The optical band gaps of various ZnO powders were calculated from UV-visible diffuse reflectance spectroscopic studies. Antibacterial activity of ZnO nanoparticls is tested against gram positive and gram negative bacteria by using agar-well method.

Keywords: Nanoparticles, XRD, SEM, Antibacterial Activity.

I. INTRODUCTION

Nanotechnology research has gained momentum in the recent years by providing innovative solution in the field of biomedical, materials science, optics and electronics. Nanotecnology can be useful in diagnostic techniques, drug delivery, sunscreens, antimicrobial bandages and disinfectant.

The many metals are research in nanomaterials like TiO2, Fe_2O_3, CdO, ZnO, etc. But ZnO is an interesting semiconductor material due to its application on solar cells gas sensors, ceramics, catalysts, cosmetics and varistors [1]. ZnO is a wide band gap semiconductor with an energy gap of 3.37 ev at room temperature. It has been used considerably for its catalytic, electrical, optoelectronic and photochemical properties. [2-5]. ZnO nanostructures have a great advantage to apply to a catalytic reaction process due to their large surface area and high catalytic activity [6]. ZnO crystallize in hexagonal wurtzite. It is lattice symmetry properties in piezoelectricity and pyroelectricity of hexagonal ZnO [7].

Many chemical routes to synthesized ZnO have been reported in literature such as, hydrothermal methods [10], sol-gel [8], thermal decomposition [9], co-precipitation [11] and electrophoretic deposition [12]. Now we are present a simple co-precipitation method to synthesize uniform, spherically shaped and pure ZnO nanoparticles using zinc nitrate and poly-ethylene glycol as a metal precursor ammonia as a precipitating agent.

X-ray diffraction shows that the synthesized ZnO nanoparticles have hexagonal crystal structure. Scanning electron microscopy (SEM) reveals that the synthesized nanoparticles acquire uniform morphology. Energy dispersive X-ray spectroscopy (EDX) confirms that the synthesis process yields pure ZnO nanoparticls. UV-Visible absorption spectroscopy is a band gap semiconductor with an energy gap.The antibacterial activity of ZnO nanoparticles is systematically tested against gram

positive and bacteria and gram negative bacteria by using agar well method.

II. METHODS AND MATERIAL

Zinc nitrate $(Zn(NO_3)_2).6H_2O$ and poly ethylene glycol (PEG-500) were used in the experiment. The ammonia (NH3) as a precipitating agent and deionized water is used for the preparation of solutions.

The $Zn(NO)_3.6H_2O$ and polyethylene glycol (PEG-500) are mixed together and grinding using an agate mortar. Then this mixture was dissolved in 300ml deionized water. The solution was ultrasonically dispersed in ultrasonic bath and solution added excess of ammonia will get precipitate is obtained. The solution reflux for 4 to 5 hours synthesized white precipitate was isolated by filtration washed with deionized water and ethanol. Then, dried in a vacuum oven at 600 °c for 24 hours to obtain the powder.

III. RESULTS AND DISCUSSION

The prepared sample was characterized by various sophisticated Techniques. The X-ray analysis (XRD) of samples is obtained using Philips X-ray diffractometer with diffraction angle 2θ in between 20 to 80° using Cu-Kα radiation of wavelength 1·54058 Å. Surface morphology and elemental analysis of the samples were carried out using scanning electron microscopy with electron dispersion spectroscopy (SEM-EDS) characterization conducted using a JEOL-JED 2300 (LA) instrument. The light absorption by sample was carried out by using Varian Carry 5000 (UV-VIS-DRS) spectroscopy in the range 800-300 nm.

Figure 1. Shows that the ZnO nanoparticles are XRD peaks significantly broader as compared to bulk samples [15]. Briefly, a peak in the diffracted intensities has been shows formation of nanoparticles in the crystal [16]. XRD pattern of the sample has prominent peaks at two theta values of 31.730, 34.370, 36.210, 47.470, 56.550, 62.770, corresponding to (100), (002), (101), (102), (110), (103). The lattice constants of our sample are found to be a = b = 3.242 A0 and c = 5.176A0 in good agreement with the values of hexagonal ZnO. The (hkl) values are also in good agreement with the standard card of ZnO powder [13]

The particle size was calculated by Debye-Scherer's equation using most intense peak (101)

$$D = \frac{k\lambda}{\beta \cos\theta}$$

Where D is the crystallite size, K is a constant (shape factor about 0.9) λ is the x-ray wavelength (1.5405 nm), β is the full width at half maximum (FWHM) of the diffraction line, and Θ is the diffraction angle. Average particle size was found to be 10.71 nm.

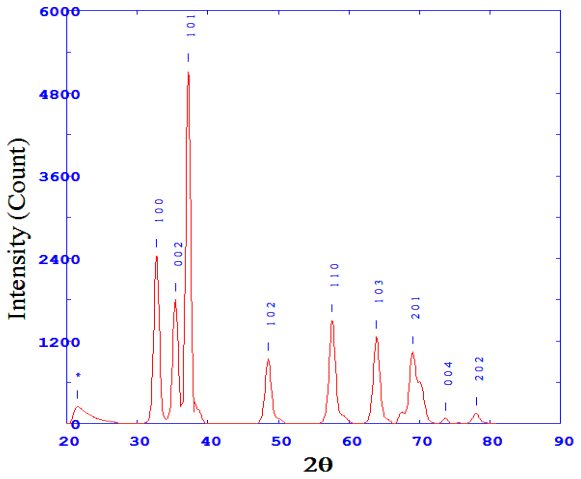
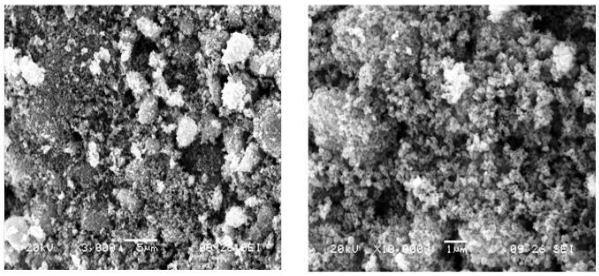


Figure 1. XRD Pattern of zinc oxide calcined at 500°C

The SEM image of ZnO nanoparticle is shown in figure 2 a high degree of agglomeration is clearly visible in agreement with previous studies [14]. The observation of some larger nanoparticles in SEM image is attributed to agglomeration [17]. EDX spectrum of ZnO nanoparticles. A sample is shown in fig (3). The names and percentages of the elements further ZnO sample are shown in the labeling clearly, Zn and O are the main constituents of the sample [20].and no trace of impurities could be found within the detection limit of EDX.



ZnO -A ZnO -B

Figure 2. SEM images of ZnO nanoparticles calcined at 500 °C with PEG as a surface directing agent.

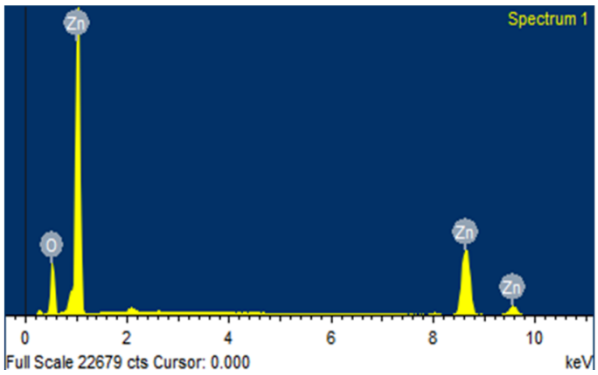


Figure 3. EDX Pattern of ZnO nanoparticles

UV-Visible diffusion reflectance spectroscopy (UV-DRS). The band gap energies (Ebg) of the ZnO nanoparticles were probed by UV-Visible diffusion reflectance spectroscopy. (DRS) determine band gap of the prepared materials. Fig.(4) shows that The diffusion reflectance spectra were recorded and cut off wave length at which absorption sharp edge rises were determined by drawing a tangent on this curves (394). The band gap energies were calculated by using cut off wavelength are represented in the following table. The band gap energy is calcuted by using the hollowing equation Ebg =1,240/λ. Where **λ is** the wavelength in nanometer and Ebg is the band gap energy of synthesized nanomaterial was found to be 3.14 ev.

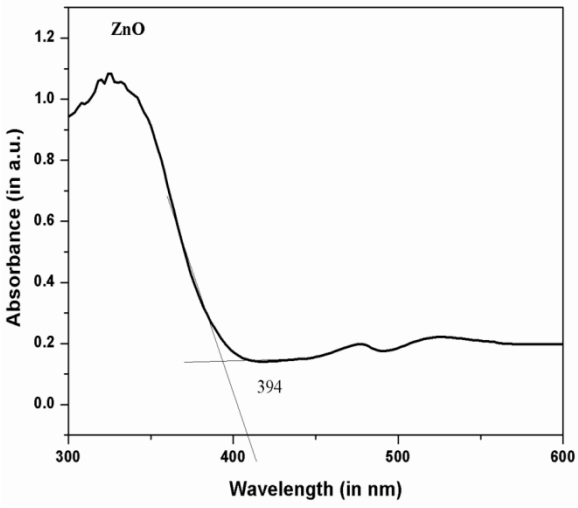


Figure 4. DRS image o f ZnO nanoparticles.

The antimicrobial test of ZnO nano particles was taken against following micro-organism. S. aureus, B. cereus, E. coli, P. aeruginosa. Anti-microbial activity was determined by Agar Cup Diffusion Method. The ZnO nanoparticle were dissolved in DMSO i.e. di-methyl sulphoxide and ultrasonicate.

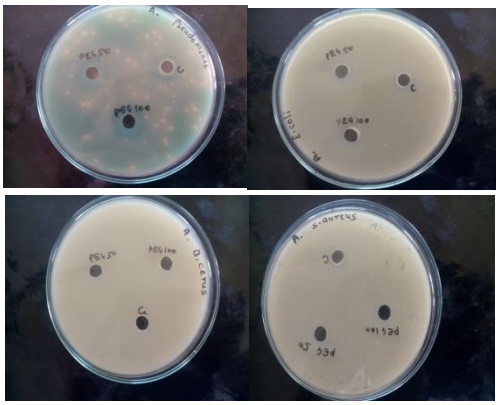


Figure 5. Antimicrobial activity.

Table 1.

Sr. No.	Test microorganisms	Sample I Zone of inhibition in'mm'	
		50µg/ml	100µg/ml
1	S. aureus	**15**	**13**
2	B. cereus	-	-
3	E. coli	**09**	**14**
4	P. aeruginosa	**08**	**10**
5	Negative control	-	-

IV. CONCLUSION

ZnO nanoparticles were prepared by a simple, fast and low cost co-precipitation method. The synthesis of ZnO nanostructures (spherical morphology) EDX shows the elemental composition of synthesized nanoparticles. Synthesized nanoparticles show 3.14 ev band gap energy which slightly lower than commercial ZnO (3.17 ev). The absorption spectra due to Zn-O stretching were observed at 3388 cm^{-1}, 1629 cm^{-1}, 760 cm^{-1}. The synthesized nonomaterial shows excellent biological activity for S. aureus, E. coli and P. aeruginosa.

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