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STUDY OF ANTIBACTERIAL ACTIVITY OFPURE AND AL DOPED ZnO NANOCRYSTALSON *BACILLUS THURINGIENSIS NCIM2130 AND PSEUDOMONAS CF. MONTEILII 9 CULTURES*

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ABSTRACT :

Hexagonal wurtizenano crystalline powder of pure and Al doped ZnO nanostructure have been prepared from reagent grade zinc acetate and sodium hydroxide using Sol Gel chemical precipitation process. XRD pattern of pure and Al doped ZnO represent the wurtize hexagonal crystal structurs. DopedZnO(Al) sample show decrease in grain size as compared to grain size of pure ZnO. Significant tuning ofoptical band gap and of particle size was observed on calcinations of ZnO(Al) samples.SEM images shows Varying Morphology of Al doped ZnO. The Varying morphology of hexagonal ZnO(Al) nanoclusters may be applicable as an antibacterial and antifungal agent.The antibacterial activity of pure and Al doped ZnO nanoparticles have been tested against Bacillus thuringiensisNCIM2130 and Pseudomonas cf. monteilii 9 cultures by using slightly modified Agar Well Diffusion method. The both the samples shows antibacterial activity against Bacillus thuringiensisbut Al doped ZnO shows more effect as compared to pure ZnO.

KEYWORDS : *ZnO NP's, Al doped ZnO nanostructure, Pl spectra, Antibacterial activity.*

1. INTRODUCTION

Zinc Oxide is an important II-VI group wide energy band gap semiconductor with optical properties that permits stable emission at room temperature having immense application in sensors, field emission and photonic devices. It exhibits a wide variety of morphologies in the nano regime that can be grown by tuning the growth habit of the ZnO crystal[1].Zinc oxide (ZnO) is an extensively studied nowadays, since it is a multifunctional direct wide band gap II–VI semiconductor (3.37 eV), which exhibits attractive properties such as large exciton binding energy (60 meV), The wide-band gap energy of ZnOsemiconductor enables huge potential for electronic and optical applications. It has unique piezoelectric properties that are very essential to enhance the performance of electromechanical devices. It is a biodegradable material suitable for medical and biological applications. [2]. The multiple morphologies of Zinc Oxide such as belts, ribbons, cables, rods, tubes, rings, springs, helices, bows, tetra pods, spirals, needles and films was forms the basis of its versatile applications[3].

ZnO exhibits significant antimicrobial activities when particle size is reduced to the nanometer range, then nano-sized ZnO can interact with bacterial surface and/or with the bacterial core where it enters inside the cell, and subsequently exhibits distinct bactericidal mechanisms. The reduced particle size was leading to enhance surface reactivity. ZnO is a bio-safe material that possesses photo-oxidizing and photocatalysis impacts on chemical and biological species. ZnO Nanoparticle of size 20-25 nm were used against pathogenic bacteria Staphylococcus aureus (Gram positive) and Salmonella typhimurium (Gram negative) and also first time against two plant fungi Aspergillus strain of flavus and fumigatus. The growth analysis data indicated that the ZnO NPs have significant bactericidal effect on both the bacteria. [4-5].

Zinc Oxide (ZnO) in nanostructure has unique physical and optical properties which can be used in variety of application such as a oxide coating for solar cell, gas sensors, catalysts photo-electronic device,

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optoelectronics, UV photo diode, photo chemical, electronics, photo detectors, schottky diode, LED device, because ZnO exhibit near-band-edge PL emission that originates from exciton transitions [6].

ZnOexhibit PL spectra under 325nm continuous excitation give significant UV emission band with a peak at 3.25 eV and a band width of 160 meV. PL spectra under 355-nm, 35-ps pulse excitation exhibited a spectrally narrowed emission band with a peak at 3.20 eV and a spectral width of 35 meV. The lasing phenomena is ascribed to the amplified spontaneous emission (ASE)by coupling of the micro cavity effect of ZnOnanorods and high intensity excitation, leads to be used in construction of laser [7].

Many of the workers across the world were doing research on this important ZnO nanostructure and their use in antibacterial activity

In this paper we report the synthesis of hexagonal symmetry pure ZnO Nano clusters and Aluminum doped ZnO nanostructures using Sol Gel chemical Precipitation method,the influence of their structural and morphological changes on Uv-Visible absorption, Photoluminescence emission propertieson sintering has been reported.

The particle size of the ZnO grains is calculated by using well known Scherer's equation (1) [8]

$$D = \frac{0.94\lambda}{\beta COS\theta} \text{---------- (1)}$$

Where **D** is the crystalline size nanoparticles (nm), λ is the wavelength of incident x-ray (nm), ß is the full width at half maxima and θ is the diffraction angle. The lattice constants a, b and c have been estimated by using relation (2) [9].

$$\frac{1}{d^2} = \frac{4}{3}\left\{\frac{(h^2+hk+k^2)}{a^2}\right\} + \left\{\frac{l^2}{c^2}\right\} \text{----------(2)}$$

Estimation of energy band gap(Eg) from statistical analysis of optical spectra equation (3) have been used.

$$\mathbf{Eg} = \frac{hc}{\lambda} \text{---------- (3)}$$

Where h is planks constant 6.63×10^{-34}Jule-sec., C is velocity of light and λ is wavelength at near band edge.

2. MATERIALS AND METHODS

Economically cheap and simple Chemical precipitation called as Sol-gel preparation is used for synthesis of Al doped zinc oxide nanoparticles ZnO(Al). High purity zinc acetate and sodium hydroxide were used as starting materials.. Initially appropriate molarity (0.75M) solutions of Zinc acetate and sodium hydroxide were prepared. These solutions were individually stirred for two hours. In zinc acetate solution 5% of Al salt solution was added slowly. To this solution sodium hydroxide solution was added slowly drop wise in a molar ratio of 1:2 with stirring continuously at appropriate temperature (50 °C). The stirring was continuous for two hours maintaining temperature constant until formation of gel. The obtained gel is kept overnight at room temperature andcentrifuged at 5000 rpm. The precipitate was washed thoroughly with distil water thrice. The white colored residue of Al doped ZnOwas dried in oven at 100°C for two hours. The dried product was ground to fine powder using agate mortar and pestle. The samples were calcined at different temperatures (200, 4000, 600°C). The as-prepared and sintered samples were characterized and results were discussed.

3. RESULTS AND DISCUSSION:

3.1 Structural Investigations:

The XRD spectra for pure ZnO was recorded in the range 2 θ=20-80° and presented in fig. 1.Pure ZnO sample show evidence of wurtize hexagonal crystal structure. XRD pattern is well matched with standard JCPDS card number (36-1451).Sharp peaks at different diffracting angles (2θ) correspond to different

reflections from the planes (100), (002), (101), (102), (110), (103) and (200). Similar structure of hexagonalZnOnano particles was investigated in the literature [10]. The high intensity and narrow line width indicate the good crystallites [11] of ZnO-NPs. The particle size nm estimated from XRD data by using relation (1) was 48 nm.

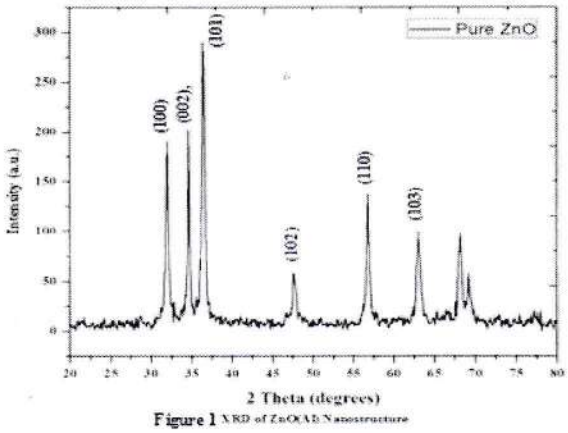

Figure 1 XRD of ZnO(Al) Nanostructure

XRD pattern of Al doped ZnOnanoparticles was shown in fig. 2. As-prepared and calcinedZnO(Al) exhibit wurtzite crystal symmetry similar to pure ZnO. The crystallity was observed improved on increasing temperature of calcinations. XRD pattern assigned to wurtzite phase and no other common secondary crystalline phases such as Al_2O_3 aluminum oxide or graphite were noticeable.

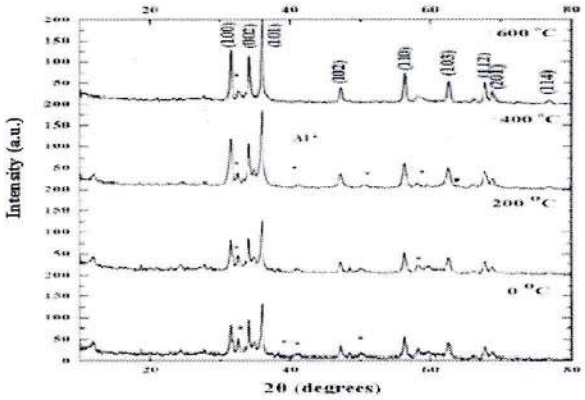

Figure 2. XRD Of ZnO(Al) Nanoparticles

On increasing the calcination temperature some peaks assigned to Al were noticed in the XRD spectra. This concluded that Al was substituted at theZn site on replacing Zn. The average grain size of the samples was estimated with the help of equation (1) using the full width at half maxima of (100), (002), (101), (102), (110), (103), (200), (112), and (201) of the x-ray diffraction peaks and displayed in table 1. The average crystalline size was increased with increase in temperature of calcinations. The particle size estimated was founddecreased as compared to particle size of pure ZnO samples. The lattice constants of pure and as-prepared and calciniedZnO(Al) was calculated by using equation (2) shown in table 1. The values of lattice constants a and b was found to decreased as calcination temperature increased.

Table 1. Grain size estimated using XRD data and SEM scans and Lattice Parameters with reference to sintering temperature of ZnO(Al)					
Sr. No.	Samples	a (A°)	c (A°)	Grain Size D (nm)	Average Grain Size SEM (nm)
1	ZnO(Al)0°c	3.2785	5.2639	36	60.48
2	ZnO(Al)200°c	3.278	5.1429	38	76.71
3	ZnO(Al)400°c	3.282	5.2568	44	76.78
4	ZnO(Al)600°c	3.1806	5.2504	47	76.80

a. Surface Morphology Study:

The SEM images of Al doped ZnO-NPs were shown in fig.3 exhibitsdistinct, nonhomogeneous porous network with an average grain size 68 nm that wascomputerizedand visualize in micrographs. The small crystalline agglomerated to form nano fused clusters that was seen in the surface morphology of ZnO(Al). The microstructure was observed changes to hexagonal nanorods at high temperature. The size of nanorods and nanograinswere varying from 37 to86 nm. The average grain size ranges from 68.48 to 76.80 nm was displayed in table 1 above. ZnO(Al) nanostructures exhibit significant varying morphology and may be used as antibacterial agent.

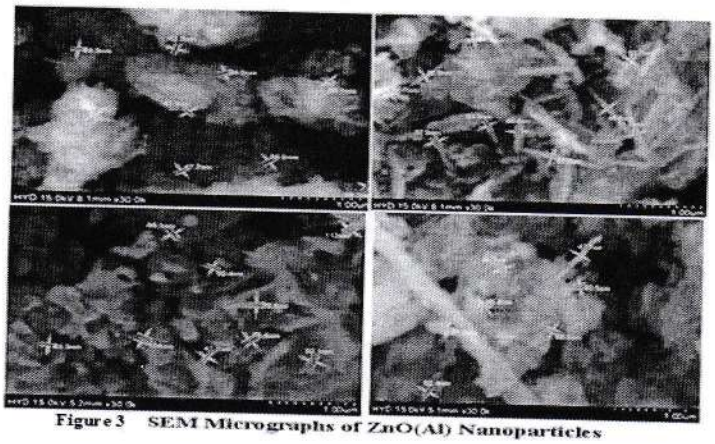

Figure 3 SEM Micrographs of ZnO(Al) Nanoparticles

b. UV-Visible Absorption Study:

The absorption spectrum of pure ZnO, and as prepared and calcinedZnO(Al)nanopowder is shown in fig.4. Pure ZnO sample exhibit a strong absorption peak at about 378 nm.The significant blue shiftof 378 nm peak to 358 nm was observed for Al doped ZnO samples. This confirms the decrease in particle size on doping. The band gap was calculated using equation (3). The estimated band gap and near band edge wavelength is tabulated in table 4. On calcination at different temperatures effectively tuned the band gap. This is attributed to change in microstructure of ZnO(Al) and grain size.

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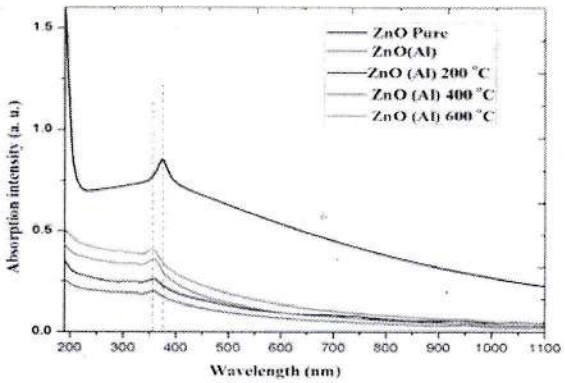

Figure 4 Uv-Visible Spectra of pure ZnO, as-prepared Zno(Al) and sintered ZnO(Al) Nanoparticles

As prepared ZnO(Al) exhibit 3.47 eV significant band gap as compared to pure ZnO nanostructure (3.28 eV). The band gap was observed decreased on increasing temperatures of calcinations (200 to 600 °C). The decrease of band gap is attributed to increase in grain size on increasing temperature. The pure and Al-doped ZnO nanostructure exhibit higher band gap and lower particle size may be used in optoelcectronics, photoelectrochemical cells, and for biological applications[2].

Table 4. Variation between band gap(Eg)and sintering temperature.

Sample	λ at near band edge (nm)	Energy band gap (eV)
ZnO (Al) °C	**358**	**3.47**
ZnO (Al) 200 °C	**365**	**3.40**
ZnO (Al) 400 °C	**370**	**3.36**
ZnO (Al) 600 °C	**375**	**3.31**
ZnO Pure	**378**	**3.28**

3.4 Antibactrial Activity of pure ZnO and doped ZnO

In order to study the antibacterial activity of doped ZnO nanoparticles against*Bacillus thuringiensis*NCIM2130 and *Pseudomonas cf. monteilii 9* cultures slightly modified Agar Well Diffusion method was used.The pure ZnO andAl doped ZnOnanopowder were calcined at 600 °C and used for antibacterial study. The inhibition zone size was measured. Pure ZnO shows inhibition zon size of 16 mm while Al doped ZnO shows increase in zone size up to 26mm. The pure ZnO and Al doped ZnO never showantimicrobial activity against *Pseudomonas cf.monteilii* 9 and not presented in form of photoplate.While comparing inhibition zone of pure ZnO with respect to ZnO(Al)) against *Bacillus thuringiensis* NCIM2130 and *Pseudomonas cf.monteilii* 9, Al doped compound is more active than pure ZnO. The photo plate of Al doped ZnO nanostructures calcined at 600 °C have been shown in figure 5.

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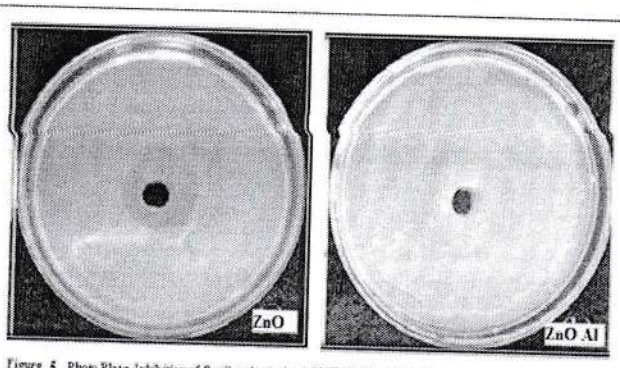

Figure 5 Photo Plate Inhibition of *Bacillus thuringiensis* NCIM2130 by ZnO Al in comparison with ZnO pure

4. CONCLUSION:

Pure and Al doped zinc oxide nanostructureexhibithexagonalwurtize crystal symmetry with different surface morphology (porous nano clusters to nanorods) were successfully prepared by Sol-Gel PrecipitationMethod. SEM images concluded that surface morphology effectively tuned on calcinations ofZnO(AL) powder from nanocluster to nanorods. XRD spectra exhibit single phase wurtizenano crystalline structure of ZnO(Al). The band gap was increased by small doping % of Al from 3.28 to 3.47 eV. The calcination temperature significantly tuned the band gap of ZnO(Al). The pure and Al- doped ZnO nanostructure exhibiting higher band gap and lower particle size with modified surface morphology may be used in optoelcectronics, devices and for biological applications. The antiracial study concluded that Al doped ZnOcalcined at 600 °C can be used as antibacterial agent against *Bacillus thuringiensis*NCIM2130.

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