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RESEARCH ARTICLE

Sol – Gel Precipitation Synthesis of ZnO Nanoparticles, their Morphological Changes after Calcinations and Antibacterial Properties

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ABSTRACT

Zinc oxide nanoparticles (ZnO) were synthesized by using a simple chemical sol-gel precipitation method with zinc acetate and sodium hydroxide in distilled water as a starting material. The synthesized sample was calcinated at different temperature for 1hour and different morphological changes occurred in samples were observed. The samples were characterized by X-ray diffraction (XRD), Scanning Electron Microscope (SEM) which showed various morphological changes of ZnO. UV shows absorption spectra of ZnO nanoparticles. ZnO nanoparticle has shown a wide range of antibacterial activity. ZnO nanoparticle inhibits the growth of *Bacillus thuringiensis* NCIM2130 upto 30 mm and *Pseudomonas cf. monteilii 9* upto 20 mm which more effective than the antibiotic used for comparative testing.

Keywords: Sol – Gel precipitation, ZnO nanoparticles, XRD, SEM, UV, *Bacillus thuringiensis* NCIM2130 and Antibacterial properties.

INTRODUCTION

Zinc oxide nanoparticles (ZnO) is a commercially important material used in paints, rubbers, concrete, electronics, lasers, transistors, photo-detectors, gas and biosensors, piezoelectric and solar cells, optoelectronics, photo catalysts, cosmetic, biomedicine, food industry, anticorrosive coating, antibacterial and antifungal agents (Jin et al., 2009, Zvekić et al., 2011). These wide varieties of prominent applications require the fabrication of special morphological and functionalization of ZnO nanostructures surface (Fakhroueian *et al.,* 2013). Different methods have been used for the production of ZnO nanoparticles such as 1) Chemical synthesis 2) Hydrothermal method 3) Electrophoretic deposition 4) Co-precipitation 5) Mechanochemical-thermal synthesis 6) Chemical vapour depositions 7) Thermal decomposition 8) Sol-Gel method 9) Electrochemical depositions and 10) Anodization (Fakhroueian *et al.,* 2013).

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The present study was aimed to i) Synthesis ZnO nanoparticle using simple Sol – Gel precipitation method by utilizing Zinc acetate and sodium hydroxide in distilled water, ii) study the morphological changes after calcinations at different temperature and iii) detect the antibacterial activity of ZnO nanoparticles.

EXPERIMENTAL METHODS

The chemicals used for this work were of analytical grade obtained from Merck (Mumbai).

Sol – Gel Precipitation Method for preparation of ZnO Nanoparticles:

A modified method of Fakhroueian *et al.,* 2013 was used where Zinc acetate and sodium hydroxide and distilled water used in the preparation of ZnO nanoparticles. Zinc acetate and sodium hydroxide was added slowly drop wise in a molar ratio of 1:2 under vigorous stirring, and stirring was continued for 4 hours. First Zinc acetate stirred for 2 hours and same time sodium hydroxide for 2 hours. Then the mixture of zinc acetate was slowly added drop wise in the solution of sodium hydroxide by continuous string for 2 hours. The precipitate obtained was filtered. The precipitate was dried in an oven at 100°C and ground to fine powder using agate mortar. The powder obtained from the above method was calcinated different temperatures such as 100°C, 200°C, 300°C, 500°C, 700°C for 2 hours. The prepared sample was then characterized for morphological changes by X – ray diffraction (XRD), UV absorption spectra and Scanning Electron Microscopy (SEM).

Characterization of ZnO Nanoparticles:

1. **X – Ray Diffraction Studies (XRD)**:

The X – ray diffraction (XRD) data were recorded by the intensity data collected over a 2θ range of 20-80 degrees. The average grain size of samples was estimated with the help of Debye-Scherer's equation using the full width at half maximum of 100, 002 and 101 of the x-ray diffraction peaks (as shown in Figure 1) .The average crystalline size increases with increase in calcite temperature. A significant increase in crystalline size is observed for the sample calcite of 100°C, 200°C, 300°C, 500°C, and 700°C.

$$D = 0.94\,\lambda / \beta \cos\theta$$

Where D is size of crystalline nanoparticles (nm), **λ** is wavelength of incident X – ray (nm) **ß** is the full width at half maximum and θ is the diffraction angle.

X- ray diffraction studies confirmed that the synthesized were ZnO all the diffraction peaks with the report (Origin 8.0 software) data and no charactertics were observed other than ZnO. The mean grain size of the particles was determined from the XRD line boarding measurement using Debye-Scherer equation. The diffraction peaks indicates that the synthesized material was in nanometer range. The grain size was found to be in the range 32nm to 124nm depending on the calcination condition (as shown in Table 1). The lattice parameters calculated were also in agreed with report values. The reaction temperatures greatly influence the particles morphology of prepared ZnO nanoparticles. The size of ZnO nanoparticles increased as the temperature for the hydrothermal synthesis increases. This is due to the change of growth between the different crystalline planes.

Table 1: Size of ZnO obtained from XRD using Debye's- Scherer's equation.

Calcinations Temperature (°C)	Crystalline size (nm) of ZnO Nanoparticles
ZnO 0°C	32
ZnO 100°C	42
ZnO 200°C	62
ZnO 300°C	78
ZnO 500°C	92
ZnO 700°C	128

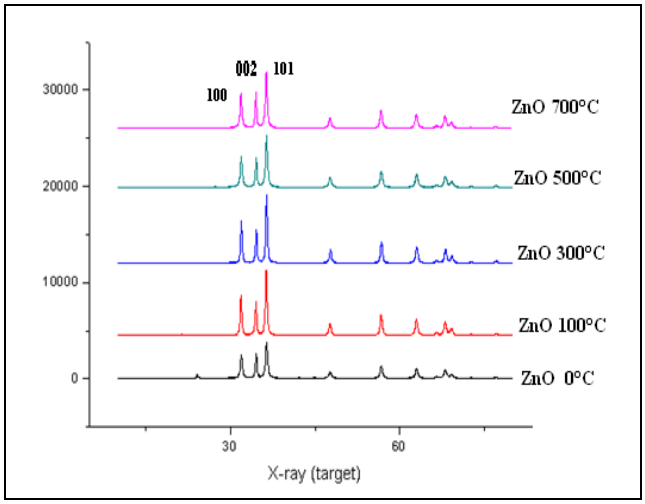

Figure 1: XRD Pattern of ZnO nanoparticles at different calcination.

UV Absorption Study:

Ultra Violet visible absorption spectrum of the ZnO nanoparticle was shown in Figure 2. The room temperature spectra exbit strong excitation absorption peaks 372 nm to 378 nm for samples. All samples have a strong absorption maximum below 400 nm.

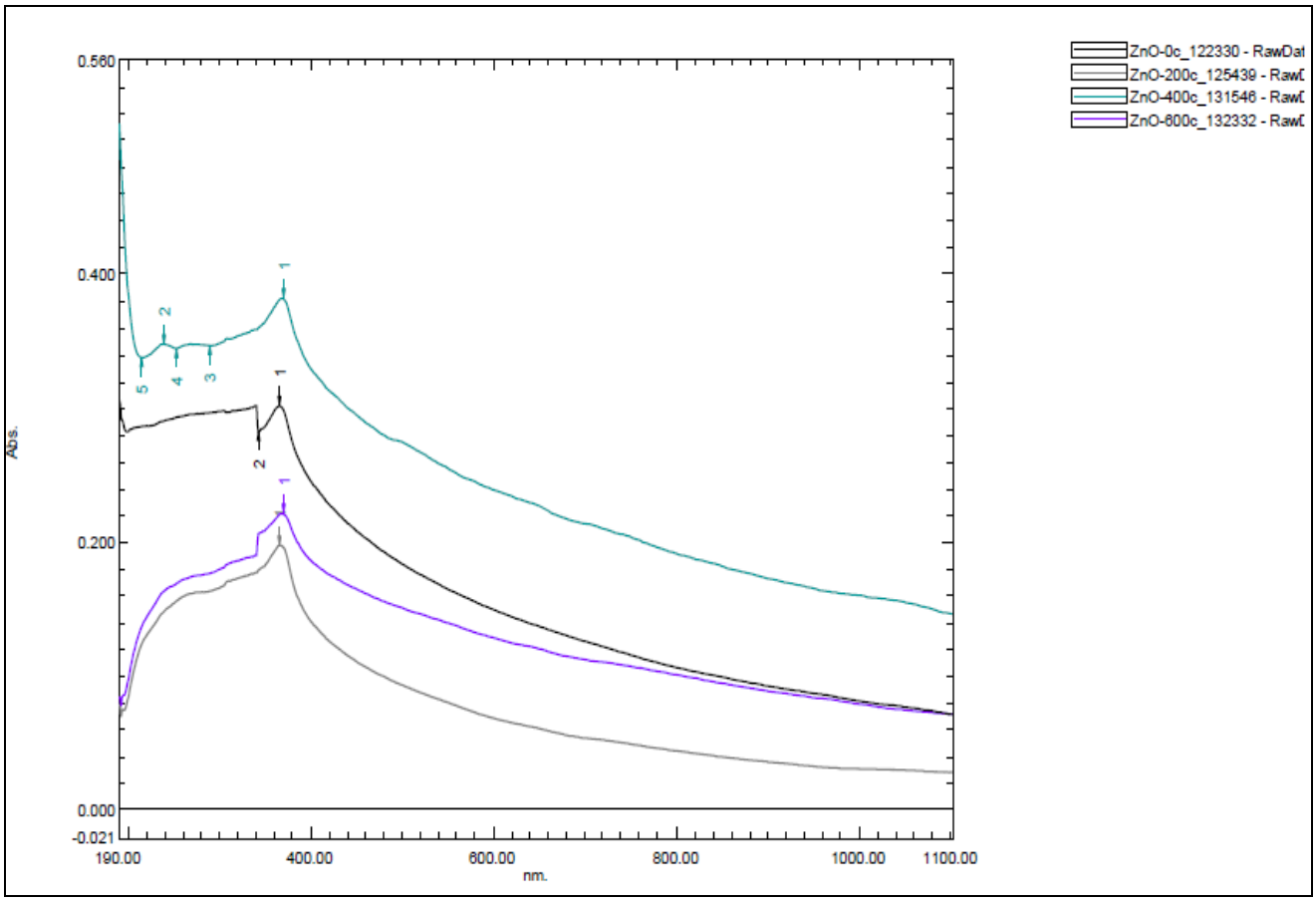

Fig 2: UV- Absorption of the ZnO Nanoparticles

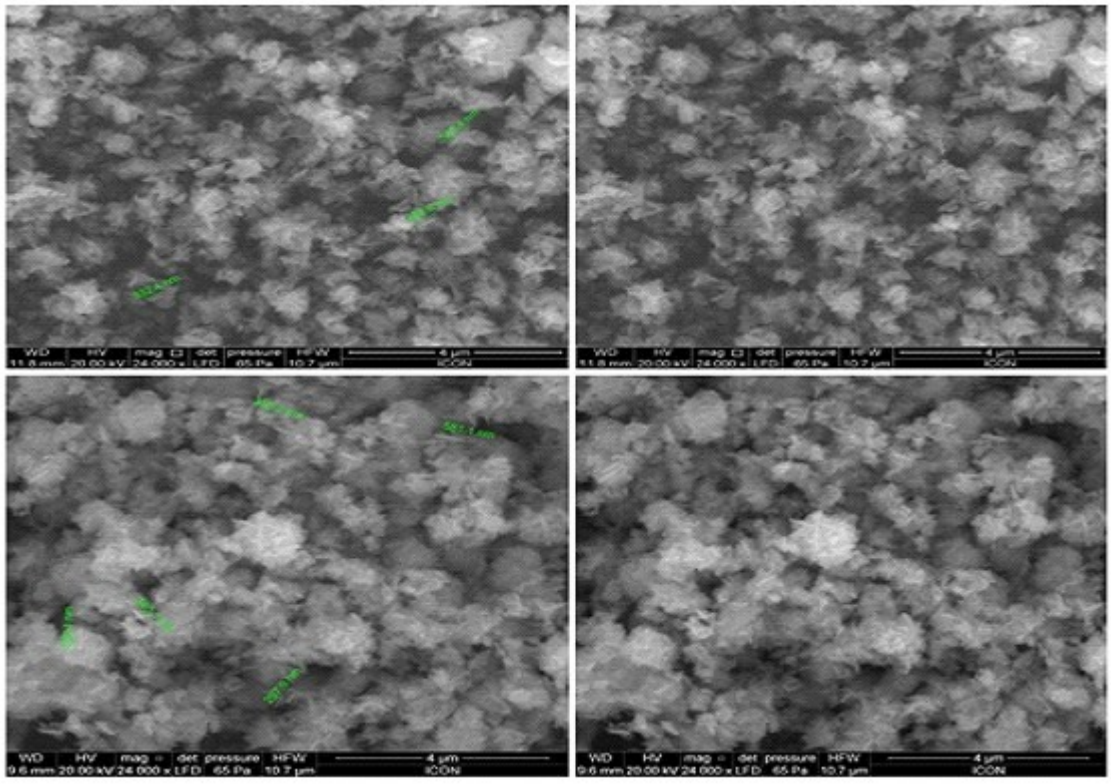

Figure 3: Scanning Electron Microscopic Images of ZnO nanoparticles

Scanning Electron Microscopic Study (SEM):
The Scanning Electron Microscopic images (SEM) of the samples also shows that (Figures 3) the physical structure of ZnO was changed with calcinations temperature. The samples calcinate at 0°C, 100°C, 200°C, 300°C, 500°C, 700°C. The SEM images of ZnO samples show that the agglomeration particles are much less in this method of preparation. High resolution SEM images of ZnO calcinate show the presence of nanoparticles.

Antibacterial Activity of ZnO nanoparticles:
Microbial Strain:
To test the antibacterial activity of ZnO nanoparticle, *Bacillus thuringiensis* NCIM2130, *Pseudomonas cf. monteilii 9* cultures was used which was grown on nutrient broth for 24 hour at 37°C.

Disc Diffusion Method:
Disc Diffusion Method was performed for testing the antibacterial activity of ZnO nanoparticles (Bauer *et al.,* 1959). First Muller Hinton agar plates were spreaded by bacterial cultures grown earlier in nutrient broth with the help of sterile cotton swabs. Allow the agar surface to dry for 5 minutes. By using the flamed sterile forceps, pick up a sterile filter paper disc and dip the disc in the ZnO nanoparticle dilutions prepared of different calcinated temperature. The dipped disc was placed near the edge of the agar surface of the inoculated plate and pressed gently to ensure firm contact of the disc with agar surface. Another disc dipped with another dilution of ZnO nanoparticle of different calcinated temperature. All the inoculated plates were incubated at 37 °C for 24 to 48 hours in an inverted position. After incubation at 37°C a clear zone around the disc was an evidence for antimicrobial activity (as shown in table 2).

Determination of minimum inhibitory concentration (MIC):
The minimum inhibitory concentration (MIC) of the ZnO nanoparticle was determined according to methods described by Bauer *et al.,* 1959. ZnO nanoparticle of different calcinated temperatures were diluted to concentrations ranging from 100 to 0.78 mg/ml in distilled water. On Mueller Hinton agar plate, bacterial culture was spreaded with sterile swab. By using sterile forceps, sterile disc was dipped in each dilution tubes and placed on Mueller Hinton agar plate inoculated earlier with bacterial culture. Control plate contains only bacterial culture spreaded on Mueller Hinton agar plate without any dilution of nanoparticle. All the plates were incubated at 37°C for 24 hours. The lowest concentration of the nanoparticle that produced no visible bacterial growth was recorded as the MIC. Inhibition of bacterial culture by ZnO nanoparticle was compared with antibiotic Gentamycin.

Table 2: Effect of ZnO nanoparticles on Gram Positive and Gram Negative bacteria

Bacterial Culture	Reference	Inhibition zone of bacterial culture by ZnO nanoparticle (mm)				
	Gentamycin (mm)	ZnO 0°C	ZnO 100°C	ZnO 300°C	ZnO 500°C	ZnO 700°C
Bacillus thuringiensis NCIM2130	10	12	13	15	22	30
Pseudomonas cf. monteilii 9	9	9	10	12	18	20

Fig. 4: Disc Diffusion Method for antibacterial activity at different calcinated temperature for ZnO

Table 2 and Figure 4 showed that all ZnO nanoparticles prepared at different calcinated temperature were capable of inhibiting *Bacillus thuringiensis* NCIM2130 upto 30 mm and *Pseudomonas cf. monteilii 9* upto 20 mm which was far effective than the antibiotic used for comparative testing. These findings also supported by findings of Fakhroueian *et al.,* 2013, where ZnO nanostructures such as nanorods, nanowires, nanorings, nanoflowers, nanospheri-cal, nanotubes, nanodisks, nanodumbbells, nanoneedles, nanowhiskers, nanonail, nanobelts, nanosheets, nanosprings, nanoribbon found effective in inhibiting the growth of *Bacillus anthracis* and *Pseudomonas aerogenes Escherichia coli (RTCC1330), Klebsiella pneumonia (RTCC1249), Pseudomonas aeruginosa (RTCC1547), Enterobacter aerogenes (RTCC1145), Klebsiella pneumonia (RTCC1249) and various gram-positive bacteria like: Staphylococcus aureus (RTCC1885), Listeria monocytogenes (RTCC1293), Enterobacter aero-genes (RTCC1145), Bacillus anthracis (RTCC1036), Bacillus anthracis (RTCC1036), Enterococcus faecalis (RTCC2121), Bacillus cereus (RTCC1040) and Staphylococcus epidermidis (RTCC1898).* Similar finding also recorded from other researchers Schwartz *et al.,* 2012, Tariq *et al.,* 2013, Stankovic *et al.,* 2013, and Sirelkhatim *et al.,* 2015

CONCLUSIONS

In this study, ZnO nanoparticles and QDs nanostructures as hexagonal and nanorods arrays were fabricated by sol-gel-chemical, method. In this case, they can have more contact with bacteria and the efficiency will enhance. These studies demonstrate that the especial ZnO QDs nanoparticles including blue shifts spectrums and complex defects on surfaces exhibit.

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