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Research Article

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Synthesis of nickel doped ZnO nanoparticles by hydrothermal decomposition of zinc hydroxide nitrate and its antimicrobial assay

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ABSTRACT

Zinc hydroxide nitrate is an anionic exchanger material which is synthesized by a pH controlled precipitation method. It was doped with nickel (2-6%) and the control was kept as undoped. Then ZnO nanoparticles are synthesized by the hydrothermal decomposition, including the doped and undoped zinc hydroxide nitrate. X-ray diffraction, Fourier Transform Infrared Spectroscopy and UV-visible spectrophotometry confirmed the synthesis of nickel-doped layered material as well as the ZnO nanoparticles. Then antimicrobial assay was done with two strains of bacteria by using the inhibition kinetics.

Keywords: *Nickel doped zinc oxide, nanoparticle, and chemical synthesis.*

INTRODUCTION

A microscopic particle with at least one dimension less than 100 nm can be considered as a nanoparticle. Nanoparticle research nowadays is an intense area of interest as it has got a wide variety of potential applications in biological sciences. Especially, the metal oxide nanoparticles have several advantages in the biological field like for antimicrobial susceptibility, for therapeutic purpose, production of gas sensors, solar cells and some of them are anti-cancerous agents too. For example, zinc oxide. Now the question comes why we are concerned about the synthesis of the nanoparticles of zinc compounds. First of all it is non-toxic and it is easy to synthesize. Especially zinc oxide has got a high potential in antimicrobial activity studies showed that it can disrupt the cell wall of bacteria, and moreover it is safe for animals and human beings.

ZnO is an n-type semiconductor with a wide band gap of 3.37 eV and it is chemically and thermally stable. It s also cheap and environmentally friendly compared to other metal oxides. Because of these properties it has found applications in fields like gas sensors[5], solar cell[6], varistors[7], light emitting devices[8], photocatalyst[9] antibacterial activity[10] and cancer treatment[11]. Zinc hydroxide nitrate (ZHN) is a synthetic layered material whose thermal collapsing of layered ZHN leads to the production of nanocrystalline zinc oxide[4]. Zinc oxide is often doped with a metal to modify its properties for various applications like solar cells, sensors, photocatalyst, optoelectronic property and others. The doping can be done with several metals like copper, cobalt, nickel and various other metals.

This study is concerned about the synthesis of doped and undoped ZnO nanoparticles with various molar percentages of nickel (2%, 4%, and 6%) by hydrothermal decomposition of zinc hydroxide nitrate following the characterization using the analytical techniques like FTIR and XRD.

MATERIALS AND METHODS

Preparation of Zinc hydroxide nitrate both undoped and doped with nickel

Distilled de-ionized water was used throughout the whole experiment and all the chemicals were used that were received. Zinc hydroxide nitrate was synthesized by the precipitation method.

In 50mL 0.2 M of $Zn(NO_3)_2$, or $Zn(NO_3)_2$ and $Ni(NO_3)_2$, at various molar percentages of nickel (2-6%), 50mL 0.5M NaOH was added dropwise with vigorous stirring so that the pH could be brought up to 7.0 ±0.05. The products were named as ZHN (for the control or bare ZHN), ZHN (2), ZHN (4) and ZHN (6) for the doped ZHN for 2%, 4% and 6% molar percentages respectively.

Hydrothermal decomposition of Zinc hydroxide nitrate both doped and undoped

Now to obtain the doped and undoped Zinc oxide nanoparticles, 5.0 mL of ammonia solution (10%) and 0.05g of sodium dodecyl sulphate (SDS) were dissolved in 200mL ethanol. After that, 0.5g of the prepared doped and undoped ZHN was added to this solution and the mixture was then refluxed and then cooled. The filtration of the solid was done and it was washed about two to three times with water as well as acetone and dried overnight at 80^0C in an oven. The hydrothermally decomposed products were labelled as ZO (For the undoped), ZO (2), ZO (4) and ZO (6) for the doped nickel molar percentages of 2%, 4% and 6% respectively.

After that, the analytical technique analysis was done using XRD, FTIR and UV-visible spectrophotometry.

Antibacterial activity assay by inhibition zone method

To examine the antibacterial activity of undoped as well as nickel doped ZnO nanoparticle of different molar percentages, at first they were suspended in normal sterile saline water and two petriplates were prepared containing Mueller-Hinton agar. 0.1 mL of culture broth was poured into the plates, both for *Pseudomonas aeruginosa* and *Bacillus subtilis* in two different plates and spread plate was prepared carefully. Using a cork borer, three wells were made in each of the petriplates, one for undoped ZnO nanoparticle and rest of them for the doped ZnO nanoparticle of different molar percentages (4% and 6%). Then, carefully, 100 microliters of each of the nanoparticle solution is poured in different wells in each of the petriplates and they were kept in the incubator overnight for 24 hours at 37^0C.

RESULTS AND DISCUSSION

XRD study of undoped and nickel-doped zinc hydroxide nitrates

X ray diffractogram of the powder taken from diffractometer was analysed to obtain information about various crystalline aspects. The XRD patterns of synthesised undoped and nickel doped nanoparticles are shown in fig.1 and 2. The major peaks observed at 37°, 46.149°, 61.63°, 72.45° and 75.84° confirms nickel as doping agent (card 75-0273).the XRD peaks for (1 0 0) and (1 0 1) planes indicates the formation of phase pure wurtzite structure of ZnO . The high intensity of (1 0 1) peak suggests that the growth of nanoparticles has taken place along the easy direction of crystallization of ZnO and the similar results were reported by Lin et al., 2005. No peaks due to impurities were observed. The mean crystallite size of synthesized nano particles was calculated using Scherrar's formula: (Singhal et al., 2010)

$$d = \frac{0.9\lambda}{\beta Cos\theta}$$

where d is the average crystallite size, λ is the wavelength of the incident X-ray beam (1.54 Å), θ is the Bragg's diffraction angle and β is the angular width of the diffraction peak at the half maximum in the radian on 2θ scale. The average crystallite sizes of the sample have been found to be 28.3 and 32.6 nm for doped and undoped nanoparticles respectively.

Fig 1: XRD patterns of ZnO nanoparticle

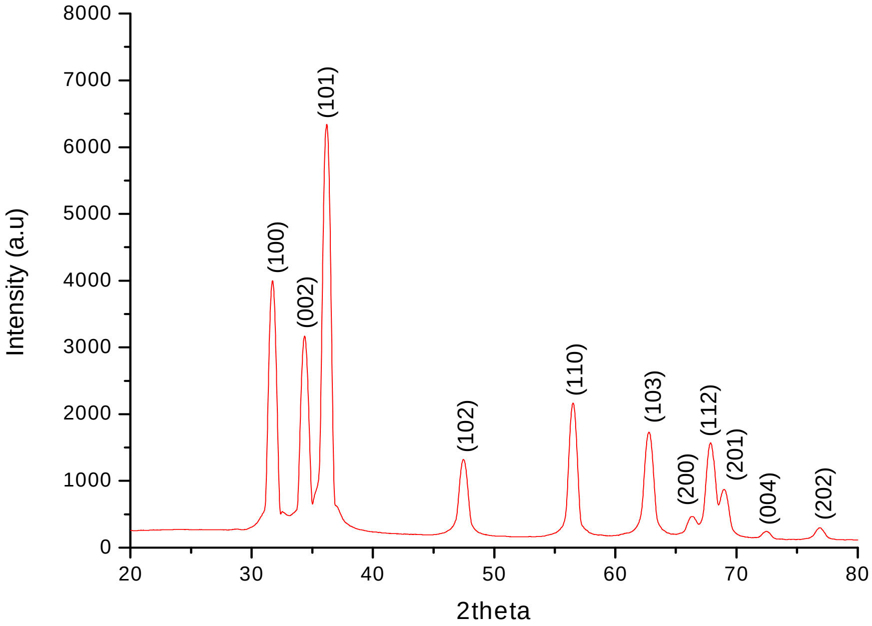
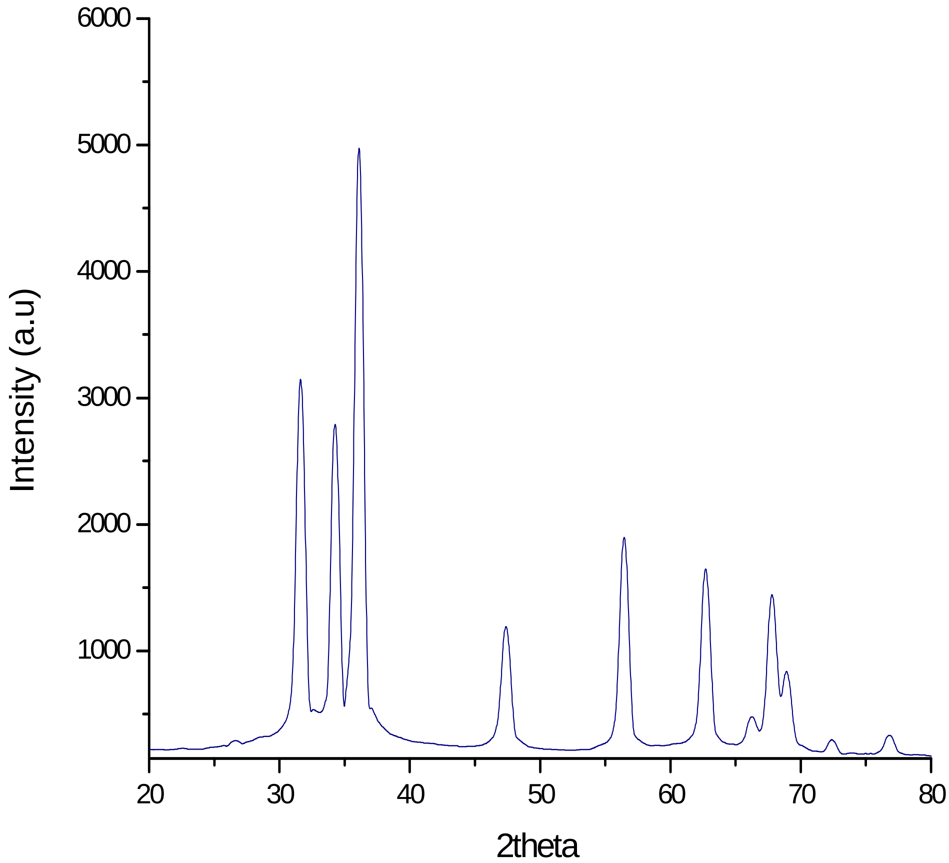
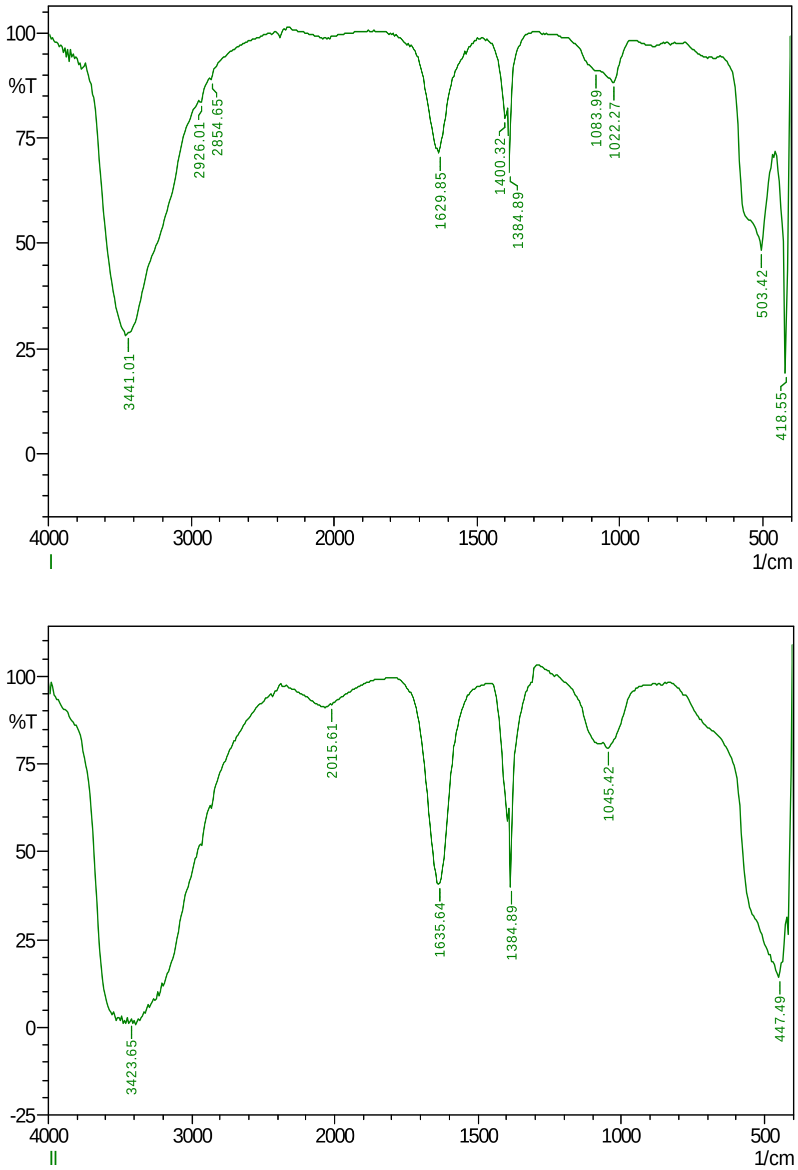


Fig 2: XRD pattern of Ni doped ZnO



FTIR study of undoped and nickel-doped zinc hydroxide Nitrates

FT-IR spectrum of hydrothermally treated nickel doped ZnO nanoparticle and undoped ZnO nanoparticle is shown in figure 3 and 4. An intense peak which was observed at 447 cm^{-1} corresponds to Zn-O stretching vibration. The broad band around 3423.65 cm-^{1}corresponds to adsorbed water molecule. An intense sharp peak at 1384.89 cm^{-1} and broad peak at 1045.42 cm^{-1}corresponds to nitrate groups are observed which implies presence of nitrate groups in the material. This might be due to adsorbed nitrate group on the surface of zinc oxide in trace amounts during hydrothermal treatment of the sample.



FT-IR patterns of ZnO and Ni-doped ZnO nanoparticles

UV-visible spectrophotometry analysis

Using the absorption spectra of doped and undoped ZnO obtained in the range of 300-800 nm, the optical band gap (E_g) was calculated from the following formula:

$$(Ahv)^{n} = B\,(hv - E_g)$$

where *A* is the absorbance, *B* is characteristic of the material and the exponent n is either 2 for allowed direct transition or ½ for allowed non-direct transition[2,3].

The structural and optical parameters for the synthesized nanoparticles are shown in in Table 1 .The band gap for the obtained pure zinc oxide is 3.2 eV and this has decreased to 3.09 eV for nickel doped ZnO. This shows that the optical band gap values of zinc oxides are decreased on the doping. Similar result has also been observed for cobalt and manganese doped zinc oxides[2].

Table 1: Structural and optical parameters of synthesized nanoparticles

Sample	c^a (A°)	$D_{101}{}^b$ (nm)	a^c (A°)	$D_{100}{}^d$(nm)	Band gap (eV)
ZO	4.9562	32.6	5.634	34.9	3.23
Ni-ZO	4.9628	28.3	5.643	27.9	3.09

Antimicrobial activity

The antimicrobial activity of ZnO and Ni-doped ZnO nanoparticles against *Pseudomonas aeruginosa and Bacillus subtilis* is shown on the basis of inhibition zone (mm). In both the cases the diameter of the inhibition zone was found to be higher (9-14 mm) for Ni-doped ZnO compared to undoped ZnO nanoparticles (4-8 mm). The above results show that doping of Nickel increased the antimicrobial activity of the synthesized ZnO nanoparticles against *Pseudomonas aeruginosa and Bacillus subtilis.*

CONCLUSION

Pure Nickel doped ZnO nanoparticles were synthesized by using hydrothermal decomposition and XRD result shows information about various crystalline aspects. The FTIR analysis had shown the presence of the chemical groups with every detail and the UV-visible spectrophotometric analysis showed the optical band gaps. Moreover the antibacterial study provided the information about the potential of the nanoparticle to inhibit the growth of bacteria that is evident from the inhibition zone.

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