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RESEARCH ARTICLE

ANTIBACTERIAL STUDY OF ZnO AND $Zn_{0.5}Mg_{0.5}O$ NANOPARTICLES SYNTHESIZED BY CO-PRECIPITATION METHOD

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ABSTRACT

In the present study, zinc oxide (ZnO) and magnesium doped zinc oxide ($Zn_{0.5}Mg_{0.5}O$) nanoparticles were synthesized by simple and cost-effective co-precipitation method. The synthesized materials were characterized by X-ray Diffraction (XRD), Fourier Transform Infrared (FTIR) spectroscopy, Field Emission Scanning Electron Microscopy (FESEM) with Energy dispersive X-ray spectrum (EDAX). Finally, the effect of magnesium (Mg) doping on the structural, morphological and anti-bacterial property of ZnO nanoparticles was analyzed. From the XRD results, it was found that there was a formation of hexagonal structured ZnO and the average crystallite size of ZnO and Zn0.5Mg0.5O was calculated to be 71 nm and 36 nm respectively. The FTIR analysis confirmed the existence of possible functional groups in ZnO and $Zn_{0.5}Mg_{0.5}O$. There was formation of almost spherical shaped particles as evident from FESEM images and agglomeration of particles was observed upon doping Mg into ZnO. The EDAX spectra of the prepared nanoparticles provided the composition of Zn, O in ZnO and Zn, Mg, O in $Zn_{0.5}Mg_{0.5}O$. No other elements have been found in the EDAX spectra of ZnO and $Zn_{0.5}Mg_{0.5}O$ that confirmed the formation of pure materials. Finally the anti-bacterial study demonstrated that ZnO and $Zn_{0.5}Mg_{0.5}O$ was effective in inhibiting *E. coli* bacteria.

Keywords: Zinc oxide; Magnesium; Dopant; Anti-bacterial; Co-precipitation.

1. INTRODUCTION

The bacterial infections are increasing day by day worldwide. The development of antibiotic resistance of bacteria is one of the major problems in the health sector. Nanotechnology plays a crucial role in developing efficient antibiotics [1]. Antibacterial as well as antiviral activity of a molecule is completely associated with the compounds that destroys the bacteria and virus or slow down their rate of growth, without affecting the nearby tissues. Antibacterial agents are of two types namely organic and inorganic. Organic antibacterial materials are less stable especially in high temperature. But inorganic materials like metals and metal oxides can withstand extreme conditions [2, 3]. ZnO is an inorganic compound appears as a white powder which is insoluble in water, inexpensive and can be easily produced. ZnO has been found to possess good antibacterial property [4]. ZnO nanoparticles possess optical, electrical and photocatalytic property [5,6]. In recent years, ZnO nanoparticles have worldwide usage that has driven attention towards research in biomedical applications focusing on cancer cell imaging [7]. Additionally, it is used as sun screen cream, rubber production and water repellent clothing [8-10]. In the present work, zinc oxide (ZnO) and magnesium doped zinc oxide ($Zn_{0.5}Mg_{0.5}O$) nanoparticles were synthesized by simple, cost-effective co-precipitation method. The synthesized materials were characterized by X-ray Diffraction (XRD), Fourier Transform Infrared (FTIR) spectroscopy, Field Emission Scanning Electron Microscopy (FESEM) with Energy dispersive X-ray spectrum (EDAX). Finally, the effect of magnesium doping on the structural,

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morphological and anti-bacterial property of ZnO nanoparticles were examined. Doping of Mg (Magnesium) into ZnO is expected to modify the properties like crystallite size, surface to volume ratio, antibacterial properties etc [11].

2. MATERIALS AND METHODS

All the materials were purchased and used without further purification.

2.1. Synthesis of zinc oxide (ZnO) nanoparticles by co-precipitation method

For the preparation of ZnO, stoichiometric amount of the starting precursors such as zinc nitrate hexahydrate $(ZnNO_3)_2.6H_2O$ and sodium hydroxide (NaOH) were taken. Initially, zinc nitrate hexahydrate was dissolved in 30 ml of distilled water. It was stirred for around 15 minutes. Meanwhile, sodium hydroxide was dissolved in 30 ml of water. After 15 minutes sodium hydroxide solution was added drop by drop to the aqueous zinc nitrate hexahydrate solution. It was then stirred for another 30 minutes. No specific temperature condition is applied for stirring. The precipitate was formed. The precipitate was centrifuged at 2000 rpm for 10 minutes. This centrifugation was done several times in order to remove the impurities. Then the remaining precipitate was dried at 100^oC in the hot air oven for 2 hours. This dried sample was then placed in a muffle furnace at 500^oC for 3 hours. The obtained material was ground using a mortar and pestle. Thus, ZnO nanoparticles were obtained by co-precipitation method. The obtained ZnO nanoparticles were used for further characterization.

2.2 Synthesis of Mg doped ZnO ($Zn_{0.5}Mg_{0.5}O$) nanoparticles by co-precipitation method

The synthesis of Mg doped nanoparticles ($Zn_{0.5}Mg_{0.5}O$) by co-precipitation method involves the same procedure as ZnO synthesis. But here, in addition to $(ZnNO_3)_2.6H_2O$ and (NaOH), magnesium nitrate hexahydrate $(Mg(NO_3)_2.6H_2O)$ is taken in stoichiometric amount and the same synthesis procedure is followed. For the preparation of $Zn_{0.5}Mg_{0.5}O$, $(ZnNO_3)_2.6H_2O$ and $(Mg(NO_3)_2.6H_2O)$ were dissolved in distilled water separately. These aqueous solutions are mixed and stirred for around 15 minutes. After that, the aqueous sodium hydroxide was added into the above mixture and then again stirred for another 30 minutes. The precipitate was centrifuged, dried and calcined at 500^oC for 3 hours to obtain $Zn_{0.5}Mg_{0.5}O$.

The XRD was performed using X'Pert Pro PANalytical with CuKα radiation (λ=1.5405Å) at 30 mA and 40kV. The fourier transform infrared (FTIR) spectroscopy was performed using SHIMADZU (miracle 10) in the wave number range of 4000-400 cm^{-1}. The field emission scanning electron microscopic (FESEM) images coupled with Energy dispersive X-ray spectrum (EDAX) was recorded using Zeiss FESEM SIGMA VP 03-04 model.

The preparation of the bacterial inoculums for anti-bacterial study is given. At first, the stock cultures were maintained at 4°C on slopes of nutrient agar and potato dextrose agar. The active culture for experiments were prepared by transferring a loop full of cells from stock cultures to test tubes (50ml nutrient broth bacterial cultures) were incubated with agitation for 24hours (37°C) on shaking incubator for 3-5 days. Each suspension of test organism was consequently stroke out on nutrient agar media and potato dextrose agar. These stock cultures were kept at 4°C. For use in experiments, a loop of each test organism was transferred into 50ml nutrient broth and incubated separately at 37°C for 18-20 hours for bacterial culture.

Antibacterial activity was performed by agar diffusion method. The stock culture of *E. coli* bacteria were received by inoculating in nutrient broth media and grown at 37 % for 18 hours. The agar plates of the above media were prepared. Each plate was inoculated with 18 hours old cultures the bacteria were swab in the sterile plates. The extracts were taken in the ratio of 25µl, 50 µl, 75 µl, 100 µl. All the plates were incubated at 37^oC for 24 hours and the diameter of inhibition zone was noted in Cm.

3. RESULTS AND DISCUSSION

3.1 X-ray Diffraction

The XRD pattern of ZnO and $Zn_{0.5}Mg_{0.5}O$ is shown in figure 1. The XRD peaks are sharp that confirm the crystalline nature of both pure and Mg doped ZnO. The diffraction peaks observed at 2θ of 31.8236^o, 34.5707^o, 36.3051^o, 47.5819^o, 56.6296^o, 63.0795^o, 67.9971^o and 69.1325^o corresponds to (100), (002), (101), (102), (110), (103), (112) and (201) planes that corresponds to hexagonal structure of ZnO [12]. The observed peaks are matched with JCPDS card number 79-0205. The crystallite size of the synthesized nanomaterials is calculated using Scherer's formula. The average crystallite size of pure ZnO and Mg doped ZnO is found to be 71 nm and 36 nm respectively. It is evident from the XRD pattern that upon dopant

addition, the crystallite size is decreased. The well-known fact is that upon decrease in crystallite size, the surface area is enhanced. The improved surface area upon Mg doping will be beneficial for various applications including anti-bacterial activity.

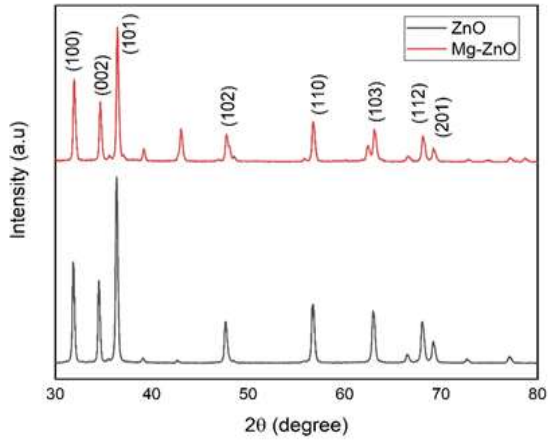

Fig. 1. XRD pattern of ZnO and $Zn_{0.5}Mg_{0.5}O$ (Mg-ZnO) nanoparticles

3.2 Fourier Transform Infrared Spectroscopy:

FTIR spectroscopy is a characterization technique used for the detection of functional groups present in the compounds. The FTIR spectra of ZnO and $Zn_{0.5}Mg_{0.5}O$ are provided in Figure 2.

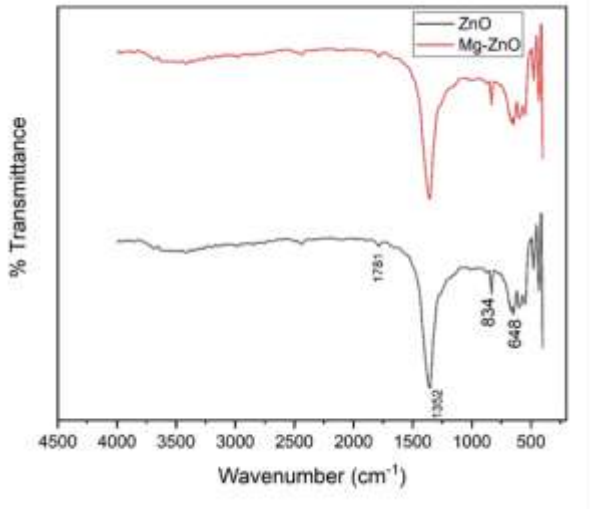

Fig. 2. FTIR spectra of ZnO and $Zn_{0.5}Mg_{0.5}O$ (Mg-ZnO)

The synthesized ZnO showed main bands around 490 cm^{-1} that attributes to Zn-O stretching peak [13]. A slight shift in the band in $Zn_{0.5}Mg_{0.5}O$ illustrates that Mg was successfully incorporated into ZnO lattice. The C-C-O symmetric stretching was observed 834 cm^{-1} [14]. The broad band at 1352cm^{-1} is due to the presence of phyto-active compound bound to the surface of ZnO nanoparticles [15]. The FTIR analysis confirmed the presence of functional groups in ZnO and Mg doped ZnO.

3.3. Field Emission Scanning Electron Microscopy:

The morphological analysis of ZnO nanoparticles was carried out using field emission scanning electron microscopy (FESEM). Figure 3 (a,b) shows the FESEM image of ZnO in different magnifications. There was formation of almost spherical shaped particles. The size of the particles is in the range of 50-100 nm. The elemental compositional analysis of ZnO was carried out using the energy dispersive X-ray analysis and the corresponding EDAX spectra in provided in figure 3 (c). It is apparent from the EDAX spectrum that the prepared nanoparticles are the composition of Zn and O alone and no other elements have been found in the EDAX spectrum. The observed intense peaks correspond to the composition of Zn and O and rest of the peaks arises by carbon tape. The weight percentage of ZnO was found to be 31.81% of O and 68.19% of Zn respectively.

Figure 4 (a,b) shows the FESEM image of $Zn_{0.5}Mg_{0.5}O$ in different magnifications. There was agglomeration of spherical particles observed after the addition of magnesium. The elemental compositional analysis of $Zn_{0.5}Mg_{0.5}O$ carried out using the energy dispersive X-ray analysis showed that the prepared nanoparticles possess only Zn, Mg and O and no other elements have been found in the EDAX spectrum. The observed peaks correspond to the composition of Zn, Mg and O, the weight percentage of ZnO was found to be 43.61% of O, 19.99% of Mg and 36.40% of Zn respectively. The EDAX spectrum conform the successful doping of Mg in pure ZnO.

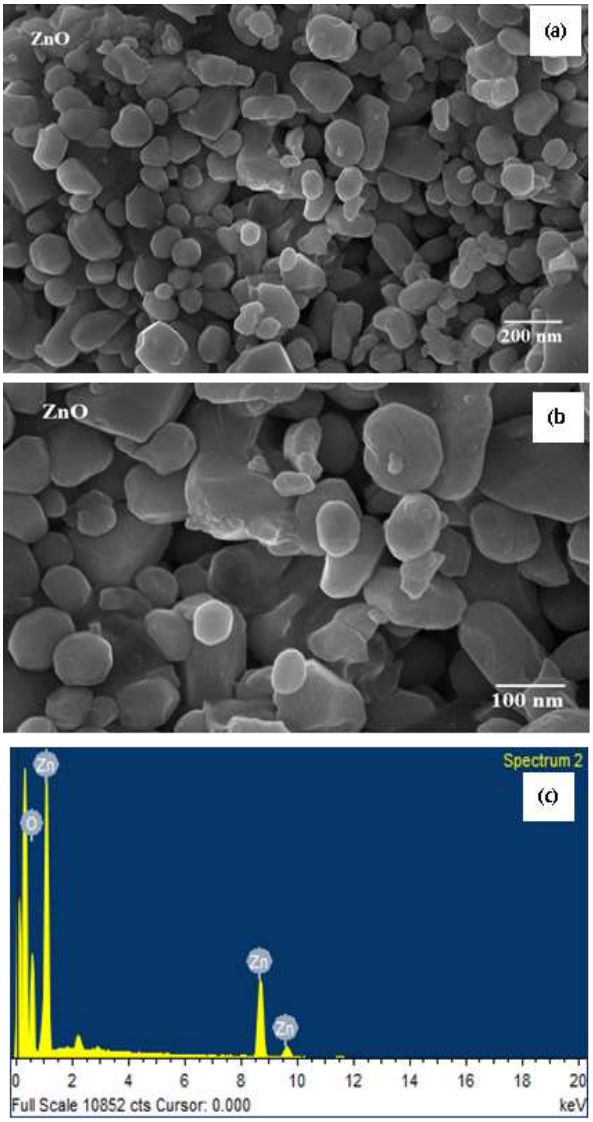

Fig. 3 (a, b) FESEM image (c) EDAX spectrum of ZnO

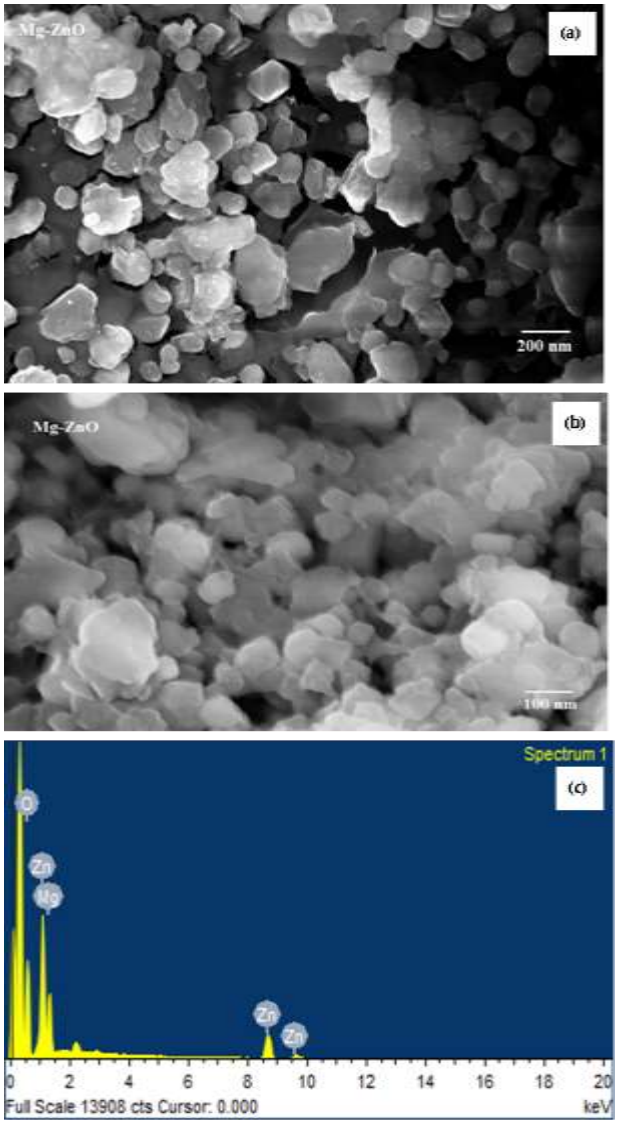

Fig. 4 (a, b) FESEM image (c) EDAX spectrum of $Zn_{0.5}Mg_{0.5}O$ (Mg-ZnO)

3.4 Antibacterial study

Figure 5 (a) and (b) shows the antibacterial activity of ZnO and Mg doped ZnO against *E. coli* bacteria respectively. From the figure we infer that zone of inhibition to the bacteria is present for both the synthesized nanomaterials and hence we can confirm that the synthesized material inhibits the growth of *E. coli* bacteria. The zone of inhibition is measured for different concentrations of the extract and it is tabulated below.

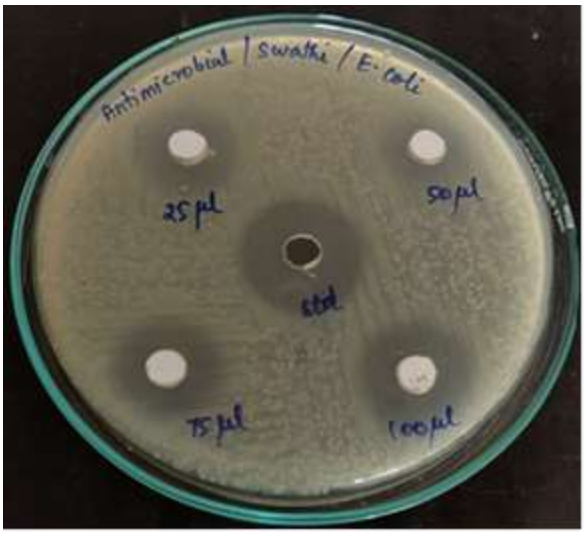

Fig. 5 (a) Antibacterial activity of ZnO nanoparticle against *E. coli*

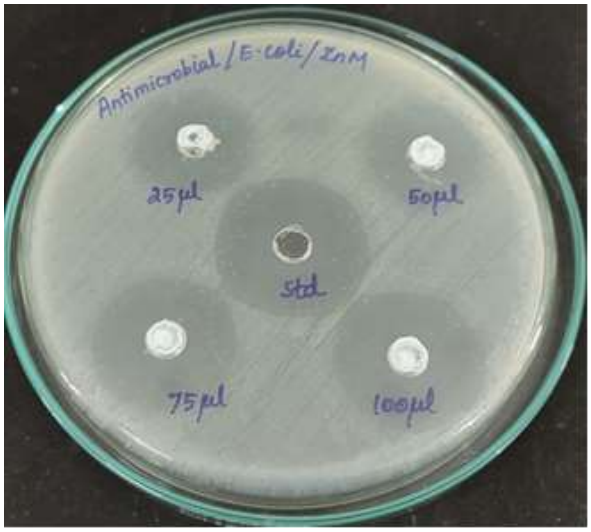

Fig. 5 (b) Antibacterial test of $Zn_{0.5}Mg_{0.5}O$ (Mg-ZnO) nanoparticle against *E. coli*

Concentration	Zone of inhibition
25 µl	0.6 cm
50 µl	0.7 cm
75 µl	1.0 cm
100 µl	1.5 cm
Standard (Chloramphenicol)	1.7 cm

Concentration	Zone of inhibition
25 µl	0.5 cm
50 µl	0.6 cm
75 µl	0.9 cm
100 µl	1.2 cm
Standard (Chloramphenicol)	1.5 cm

From the above table the zone of inhibition, it is evident that ZnO and $Zn_{0.5}Mg_{0.5}O$ are proven to have better antibacterial activity against *E. coli*

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4. CONCLUSION

ZnO and $Zn_{0.5}Mg_{0.5}O$ nanoparticles were prepared by simple and cost-effective co-precipitation method. The formation of hexagonal phase ZnO nanoparticles were confirmed from XRD analysis. There was a decrease in crystallite structure upon Mg doping which ultimately led to increase in surface area of ZnO nanoparticles. The average crystallite size of ZnO and $Zn_{0.5}Mg_{0.5}O$ was 71 nm and 36 nm respectively. The functional groups present in ZnO and $Zn_{0.5}Mg_{0.5}O$ was confirmed from FTIR analysis. There was a formation of nearly spherical shaped morphology of ZnO nanoparticles. An agglomeration of particles observed upon doping Mg in ZnO nanoparticle. ZnO and $Zn_{0.5}Mg_{0.5}O$ was effective in inhibiting *E. coli* bacteria.

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