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Green synthesis of Mg doped zinc oxide nanoparticles using aloe vera plant extract and its characterization

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*Corresponding author: E-mail addresses: a.angelezhilarasi@gmail.com; Phone: 9751781363 ABSTRACT

A novel green and ecofriendly technique is reported in this paper using aloe vera plant extract for the synthesis of pure and metal doped zinc oxide nanoparticles. Biosynthesis of pure and 1.5 wt % Mg doped nanostructured zinc oxide was achieved by microwave method using high purity metal nitrates and aloe vera plant extract. The synthesized doped zinc oxide nanoparicles were characterized by Diffuse reflectance spectroscopy (DRS) X-ray diffraction technique (XRD), Fourier transform infrared spectroscopy (FT-IR), High resolution scanning electron microscopy (HR-SEM), and Photoluminescence (PL) spectroscopy. The formation of Hexagonal wurtzite structure of zinc oxide was confirmed by XRD studies. The formation of zinc oxide phase was also confirmed by FT-IR. The optical properties were determined by DRS and PL. It was observed that the band gap 1.5 wt % Mg doped zinc oxide had an increased band gap than that of pure zinc oxide.

Keywords: green synthesis, optical properties, aloe vera, ZnO nanoparticles, microwave irradiation.

1. INTRODUCTION

In recent years, zinc oxide (ZnO), with remarkable technological and scientific interest possessing a direct wide band gap (3.37 eV, 387 nm, deep violet/borderline ultraviolet (UV) and a large exciton-binding energy (60 meV), is a preferred multitasking metal oxide due to its unique and tunable optical and electrical properties. (Vayssieres et al, 2001; Konenkamp et al, 2002). It also finds applications in UV absorption, solar cells, catalysis, optoelectronics, gas sensors, field emission displays, light-emitting diodes, cosmetics, rubber and textile industry (Wang, 2004; Cao, 2008) Aloe vera plant-extract provides an efficient, simple, pollution free and green pathway for the synthesis of nanostructured particles. A vast work has been reported on the synthesis of various nanoparticles using Aloe vera plant extract (Chandran, 2006; Maensiri, 2008; Phumying, 2013; Klinkaewnarong, 2010; Laokul, 2009; Laokul, 2011). Aloe vera plant extract is an environmentally, non-polluting compatible solvent system, which acts as an eco-friendly reducing agent and a nonhazardous gelling agent for stabilizing the nanostructures (Visinescu, 2011; Varma, 2012; Sheppard, 1988). Microwave use is a ecofriendly technique, with wide advantages like heating source, less time and energy consumption and the ability to synthesize microstructures (Clark, 2000; Palchik, 2000) Microwave heating is varied from conventional heating as the heat is generated within the material internally instead of originating from external sources. The heating is very fast as the material is heated by energy conversion rather than by energy transfer, which occurs in conventional methods (Chandran, 2006).

2. EXPERIMENTAL

Preparation of aloe vera plant extract: 25 grams of thoroughly washed aloe vera plant were finely cut, and the gel obtained was dissolved in 50 ml of deionized water and stirred for 30 min till a clear solution is obtained. The resulting extract was used as the aloe vera plant extract solution.

Preparation of pure and Mg doped zinc oxide by microwave heating method: Zn(NO₃)₂ (99%, Merck Chemicals, India) and Mg(NO₃)₂ (98%, Merck Chemicals, India) were dissolved in the concentration of 1.5 wt % and the above clear transparent solution was placed in a domestic microwave oven (2.45 GHz, 800 W) for 10 min. The solution boiled and underwent dehydration followed by decomposition with the evolution of gases. From the XRD and FT-IR the formation of zinc oxide phase without any impurity was confirmed.

Characterization: The structural studies of pure and 1.5 weight % Mg doped zinc oxide prepared by Microwave method were carried out using a Philips X'pert diffractometer for $2\theta=10^\circ-80^\circ$ using Cu $K\alpha$ radiation at $\lambda=0.154$ nm. . A Perkin Elmer infrared spectrophotometer was used for the determination of the surface functional groups. The morphology of pure and 1.5 weight % Mg doped zinc oxide were performed using JOEL JSM6360 HR-SEM. The emission properties were recorded using a Varian Cary Eclipse fluorescence spectrophotometer. The diffuse reflectance UV-Visible spectra (DRS) of the nano sized materials were recorded using a Cary100 UV-visible spectrophotometer.

3. RESULTS AND DISCUSSION

Structural investigations of pure and Mg-doped ZnO NPs: The structural information and crystallinity of pure and 1.5 weight % Mg doped ZnO nanocrystals prepared by microwave method is given in the XRD pattern as shown

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in figure 1. The strong intense and sharp peaks of ZnO indicate that the nanoparticles are highly crystalline. The samples exhibited at 2θ =18.86°, 31.25°, 36.65°, 44.45°, 55.35°, 59.24°, and 65.53° are associated with the 111, 220, 311, 400, 422, 511, and 440 planes respectively.

The XRD patteren exhibited only zinc oxide phase and no other impurity peaks were observed. The crystallite size of the pure and 1.5 wt % Mg doped ZnO nanocrystals were estimated from higher intense peak of XRD pattern using Debye Scherrer's equation

i.e., D= $0.89 \lambda / \beta \cos \theta$.

Where D is the average crystal size, λ – the wavelength of the X-ray radiation and β – the full width at half maximum (FWHM). The calculated average crystallite size from the high intense plane of (101) is 22.51nm.

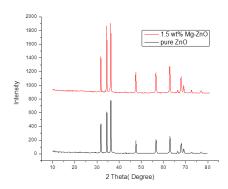


Figure 1.XRD images of pure and Mg doped ZnO prepared by microwave heating

Size and morphological investigations of pure and Mg-doped ZnO NPs: Morphological and structural characterizations of the prepared pure and 1.5 wt% Mg doped ZnO NPs were performed by HR-SEM. HR-SEM image of pure ZnO, 1.5 wt% Mg doped ZnO prepared by microwave are shown in Figure 2 and Figure 3. Pure ZnO NPs are nearly spherical in shape and are highly agglomerated. 1.5 wt% Mg -doped ZnO NPs and self-assembled as flake like morphology.

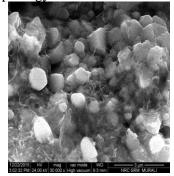


Figure.2.Pure ZnO Prepared by microwave method

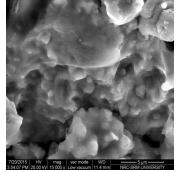
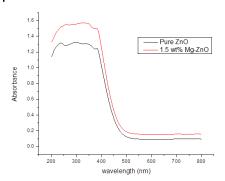


Figure.3. 1.5 wt % Mg doped ZnO Prepared by microwave method

Optical absorption and photoluminescence investigations of pure and Mg-doped ZnO NPs: Figure 4 shows diffuse reflectance spectra of pure and 1.5 wt % Mg doped ZnO NPs by microwave method. A sharp absorption edge at about 381 nm (energy band gap - 3.25 eV) corresponds to pure ZnO. It can be clearly seen from Figure 4 that the maximum of the absorbance band shifts towards lower wavelength in increasing the Mg concentration from 381 nm to 379 nm refers to the band gap energy of 3.25 ev to 3.36 ev. This could be mainly attributed to the quantum size effect.

The obtained PL spectra of pure and 1.5 weight% Mg doped ZnO prepared by microwave method is shown in figure 5. The emission at 361 nm is a weak UV band emission, a result of recombination of an exited electron. The visible or deep trap state emissions at 409,446,490,619,694, nm refers to the recombination of the electron-hole pairs from localized states with energy levels deep in the band gap, resulting in lower energy emission. The results indicate that impregnated Mg does not eliminate the emission peaks of ZnO, but confound the recombination of photo induced electron – hole pairs.

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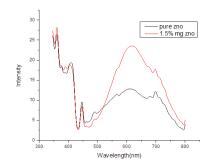


Figure.4.DRS images of pure and Mg doped ZnO prepared by microwave heating

Figure.5.PL spectra of pure and Mg doped ZnO prepared by microwave heating

Fourier transform infrared spectroscopy (FT-IR) studies: Figure 6 shows the FTIR spectra of pure and 1.5 wt% Mg doped ZnO nanoparticles synthesized by microwave combustion method. The strong band observed at wave number 435 cm clearly shows the presence of Zn-O stretching mode. Other bands at 3425, 2938, 2345, 1389, 1108 cm_1 corresponds to the presence of hydroxyl group, carboxylic acid O-H stretching mode , C=O stretching mode, N-H bending vibration for primary and secondary amines of proteins present in plant extract and C-H stretching mode for alkanes.

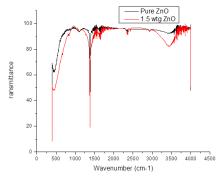


Figure.6.FTIR spectra of pure and Mg doped ZnO prepared by microwave heating

4. CONCLUSION

Pure and 1.5 wt % Mg doped ZnO was prepared by microwave method using aloe vera plant extract. ZnO nanoparticles obtained were highly crystalline and found to have crystal defects which were confirmed by their tunable optical properties. The average crystallite size of ZnO nanoparticles prepared by microwave method was 22.51 nm. The formation of ZnO phase was confirmed by XRD and FT-IR results UV studies confirmed the increase of band gap as Mg was doped in ZnO nanoparticle phase. Hence a simple green pathway has been developed to synthesize pure and Mg doped ZnO nanosized structures with tunable properties.

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