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RESEARCH ARTICLE

SYNTHESIS AND CHARACTERIZATION OF ZINC OXIDE NANOPARTICLES DOPED WITH DIVALENT MANGANESE BY GREEN ROUTE USING PLANT EXTRACT

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ABSTRACT

This work describes a novel green and ecofriendly technique for manufacture of pure and metal doped zinc oxide nanoparticles utilising aloe vera plant extract. The microwave approach was used to biosynthesize pure and 1.5 wt% Mg doped nanostructured zinc oxide utilising high purity metal nitrates and aloe vera plant extract. Diffuse reflectance spectroscopy (DRS), X-ray diffraction method (XRD), Fourier transform infrared spectroscopy (FT-IR), High resolution scanning electron microscope (HR-SEM), and Photoluminescence (PL) spectroscopy were used to characterise the synthesized doped zinc oxide nanoparicles. XRD analyses supported the formation of the hexagonal wurtzite structure in zinc oxide. The FT-IR method also supported the creation of the zinc oxide phase. DRS and PL were used to determine the optical characteristics. The band gap of 1.5 wt% Mg doped zinc oxide was found to be greater than that of pure zinc oxide.

INTRODUCTION

The remarkable technological and scientific interest in zinc oxide (ZnO), which has a direct wide band gap of 3.37 eV, a deep violet/borderline ultraviolet (UV) wavelength of 387 nm, and a significant exciton-binding energy of 60 meV, has made it a popular multipurpose metal oxide in recent years due to its distinct and adaptable optical and electrical properties (Vayssieres, 2001; Konenkamp, 2002). Additionally, it is utilised in the production of UV absorbers, solar cells, catalysis, optoelectronics, gas sensors, field emission displays, light-emitting diodes, cosmetics, rubber, and textiles (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 a non-toxic, ecologically friendly solvent that can be used to stabilise nanostructures (Visinescu, 2011; Varma, 2012; Sheppard, 1988) by acting as a non-hazardous gelling agent, eco-friendly reducing agent, and non-polluting solvent system .The use of microwaves is a green method with many benefits, including heating source, reduced time and energy consumption, and the ability to synthesise microstructures (Clark, 2000; Palchik, 2000).

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In contrast to conventional heating, microwave heating produces heat internally within the material, as opposed to coming from outside 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).

EXPERIMENTAL

20 grams of properly cleaned aloe vera plant were finely cut, and the resulting gel was diluted in 40 ml of deionized water and agitated for 30 minutes to produce a clear solution. The resultant extract served as the aloe vera plant extract solution. $Zn(NO_3)_2$ (99%, Merck Chemicals, India) and $Mg(NO_3)_2$ (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 mixture began to boil, dehydrate, and then decompose with the evolution of gases.

Characterization: From the XRD and FT-IR the formation of zinc oxide phase and its purity was confirmed. 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 $2\theta = 10°–80°$ using Cu Kα 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 (Angel Ezhilarasi, 2016)

RESULTS AND DISCUSSIONS

X-Ray Diffraction Analysis: The XRD pattern of pure and 1.5 weight% Mg doped ZnO nanocrystals generated by microwave technique provides structural information and crystallinity, as illustrated in figure 1. The highly crystalline nature of the nanoparticles is shown by the strong, intense, and sharp peaks of ZnO. 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.

There were no other impurity peaks visible in the XRD pattern, which exclusively showed the zinc oxide phase. Using Debye Scherrer's equation (1), the crystallite size of the pure and 1.5 wt% Mg doped ZnO nanocrystals was calculated from the higher intensity peak of the XRD pattern.

$$D = 0.89 \lambda / \beta \cos \theta \quad \text{.....(1)}$$

Where D is the average crystal size, λ – the wavelength of the X-ray radiation and β – the full width at half maximum .The calculated average crystallite size from the high intense plane of (101) is 22 nm

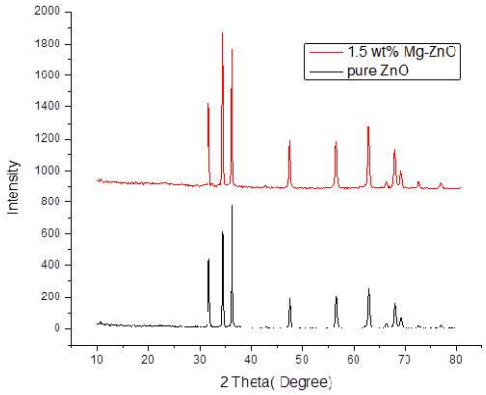

Figure 1. XRD images of pure and Mg doped ZnO prepared by microwave heating

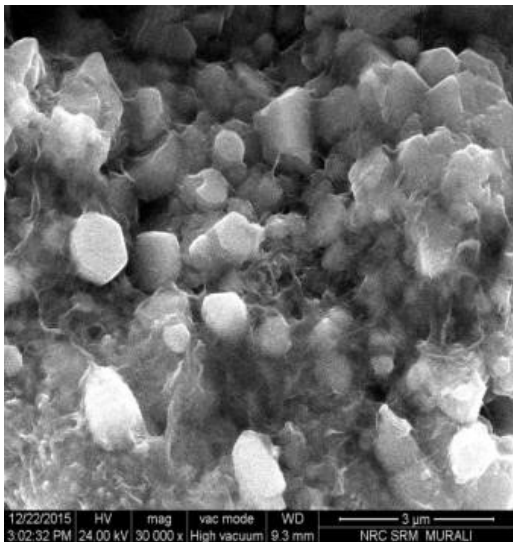

Figure 2. SEM of Pure ZnO prepared by microwave method

SEM Analysis: The produced pure and 1.5 wt% Mg doped ZnO nanoparticles underwent morphological and structural characterizations by HR-SEM. Figures 2 and 3 depict HR-SEM images of microwave-prepared pure ZnO and ZnO with 1.5 weight percent Mg doped. Pure ZnO particles are nearly spherical in shape and are highly agglomerated. 1.5 wt% Mg - doped ZnO particles and self-assembled as flake like morphology.

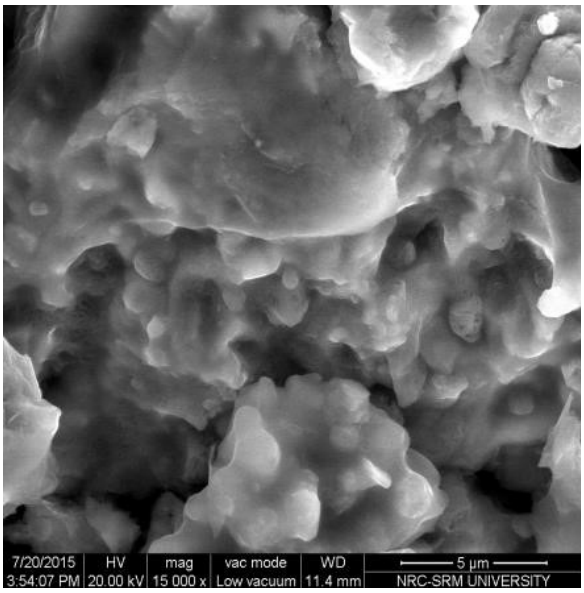

Figure 3. SEM of 1.5 wt% Mg doped ZnO prepared by microwave method

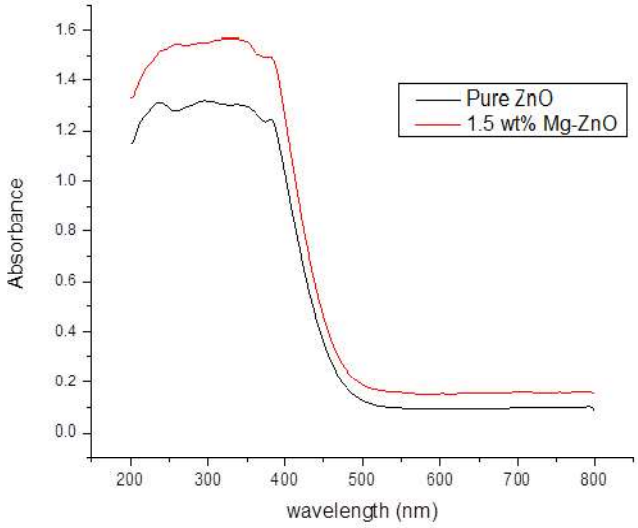

Figure 4. DRS images of pure and Mg doped ZnO prepared by microwave heating

Optical absorption and photoluminescence investigations of pure and Mg-doped ZnO: 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 nmto 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 shownin Figure 5.

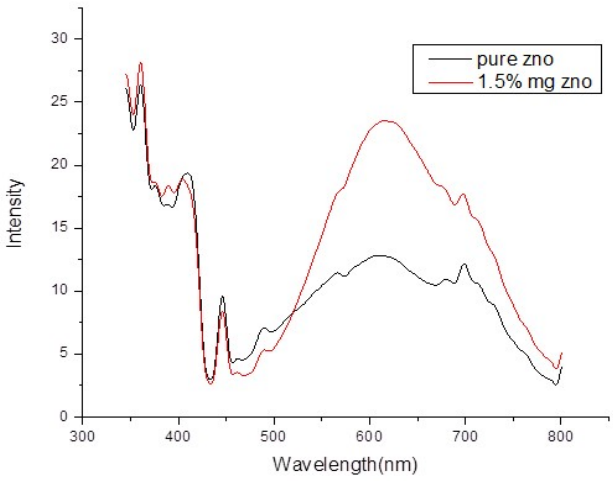

Figure 5. PL spectra of pure and Mg doped ZnO prepared by microwave heating

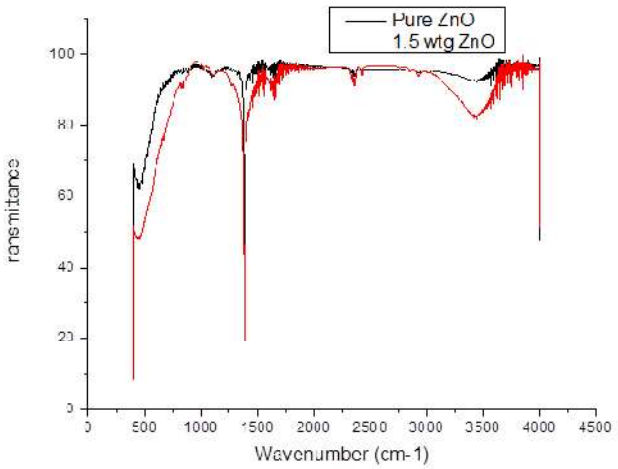

Figure 6. FTIR spectra of pure and Mg doped ZnO prepared by microwave heating

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 and 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.

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 435cm^{-1} clearly shows the 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.

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 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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