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RESEARCH ARTICLE

SYNTHESIS AND CHARACTERIZATION OF UNDOPED AND DOPED ZnO PHOTOCATALYST

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ABSTRACT

Undoped and doped ZnO photocatalyst where characterized by Fourier transform infra red (FTIR), Diffuse reflectance spectra (DRS), Scanning electron microscope (SEM), Brunauer-Emmett-Teller (BET) and X-ray diffraction (XRD). From these studies we investigated the optical absorption (λ_{max}), functional group, surface morphology, particle size and elementary composition of undoped and doped ZnO. The dopants used where four atomic percentage of Mn^{2+}, Fe^{2+}, Co^{2+} and Ni^{2+}.

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INTRODUCTION

Wide band-gap oxide semiconductors, when doped with transition metal ions (Mn, Fe, Co and Ni) have attracted much attention for their promising versatile applications. ZnO is an n-type semiconductor with wide direct bandgap energy (3.37 eV) and a larger binding energy (60 meV). Due to its unique characteristics like low cost, non toxicity, abundance in nature, suitability to doping, this material has got wide applications in electronic and optoelectronic devices such as ultraviolet light-emitters, piezoelectric transducers and solar cells (Bao *et al*., 2006; Cheng *et al*., 2007; Sahay and Nath, 2008; Xu *et al*., 2005). In this paper, we present our investigation to understand the pure ZnO and metal doped ZnO photocatalyst prepared through a hydrothermal method. The synthesis and metal doped to understand the photocatalyst different characterization.

MATERIALS AND METHODS

Photocatalyst preparation

Zinc oxide, were purchased from sigma Aldrich and was of analytical reagent 95 grade and used without further purification. Deionised water was used in all experimental preparations. The samples were prepared by hydrothermal method (Dhanalakshmi *et al*., 2008). High temperature sintering method was adapted for the preparation of the photocatalyst. In the case of doping, a common procedure was followed: An aqueous slurry of the semiconductore powder containing calculated amounts of transition metal salt was stirred magnetically (REMI-magnetic stirrer-2MLH) for 2 hours to distribute the metal ions uniformly upon the semiconductor powder. This slurry was then evaporated in an air oven at 100-200^{o}C. The dried samples were ground to fine powder, loated in a silica boat and introduced into a muffle furnace for sintering. Sintering was carried out at 450^{o}C for all the samples. Stepwise increases of temperature increased the effectiveness of doping (Mori *et al*., 1985; Prabu and Anbarasan, 2014). Sintering of samples was carried out in an inert atmosphere, after cooling to room temperature, the sintered samples were ground to fine powder.

Instrumentation

Fourier transform infrared spectra (FT-IR) were recorded in a Bruker 3000. UV-vis diffuse reflectance spectra (DRS) were determined by recorded on Varian Cary 5000 model UV spectrometer. Scanning electron microscopy (SEM) measurements were performed on 'ZEISS'. The surface areas of the photocatalyst powders were measured using a BET apparatus. X-ray diffraction patterns were recorded using computer controlled XRD units (X-ray generator: PW 1130: Vertical diffractrometer: PW 1050; Diffractrometer control PW 1710; Philips, Holland).

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RESULTS AND DISCUSSION

Characterization studies

Fourier Transform-Infrared (FT-IR)

FTIR spectra of pure and metal doped ZnO photocatalyst are shown in the Fig. (1). The broad peak in higher energy in the region at 3400 - 3600 cm^{-1} is due to OH stretching or it may be due to the M-OH-M. The peak in the range 1400 - 1757 cm^{-1} is due to OH bending of adsorbed moisture in the sample and all other peaks are attributed to the characteristic of the material. The main absorption band is due to Zn-O stretching of ZnO in the range of 600 - 400 cm^{-1}. FTIR spectra of pure sample of the present investigation are similar to that of Cr doped ZnO samples and are in good agreement with the reported values (Kurian *et al*., 2004; Rema Devi *et al*., 2007; Maensiri *et al*., 2006; Suwanboon, 2008; Dole *et al*., 2011). (ZnO and Fe-ZnO), the peaks in the range of 400-700 cm^{-1} were attributed to ZnO stretching modes (Saleh *et al*., 2013). Also an additional peak was obtained in the range from 800 – 1500 cm^{-1}, which could be attributed to the incorporation of Fe^{3+} ions into the lattice position of the ZnO nano-structures (Pandiyarajan *et al*., 2012).

Diffuse Reflectance Spectra (DRS)

The UV-Visible diffused reflectance spectra (DRS) of the undoped and Cd, Fe, Cr doped ZnO samples are shown in Fig. (2). The band gap (E_g) of ZnO nanoparticles were calculated by using $E_g = hc/\lambda$, where h = plank's constant, c = velocity of light and λ = wavelength. The maximum absorption for all samples are observed in 380 nm from which the approximate band gap of 3.2 eV is calculated.

Scanning Electron Microscope (SEM)

The SEM is used to study the surface morphology of as-synthesized samples. The SEM micrographs of as-synthesized samples of pure ZnO and metal doped ZnO are shown in fig**.** (3). As synthesized samples of undoped particle size on the surface of small. The doped samples particle size is very large is observed.

Surface area measurements (BET)

The surface area of undoped ZnO is found to˷ 4.6 m^2/g. It is generally observed that the surface areas of the doped samples slightly increased from those of the undoped samples.

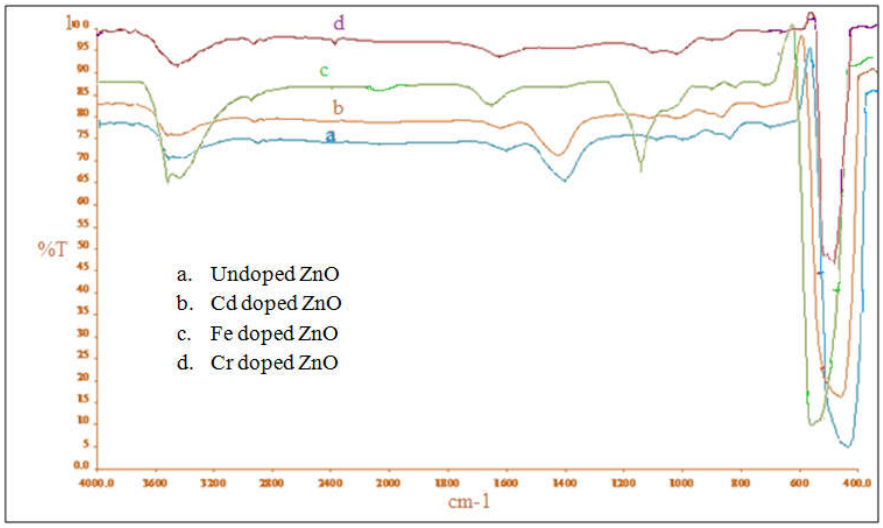

Fig.1. IR Spectra of undoped and metal ions ZnO

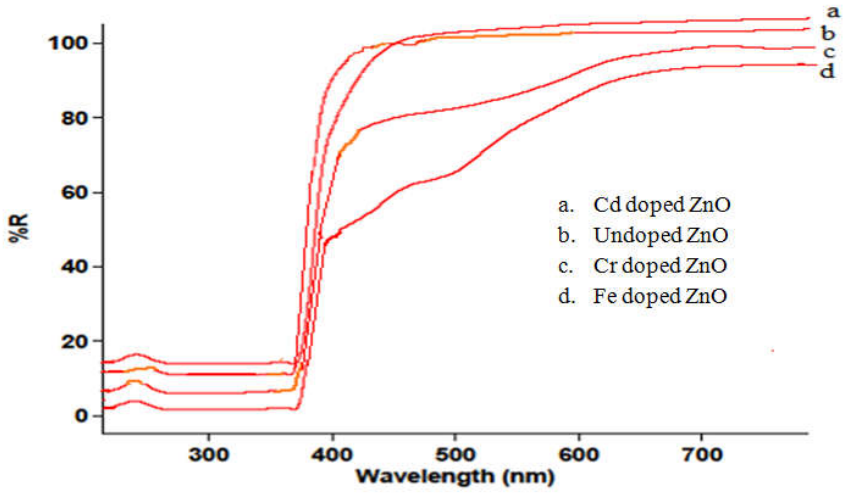

Fig.2. Diffuse absorption spectra of undoped and metal ions doped ZnO

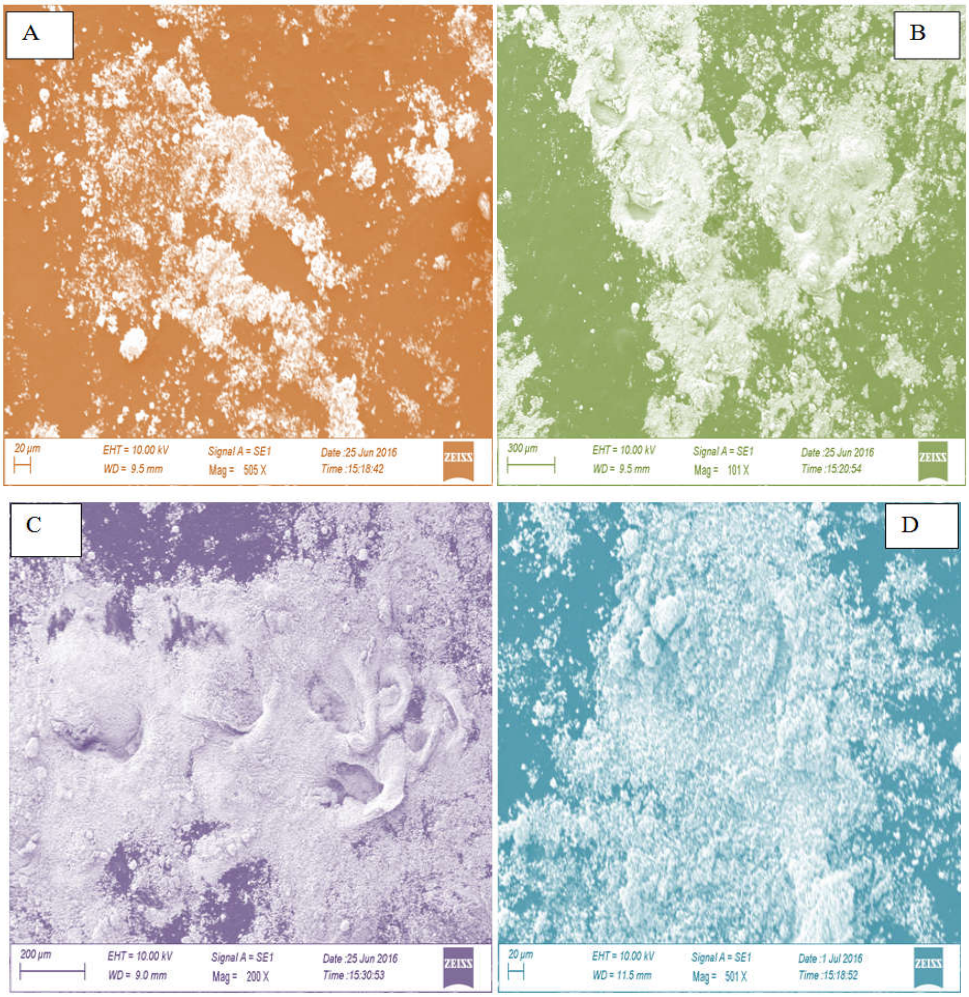

Fig.3. Scanning Electron Micrographs of (a). Undoped ZnO (505 X) (b). Four atomic percentage Cd doped ZnO (101 X) (c). Four atomic percentage Cr doped ZnO (200 X) (d). Four atomic percentage Fe doped ZnO (501 X)

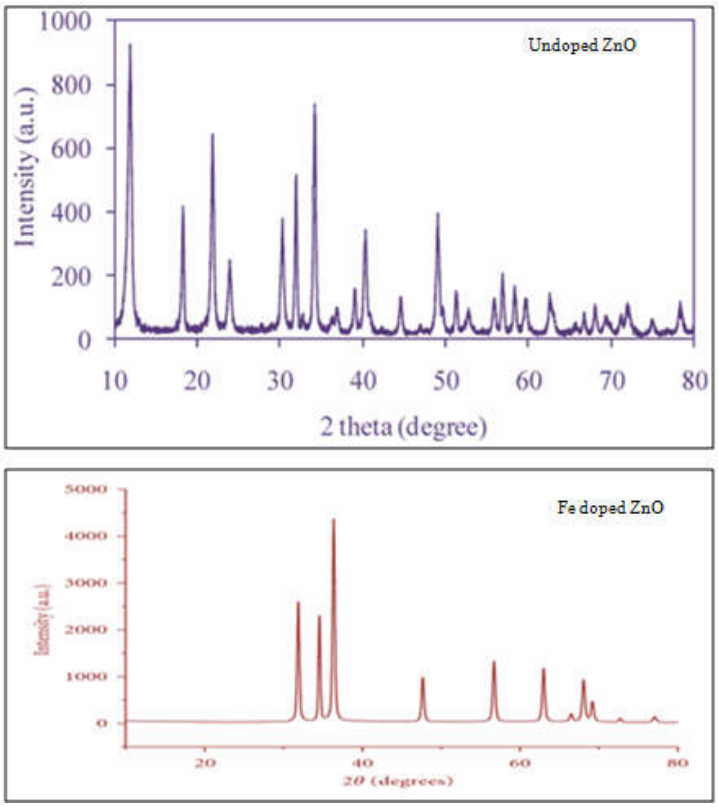

Fig.4. X-diffractogram of undoped and Fe doped ZnO

However, the difference in the surface areas of each sample is almost negligible. Hence, the differences in the photocatalytic efficiencies of these undoped and doped samples may not be due to the small differences in the surface area but due to the other factors such as dopant nature, dopant concentration, etc.

X-ray diffraction (XRD)

X-ray diffraction measurements have been taken in the range, $2\theta = 20^0 - 80^0$ for the undoped and doped (Cd/ZnO, Fe/ZnO and Cr/ZnO) photocatalysts. The 2θ values at which major peaks appear have been found to be almost the same for both undoped and doped samples except the intensities of the peaks. It is interesting to the crystallinity has been increased as evidenced from the more intense peaks observed in the doped samples. This observation is also supported by XRD spectrum Fig. (4) Where more intense and sharp peaks are observed for the doped samples.

Conclusion

This present study showed a very simplest technique for metal doping four atomic percentage Cd^{2+}, Fe^{3+} and Cr^{3+} the chemical groups of the samples have been identified by FTIR spectra. The cut-off wavelengths were identified by UV-Visible (DRS) analysis and the band gap energies of the undoped and doped products are lies between 3.2 eV. The SEM image of particle size confirmed that doped and undoped morphology of the products. This observation is also supported by the XRD spectrum where more intense and sharp peaks are observed for the doped samples.

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