

# Structural and morphological study of Mn doped ZnO nanoparticles prepared by sol-gel method

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## ABSTRACT

1, 3 and 5 at. % Mn doped ZnO nanoparticles have been synthesized by the sol-gel method. The effect of different Mn doping concentrations on the structural and morphological properties of ZnO was studied by using XRD, SEM, TEM and EDAX. The XRD analysis reveals that the samples are polycrystalline with hexagonal wurtzite structure. The SEM images of ZnO nanoparticles show that the grain size decreases with an increase in the Mn content. EDAX study reveals there is no impurity peak present in prepared samples and shows the samples are in stoichiometric.

**Keywords:** Mn doped ZnO nanoparticles, XRD, SEM, TEM and EDAX

## INTRODUCTION

ZnO is a well-known wide band gap (~3.3 eV) semiconductor material, It is also gaining importance because of their potential applications and desired properties such as low cost and non-toxicity. In particular, ZnO thin films can be applied to acoustic-wave devices due to their large piezoelectric constant. Recently, a number of ZnO films doped with various metallic ions to study extensively for the manipulation of their optical and electrical properties (Lu *et al.* 2007). Zinc oxide (ZnO) is one of the few oxides that show quantum confinement effects. it is also used in broad areas such as catalysts, microsensors, photoelectrochemical cells, piezoelectric transducers, photo-detector, UV-detector and actuators. ZnO is also a green material hence it is bio safe, biocompatible and biodegradable. It is widely used in medical applications and environmental science (Li *et al.* 2011). ZnO is a piezoelectric material and has been used for transparent thin film transistors, blue and UV light-emitting diodes and laser diodes. Because of its cheapness and abundance ZnO attracts much attention in recently (Chiena *et al.* 2004). Zinc Oxide (ZnO) is a group II-VI semiconductor material widely applied in optoelectronic, high chemical stability, and high order transmittance and piezoelectric properties.

Development electronic devices such as surface acoustic wave devices, gas sensors, FET, and solar cells ZnO are used. In solar cell device technology, ZnO is most important material used as the window layer because of its transparency to the solar radiation spectrum centered at 1.5 eV. ZnO is considered to be the most promising TCO material in the view of low cost, excellent transmittance coefficient and stable chemical material properties (Gupta *et al.* 2014). Chao Li et al. studied gas sensing property of honeycomb-like ZnO by direct precipitation method (Li *et al.* 2008). Duy-Thach Phan et al studied Effects of defects in Ga-doped ZnO nanorods formed by a hydrothermal method (Phan *et al.* 2013). Dar et al. (2012) studied Ce-doped ZnO nanorods for the detection of hazardous chemical.

For metal oxides, doping with suitable cation/anion modifies the bandgap which may modulate their electrical properties. Band gap of nanocrystals can be tuned by altering the size, shape of the materials through alloying, doping, strain tuning, band gap edge warping techniques. Injection and transportation of photo-excited electrons within the cell plays important role in determining the efficiencies of DSSCs. Introducing cations as dopant in photo-anode materials (metal oxides) exerts larger dipole moment that changes the interface energetic for electron transfer (Sengupta *et al.* 2016). Recently, transport properties, especially the anomalous Magneto-resistance (MR) of transition metal (TM)-doped ZnO, have attracted much interests. A few models have been proposed to describe the observed anomalous MR behavior. In most cases, the positive component of MR is thought to result from a giant spin splitting of band states caused by the s-d exchange interaction in DMSs (Ji *et al.* 2009).

Transition metal (TM) doped ZnO has recently attracted considerable attention for the potential application in spintronic devices because its Curie temperature is theoretically predicted to be well above room temperature and room temperature ferromagnetism (RTFM) has been observed experimentally in Co-,3 Fe-,4 Mn-,5,6 and Cu-doped7 ZnO systems (Xu DH *et al.* 2012). Hence in present investigation, ZnO nanoparticles have been prepared by the sol-gel method. The purpose of present study is to investigate the effect of Mn doping on to the structural and morphological properties of ZnO nanoparticles and to improve the materials performance for its various applications.

## METHODOLOGY

### Synthesis of ZnO nanoparticles

Zinc oxide nanoparticles were prepared by sol-gel method. For this 0.2 M zinc acetate dihydrate solution, prepared in distilled water. The ammonium hydroxide added drop wise to this prepared solution till pH becomes 8 with continuous stirring and formation of sol take place. This sol of zinc hydroxide is dried at 80 °C for 6 h to get gel. The obtained gel is calcinated at 400 °C for 3 h in a muffle furnace which forms pure Zinc oxide nanoparticles. Same procedure followed for doped ZnO synthesis using dopant Mn acetate of 1, 3 and 5 at. % which is added with zinc acetate precursor to form doped ZnO nanoparticles.

Finally, the sample was obtained after cooling down at room temperature in air. The XRD patterns were recorded on Bruker D2 PHASER XRD. SEM and EDAX image was obtained on JEOL JSM-6360. TEM image was obtained on JEOL JEM 2100.

## RESULTS AND DISCUSSION

### 1. Structural properties (XRD)

Fig. 1 shows the X-ray diffraction patterns of pure ZnO and 1%, 3%, 5% Mn doped ZnO obtained with Cu-K $\alpha$  (1.54056 Å) radiation. It has been observed that all of peaks of XRD pattern belong to the hexagonal lattice of ZnO with three most preferred orientations namely (100), (002) and (101). Most importantly, all of the XRD peaks were attributed to ZnO and no other undesired peaks were observed due to secondary phases or impurity phases within the detection limit of the X-ray diffractometer. From Fig1. it is seen that the all samples are polycrystalline in nature with no impurity peak present. The peaks obtained are indexed by using JCPDS card no. 36-1451. The lattice constant 'a' is calculated by using the following formula:

$$d = \frac{a}{\sqrt{h^2 + k^2 + l^2}}$$

Where, (hkl) are miller indices, 'd' is interplaner spacing and 'a' is lattice parameter. The average crystallite size 'D' of the samples was calculated using the Debye-Scherrer formula

$$D = \frac{0.9\lambda}{\beta \cos\theta}$$

Where ' $\lambda$ ' is the wavelength of X-ray (1.5406 Å), ' $\beta$ ' the full-width at half-maximum in radian, and ' $\theta$ ' the angle

of diffraction. It is seen that crystallite size (D) varies in between 24 and 33 nm.

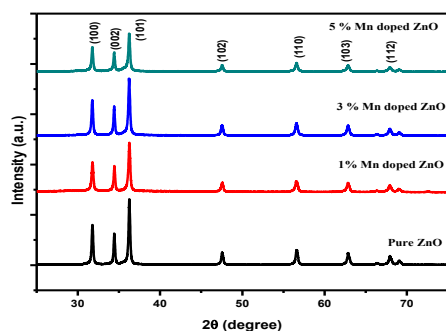


Fig.1-XRD patterns of pure ZnO and 1%, 3%, 5% Mn doped ZnO Nanoparticles

## 2. Morphological Properties (SEM and TEM)

Fig 2. shows the SEM images of pure ZnO and 1%, 3% and 5% Mn doped ZnO nanoparticles. The SEM images show that the formation of cube like structure of the grains. Results obtained from SEM are in good agreement with results obtained from XRD. The variations in XRD results are well supported by TEM micrographs. Fig 3. represents the low resolution TEM images of all synthesized samples. The morphology of all the samples has been found to be roughly spherical with irregular particle size distribution. Particle diameter has wide size distribution ranging from 24 and 33 nm for samples.

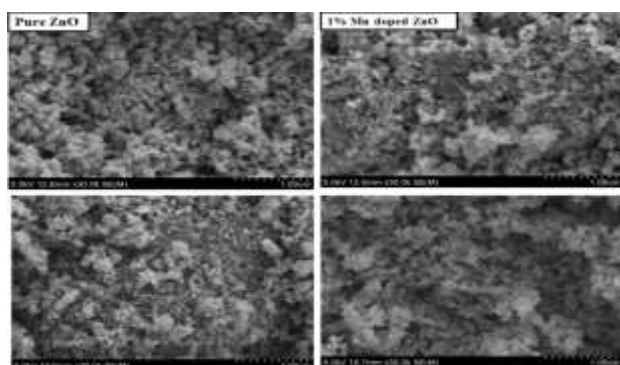


Fig.2- SEM images of pure ZnO and 1%, 3%, 5% Mn doped ZnO Nanoparticles

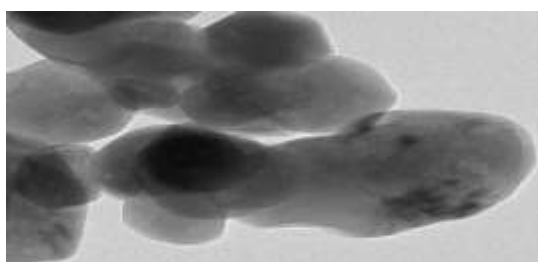


Fig.3- TEM image of ZnO Nanoparticles

## 3. EDAX

Fig. shows the EDAX spectrum of pure ZnO and 1%, 3% and 5% Mn doped ZnO nanoparticles. The result confirmed the presence of the required elements in the prepared composition with almost all the peaks associated with elements such as those of Zn, Mn and O. From the EDAX data it is seen that there is no major impurity during formation of the ZnO nanoparticles.

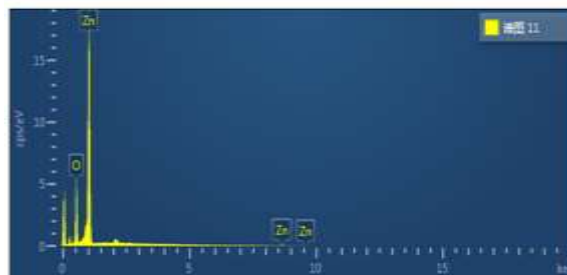


Fig.4 (a) - EDAX image of pure ZnO Nanoparticles

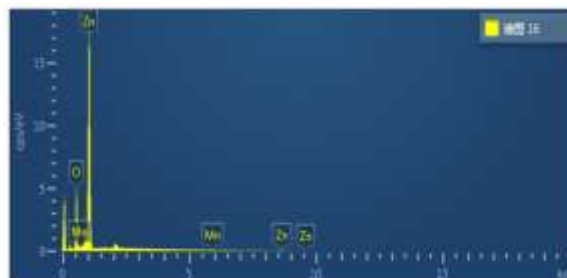


Fig.4 (b) - EDAX image of Mn doped ZnO Nanoparticles

## CONCLUSION

Sol-gel synthesis route is simple, cost and time effective, non-toxic method for synthesize Mn-doped ZnO nanoparticles. The XRD pattern reveals the formation of hexagonal wurtzite structure with average crystalline size of 24 and 33 nm. SEM and TEM images reveal the nanoparticles are in cubic shape with the particles are uniformly dispersed and the grain size decreases with an increase in the Mn content because the atomic radius of Mn (117pm) is less than Zn (125pm). From EDAX result it is confirmed that the presence of the required elements in the prepared composition and the prepared samples are well stoichiometric. So, Morphological changes associated with the Mn doped ZnO which helps to control surface area of ZnO nanoparticles for different applications such as dye sensitized solar cell devices, catalysis and gas sensing.

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