Indian Journal of Pure & Applied Physics
Vol. 63, January 2025, pp. 77-81
DOI: 10.56042/ijpap.v63i1.13592

Improvement in Structural and Optical Properties of $ZnFe_2O_{3-\delta}$ in Nano Structured Materials with Fe_2O_3 Concentration

Tanuja Kumari[a*], AnitaYadav[b] & Rajesh Sharma[b]

[a]Baba Mastnath University, Asthal Bohar, Rohtak, Haryana 124 001, India
[b]Faculty in Physics, MNS, Govt. College, Bhiwani, Haryana 127 021, India

Received 8 September 2024; accepted 19 December 2024

At present time, technologies of particles at nanoscale are a blazing field for today's researchers. The transition metal oxide (TMO) nanomaterials are used immensely because of their colourful properties caused by their valence d-orbital electrons. The ZnO and Fe_2O_3 nano particulates have promised properties such as electrical, magnetic, antibacterial and photo-catalytical etc. In present work, researchers synthesized pure ZnO and Fe doped ZnO nano particulates by microwave irradiated chemical co-precipitation method. The as-synthesized samples were calcined at 600 ºC for 2 hrs. The various concentration (5, 10 & 20 %) Fe doped samples were examined through XRD, FTIR, FESEM and HRTEM tools. The XRD results revealed that calcined samples were formed of hexagonal wurtzite structure of ZnO parental structure with crystallite size 26 nm (5%), 44 nm (10%) and 50 nm (20%) respectively. The presence of Fe^{3+} ions produces a uniform strain in crystals. The significant IR peaks at position at 853 cm^{-1} and 514 cm^{-1} may be attributed by O-Fe-O & O-Zn-O of Fe_2O_3 and ZnO respectively. The morphology of nanoparticles was examined through electron microscopy *i.e.* FESEM and HRTEM. FESEM results showed that nanoparticles are irregular in size with stone-like polygon structure and porous in nature. HRTEM images revealed that particles are polycrystalline in shape, agglomerated in nature and nano in size. The average grain size measured by Image J software was 42.3nm and also supports the XRD results. The UV visible spectroscopic results of samples shows that newer materials are highly sensitive in the wavelength range of visible spectra and have ΔE – 1.8 eV respectively. So, newer materials may have recommended key material for fabrication of photo induced properties devices such as solar cell aided photovoltaic and photodiode. The PL spectra of the Fe doped ZnO nano particulates reveals that their excitation wavelength was 303 nm which is located in the strong UV emission region and has a solid emission peak at 665 nm which lies in the red region of white spectrum.

Keywords: XRD; FTIR; FESEM; HRTEM; Pl; ZnO; Fe doped Nanoparticles

1 Introduction

The recent study shows that particles exhibit different properties with change in its shape, size and condition during their synthesis. The properties of solid material drastically change at nanoscale so that study of innovative technological applications of these particles have priority for today 's researchers. Among transition metal oxide, ZnO is a promising candidature due to its semiconducting properties with band gap ΔE -3.37eV and size dependent magnetic behaviour. The past study shows Zn and ZnO have diamagnetic behaviour over a wide range of applied external magnetic fields. Whereas, at nano size ZnO and Fe doped ZnO transformed their behaviour from diamagnetic to super paramagnetic with size dependency. Moreover, the iron oxide nanoparticles (IONPs) also have unique physicochemical, magnetic and photo-catalytical properties due to optimum d-orbital valencies and spin magnetic moment. Therefore, the advanced properties of Fe_2O_3 nanoparticles make its superiority among other fields of technological application such as MRI's, supercapacitor, memory storage devices and other industrial applications. In present work, the author aimed to synthesize ZnO NPs, Fe_2O_3 NPs and Fe_2O_3 doped ZnO nanocomposite with different concentrations and calcinations temperature via microwave irradiated chemical co-precipitation method. The newer material may have adhered cumulative properties of host (ZnO) and guest (Fe_2O_3) materials. The effect of Fe concentration in ZnO lattices in respect of structural and optical behaviour were summarized.

2 Synthesis Mechanism

ZnO pure and Fe-doped ZnO samples were prepared via microwave irradiated co-precipitation

---

*Corresponding author:
(E-mail: tanujavashistha27@gmail.com)

protocols. All the chemicals used in present study were highly pure in nature and researchers declared that no further purification was made at laboratory scale. The appropriate concentration of Zinc Nitrate and Ferric Chloride were taken as source material and mixed with 100ml doubly deionised water. The solution was mixed reflex by a magnetic stirrer so that light reddish colour solution was obtained. The 2.0 M NaOH solution was used as surfactant and added dropwise to raise Ph of ZnO pure and Fe doped ZnO solutions. And resulting precipitation was formed at Ph of solution at 8.0 and above. The optimum precipitation with small size was seen at Ph 9.0 and kept at consideration in this research. The precipitates (ppts) were collected via filtration Whiteman filter paper and ppts washed multiply with ethanol and doubly deionised water. The wetty cakes of various samples were irradiated with a microwave in an oven. The dried cake was calcined at 600 °C for 2hr in a muffin furnace and crushed in agate mortar so that finally powdered samples were in formation. The various calcined samples were examined through various characterization tools such as X-Ray diffractometer, IR Spectrometer, FESEM, HRTEM and Pl *etc*.

3 Results and Discussion

3.1 X-Ray Diffraction

The diffraction pattern of the beams was examined using X-rays and the source of X-rays was copper CuKα = 1.5406 Å. The scanning rate was maintained at 57.3 minutes per second for the wide-angle diffraction with a range of 2Θ. Fig. 1 displayed the X-ray diffraction patterns of both pure and FeO/ ZnO films by using X-ray diffractometer (Bruker D8 Advance) in CIF, LPU, PHAGWARA(PUNJAB).

The XRD results revealed that no phase transformation occurred with doping concentration Fe_2O_3 in ZnO. However, broadening of peaks occurred at 5% concentration of Fe_2O_3, it might be due to distortion in crystal lattice occurring with small doping level and thereafter, broadening observed in lesser amounts with further increase of dopant concentration of Fe_2O_3 in ZnO. It might be due to strain arising in the crystal due to presence of Fe^{3+} ion and Fe^{2+} ion and the same were signified by additional peaks occurring at 22.48, 29.41, 18.36, 22.56, 26.04. Results showed that calcined samples were polycrystalline associated with the crystal structure of hexagonal wurtzite. Each observed peak extremely matches the peaks in the JCPDS card no.36-1451.

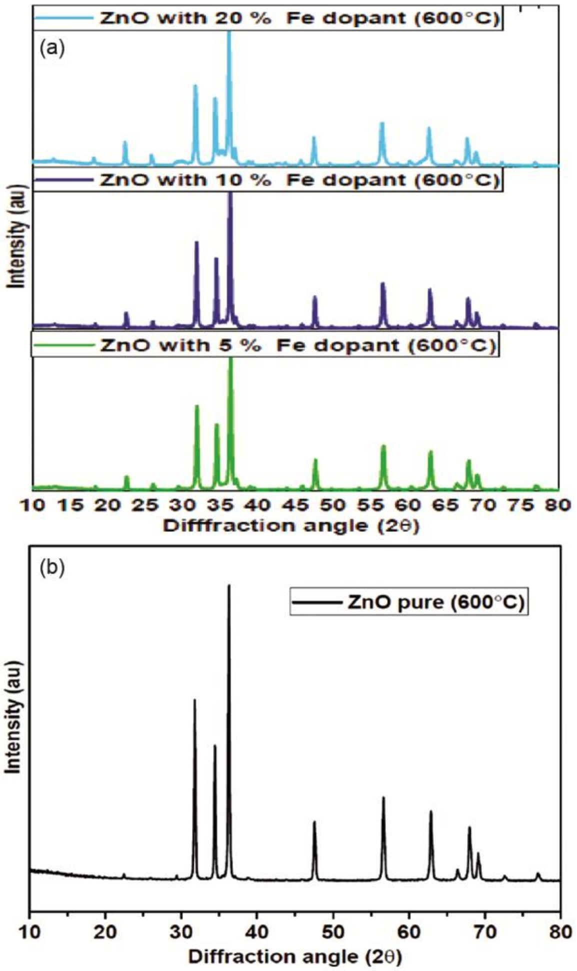

Fig. 1−XRD of (a) Fe doped ZnO (5 %, 10% & 20%) and (b) ZnO (pure) calcined for 2hrs at 600 °C

The variation in the maximum intensity at 36° is shown in the Table-1. The outcomes revealed that the intensity rises with doping concentration.

The range of the measured D values was 40-50 nm. The size of the FeO/ZnO crystallites initially decreases and then increases as the doping concentration increases as displayed in Table -1.This indicates that there are more strains and defects caused by the Fe doping in ZnO.

Saleh et al. also observed that FeO / ZnO produced by co-precipitation process reduced the size of the crystallites[15].

With help the Debye Scherrer formula, the size of the calcined samples was established.

$$d = 0.89\lambda/\beta \cos \theta \qquad \ldots (1)$$

where d -crystallite size and θ, λ and β have as usual scientific notation.

3.2 IR Spectroscopy findings

IR peaks corresponding to vibration of various oxide nanoparticles were exhibited at position 0-4000 cm^{-1} by using Perkin Elmer Frontier FTIR. The infrared spectroscopy was employed to study the elemental analysis of all the calcined ZnO pure and Fe (5%, 10% & 20%) doped ZnO calcined at 600 ºC for 2hrs. Fig. 2 shows the corresponding chemical bonding between the constituent elements and their mode of vibration in the calcined pure and 5-20% FeO/ ZnO nano particulates. The peaks at position 411, 544 and 570 cm^{-1} were attributed to ZnO stretching mode[16].

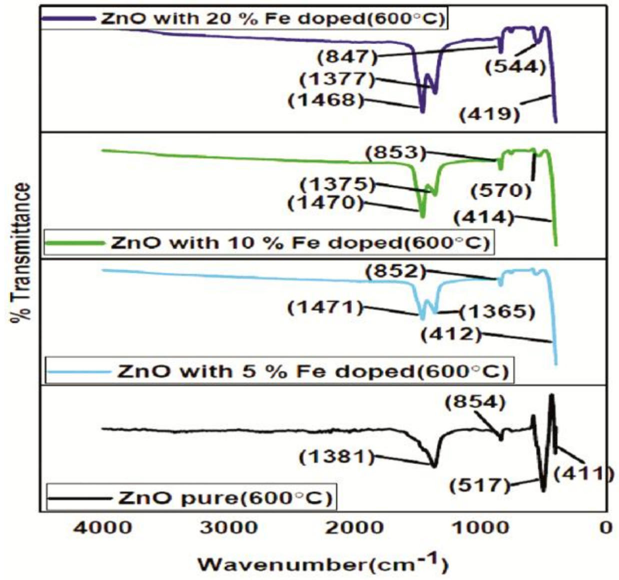

Fig. 2 — IR spectra of ZnO (pure) and Fe doped ZnO (5 %, 10% & 20%) calcined for 2hrs at 600 ºC

The peaks at position 847, 852, 853 and 854 cm^{-1} indicate the Fe^{3+} ion incorporated successfully into the ZnO matrix. ZnO doped with high concentration of Fe also exhibited comparable spectra with faint peaks.

3.3 FESEM Image

The scanning of samples through electron (e^-) microscopy was done by field effect scanning electron microscope JEOL JSM-7610FPLUSEDS at LPU, PHAGWARA, PUNJAB with magnification 100 nm and 1µm.The as received FESEM images of FeO/ZnO NPs calcined for two hours at 600 ºC at various magnifications were shown in Figs. 3(a & b). The FESEM images showed that each sample examined some irregular and porous shaped polycrystalline nanoparticles due to the clustering of initial nuclei.

Images showed that the nanoparticles were not all uniformly spherical in shape. The images exhibited agglomerated nature even at 1µm.

3.4 HRTEM images

The morphology and microstructure of calcined samples were examined by using HRTEM JEOL, JEM2100 in SAIF, PU, CHANDIGARH.

The as received images from SAIF were displayed in Fig. 4. The results showed that agglomerated and randomly distributed truncated-shaped nanoparticles

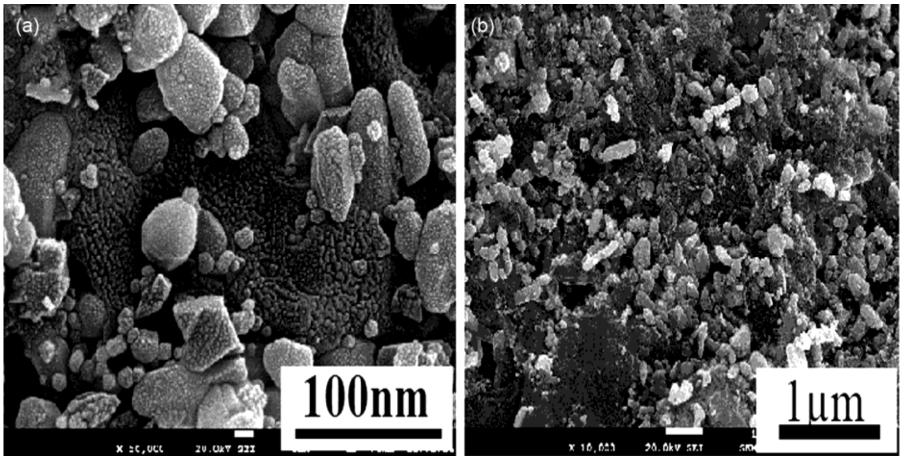

Fig. 3 — FESEM images of Fe doped ZnO nanoparticles calcined for 2hrs at 600 ºC for 2hrs

Table 1 — Crystalline size of nano-particulates of XRD samples of Fe doped ZnO

Samples	Temp^r	Peak position(2Θ)	d-spacing	Intensity	FWHM	Crystalline size(nm)
ZnO (pure)	600 °C	36.26	2.475	761	0.205	40.32
ZnO/FeO (5%)	600 °C	36.37	2.468	663	0.307	26
ZnO/FeO (10%)	600 °C	36.31	2.472	742	0.184	44
ZnO/FeO (20%)	600 °C	36.12	2.484	769	0.164	50

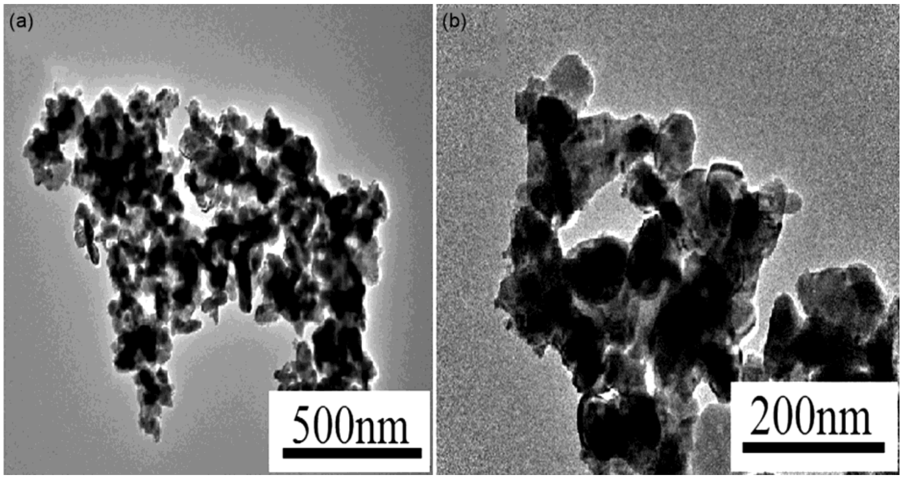

Fig. 4 — HRTEM images of Fe doped ZnO nanoparticles calcined for 2hrs at 600 ºC

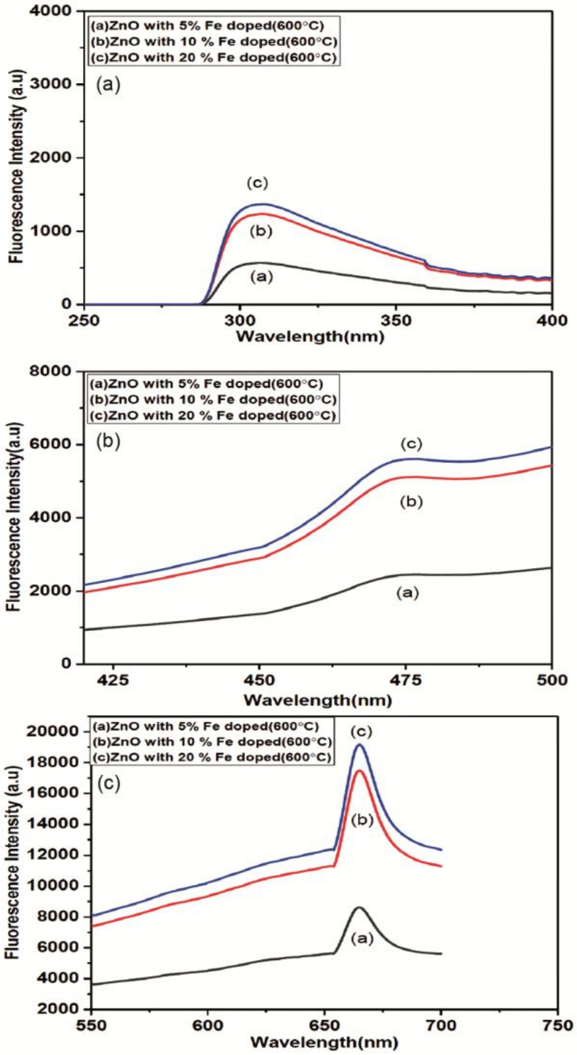

Fig. 5 — PL spectrum of FeO/ ZnO NSM (a) λ= 250-400 Å (b) λ =400-500 Å (c) λ =550-700 Å of calcined for 2hrs at temperature 600 °C

were obtained and also images were concreted in nature.

3.5 PL Intensity analyses

The visible region (400–700 nm) of doped ZnO nanoparticle samples was investigated using PerkinElmer Spectrum FL Version 1.0.0. A "double beam 365 UV-Visible IR Spectrometer" that scanned a wide range of UV wavelengths at a rate of one scan per second was used to analyze the calcined samples. In Fig. 5, the data obtained from the laboratory were plotted.The photoluminescence (PL) spectra of all the prepared FeO/ZnO nano particulates at ambient temperature were recorded by using 303 nm as the excitation wavelength. The UV light generated by free excitons recombining through an exciton-exciton collision process[18]. The doped ZnO layers' PL spectra showed a strong emission peak at 665 nm which is located in the red regions.

As seen in Fig. 5, intensity of 20% Fe doped ZnO is maximum. The results reflect that our transition occurred within visible light wavelength (1.8 to 3.1eV) and novel materials are photosensitive properties and may be used for fabrication of solar cells.

4 Conclusion

The calcined samples of pure ZnO and Fe (5%, 10% & 20%) doped were synthesized successfully by microwave irradiated chemical co-precipitation techniques. The XRD results of study concluded that ZnO parental hexagonal structure was in formation in all calcined samples. The crystallite size of nanoparticles continuously increases with increases of dopant concentration. However, small decrement was noticed with pure ZnO and 5% Fe doped ZnO nano particulates. The IR peaks at wavenumber 411, 544 and 570 cm^{-1} were corresponding to O-Zn-O vibration and sharp peaks occurred at position 847, 852 and 854 cm^{-1} were attributed by O-Fe-O vibration of ZnO and Fe_2O_3 molecules respectively. The IR results conclusively support the XRD results of study. The electron microscopy image shows that crystal stone-like structures were obtained with multiple grain sizes at nanoscale. The PL study of samples indicated that newer materials are highly photosensitive properties in visible and ultraviolet regions. Hence forced, newer materials have approachable applications in fabrication of solar cells and electrodes of photovoltaic cells.

Acknowledgement

The Authors gratefully acknowledged to Principal and Technical Staffs of M.N.S. Govt. College Bhiwani provides laboratory facilities for manufacturing mechanisms and also authors extend their sincere gratitude to Technical Staff SAIF PU, Chandigarh and CIL LPU, PHAGWARA(PUNJAB) for characterization of samples.

References

1 Al-Dhabi N A & Arasu MV, *Nanomaterials,* 8 (2018) 1.

2 Dai J, Suo Z, Li Z & Gao S, *Res Phys,* 1 5(2019).

3 Anjum, Nazli H, Bashir F & Mahmood K, *Mater Today Proc,* 2 (2015) 5552.

4 Agarwal S, Jangir L K, Rathore K S, Kumar M & Awasthi K, *Appl Phys,* 125 (2019).

5 Singh P, Kumar R & Singh R K, *Indus Eng Chem Res,* 58 (2019) 17130.

6 Vinitha V, Preeyanghaa M & Vinesh V, *Emergent Mater,* 4 (2021) 1093.

7 Carofiglio M, Barui S, Cauda V & Laurenti M, *Appl Sci,* 10 (2020).

8 Kumari V, Mittal A, Jindal J, Yadav S & Kumar N, *Front Mater Sci,*13 (2019).

9 Kandapal N D, Sah N, Loshani R, Joshi R & Prasad J, *J Sci Indus Res,* 73 (2014) 87.

10 Nguyen T B, Huang C P & Doong R, *Science of the Total Environment*, 646 (2019) 745.

11 Kumari T, Dahiya S & Sharma R, *Int J All Res Educat Sci Meth (IJARESM),* 11 (2023) 1573.

12 Kumari T, Dahiya S & Sharma R, *Int J All Res Educat Sci Meth (IJARESM),* 11 (2023) 465.

13 Kumar R, Sharma A & Kishore N, *IJPCMF Int J Phys Chem Math Fundament,* 3 (2013) 1.

14 Yadav A, Sharma R & Choudhary N, *J Renewable Rnergy Exchange,* 9 (2021) 340.

15 Castro-Lopes S & Guerra Y, *Solid State Sci,* 109 (2020) 106438.

16 Raja K, Ramesh P S & Geetha D, *Spectrochem. Acta A – Mol. Biomol Spectrosc*, 131 (2014) 183.

17 Ba-Abbad M M , Kadhum A A H , Mohamad A B, Takriff M S & Sopian K, *J Alloy Compd*, 550 (2013) 63.

18 Srinivasulu T, Saritha K & Reddy K T R, *Mod Electron Mater*, 3 (2017) 76.