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STRUCTURAL CHARACTERIZATION AND OPTICAL PROPERTIES OF Fe & Ni-DOPED ZINC OXIDE NANOPORED PARTICLES, SYNTHESIZED USING MICROWAVE METHOD

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ABSTRACT

Nano-sized Fe and Ni doped ZnO powders were prepared by a simple, time conserving microwave method which yield doped Zinc Oxide nanoparticles of uniform size, shape and morphology. A mixture of Zinc nitrate, citric acid with Ferric nitrate and nickel nitrate was used to synthesize the Fe and Ni doped ZnO nanoparticles. X-ray diffraction (XRD), scanning electron microscopy/energy-dispersive analysis (SEM/EDS), and transmission electron microscopy (TEM) techniques were used to characterize the nanometer size pored materials. Compared with the photoluminescence (PL) spectrum of the pure ZnO nanoparticles, the peak position of emission of these doped ZnO nanoparticles exhibited blue shift. Moreover, energy band gap evaluation of these particles has been done by using UV–Visible absorption spectroscopy.

Keywords: Doped -Zinc-Oxide, SEM, TEM, UV-Vis, PL.

I. INTRODUCTION

Microwave irradiation has shown very rapid growth in its application to material science due to its unique reaction effects such as rapid volumetric heating and the consequent dramatic increase in reaction rates, etc. Compared with the conventional methods, the microwave synthesis has the advantages of short reaction time, small particle size, narrow particle size distribution and high purity. [1]. Zinc oxide has a stable wurtzite structure. Based on these remarkable physical properties and the motivation of device miniaturization, large effort has been focused on the synthesis, characterization and device applications of Zinc Oxide nanomaterials[2].Doping was intentionally introduced to add impurities, in this case: iron and nickel. The nitrates act as an oxidation agent and the citric acid act as a reducing agent and change the gel to a fine and intensively porous substance [3]. This method is a modified sol-gel method which uses a microwave. Fe and Ni in 10 % molecular weight ratio were taken. A comparison between the optical properties of pure, Fe-doped, and Ni-doped ZnO was done to illustrate the effect of doping.

II. EXPERIMENTAL

For the synthesis of iron doped zinc oxide, zinc nitrate and ferric nitrate with the citric acid was taken. A microwave oven with 650 W power was used. One molar Zinc nitrate was dissolved in the 25ml distill water

and 10% one molarferric nitrate was dissolved in other 25ml distill water and citric acid was added in the mixture of two. Then this mixture was steered for few minutes. The whole mixture was put in to the microwave oven for the microwave synthesis. Mixture was heated non continuouslyin cycles until reaction get complete with gaps of 10 seconds each. As the result of the reaction brownish color iron doped zinc oxide was formed in the ash form. The product was pulverize to powder using Mozart crystal and further washed with distill water for several times , then it is filtered and dried to get the powder form of the synthesized material.

The synthesis of nickel doped zinc oxide was done by the same process, except the starting material was now zinc nitrate and nickel nitrate. The product was similarly pulverized, washed, and then dried to get the final material. As the result, brownish yellow color nickel doped zinc oxide was formed.

These materials were characterized by XRD, SEM/EDS ,TEM, PL , and UV-Vis.

III. RESULT AND DISCUSSION

3.1 XRD Analysis

The as-prepared ZnO, Fe doped ZnO and Ni doped ZnO were characterized by X-ray powder diffraction. XRD pattern of the iron doped zinc oxide and nickel doped zinc oxide are compared with the pure zinc oxide, prepared by this method (shown in fig. 1). In this analysis, peaks were found to be sharp implying the crystalline nature of the sample. After doping the crystallity of material is still maintained.

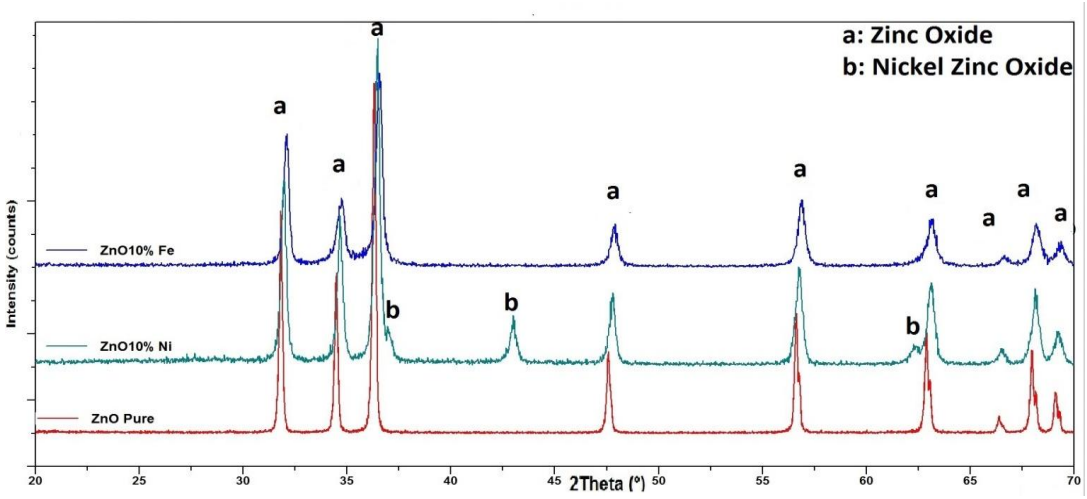

Fig. 1 XRD Graph of Pure, 10%-Iron Doped, and 10%-Nickel Doped Zinc Oxide

The profile of Fe-doped ZnO shows degradation of peaks as observed, implying the introduction of impurity to the host ZnO lattice. No additional phase is observed in this case. The crystal is in same phase of ZnO as compared to the JCPDS file. Whereas, the profile of Ni-doped ZnO exhibits both degradation (shifting of peaks) and formation of new phase. Additional peaks can be seen at 2thata = 36, 43, and 62, which is diffraction from the nickel crystal system.

3.2 SEM/EDS

The scanning electron microscopy (SEM) was used to investigate the morphology of the doped zinc oxide powders. SEM images of iron doped zinc oxide and nickel doped zinc oxide was done on SEM (ZEISS, model EVO-18) and are shown in the fig. 2 and 3.

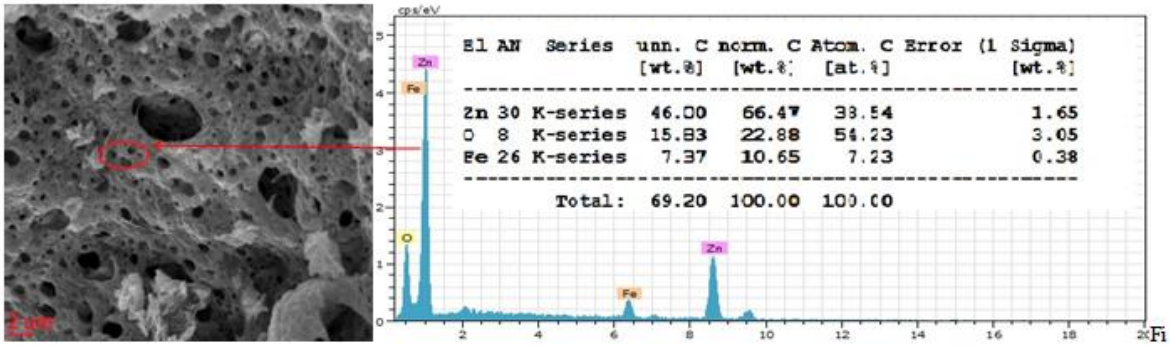

Fig. 2 SEM image of Fe-doped ZnO with EDS.

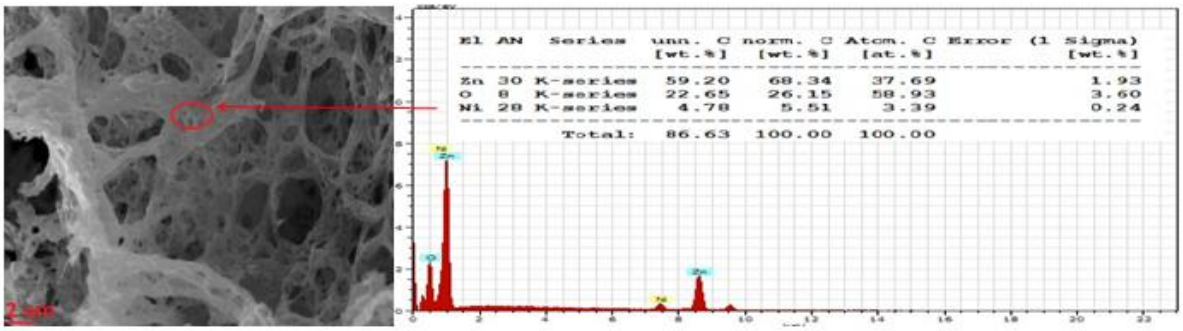

Fig. 3 SEM image of Ni-doped ZnO with EDS.

It can be seen from the SEM images, that both materials have porous morphology. The EDS profile of the materials are given beside the SEM images. The energy dispersive X-Ray justifies the presence of dopants in the host compound. Quantitative analysis of the sample is also shown in the figure, from which it can be concluded that no extra impurities are found.

3.3 TEM

Transmission electron microscopy was used to analyse the morphology and size of the particles. The TEM image (fig. 4 and 5) reveals that the product consists of hexagonal shaped particles with a regular morphology and narrow size distribution. The average size of the iron doped zinc oxide particles was found to be nearly 75 nm. Whereas the average size of nickel doped zinc oxide was found to be 80 nm.

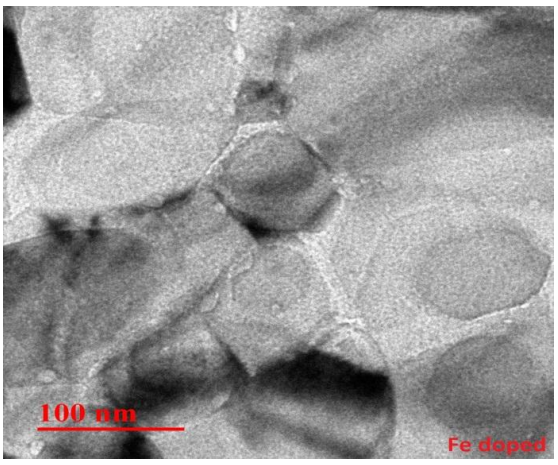

Fig.4 TEM image of 10% Fe doped ZnO

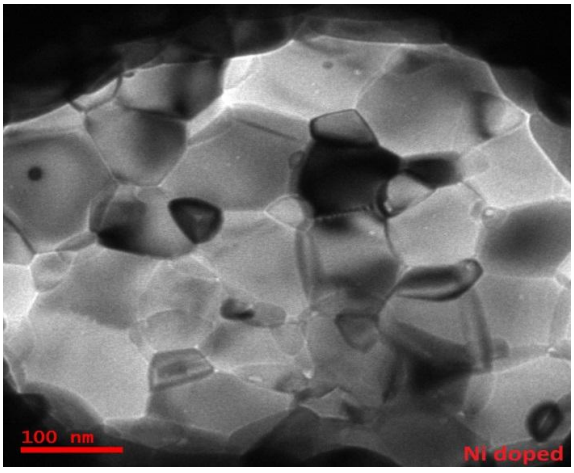

Fig.5 TEM image of 10% Ni doped ZnO

3.4 UV-Vis

Fig. 6 shows the UV–Vis absorption spectra of ZnO nanoparticles as a function of wavelength in the wavelength range from 200 to 460nm. By comparing the absorption position of the spectra, a blue-shift can be seen along with variation of dopants. This blue shift can be inferred to particle size confinement when doping is introduced.

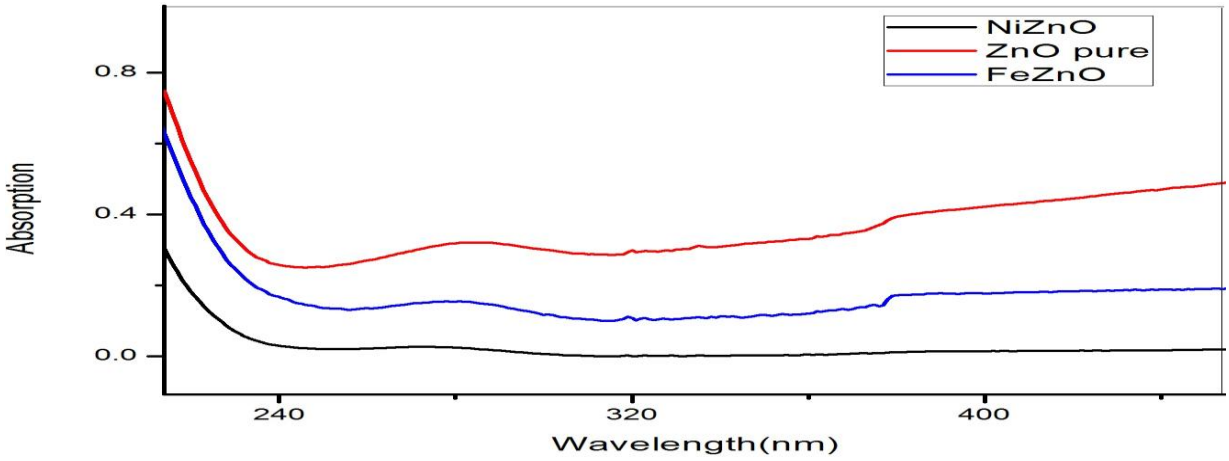

Fig. 6 Shows the UV–Vis Absorption Spectra of Pure and Doped ZnO.

The direct band-gap energy of the prepared nanoparticles is estimated by extrapolating the straight line plot of $(\alpha h\nu)^2$ vs. the photon energy ($h\nu$) (fig. 7). As the doping is done the energy band gap is increased. This increased energy band gap is the result of the decreased size of the nanoparticles. The band gap of ZnO prepared by the microwave method is found to be 5.1 eV, while the doped ZnO particles has larger band gap as Fe doped- 5.3 eV and Ni doped – 5.6 eV.

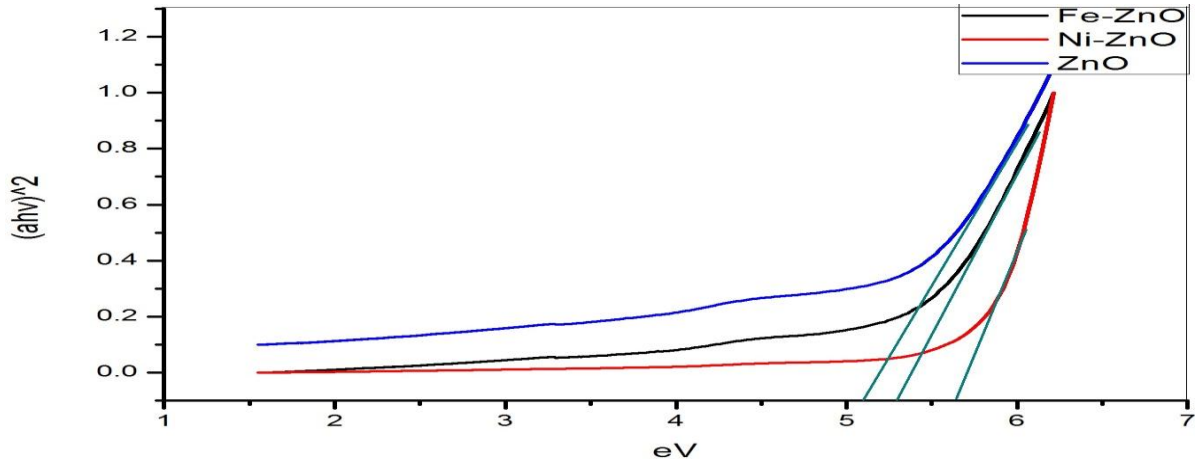

Fig. 7 TheTauc's plot

3.5 PL Emission

The photoluminescence (PL) measurements were performed using the Shimadzu 1501 RF .fig. 8 shows the PL emission of the pure, Fe-doped, and Ni-doped ZnO. As observed ,on doping the ZnO nanoparticle's emission wavelength has been decreased. This shifting is resulted due to the increased band gap of the nanoparticles. The

intensity of doped ZnO peaks are observed to be stronger than the pure zinc oxide. The blue shift is due to the size confinement of the particles.

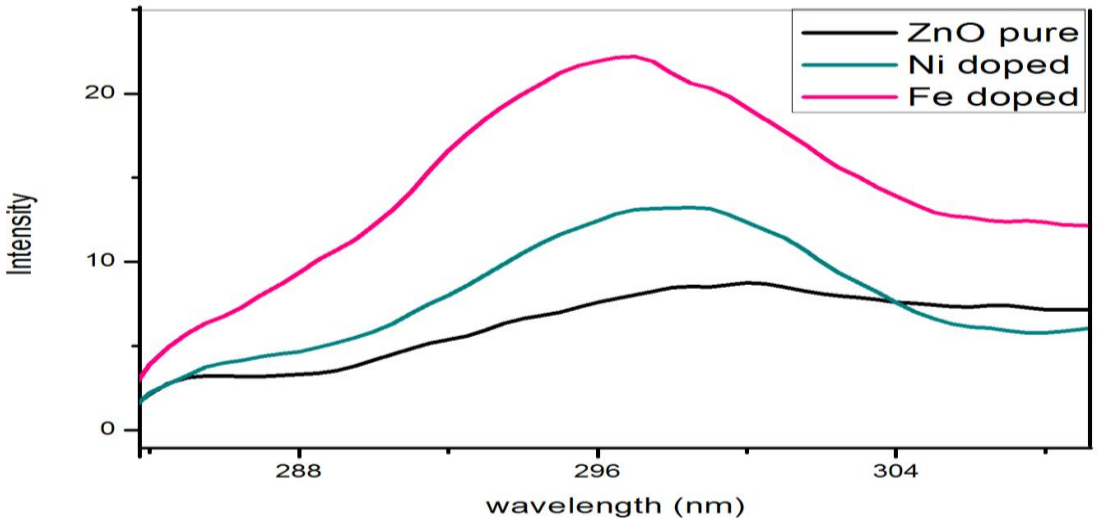

Fig. 8 The PL Emission of Pure and Doped ZnO.

IV. CONCLUSION

Iron doped and Nickel doped ZnO nanoparticles were synthesized using microwave method; a modified sol-gel method which uses the microwave irradiation. The structure and phase of the samples were determined using X-ray diffraction method.XRD and EDS graphs justifies the doping. XRD of Fe doped ZnO exhibits the shifting of peaks, while XRD of Ni doped zinc oxide particles exhibits degradation of peaks and formation of new phase. The surface morphology of prepared nano powders was observed from SEM and TEM studies, found to be porous and hexagonal shape. The band gap increases as the crystallite sizes reduces pure- 5.1eV, Fe-doped 5.32eV, and Ni-doped 5.64eV.The PL emission shows a blue-shift with the doping in the zinc oxide. The blue-shift observed in the UV–Vis spectrum is a typical signature of size confinement in doped ZnO nanoparticles. Material with a such tremendous band gap can be used as a window in optical devices.

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