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OPTICAL PROPERTIES OF CHROMIUM & COBALT DOPED ZINC OXIDE POWDER PREPARED BY SOL - GEL COMBUSTION METHOD, WITH THE ASSISTANCE OF MICROWAVE RADIATIONS

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ABSTRACT

Zinc Oxide doped with chromium and cobalt, in form of powder with nanometer size has been prepared by typical sol- gel route, with assistance of microwave radiations. The as prepared material's structure was characterized under X- Rays Diffraction(XRD) and Scherrer formula was used to find the crystal size. The materials also have been characterized under Scanning Electron Microscope(SEM) and Transmission Electron Microscope(TEM) for morphological information, where TEM verifies the size of particles found out by XRD. The optical properties of prepared doped zinc oxide powder were found by using the Ultra Violet-Visible(U.V-Vis) and the Photoluminescence(PL) spectrometers.

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

I. INTRODUCTION

The gel combustion method is a method based on combustion of sol to gel which is normally done by taking the raw materials like nitrates and a fuel compound. It is commonly based on combustion of oxidising agent where a fuel as reducing agent is used. It is an exothermic reaction which requires the heating of sample at initial stage[1]. This heat is provided by using microwave radiations in our experiment. Zinc Oxide (ZnO) based on its stable wurtzite structure has been used for device miniaturization, which is assuaged with ZnO's different nanomaterials [2]. Doping is intentionally introduced to modify the properties of material. In this paper ZnO was doped with the chromium (Cr) and cobalt (Co) in the sole purpose of manipulating the properties of ZnO. Microwave radiation which has a Monique effect of frequent volumetric heating[3]. The purpose of microwave radiation was fulfilled by using a microwave oven here. Homogenous, high purity and quality nanometre range powder is the product of a sol – gel combustion reaction. It can be due to the possibility of stoichiometric control offered by this reaction method [4].

The optical properties of iron and nickel doped ZnO prepared by same method has been checked earlier[5]. In this paper the optical properties of Cr and Co – doped ZnO, using the Uv-Vis and PL spectrometer are found.

The motive materials were prepared by using the nitrates of zinc, chromium, and oxide with citric acid as a fuel agent.

II.EXPERIMENTAL

The synthesis of pure zinc oxide was done by the simple sol- gel combustion method, with nitrates of zinc and citric acid as mentioned before. A 0.2 mol solution of zinc nitrate is prepared by dissolving the accurate amount in 50ml of distilled water. This solution was added with 0.2 mol of citric acid and then allowed to heat in a microwave oven. The microwave with a power of 650W was used during the whole experiment. The doping ofCr and Co was done by using the respective nitrates of doped materials in the samples. The doping was done by molar percentage method.

The doping percentage was 5% for the both doped zinc oxide's material. Heating in this method is done by giving time gaps of 10 seconds basically. The doped material after getting the reaction complete was washed with water for several times to remove the deposit gases during the closed environment heating treatment. The as prepared material was then dried and then let to the pulverization using the Mozart Crystal.

Sample of Co & Cr doped ZNO were characterized by XRD, SEM, and TEM whereas optical properties were investigated by UV-Vis and PL in present work.

III. RESULTS AND DISCUSSION

3.1 X- Rays Diffraction

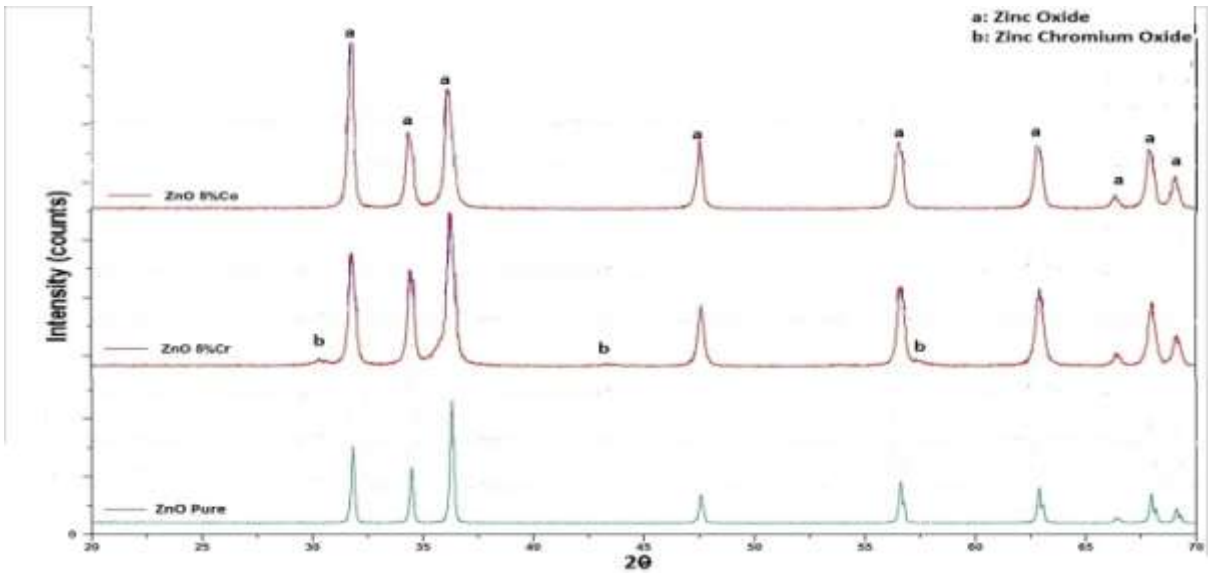

Fig. 1 XRD Graph of Pure, 5%-Cr Doped, and 5%-Co Doped ZnO.

The X- Rays diffraction plot is shown in figure 1. From the plot, it can be clearly seen that the additional peaks are only present in the Cr doped-ZnO. The Co doped-ZnO exhibits only negligible shifting of the peaks. The pure ZnO material plot is taken as a reference to observe any difference due to the doping.

The crystallinity of the prepared samples can be seen from the sharpness of the peak. After doping the crystalline nature of the material remained same. The extra peaks in the Cr doped-ZnO can be seen at 2Θ equals to 31.3, 43.1 and 57.45. The presence of these additional peaks can be explained by the formation of a new

phase called chromium zinc oxide due to the doping effect. Although, the Co doped-ZnO haven't gave any hint of new phase but, the peaks are shifted to right side as compared to the pure ZnO prepared by the same method. This shifting of peaks can be understood by the presence of doped Co in the same phase of ZnO. The figure 1 shows the different phases of the ZnO material as a: depicts the ZnO phase which is present in both the Cr and Co-doped ZnO whereas the b: phase depicts the Zinc Chromium Oxide which is present only in the Cr-doped ZnO material only. The Scherrer formula is used to find the crystal size of the materials [6]. The average size of the crystallite (L) was calculated from the full-width at halfmaximum(FWHM) Scherrer formula [7] (ignoringthe effect of strain):

$$B = k\lambda / L \cos\theta \qquad (1)$$

whereθ is the Bragg angle, λ is the radiation wavelength and k is a constant whichdepends on the peak shapes, crystallite habit and particle shape. The table 1shows the average particle size of the ZnO materials doped in this paper. It can be easily observed that the ZnO prepared by the sol-gel combustion method has crystallite size in nanometres while the size of doped ZnO materials reduced due to the doping effect.

Table 1.The average crystallite size of pure and doped ZnO sample calculated from XRD.

Material	(%) Average crystallite size (nm)
0.0 (undoped) ZnO	40
5% Cr doped ZnO	32
5% Co doped ZnO	38

3.2 SEM

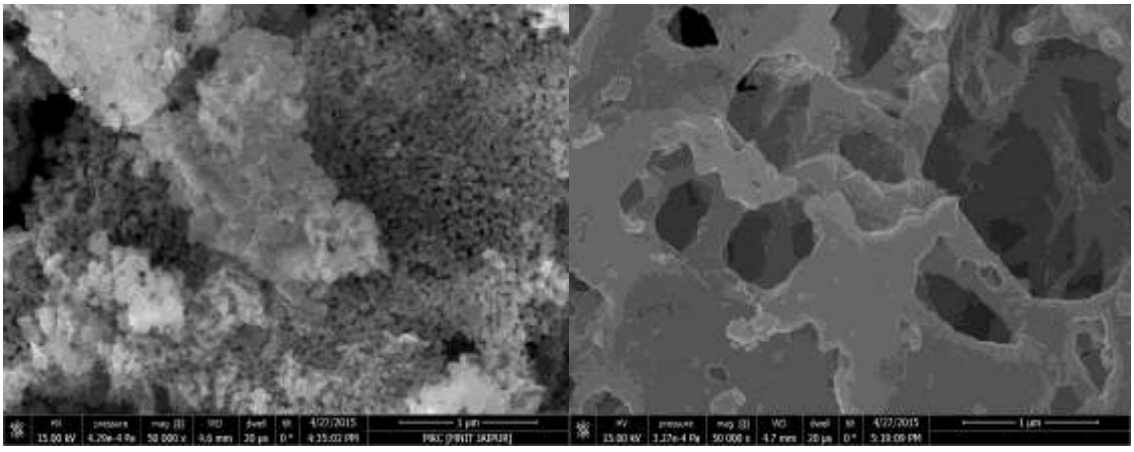

Fig. 2 SEM Image of Cr-Doped ZnO. **Fig. 3 SEM Image of Co-Doped ZnO.**

The scanning electron microscopy (SEM) was used to investigate the morphology of the doped ZnO nanomaterials. SEM images of Cr doped-ZnO and Codoped-ZnO were taken by SEM (ZEISS, model EVO-18) and are shown in the figure 2 and 3, respectively. From the images it can be considered the particles are very small in size. The Cr doped-ZnO material's particles are found to be smaller in size and well separated from each other(figure 2) as compared to the Co doped-ZnO, which also verifies the difference in size from Scherrer formula's result. The particle's form of the Co doped-ZnO is small, but is found to be agglomerated in nature(figure 3).

3.3 TEM

Transmission electron microscope(TEM) was used to analyse the morphology and size of the particles. The TEM images (figure 4 and 5) reveal that the product consists of hexagonal shaped particles with a regular morphology and narrow size distribution.

The average size of the Cr doped-ZnO particles was found to be nearly 31 nm. Whereas, the average size distribution of Co doped-ZnO particles was found to be 38 nm. The size determined by the TEM justified the size determination from the XRD method, as both showed the similar results. This justification from the TEM proves the formation of as prepared ZnO materials in the nanometres range.

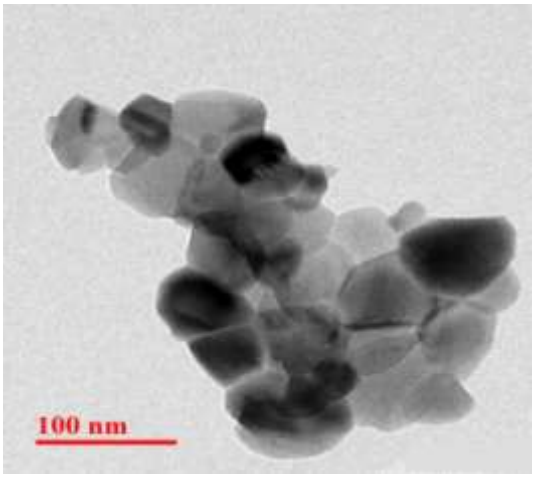

Fig.4 TEM image of 10% Cr doped ZnO

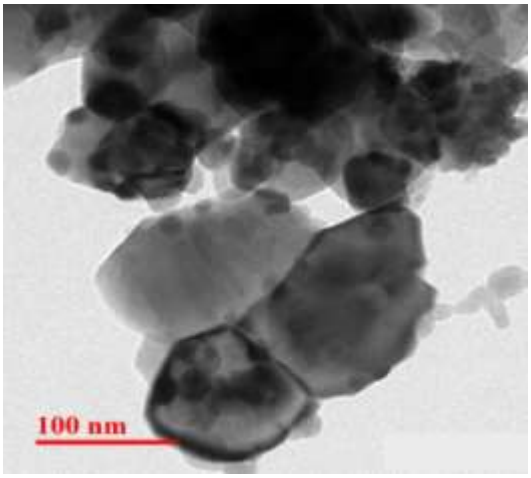

Fig.5 TEM image of 10% Co doped ZnO

3.4 UV-Vis

The optical properties of as prepared ZnO materials, when measured by the UV-Vis, gave the following plot (figure 6). In it, the ZnO's materials are shown as the function of wavelength ranging from 150 to 480 nm. On the comparison of the absorption peaks, a blue shift can be fathomed in them as the peaks moved to lower wavelengths. This blue shift aroused due to the result of doping in pure ZnO with the Cr and Co. The confinements of particle size can be considered on the act of doping with Cr and Co as depicted from this blue shift.

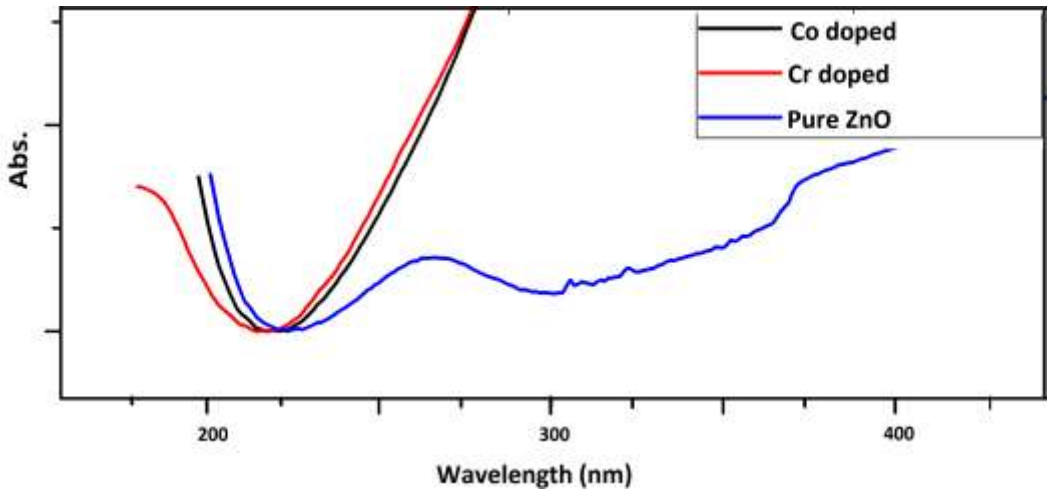

Fig. 6 Shows the UV–Vis Absorption Spectra of Pure and Doped ZnO.

3.5 PL Emission

Shimadzu 1501 RF was used to perform the photoluminescence (PL) as depicted and the results are shown in the figure 7. The figure shows the PL emission of the pure, Cr doped- ZnO and Co-doped ZnO. The intensity of doped ZnO materials as observed to be stronger than the pure ZnO. On the doping, the emission wavelength of particles has been decreased. The blue shift of the peaks can be easily observed. This blue shift also justified the shift observed under the UV-Vis results. On doping with a material having wider band gap, the band gap ZnO can be manoeuvred for different applications like LEDs. The doping of ZnO changes its electronic, optical and as well as structural properties[5,8,9,10].

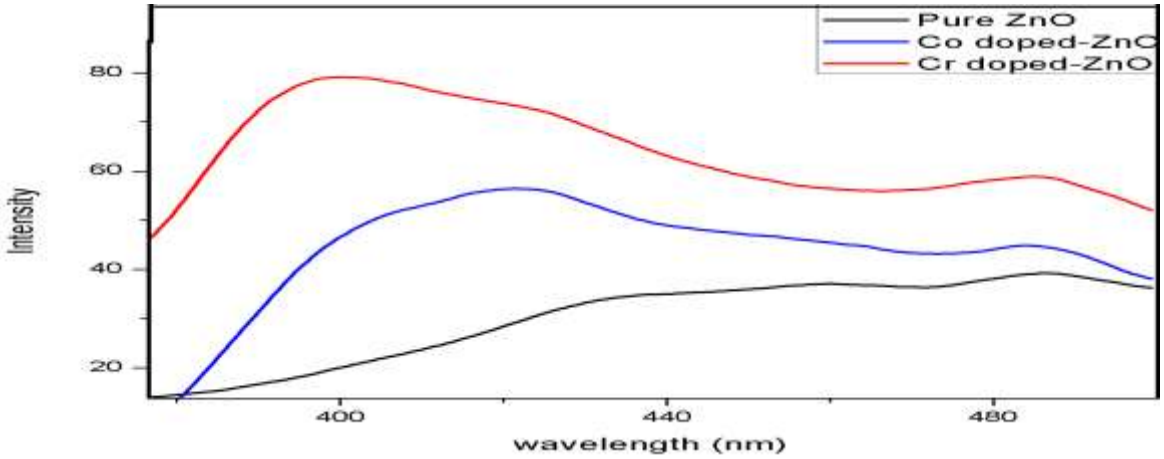

Fig. 7The PL Emission of Pure and Doped ZnO

IV. Conclusion

The Cr and Co doped-ZnO were prepared by the sol-gel combustion method with the assistance of the microwave radiations.The structure and phase of the as prepared materials were determined by using the XRD. The XRD patterns showed the shifting of peaks to high angles in case of doped ZnO as compared to the pure ZnO. The formation of new phase was seen in Cr doped-ZnO. On the other hand,in case of Co doped-ZnO the pure phase implies that the Co atoms effectively occupied the ZnO lattice. The average size of particles was measured by the Scherrer formula for the both doped and pure ZnO, and was found to be innanometres range. The average size of the prepared material crystals was later verified by the TEM analysis as it was similar to the XRD results. The morphology and structural analysis was done by the SEM. The particles were found to be porous in morphology. With justifying the particle size, TEM also gave the shape information about the ZnO particles as to be hexagonal. The PL emission showed a blue-shift with the doping in the zinc oxide. The blue-shift was also observed in the UV–Vis spectrum which is a typical signature of size confinement in doped ZnO nanoparticles. Both the Uv-Vis and the PL results verified the shift in doped ZnO particles indicating the change in optical property of doped materials.

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