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# Synthesis and Characterization of Pure and $Cu^{2+}$ Doped Zinc Oxide Nanoparticles Using Sol-Gel Method

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**Abstract:** ***Pure and $Cu^{2+}$ doped ZnO nanoparticles of size less than 50nm are synthesized by simple Sol-gel method. The prepared nanoparticles were characterized by X-ray Powder Diffraction (XRD), Fourier Transform Infrared Spectroscopy (FTIR) and UV-Vis spectroscopy. The structure, crystallite size, lattice parameters and microstrain of the prepared samples were studied using XRD analysis. The XRD spectra indicated hexagonal wurtize structure for both pure and doped samples. Also, the precise value of crystallite size and microstrain of the prepared samples were found using W-H plot. The metal oxide formation and phase purity of the prepared samples were further confirmed using FTIR analysis .The bands corresponding to ZnO formation are found around 495 $cm^{-1}$ and that corresponding to Cu-O stretching vibration are found around 553 $cm^{-1}$. The optical studies of the prepared samples were done using UV-Vis spectroscopy which showed indirect allowed type of transition for all samples. Also, the band gap of the prepared samples were found using Tauc plot as 3.20eV, 3.33eV and 3.31eV for pure, 1 wt. % $Cu^{2+}$ and 3 wt. % $Cu^{2+}$ doped ZnO nanoparticles respectively.***

**Keywords:** ZnO nanoparticles, XRD, FTIR, UV-Vis, sol-gel method

## 1. Introduction

Zinc oxide is the one of the most important n-type semiconductor materials with a 3.37eV band gap at room temperature and 60meV excitation binding energy [S.R.Brintha.et. al, 2015]. Zinc Oxide is due to its variant morphologies like nanorods, nanoflowers, nanowires, nanodendrites and nanoparticles have diameter in the range of tens of hundred of nanometer. This reduction in size improves its physical properties and hence gives different results as compared to the bulk Zinc Oxide [Robina Ashraf.et. al, 2013]. ZnO nanopowders have applications in ultraviolet filtering, catalytic, anti- corrosion and anti-bacterial properties. Other applications of Zinc Oxide nanopowder include electro photography, photo printing, capacitors, protective coatings, antimicrobial and conductive thin films in LCDs, solar cells and blue laser diode [J.N. Hasnidawani.et.al, 2015]. Among various methods available for the preparation of nanopowders, the sol-gel method provides high purity and quality with homogenous distributions of nanopowder [Deepak Davis.et.al, 2016]. In the present work Pure and Cu doped ZnO nanoparticles are prepared using sol gel method.

## 2. Experimental

Pure and Cu doped Zinc Oxide nanoparticles were synthesized by using sol-gel method. In order to prepare a sol, stoichiometric amount of Zinc acetate Dihydrate and Sodium hydroxide were dissolved in 50 ml of distilled water.

The Solution was then mechanically stirred for about 15 minutes. As a result, a white precipitate was formed, which is then dried to form ZnO nanopowder. Also 1Wt%$Cu^{2+}$ and 3Wt% $Cu^{2+}$ doped ZnO nanoparticles was synthesized by using Zinc acetate Dihydrate and Sodium hydroxide as starting precursors. They are dissolved in 50 ml of distilled water and then copper nitrate in 1 and3 Wt% were added as dopants to the solution respectively. Then the solution was mechanically stirred and a white precipitate formed to get dried Cu doped ZnO nanopowder.

## 3. Results and Discussions

### 3.1 XRD Studies

For Phase identification/structural characterization of as synthesized Zinc Oxide material, the most appropriate technique (ie) X-ray diffraction was employed. The XRD pattern of Pure ZnO, ZnO+1Wt%$Cu^{2+}$ and ZnO+3Wt%$Cu^{2+}$ nanoparticles prepared by sol-gel method is shown in the fig:3.1(a).

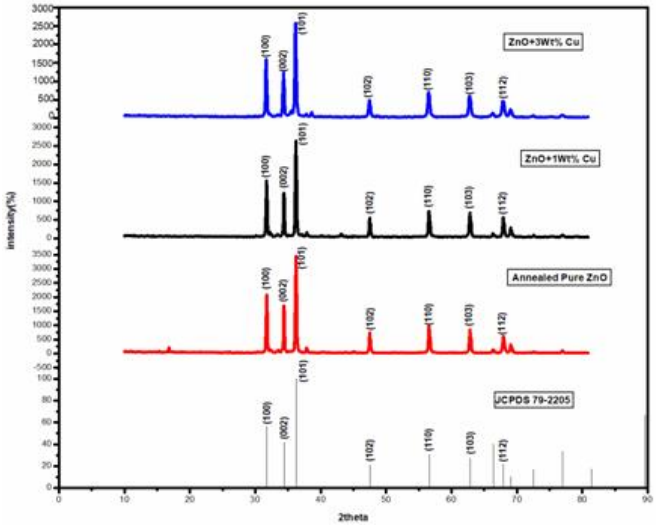


**Figure 3.1 (a)** XRD Pattern of Pure and Cu doped ZnO nanoparticles

The diffraction peaks of pure ZnO were identified to originate from (100), (002), (101), (102), (110), (103) and

(112) planes. The main diffraction peak is observed at 2Θ value of 36.2363˚. The value of (β) observed for Pure ZnO is 0.1968. This peak is identified to originate from (101) plane of the Pure ZnO.

The diffraction peaks of 1Wt% and 3Wt%Cu doped ZnO nanoparticles was identified to originate from (100), (002), (101), (102), (110), (103) and (112) planes. All the peaks are indexed to wurtize structure of ZnO having hexagonal phase, which is in good agreement with standard JCPDS (card no.79-2205). The intensity of the diffraction peaks are increased on 1Wt. % and 3Wt. % Cu doped ZnO nanoparticles showing the degradation of crystallinity. The crystalline size of the nanopowder are estimated using the Scherrer's formula [Mahmoud.W.E.et.al.2013],

$$D = 0.9\lambda/\beta\cos\Theta \quad \text{----------} \quad 1$$

Where, λ is the wavelength
β is the full width half maximum
Θ is the Bragg's angle.

Based on the Scherrer's formula the average crystallite size of Pure ZnO, 1Wt. % and 3Wt. % Cu doped ZnO are observed as 43nm, 41nm and 34nm respectively. The micro strain of the prepared nanoparticles were found by using the formula,

$$E = \beta/4\tan\Theta \quad \text{----------} \quad 2$$

Based on this formula, the average strain of the Pure ZnO, 1Wt. % and 3Wt. % Cu doped ZnO are observed as 0.24514, 0.300174 and 0.228176 respectively. Williamson-Hall plot (fig. 3.1(b)) was constructed to find the precise value of crystallite size and microstrain.

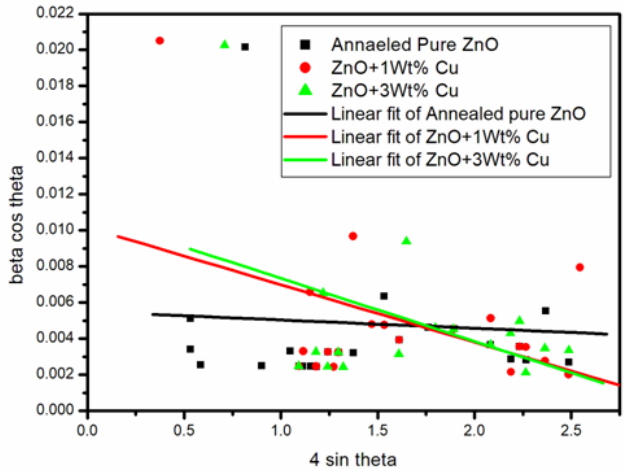


**Figure 3.1: (b)** Williamson–Hall plot for pure and Cu doped ZnO nanoparticles

From the plot, the crystallite size and microstrain of the Pure ZnO, 1Wt. % and 3 Wt. % Cu doped ZnO are observed as shown in the table 3.1 respectively

**Table 1:** Structural parameters of prepared sample

| S.NO | Sample details | Lattice parameter | | Crystalline size | | Microstrain | |
|---|---|---|---|---|---|---|---|
| | | a | c | XRD | W-H Plot | XRD | W-H Plot |
| 1. | Pure ZnO | 3.25142 | 5.20903 | 43nm | 25nm | 0.24514 | 4.63599E-4 |
| 2. | ZnO+ 1Wt% Cu | 3.25196 | 5.21014 | 41nm | 14nm | 0.300174 | 0.00319 |
| 3. | ZnO+ 3Wt% Cu | 3.22999 | 5.22999 | 34nm | 13nm | 0.228176 | 0.00349 |

### 3.2 FTIR Studies

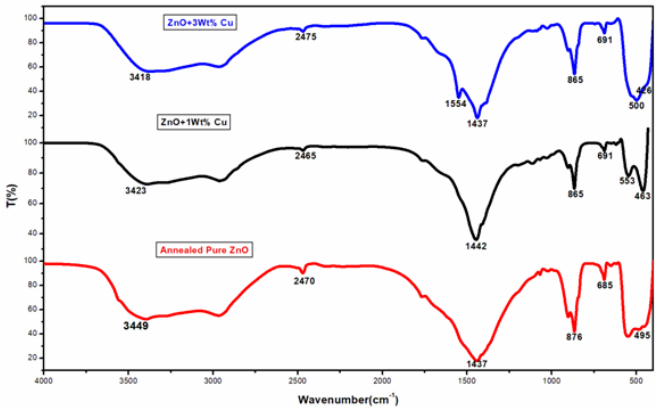


**Figure 3.2:** FTIR spectra of Pure Zn0 and Cu doped ZnO nanoparticles

Fig. (3.2 ) shows the FTIR spectrum of the pure, 1wt.% $Cu^{2+}$ doped and 3wt.% $Cu^{2+}$ doped ZnO nanoparticles in the range of 400-4000 $cm^{-1}$. Strong absorbance below 800 $cm^{-1}$ is the characteristics of metal oxides. Here, in all the three samples, bands at around 495 $cm^{-1}$ are observed that corresponds to the ZnO stretching vibration. Also, the absorption bands at around 606 $cm^{-1}$ are due to C-H bending and that at 876 $cm^{-1}$ is due to O-H bending. The band at 1437 $cm^{-1}$ and 2470 $cm^{-1}$ correspond to C=OH stretching of carboxylate and C-H stretching respectively. A broad absorption band found at around 3449 $cm^{-1}$ can be assigned to O-H stretching. In the $Cu^{2+}$ doped ZnO samples, the bands around 553 $cm^{-1}$ and 500 $cm^{-1}$ are observed which corresponds to the Cu-O stretching vibration. In those samples the intensities of bands corresponding to ZnO are found to decrease which indicates the impregnation of $Cu^{2+}$ in the doped samples.

### 3.3 UV-Vis Studies

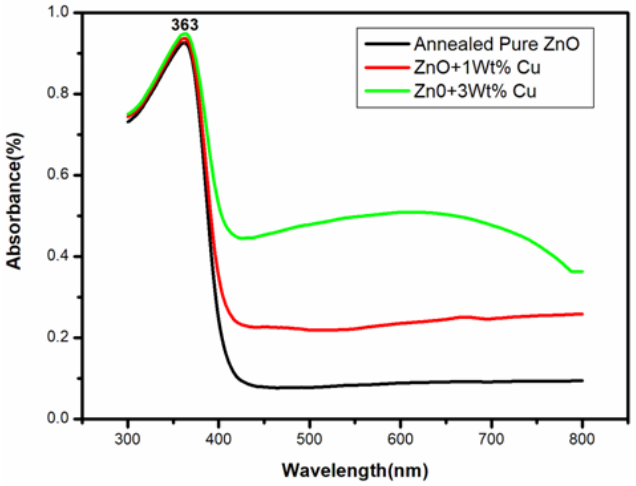


**Figure 3.3(a)** UV- Vis spectrum of Pure ZnO and Cu doped ZnO nanoparticles

The UV-Vis absorption study was carried out in order to find the optical absorbance of the synthesized pure and Cu-doped ZnO samples. Fig. 3.3(a) shows the UV- absorbance spectrum of pure ZnO, 1Wt. % and 3Wt. % Cu doped ZnO nanoparticles respectively. Strong absorption bands at around 363nm are observed which corresponds to the absorption edge of the prepared samples.

The band gap of the pure and Cu doped nanoparticles are found using Tauc's plot. The Tauc's plot is drawn for $(\alpha h\nu)^{1/2}$ along x-axis and hν along y- axis as shown in the fig.3.3(b)

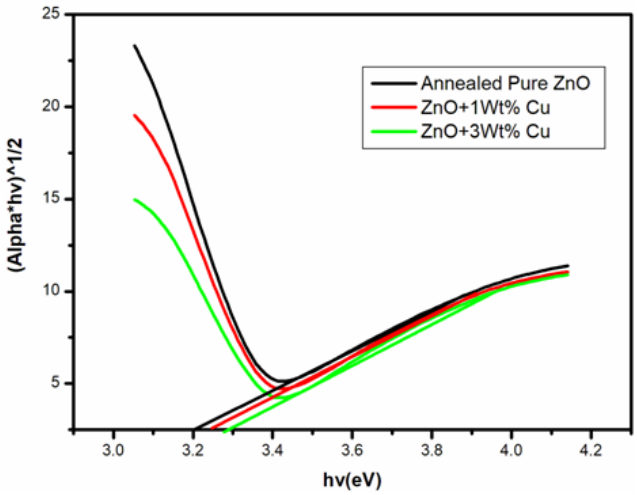


**Figure 3.3(b):** Tauc's plot for Pure ZnO and Cu doped ZnO nanoparticles.

From the figure, we found all the three samples showed the indirect allowed transition. The Band gap energies, (Eg) of pure ZnO, 1Wt. % and 3Wt. % Cu doped ZnO nanoparticles are found to be 3.20 eV, 3.33 eV and 3.31 eV respectively. The band gap is found to increase for doped samples with decreasing particle size.

## 4. Conclusion

In the present study, Pure and $Cu^{2+}$ doped ZnO nanoparticles of size less than 50nm are synthesized by simple Sol-gel method. The prepared nanoparticles were characterized by X-ray Powder Diffraction (XRD), Fourier Transform Infrared Spectroscopy (FTIR) and UV-Vis spectroscopy. The structure, crystallite size, lattice parameters and microstrain of the prepared samples were studied using XRD analysis. The XRD spectra indicated hexagonal wurtize structure for both pure and doped samples. Also, the precise value of crystallite size and microstrain of the prepared samples were found using W-H plot. The metal oxide formation and phase purity of the prepared samples were further confirmed using FTIR analysis .The bands corresponding to ZnO formation are found around 495 $cm^{-1}$ and that corresponding to Cu-O stretching vibration are found around 553 $cm^{-1}$. The optical studies of the prepared samples were done using UV-Vis spectroscopy which showed indirect allowed type of transition for all samples. Also, the band gap of the prepared samples were found using Tauc plot as 3.20eV, 3.33eV and 3.31eV for pure, 1 wt. % $Cu^{2+}$ and 3 wt. % $Cu^{2+}$ doped ZnO nanoparticles respectively.

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