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Research Article

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Synthesis and Characterization of Nanoparticles Using Co-Precipitation Method: A Comparative Study

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ABSTRACT

The ZnO and SnO_2 nanoparticles were synthesized using the co-precipitation method. The samples were calcined at 550°C. The samples were characterized by various advanced techniques such as Fourier Transform Infrared Spectroscopy (FTIR), X-ray diffraction (XRD), UV- Visible Spectroscopy and Particle Size Analyzer. The XRD studies revealed that the average crystallite size of ZnO and SnO_2 nanoparticles were found in the range of 27.8 nm and 24.6 nm. The various functional groups for synthesized nanoparticles were detected by FT-IR spectroscopy analysis. The optical properties of the samples are investigated by measuring the UV-Visible Spectroscopy. The Particle size analyzer studies confirmed that the ZnO and SnO_2 as a nanoparticles. The particle size of the ZnO was 42 nm and the particle size of the SnO_2 was 44 nm. The ZnO nanoparticle is used to make nanorod sensor and SnO_2 nanoparticle is used for catalyst for photodegradation of dyes and illumination with UV light.

Keywords: Nanomaterials; Co-precipitation method; XRD; FTIR; UV and particle size analyzer; Photodegradation of dyes

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INTRODUCTION

Among the metal oxides (ZnO, TiO_2, WO_3, CeO_3 and SnO_2 etc.), ZnO and SnO_2 are an important n-type semiconductors with wide energy bandgap (3.6 eV and 3.37 eV) from experimental calculations [1-3]. Because of its optical transparency in the visible region, it has a wide range of applications in gas sensors, optoelectronic devices, dye base solar cells, secondary lithium batteries and catalysts. Many methods have been developed to prepare ZnO and SnO_2 particles, including the Sol-gel and microwave method [4,5], evaporative decomposition of solution [6], template-assisted growth [7], wet chemical synthesis [8] and gas-phase reaction [9,10]. Among these methods we have adopted chemical co-precipitation method for the synthesis of ZnO and SnO_2 nanoparticles because it is most effective and simple due to its capability in controlling the structural and surface properties of nanoparticles. In this paper, the comparative study of ZnO and SnO_2 nanoparticles has been prepared by Co-precipitation method. XRD, FTIR (SHIMADZU), UV-visible spectra and Particle Size Analyzer (Model SHIMADZU 2300) techniques are used to characterize the structural, Chemical and Optical properties of ZnO and SnO_2 nanoparticles. A detailed discussion about the ZnO and SnO_2 nanoparticles are given.

EXPERIMENTAL SECTION

Pure zinc oxide and tin oxide nanoparticles samples have been synthesised by Co- Precipitation method. The starting materials are zinc acetate dehydrate Zn $(CH_3COO).2H_2O$ (ZnO – A) / Zinc Chloride $ZnCl_2$ (ZnO – B) / Tin chloride $SnCl_2.2H_2O$ and Sodium hydroxide (NaOH). We dissolved 1 M of Zn $(CH_3COO)2H_2O/ZnCl_2/SnCl_2$ in 100 ml H_2O under heating and continuous stirring of 30 minutes. NaOH (0.5 mol) was dissolved in 100 ml of distilled water and

added drop wise to the stirring solution of Zinc acetate dyhydrate and the mixture was stirred for 2 hours. The precipitate was filtered and anneled at 80° C. The dried sample was also calcined at 550° C.

RESULTS AND DISCUSSION

X-ray Diffraction

X-ray diffraction patterns recorded for the synthesized ZnO and SnO_2 nanoparticles are presented in Figures 1-3. Figures 1 and 2 shows 2θ values at 32.9º, 34.5º 36.4º, 47.9º, 63.2º and 77.3º equivalent to (100), (002), (101), (102), (103), and (202) planes in that order. The diffraction pattern of both the samples was compared with JCPDS data (card no: 891397) and indexed, which confirmed that the ZnO nanoparticle are of wurtzite hexagonal type structure [11]. The X-ray diffraction (XRD) of SnO_2 nanoparticles power is shown in Figure 3. The peaks at 2θ values at 26.1º, 33.2º, 37.4º, and 50.2º can be associated with (110), (101), (200) and (211) respectively. Peaks can be ascribed to tetragonal rutile stucture (JCPDS card No. 41-1445) without any secondary phases [12].

The average crystallitesize can be calculated using Debye- Scherrer's formula:

$$D = K\lambda / \beta\cos\theta$$

Where D is the mean crystalline size, K is a grain shape factor (0.9), λ is the wavelength of the incident cu- kα beam. θ is a Bragg reflection and β is the full width at half maximum. The average crystallite size ZnO-A and ZnO-B nano particles were found to be in the range 27.8 nm and 29 nm. The average size SnO_2 nanoparticle was found to be in the range 24.6 nm.

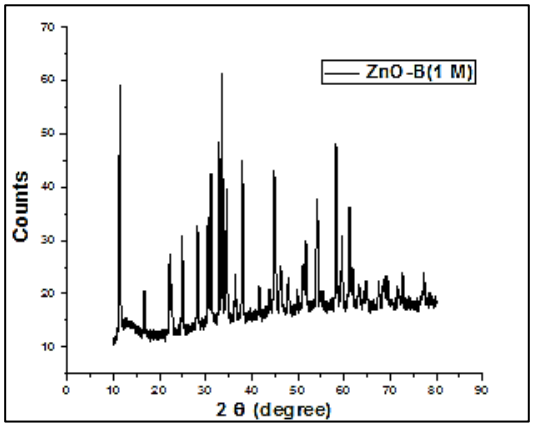

Figure 1: XRD spectrum of ZnO-A

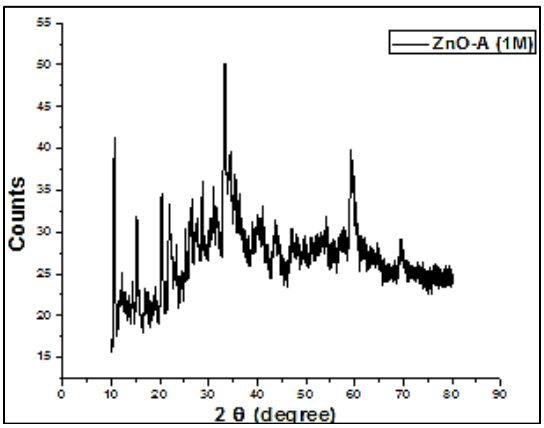

Figure 2: XRD spectrum of ZnO-B

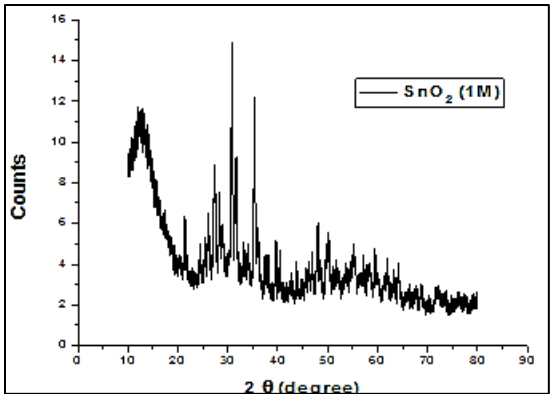

Figure 3: XRD spectrumof SnO_2 from tin chloride as a salt

Fourier Transform Infrared Spectroscopy Analysis

FTIR spectrum is used to calculate various functional groups present in ZnO and SnO_2 nanoparticles and also determined the absorption range. Figure 4 shows that the ZnO nanoparticle using as salt zinc acetate.

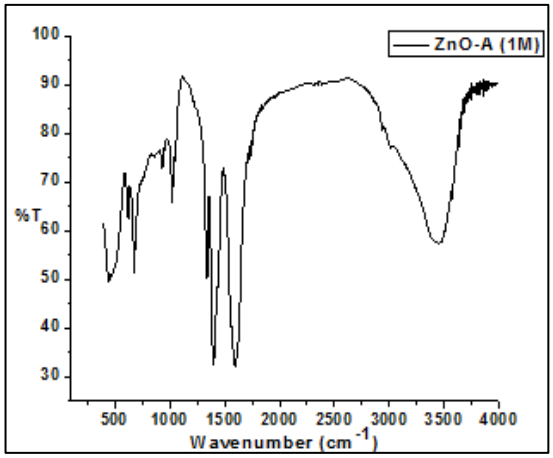

Figure 4: FTIR spectrum of ZnO-A

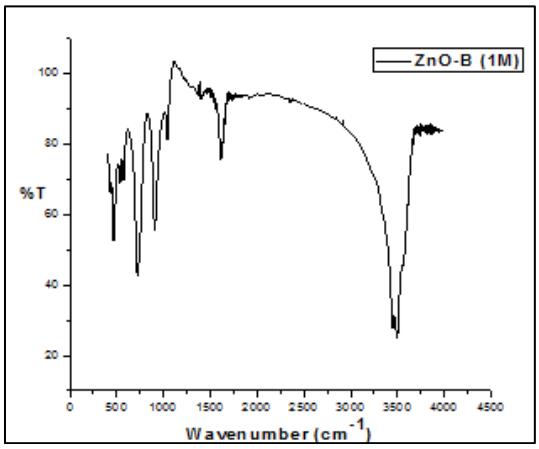

Figure 5: FTIR spectrum of ZnO-B

From Figures 4-6 shows that sharp peaks were observed in 439.74 cm^{-1}, which could be attributed to the ZnO stretching vibration mode. A wide peak was in the range of 3387.73 cm^{-1} - 3461.99 cm^{-1} that was related to the presence of hydroxyl ions (OH) in the ZnO nanoparticles [13]. It shows that sharp peaks were observed in 466.74 cm^{-1}, which could be attributed to the Zn-O stretching vibration mode. A peak was in the range of 3175.58 cm^{-1} -

3494.77 cm^{-1} that was related to the presence of hydroxyl ions (OH) in the ZnO nanoparticles. The absorption peak at 627.79 cm^{-1} arises from the bending vibration of Sn-O-Sn**.**

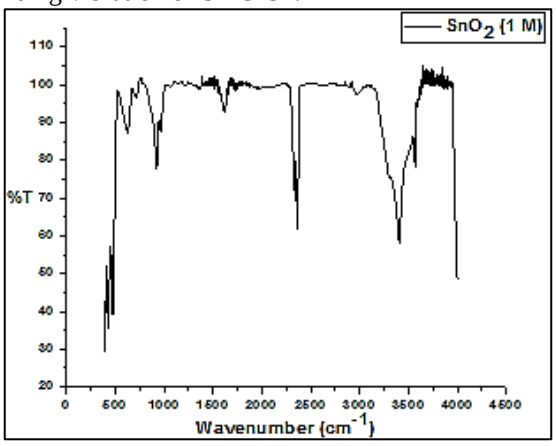

Figure 6: FTIR spectrum of SnO_2 from tin chloride as a salt

UV–Visible Spectra Analysis

The absorption spectrum of ZnO nanoparticles is shown in Figure 7. The figure shows high absorption coefficient in the UV region, whereas it's transparent in the visible region [14,15]. The optical band gap energy (Eg) of the semiconductor is calculated from Tauc relation. A plot of $(\alpha h\nu)^2$ *versus* hυ shows intermediate linear region, the extrapolation of the linear part can be used to calculate the Eg from intersect with hνaxis as shown in Figure 8. The resultant values of Eg for ZnO(Zinc acetate) is found to be about 5.54 eV. The absorption spectrum of ZnO using Zinc chloride as salt nanoparticles is shown in Figure 9. The resultant values of Eg for ZnO using Zinc chloride as a salt is found to be about 6.12 eV. The resultant values of Eg for SnO_2 are found to be about 5.08 eV (Figures 10-14).

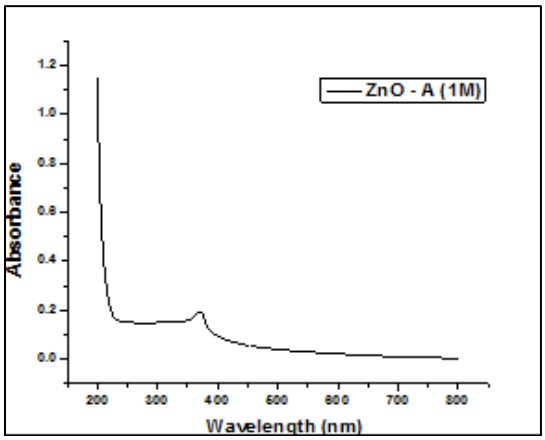

Figure 7: UV spectrum of ZnO-A

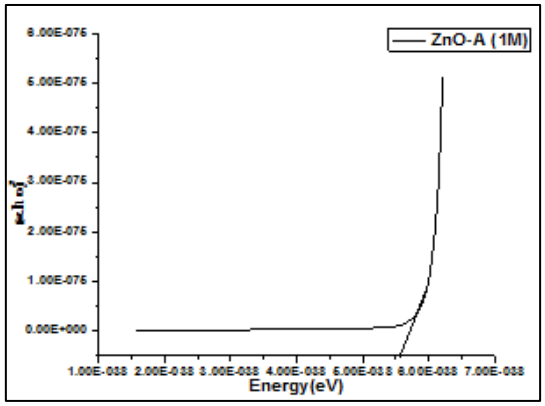

Figure 8: Band gap diagram of ZnO-A

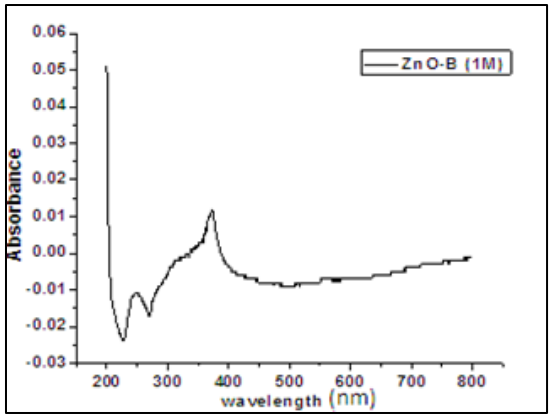

Figure 9: UV spectrum of ZnO-B

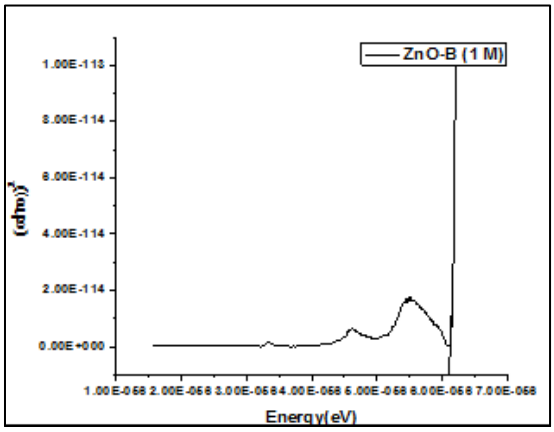

Figure 10: Band gap diagram of ZnO-B

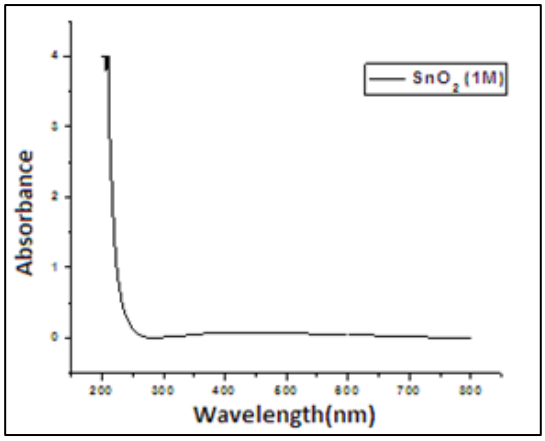

Figure 11: UV spectrum of SnO_2

Particle Size Analyzer

The Particle size of ZnO and SnO_2 has been determined using particle size analyzer (Shimadzu, Model 2300). The particle size of the Zinc oxide (ZnO) nano particle was measured at 42 nm (i.e., 0.042 μm). The particle size of the tin oxide nano particle (SnO_2) was measured at 44 nm (i.e., 0.044 μm).

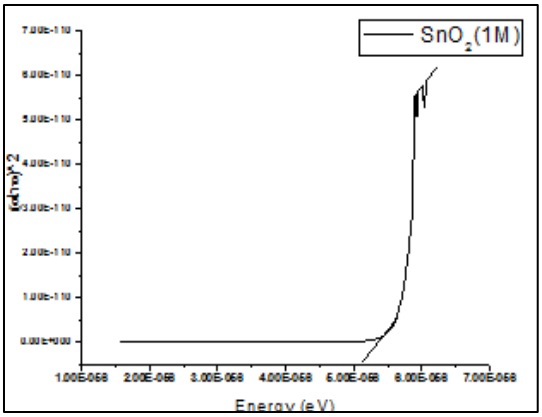

Figure 12: Band gap diagram of SnO_2

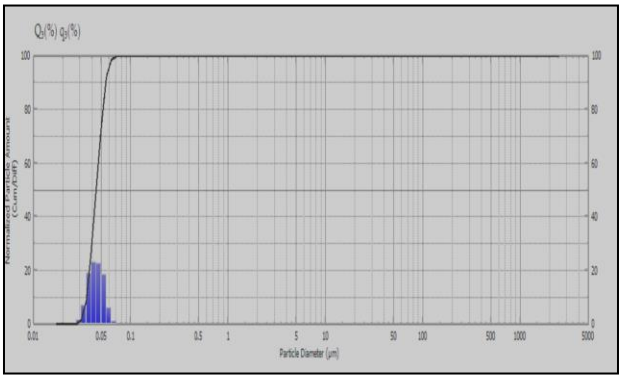

Figure 13: Particle size analyzer of ZnO

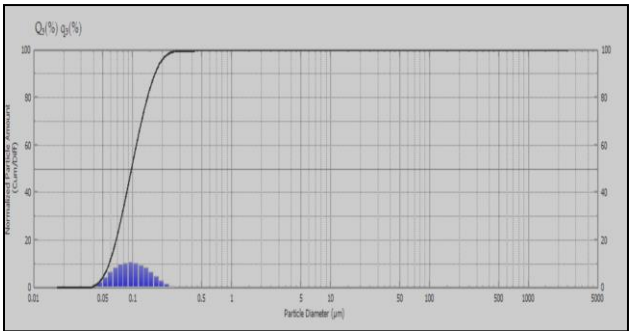

Figure 14: Particle size analyzer SnO_2

CONCLUSION

The ZnO and SnO_2 nanoparticles were synthesized using the Co-Precipitation method. The samples were calcined at 550°C. The XRD studies revealed that the average crystallite size of ZnO-A and ZnO-B nano particles were found to be in the range 27.8 nm and 29 nm. The average size SnO_2 nanoparticle was found to be in the range 24.6 nm. The Particle size analyzer studies confirmed that the ZnO and SnO_2 as a nanoparticles. The particle size of the ZnO from Zinc chloride as a salt was 42 nm and the particle size of the SnO_2 was 44 nm. The FTIR spectra proved that the presence of metal oxide stretching (cm^{-1}) with respect to zinc oxide and tin oxide nanomaterials. The UV – Visible spectral studies concluded that the optical band gap of ZnO from Zinc acetate as a salt was found to be less compared to the ZnO from Zinc chloride as a salt. The energy band gap of the SnO_2 was 5.08 eV.

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