Facile Synthesis and Characterization of nano-sized ZnO by co-precipitation method

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Abstract **- Nano-sized particles of Zinc oxide was synthesized by simple and rapid Co-precipitation method using Zinc chloride as precursor and sodium hydroxide as a stabilizing agent. The way of adding base is a factor which can affect properties of nanoparticles. It is necessary to maintain the particle size with in pH range and temperature. The size, structure and morphology of zinc oxide nanoparticles were characterized by Scanning Electron Microscopy, UV-visible spectroscopy, X-ray diffraction and infrared spectroscopy techniques. The average particle size of Zinc oxide nanoparticles was calculated from the X-ray diffraction study and atomic percentage of Zinc and oxygen in the synthesized ZnO nanoparticles was determined by EDX spectra.**

Index Terms - UV-visible spectroscopy, SEM, XRD Zinc oxide nanoparticle, Co-precipitation.

I. INTRODUCTION

Nanocrystalline materials have attracted a wide attention due to their unique properties and potential application in nano device fabrications. Zinc oxide is a wide range band gap of 3.3eV at 300 K and large excitonic binding energy of 60 meV that have made zinc oxide important both for scientific as well as industrial applications [1-4]. Zinc oxide nanoparticles are used in various applications like medical fields, electronics, antibacterial textiles [5], solar cells, gas sensors, light emitting diodes [6], antibacterial and antifungal agents when incorporated into plastics and paints [7]. Various methods have been used for the synthesis of zinc oxide nanoparticles such as sol gel method [8], solvothermal and hydrothermal method [9], Co-precipitation method [10]. Among them, co-precipitation is the most effective method for synthesizing various kinds of metal oxide due to its simplicity, rapid preparative and low cost to produce metal nanoparticles. In the present work, we mainly focus on ZnO nanoparticles synthesized by simple co-precipitation method from zinc chloride and sodium hydroxide. Due to non-toxicity of zinc oxide nanoparticles, it acts as an additive for textiles [11-12].

II. EXPERIMENTAL PROCEDURE

A. Materials

Zinc chloride ($ZnCl_2$), sodium hydroxide (NaOH) and Ethanol (C_2H_6O) were purchased and used without further purification.

B. Synthesis

Zinc oxide nanopowders were prepared by co-precipitation method. Zinc chloride (0.2M) and sodium hydroxide (0.1M) are mixed with continuous stirring at a constant temperature of 70^0C. While stirring, NaOH solution was added till the pH of solution become 12. The stirring was continued for 2 hours at a constant temperature of 70^0C. White colored precipitates was formed, filtered, and washed with ethanol. Precipitates were dried overnight at 100^0C. Than the precipitates were kept in muffle furnace at 500^0C for 2 hours.

C. Characterization

The chemical properties of formed zinc oxide nanoparticles were determined by Fourier Transform Spectroscopy (FTIR) in the region of 400-4000 cm^{-1}. The crystal structures of the products were characterized by X-ray Diffraction (XRD) over 2θ range of $20\text{-}100^0$ (Rikagu Mini-2 using CuKα1, λ=0.15406 nm radiations), UV-visible spectroscopy (Systronic-2203) in the wavelength range of 200-100cm^{-1}) and surface morphology was studied by SEM. Atomic percentage of Zinc and oxygen in the synthesized ZnO nanoparticles was determined by EDX spectra.

III. RESULTS AND DISCUSSION

A. FTIR Spectra

Figure 1 illustrates the FTIR spectra of zinc oxide nanoparticles synthesized by co-precipitation method. Various peaks are observed in different regions of FTIR spectrum. The characteristic peak near at 499 cm^{-1} indicates the formation of zinc oxide nanoparticles. The weak band at 712 and 910 cm^{-1}indicates the presence of stretching and bending of C-O in the spectrum [13].

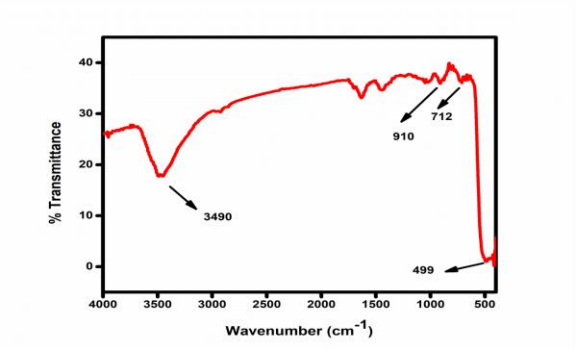

Figure 1: FTIR Spectra of Zinc Oxide Nanoparticles

B. XRD Spectra

Figure 2 shows the XRD spectra of zinc oxide nanoparticles. This diffraction pattern shows the characteristics peak for the spinel ZnO phase of zinc oxide nanoparticles (NPs).

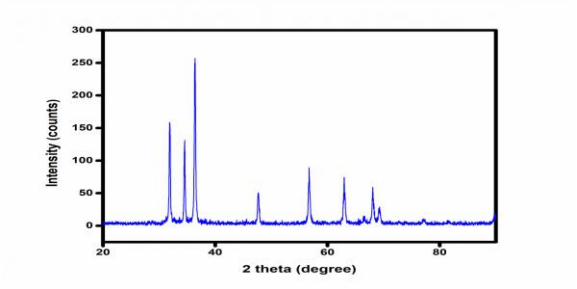

Figure 2: XRD Spectra of Zinc Oxide Nanoparticles

The X-ray diffraction pattern revealed major peaks at 2θ values of 31.84 (100), 34.54 (002), 36.4 (101), 47.7 (102), 56.7 (110), 62.98 (103), 68.8 (201), 71.08 (004) respectively. The lattice constant of a = 3.253 and c = 5.215 which are confirmed by JCPDS card no. 80-0074 and 79-2205. Therefore, the synthesized sample is ZnO nanoparticles. Average particle size of the zinc oxide nanoparticles corresponding to the highest intensity peak was found to be 30.97 nm using Debye Scherrer formula ($d = 0.9\ \lambda / \beta \cos\theta$) [14].

C. UV-Visible Spectra

Figure 3 (a) shows the UV-Visible spectra of zinc oxide nanoparticles synthesized by co-precipitation method as a function of wavelength. The UV-Visible spectra show band at 390 nm. Figure 3 (b) is the Tauc plot showing variation of $(\alpha h\nu)^2$ vs. hν and Figure 3 (c) is the Tauc plot showing variation of $(\alpha h\nu)^{1/2}$ vs. hν.

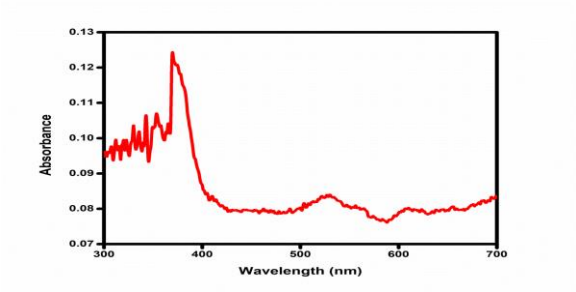

Figure 3 (a): UV-Visible Spectra of Zinc Oxide NPs

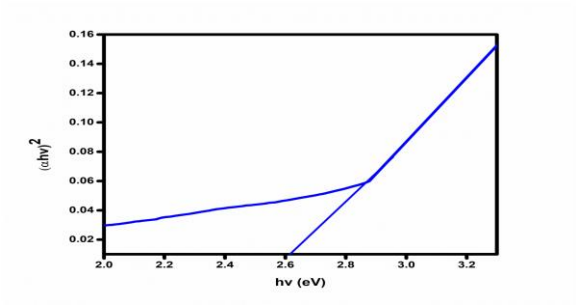

Figure 3 (b): Direct Band Gap of Zinc Oxide NPs

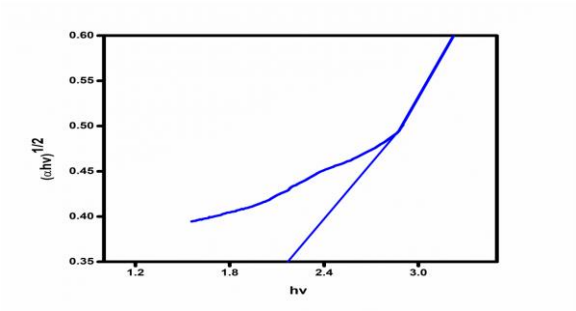

Figure 3 (c): Indirect Band Gap of Zinc Oxide NPs

These plots are calculated from UV-Visible spectra for zinc oxide NPs. The direct and indirect band gap (Eg) of zinc oxide was found to be 2.61 eV and 2.1 eV respectively as shown in Figure 3 (b) and (c).

D. SEM Images

Figures 4 (a), (b), (c) shows the SEM images of zinc oxide nanoparticles. The surface morphology of synthesized zinc oxide NPs was investigated by SEM

images shown in Figure 4 (a), (b) and (c). The three images of synthesized NPs are in higher to lower magnification i.e (5.00 µm, 3.00 µm and 1.00 µm). The as-synthesized nanoparticles are in dispersed form and 20-70 nm in size [15].

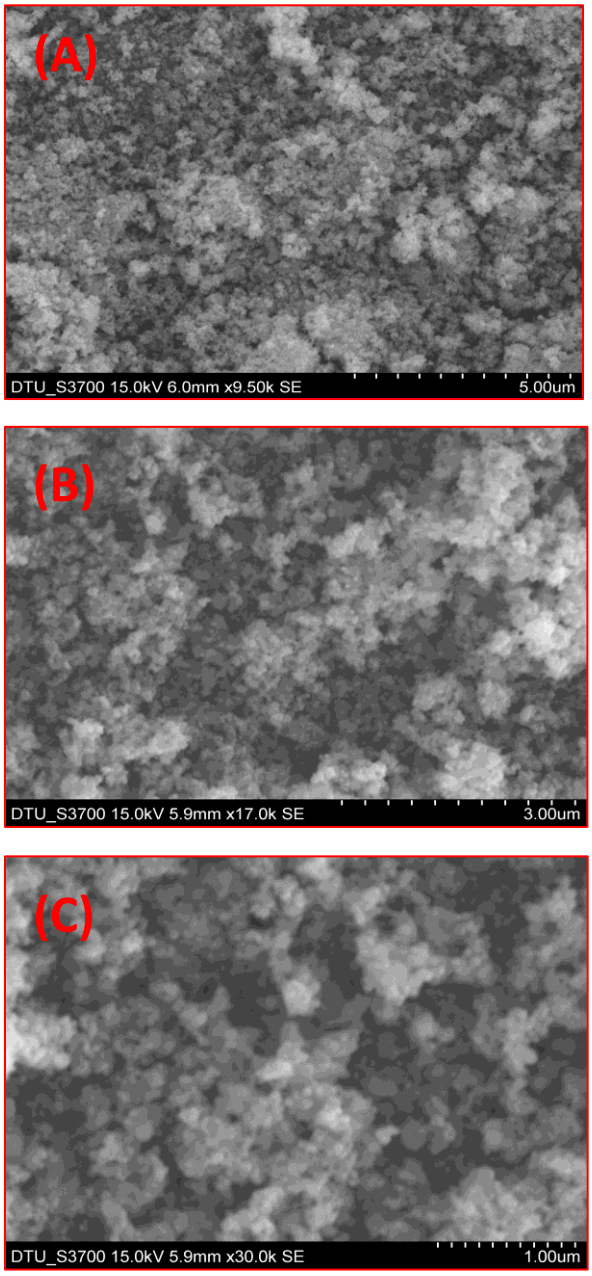

Figure 4: SEM Images of Zinc Oxide Nanoparticles (a) 5.00 µm, (b) 3.00 µm and (c) 1.00 µm.

E. EDX Spectra

Figure 5 shows the EDX spectra of zinc oxide nanoparticles synthesized by co-precipitation method. The energy dispersive X-ray spectrometry (EDX) analysis was used to know the molecular ratio of Zinc and oxygen in the synthesized ZnO NPs. The EDX spectrum of ZnO contains zinc and oxygen elements in atomic percentage of 53.49% and 46.51% respectively in the sample.

Table 1 : EDX Analysis of Zinc Oxide-NPs

Element	Weight %	Atomic %
O (K)	19.95	46.51
Zn (K)	80.05	53.49
Total	100.00	100.00

Where "K" represents 1st inner shell.

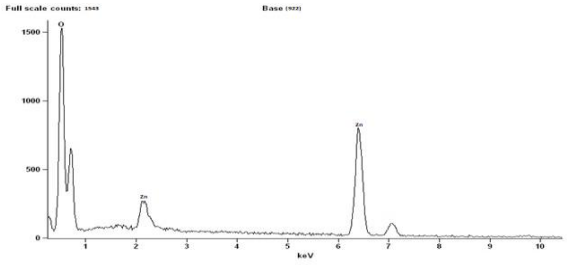

Figure 5: EDX Spectra of Zinc Oxide Nanoparticles

IV. CONCLUSION

Zinc oxide nanoparticles were prepared by co-precipitation method by using zinc chloride ($ZnCl_2$) and sodium hydroxide as a precursor. Average particle size of the zinc oxide nanoparticles was found to be 30.97 nm using Debye Scherrer formula. It is clear from SEM images that nanoparticles formed are in dispersed form and 20-70 nm in size. The EDX spectrum of ZnO shows that it contains zinc and oxygen elements in atomic percentage of 53.49% and 46.51% respectively in the sample.

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