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Characterization of ZnO Nanoparticles synthesized by wet chemical method

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Abstract: Zinc oxide nanoparticles were successfully synthesized by wet chemical method using Zinc acetate dihydrate and potassium hydroxide as a precursor materials. Ethanol was used as a solvent for homogeneity of the solution and helps to make a stoichiometric solution in order to obtain Zinc oxide nanoparticles. The highly stable colloidal ZnO nanoparticles have been prepared at room temperature without using any surfactant. The sample were characterized by field emission scanning electron microscopy (FE-SEM), transmission electron microscopy (TEM), X-ray diffraction (XRD), ultraviolet visible spectroscopy (UV-Vis) and photoluminescence spectroscopy (PL). The TEM images showed that the average size of the nanoparticles was calculated to be ~33nm. Photoluminescence (PL) studies show bright luminescence with peak maximum at 540nm and 563nm due to oxygen vacancy centres (V_o) present in nanoparticles. The optical transmission spectrum of colloidal nano particles of ZnO shows sharp absorption at 4.2eV which is blue shifted as compared to bulk ZnO (3.36eV) due to the quantum confinement effect.

Keywords: ZnO nanoparticle; XRD; UV-Vis spectroscopy; PL; SEM; TEM.

Introduction and Experimental Method

Zinc oxide has been a subject of interest for the scientists and the industry for decades. ZnO is known to be a wide band-gap semiconductor (3.37eV) with a high exciton binding energy [1]. It is a versatile material that has found applications in a variety of areas such as photo catalysis, sensors, piezoelectric transducers, solar cells, transparent electrodes and electroluminescent devices[2], gas sensing devices [3]. There are several solution based routes are available for the preparation of ZnO nanoparticles such as solvothermal, hydrothermal, sol-gel [2], micro-emulsion, vapor phase transport process [4], precipitation[5], RF magnetron sputtering [6], etc., In the present work, an attempt has been made to synthesis ZnO nanoparticles by simple method without adding any surfactant in the precursor solution.

In the present work, Zinc acetate (Merck) and potassium hydroxide (Merck) were used as precursors with ethanol as a solvent for the synthesis of ZnO nanoparticles. 0.1 M of Zinc acetate solution was dissolved in ethanol with $pH \sim 5$. The solution was stirred for 2 hours in order to obtain homogeneous mixture of Zinc salt. 0.1M of potassium hydroxide was mixed in ethanol with a pH of 11. The KOH solution was added drop wise in Zinc acetate solution under continuous stirring. The reaction was carried out at room temperature with a $pH \sim 7$. The final solution was allowed to stirred for 3 hours using magnetic stirrer and filtered through

wattmann filter paper in order to remove impurities present in the solution. The colloidal solution was characterized by optical and microscopic properties.

Results and Discussion

The X-ray diffraction spectrum of synthesized nanoparticles was recorded using CuK_{α} radiation as shown in the fig.1. The film exhibited peaks at 31.7°, 34.4°, 36.35°, 47.6°, 56.6°, 62.9°, 68.0° was well matched with JCPDS card no. 5-0664. Compared with all other peaks, (0 0 2) plane show higher intensity. The grain size was calculated by using Scherrer's equation and is found to be 52nm.

$$D = \frac{\kappa \lambda}{\beta \cos \theta}$$

where k is the shape factor of the crystalline (k=0.9), λ is the wavelength of the x-ray used (1.54060Å), β - the FWHM of (002) plane, θ is the diffraction angle.

The optical properties of ZnO particles were carried out at room temperature by using JASCO V 670 spectrophotometer. Fig 2 shows the absorption and transmittance spectra of ZnO nanoparticles. The UV.Visible spectrum shows the maximum transmittance value in the optical region of 200 nm-900nm. The bandgap of ZnO nanoparticles were found to be 4.2eV.

The PL spectrum of ZnO nanoparticles were recorded at room temperature was shown in the figure 3. Photoluminescence (PL) studies show bright luminescence with peak maximum at 540 nm and 563 nm due to oxygen vacancy centres (V_o) present in nanoparticles and the corresponding excitation wavelength for the emission peak was 250nm. The sharpness of the PL emission indicates that the ZnO nanoparticles are nearly mono dispersed in the precursor solution.

The surface morphologies of synthesized particles were investigated by SEM was shown in the Figure 4. Synthesized nanoparticles were composed of spherical grains with uniform shape and are observed to be monodisperse in precursor solution. Figure 4 shows the TEM micrograph of ZnO nanoparticles. ZnO particles are composed of randomly oriented spherical grains with an average size of 33nm. The ZnO particles were isolated in the corner of the sphere. The outer sphere was due to the formation of Zinc hydroxide or Zinc oxide formation. The mean diameter of the outer sphere was found to be 190 nm and it may decreases by adding capping agent.

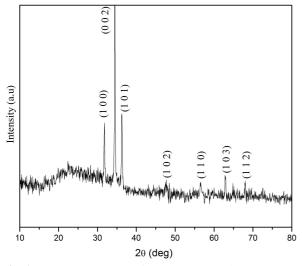


Fig 1: XRD spectrum of ZnO nanoparticles.

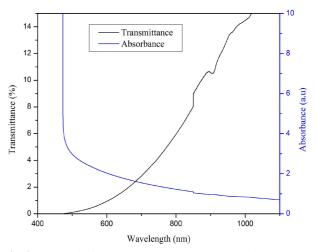


Fig 2: UV-Visible absorbance and transmittance spectrum of ZnO nanoparticles

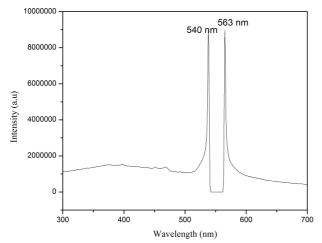


Fig 3: Photoluminescence spectrum of ZnO nanoparticles

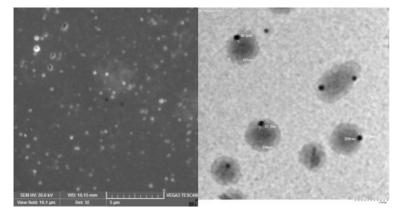


Fig 4: SEM micrograph of ZnO nanoparticles and TEM image of ZnO nanoparticles

Conclusion

A simple method was employed to synthesize Zinc oxide nanoparticles by using ethanol as a solvent without using any capping agent. From the XRD results, the formation of hexagonal phase of ZnO was confirmed and the particle size was found to be 52nm. The bandgap of ZnO nanoparticles was found to be 4.2eV. PL studies show bright luminescence with peak emission peak at the maximum at 540nm and 563nm. ZnO particles are composed of randomly oriented spherical grains are confirmed by SEM and TEM.

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