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RESEARCH ARTICLE OPEN ACCESS

COMPARATIVE ANALYSIS OF ZNO NANOPARTICLE SIZE USING VARIOUS CHARACTERIZATION TECHNIQUES

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ABSTRACT

Preparation of molar concentration varied ZnO nanoparticles was done by chemical bath deposition technique and characterized using XRD, HRTEM and PL spectroscopy. XRD reveals hexagonal structure of ZnO according to standard JCPDS data. The line broadening of ZnO nanoparticles due to small crystal size and strain was analyzed by DS formula and WH technique. HRTEM image of ZnO nanoparticles were compared with the size obtained from the above two method. The size obtained from HRTEM image is little larger than that obtained from XRD as XRD revels only the crystallite size where as combination of many crystallite forms a particle which is observed by HRTEM image. The particle size was also calculated from PL study using the Gaussian equation which also well supports our results obtained from the XRD and HRTEM study.

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INTRODUCTION

Zinc oxide (ZnO) nanoparticles is an attractive low-cost semiconductor since decade due to their high demand in wide range of applications like photo-detectors, LED, solar cells, gas sensors and also in biomedical applications. It is a wide band gap semiconductor with a band gap of 3.37 eV and also possesses a large exciton binding energy of 60 MeV [1,2]. Because of its suitability in the application of various optoelectronic devices and low toxicity, many workers prefer to synthesize ZnO employing different methods [3-7]. Thus several researchers recognized ZnO because of its unique structural, optical and electrical properties for developing many useful products. In addition to its use in electronic, cosmetic and bio-sensing product, ZnO is also acts as drug carriers, antibacterial, anti-fungal and antioxidant agents as it is cheap and exhibits a low toxicity [3]. ZnO nanoparticles are also used in treatments such as cancer, infection and control diabetes [3]. It also shows a good luminescence property and thus it is also used in bio-imaging [4]. Thus ZnO is a multi-faced material with unique morphologies which are responsible in application in several fields. Here in this work CBD technique have been used to synthesize ZnO nanoparticles because of its low cost compared to other technique and also need of low sophisticated instruments. Use of proper nontoxic capping agent is also a challenging task and so polyvinyl pyrrolidone (PVP) is used as the capping agent in the present work as it contains eco-friendly functional groups (*C=O, C-N and* CH_2) in it. It is a great stabilizer and prevents agglomerism of nanoparticles via repulsive forces which come from its hydrophobic carbon chain in it that extends into the solvent and interact with each other. The functional groups in PVP make a well quantum confinement during the synthesis and generate the as desired nanoparticles.

Synthesis and characterization technique used in the present study: All chemicals having 99% purity procured from Merck has been used in the synthesis of ZnO nanoparticles. PVP is used as the capping agent in the present work along with zinc acetate dehydrate [$Zn(CH_3COO)_2 2H_2O$], sodium hydroxide (*NaOH*) and deionized water as the aqueous medium. Figure 3.1 shows the schematic for the preparation of ZnO nanoparticles. For the preparation of ZnO nanoparticles of molarity 1M, 1M of zinc acetate solution is taken in 25 ml of deionized water that have 3% PVP stock solution. After 5 min under striation with the help of magnetic stirrer, 1 M of NaOH solution in 25 ml of water is added dropwise. The above mixture is then stirred for about 1.5 hour at 90^0C under vigorous stirring which turns the whole mixture to milky white solution. The conversion of the solution to milky white confirms the formation of ZnO nanoparticles which is then filtered and dried for about 5 to 6 hours in hot air oven. The chemical reaction involved in the formation of ZnO by using the above method is given as follows-

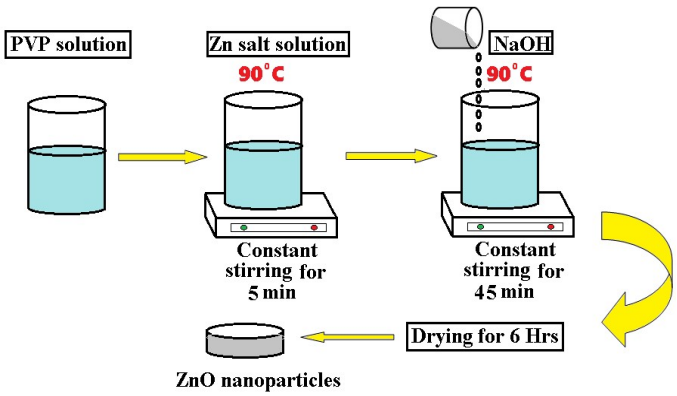

Fig. 1. Schematic for the preparation of ZnO nanoparticles

$Zn(CH_3COO)_2 2H_2O + 2NaOH$ ---------- $ZnO + 2NaCH_3COO + 3H_2O$

By using the above method ZnO nanoparticle of 1M molarity can be prepared which is coded as Z1. Similarly, ZnO nanoparticle having molarities 0.1M, 0.01M and 0.001M can also be prepared which are coded as Z2, Z3 and Z4 respectively. The above synthesis nanoparticles are characterized using different techniques. The structural and surface morphology is studied with the help of X-Ray diffractometer (XRD) and high-resolution transmission electron microscopy (HRTEM) using ULTIMA IV X-Ray diffractometer and JEOL 2100F respectively. The optical study is done by photoluminescence (PL) spectroscopy using a Jasco spectro-flurometer (FP-800). The excitation wavelength for PL study was kept at 300 nm.

RESULTS AND DISCUSSION

Results obtained from XRD studies: The XRD pattern of specific sample Z1, Z2, Z3 and Z4 having molarities 1M, 0.1M, 0.01M and 0.001M respectively is shown in Figure 2. Prominent diffraction peaks correspond to plane (100), (002), (101), (102), (110), (103), (200) and (112) are seen for ZnO nanoparticles which show hexagonal structure according to JCPDS- 00-001-1136 card number. M Jabeen in 2014 also observed similar hexagonal wurtzite structure of ZnO nanorod synthesis by using the hydrothermal method [5]. Hexagonal structure of ZnO nanopowder was also observed by S K Fatah in his study where ZnO nanopowder was prepared by using the precipitation method [6]. The lattice parameter of hexagonal structure ZnO is calculated using equation 1 which are found to be a = 3.239 *Å* and c = 5.201 *Å* which is almost similar to the above mentioned standard JCPDS data.

$$\frac{1}{d^2} = \frac{4}{3}\left(\frac{h^2+K^2+hk}{a^2}\right) + \frac{l^2}{c^2} \quad (1)$$

The average crystallite size of all the prepared samples is calculated using the DS formula

$$D = \frac{K\lambda}{\beta Cos\theta} \quad (2)$$

Where D is the average crystallite size of the sample, K is Scherrer's constant (0.94), λ is the wavelength of X-Ray used, β is full width half maximum and θ is Bragg's diffraction angle. However, while calculating the crystallite size using the DS formula, the strain of the prepared sample is not taken into consideration but to the best of our knowledge, both strain and X-ray line broadening has its contribution in determining the crystallite size of a sample. Thus Williamson-Hall (*WH*) technique has been employed to all the samples where contribution of both crystallite size (D) and strain ($\boldsymbol{\varepsilon}$) has been taken into consideration. Equation (3) known as *WH* equation is given as follows-

$$\beta Cos\theta = \frac{K\lambda}{D} + 4\varepsilon Sin\theta \quad (3)$$

The details of crystallite size and strain calculated from WH technique for all the sample is shown in Table 1. The calculated values obtained from WH method have more accuracy then DS method as it includes the contribution of peak broadening from both size and strain.

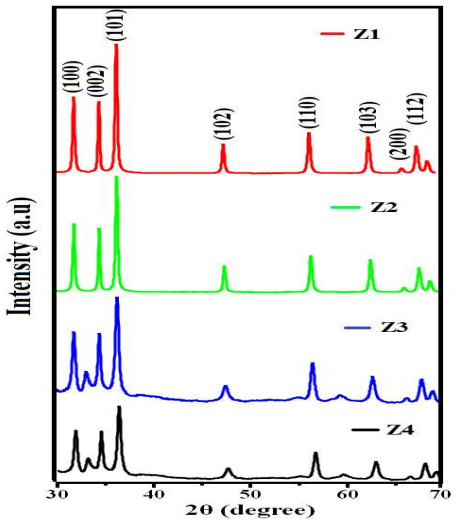

Fig. 2. XRD spectra of molar concentration varied ZnO nanoparticles

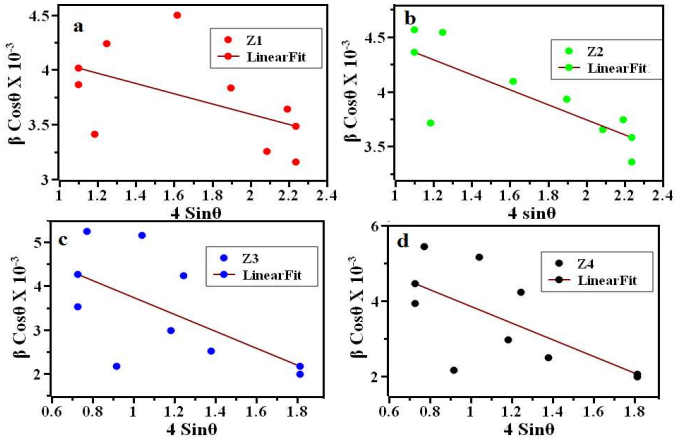

Fig. 3. WH technique of ZnO nanoparticles

Results obtained from HRTEM studies: For more clear, magnified and precise observation of prepared nanoparticles HRTEM measurements were done as shown below. Figure 4 shows HRTEM image of ZnO nanoparticles at different molarities. Well-distributions of the nanoparticles are seen over the solution. It is also observed that the particle size of ZnO nanoparticles increases with the increase in molarity which is also confirmed from the XRD study. A Hexagonal type structure is seen in the highly magnified image of all the prepared sample of ZnO nanoparticles which supports the shape found in XRD study. Hexagonal shape ZnO nanoparticles were observed in several studies in literature among them, P K Giri *et al.* [7] and V D Mote *et al.* [8] also found such structure of ZnO prepared by chemical synthesis and co-precipitation process respectively. It is seen that the particle size calculated from HRTEM study is a bit larger than that found from XRD study which may be due to the usual combination of many crystallites to form bigger structures.

Results obtained from PL studies: The PL spectra of all the prepared sample of molar concentration varied ZnO are done at room temperature with an excitation wavelength of 300 nm. Non-uniform distribution of PL spectra is observed from Figure 5 is due to presence of defect in the sample.

The presence of a defect in the sample can be confirmed by the occurrence of less intense peak in the longer wavelength side of the spectra. The prime high intense peak as seen in the curve corresponds to the band-to-band transition. This emission band is fitted with a Gaussian peak function given by equation (4) [9, 10].

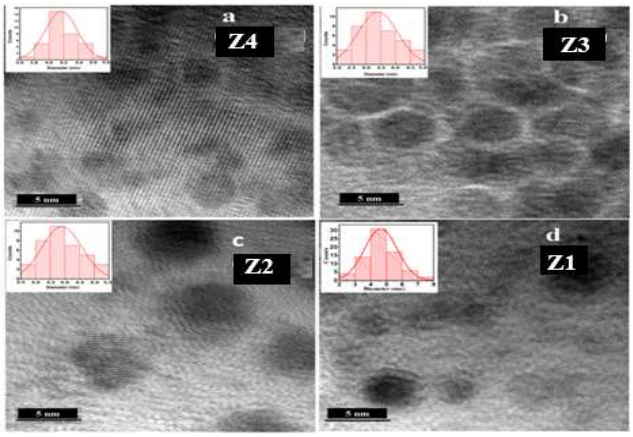

Fig. 4. HRTEM image of ZnO nanoparticles

$$P(R) = \frac{1}{\sigma_R\sqrt{2\pi}} \exp\left[-\frac{(R-R_0)^2}{2\sigma_R^2}\right] \quad (4)$$

Where R_0 = mean radius of particle and σ_R = standard deviation. Comparing equation (4) with the standard Gaussian equation, the particle size of all the samples is calculated and found to be 4.3, 3.6, 3.0 and 2.9 nm for samples Z1, Z2, Z3, and Z4 respectively. The calculated particle sizes are in excellent agreement with the measurements from HRTEM image and XRD study. An average exponential increase of size with molar concentration is observed. An enhancement of size thus may be attributed to more defects induced at the surface because of the high density of reacting ions which increases with concentration. The particle size calculated from HRTEM image and PL data became larger than those measurements from XRD as XRD measures the crystallite size of any sample which is usually agglomerated to form a bigger structure that are observed in HRTEM images. The particle size calculated from all the above method is shown in Table 1 and a correlation graph is drawn in Fig 6.

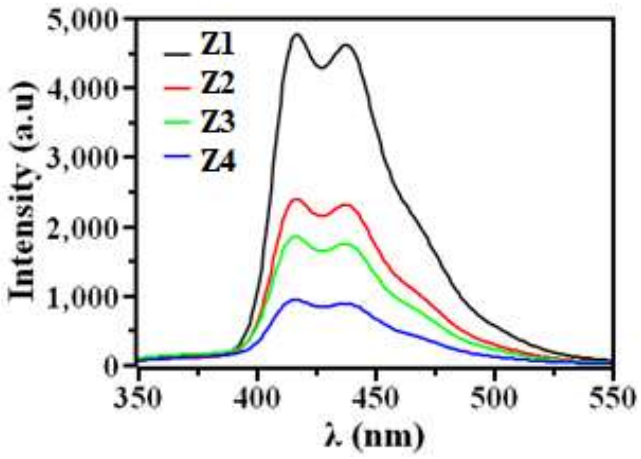

Fig. 5. PL Spectra of the molar concentration varied ZnO

Table 1. Size obtained from different methods of ZnO nanoparticles

Sample Name	D (nm)			
	DS method	WH method	HRTEM	PL
Z1	4.1	4.0	4.2	4.3
Z2	3.5	3.4	3.6	3.6
Z3	2.6	2.5	2.8	3.0
Z4	2.3	2.1	2.7	2.9

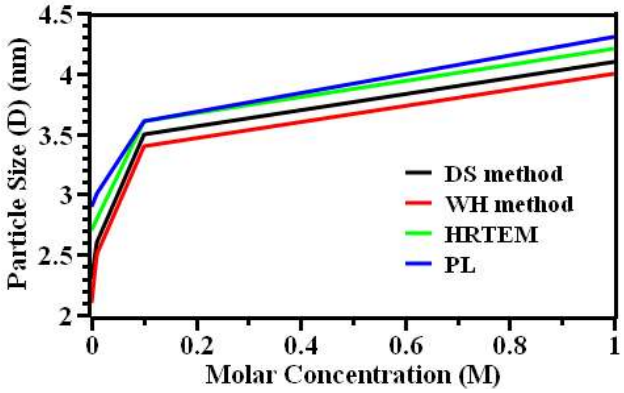

Fig. 6. Correlation graph between the size obtained by DS, WH, HRTEM, PL technique

CONCLUSION

ZnO nanoparticles were synthesized by the chemical bath deposition technique and characterized by XRD, HRTEM and PL spectroscopy. The line broadening of ZnO nanoparticles due to small crystal size and strain was analysed by Scherrer's formula. The size and strain contribution to line broadening were analyzed by the method of WH technique. HRTEM image od ZnO nanoparticles were compaired with the size obtained from the above two methods. The size obtained from HRTEM image is little larger than that obtained from XRD as XRD revels only the crystallite size where as combination of many crystallite forms a particle which is observed by HRTEM image. Particle size was also calculated from the PL study using the Gaussian equation which also well supports our results.

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