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SYNTHESIS AND CHARACTERIZATION OF ZINC OXIDE (ZnO) NANOROD BY WET CHEMICAL METHOD

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ABSTRACT

Zinc Oxide (ZnO) nanorods have been successfully prepared in Polyethylene glycol (PEG Mw=4000) by wet chemical method. Zinc Oxide (ZnO) nanorods have been characterized by X-ray diffraction (XRD) , absorption spectra, Scanning Electron Microscope (SEM) with EDAX . X-ray diffraction (XRD) indicated that the crystalline size were 45.788 nm,40.508 nm ,40.7331 nm, 29.650 nm, 12.6346nm, 23.89671 nm, 24.57666nm and 22.30592nm of (100),(002),(101)(102),(110),(103),(112) and (201) plane respectively and the average particle size was obtained 52 nm of ZnO nanorods . Surface morphology has been investigated by using the Scanning Electron Microscope (SEM). EDAX confirm the chemical composition of zinc oxide.

KEYWORDS: Absorption Spectra, Nanorods, PEG, SEM, XRD ZnO

INTRODUCTION

Zinc oxide (ZnO) is rapidly gaining reliability as a material with excellent potentiality for electronic and photonic devices and it has wide and direct band gap (3.37 eV) and large exciton binding energy (60 MeV)[1]. Owing to the semiconducting and piezoelectric dual properties, novel applications are introduced which have keen effect in many areas such as self-powdered nanodevices and nanosystems. The demonstration of room temperature ultraviolet lasers, field effect transistors and field emission arrays based on zinc oxide nanorods have stimulated great importance in developing functional nanodevices [2,3]. Moreover, the wide range of morphological heterogeneity in the nano-regime has made this material a promising candidate in the field of nanotechnology and opened up new possibilities for the fabrication of high performance devices based on these nanostructures. Among the various shapes of nanostructures, one dimensional (1D) nanostructures have received considerable attention due to their potential interests for understanding fundamental physical concepts and for efficient field emission that has tremendous commercial applications [4-6].

Literature survey reveals that a large number of techniques for preparing ZnO nanostrucures have been applied by different workers. Zinc oxide (ZnO) nanostructure have been prepared by Electrochemical deposition (ECD) [7, 8], VLS Method [9-11], Chemical method [12-16], Hydrothermal method [17], a microwave irradiation method [18], Thermal evaporation [19], Ion-beam technique [20], Sol–gel technique [21,22]. and pulsed laser ablation [23]. Compared with above synthesis processes, wet chemical approach is relatively popular since it is simple, low expense and environment protective. It is known that size of the particles can be controlled easily through the use of polymers in the system. Many polymers are known to have long hydrocarbon chain structures with hydrophobic ends. It is believed that this structure are critical in manipulating particle size, are known as capping agent. We reported a simple wet chemical approaches in which ammonia water and zinc nitrate as raw materials and Polyethylene glycol (PEG) as capping agent were employed for synthesis of ZnO nanorods. Especially, this synthesis process possesses large-scale production and facile raw materials which is favorable for the application in various fields. Furthermore, the crystal growth mechanism was discussed.

EXPERIMENT

The glass wares (canonical flask, measuring cylinder, beakers) were first cleaned and rinse with distilled water and dried at atmosphere. All the materials and solvents were weighted with help of electronic weighing balance. 0.02 mol $Zn(NO_3)_2 \cdot 6H_2O$ was dissolved in 600 ml distilled water under stirring (solution A). The mixture of PEG (Mw=4000) and $NH_3 \cdot H_2O$ was added dropwise into the solution at room temperature, resulting in a white solution and pH=10. The final mixture was rapidly heated to 70 °C and stirred vigorously. After the reaction, the precipitate was filtered and washed several times with deionized water and alcohol, and then dried at room tempreature in air overnight.

The obtained sample was characterized by X-ray diffraction (XRD), UV-Visible Absorption Spectroscopy, Scanning Electron Microscopy (SEM) with Energy dispersive X-ray analysis (EDAX).

RESULTS AND DISCUSSIONS

X-Ray Diffraction (XRD) Studies

The XRD patterns of prepared sample was taken by X-ray diffractometer using the characteristic CuKα (1.5418 Å), lines of metal anticathode. Figure 1 shows XRD patterns of as-prepared ZnO sample. The Diffraction Peak appeared in the XRD Spectra are indexed to present the wurtzite structure of ZnO (JCPDS, Card no.36-1451) and Si material. The higher intensity peak of ZnO is at 2θ= 36.85 is the characteristic of cubic (101) plane.and other diffraction peaks are the characteristics of (100),(002),(102),(110),(103),(112) and (201) planes.

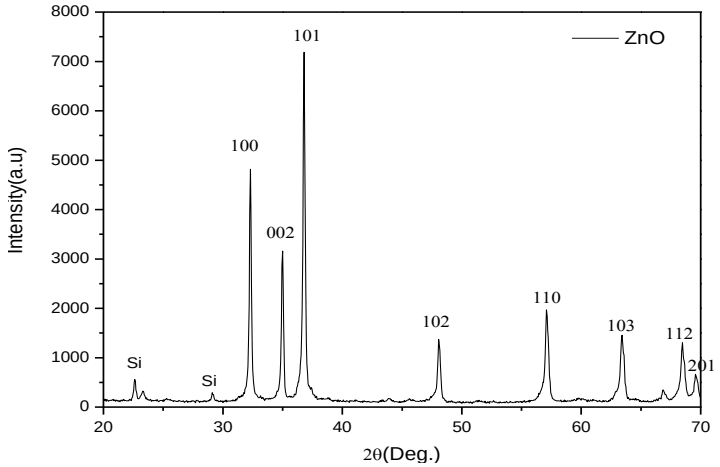

Figure 1: XRD Diffraction Pattern of as-Prepared ZnO Nanorods

The Crystallite Size "t" of the As-prepared ZnO nanorods was determined by using the Scherrer formula formula [24].

$$t = \frac{0.9\lambda}{B \cos \theta_B}$$

Where λ is the X-ray Wavelength (1.54 Å), θ_B is the bragg diffraction angle, and B is the FWHM of peak. The crystallize size of the As-prepared ZnO nanorods is shown in table 1

Table 1: Crystallize Size of ZnO Nanorods

Plane	$2\theta_B$(Degree)	B(Degree)	t(nm)
100	32.189	0.1806	45.77886
002	34.99	0.2056	40.51167
101	36.85	0.2056	40.73372
102	48.061	0.2934	29.65053
110	57.087	0.7159	12.6346

Table 1: Contd.,			
103	63.55	0.3911	23.89671
112	68.49	0.3911	24.57666
201	69.5712	0.4337	22.30592

The lattice strain and crystalline size was calculated from the following equation: [25]

$$\frac{\beta Cos\theta}{\lambda} = \frac{\tau Sin\theta}{\lambda} + \frac{1}{\varepsilon}$$

where β is the measured FWHM (full-width at half maximum), θ is the Bragg peak angle of the peak, λ is the X-ray diffraction wavelength (in this case, it is 1.54 Å), ε is the effective particle size and τ is the effective strain.

The average particle size can be estimated from the extrapolation of the plot shown in Figure 2 and the crystalline size was obtained 52 nm based on the intercept inverse, *i.e.*, $1/\varepsilon = 0.01890$, which yields $\varepsilon = 52$ nm.

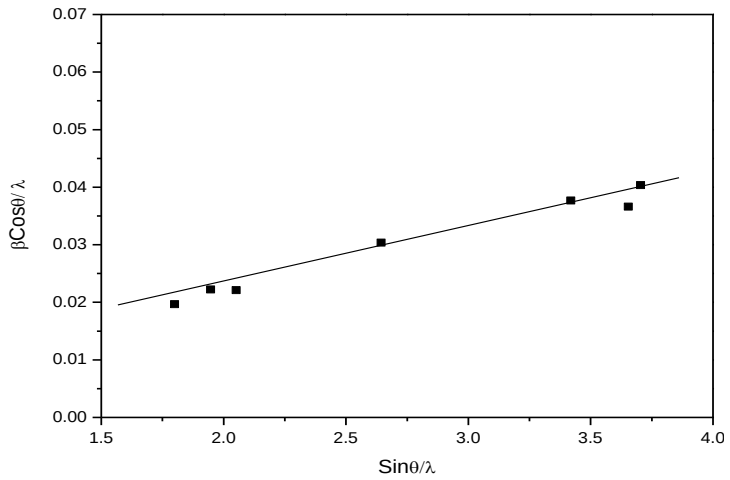

Figure 2: β Cosθ/λ vs. Sinθ/λ for ZnO Nanorods

Determination of Lattice Parameters

We can easily determine the values lattice constant 'a' and 'c' of as-prepared ZnO nanorods from the equation[26]

$$\frac{1}{d_{hkl}^2} = \frac{4}{3}\left(\frac{h^2+k^2+hk}{a^2}\right) + \frac{l^2}{c^2}$$

Where . d_{hkl} is the measured values of inter spacing distance for a particular (hkl) plane. The Measued Value of lattice constant 'c' of (002) plane and lattice constant 'a' of (100) plane is shown in table 2.

Table 2: Measured Lattice Constants from XRD

	a(Å)	c(Å)	c/a
ZnO Nanorods	3.207501	5.136758	1.601483

Optical Absorption

The optical absorption spectrum of the as-prepared ZnO nanorods was measured by using a Hitachi U-3400 UV-Vis spectrophotometer. The nanocrystallites powder has been suspended in glycerol using magnetic stirrer and the optical absorption spectra has been recorded at room temperature. The UV-Vis absorption spectrum of the ZnO nanorods is shown in figure 3. The absorption spectra have a narrow peak near the band edge in the exciton absorption region (at about 370 nm) and blue-shifted relative to the bulk exciton absorption (380 nm). The stronger exciton effect is an important character of quantum confinement effect [29].

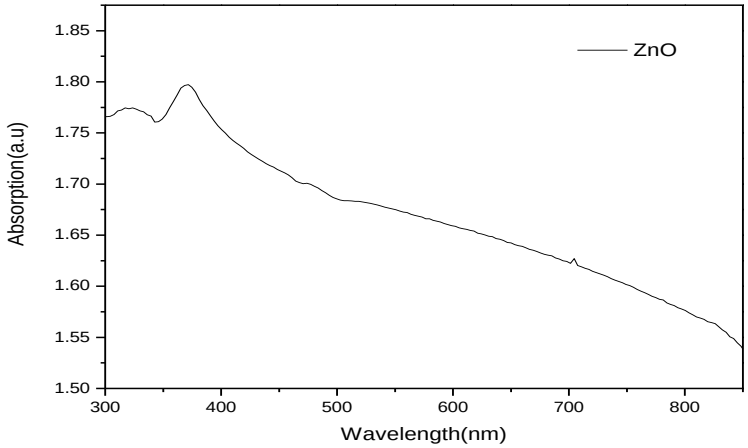

Figure 3: Absorption Spectra of as-Prepared ZnO Nanorods

Scanning Electron Microscopy (SEM) & EDAX Studies

Scanning Electron Microscopy (SEM) with Energy dispersive X-ray analysis (EDAX) is suitable technique to study the microstructre and chemical composition of nanostructured materials .Figure 4 represent the SEM Images of ZnO sample with single short and long nanorods.

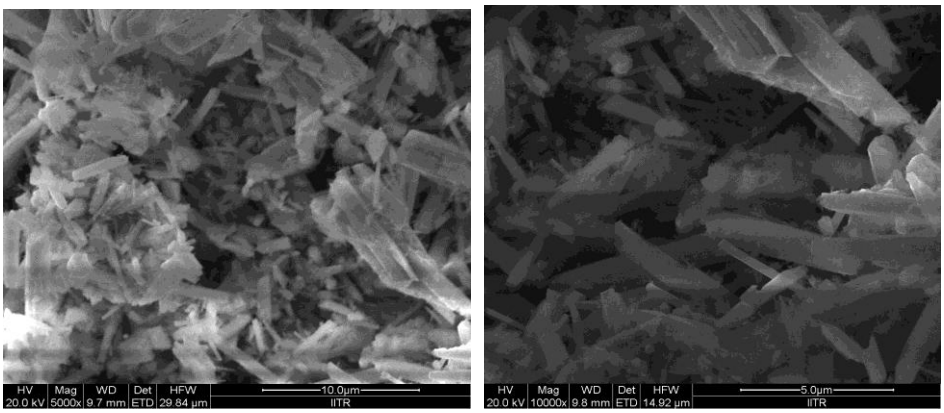

Figure 4: SEM Images of as-Prepared ZnO Nanorods

Table 3

Element	Weight%	Atomic %
O K	12.53	38.64
Si K	1.00	1.63
Zn K	85.47	59.73
Totals	**100.0**	

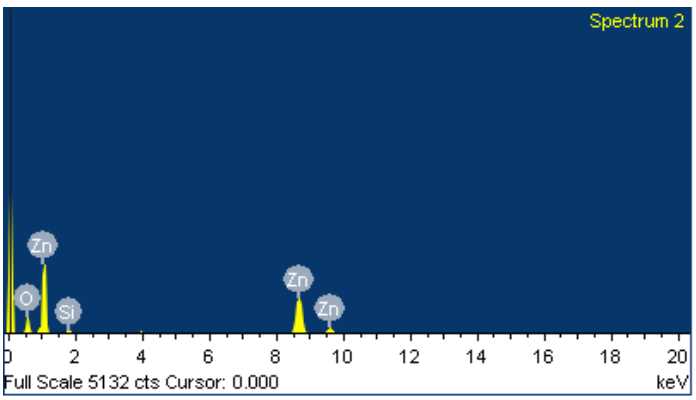

Figure 5 : Energy Dispersive X-Ray Analysis (EDAX) Pattern of as-Prepared ZnO Nanorods

EDAX spectroscopy is analytical tool to determine the composition of the sample. The composition of the ZnO nanorod was analyzed by EDAX spectroscopy as shown in Figure 5 which exhibits the presence of Zn and O with impuity of Si. The atomic percentage of Zn,and O shows excess of Zn in the material. Electron beam induced inner-shell ionization and subsequent emission of characteristic fluorescence are analyzed in order the composition. The purity and composition of the prepared sample was studied by Energy dispersive X-ray analysis (EDAX). As shown in figure 5, Zn L_α - fluorescence (L_α around 1 keV energy range), Zn K-fluorescence (K_α in the energy range 8-9 keV and K_β in the energy range 9-10 keV), O K_α- fluorescence (K_α in the energy range 0-1 keV) and), Si K_α- fluorescence (K_α in the energy range 1-2 keV).This fluorescence Spectrum shows the presence of Zn , O as the elementary components with Si as a impurity.

CONCLUSIONS

ZnO nanorods have been successfully prepared on a large scale through a very simple wet chemical method at 70 °C. X-ray diffraction (XRD) indicated that the crystallize size of ZnO nanorods were were 45.788 nm,40.508 nm ,40.7331 nm, 29.650 nm, 12.6346nm, 23.89671 nm, 24.57666nm and 22.30592nm of (100), (002) (101), (102), (110), (103), (112) and (201) plane respectively and the average particle size was obtained 52 nm. ZnO nanorods as synthesized gives blue shifted as compared to the bulk band gap value and is evident of quantum confinement. EDAX spectroscopy exhibits the presence of Zn and O with Si as impuity. The atomic percentage of Zn and O shows excess of Zn in the material.

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