STRACTURAL AND OPTICAL CHARACTERIZATION OF SENSITIZED ZnO POWDER BY CHEMICAL REACTION METHOD

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Abstract

Prepared ZnO powder has been successfully synthesized by chemical reaction method. The calcination of ZnO powder has been characterized by number of techniques such as X-ray diffraction (XRD), Scanning Electron Microscopy (SEM) and Ultraviolet and Visible spectrophotometry (UV-Vis) at room temperature. The structural characteristic was examined using XRD and the surface morphology of ZnO powder was analyzed by Scanning Electron Microscopy (SEM). XRD analysis revealed that the sample crystallite in polycrystalline nature with wurtzite hexagonal structure and average crystallite was 30.50 nm at 500 °C. The microstructural of SEM result was observed that hexagonal shape and granular in nature. The absorption spectrum indicated that the energy band gap of ZnO powder. The optical property of the ZnO powder was determined by UV-Vis absorption spectrophotometry. The energy band gap of sensitized powder was examined to be equal to 3 eV. Taking into account the results obtained, ZnO powder has been reported for photoanode of solar cell applications.

Keywords: chemical reaction method, ZnO powder, XRD, SEM, UV-Vis

Introduction

Nanotechnologies are the design, characterization, production and application of structures, devices and systems by controlling shape and size at the nanometer scale. Two approaches for the building up nanoscopic features have been investigated: the so-called top down method and bottom up method. In the top down methods, the features are written directly onto a substrate, for example, by electron beams, and then by applying appropriate etching and deposition processes, the nanoscopic features are engraved. In the bottom-up approach, nanocomponents are made from precursor in the liquid, solid or gas phase employing either chemical or physical deposition processes that are integrated into building blocks within the final material structure [Filipponi L. and D. Sutherland, 2013].

ZnO is an important II-VI compound semiconductor material due to its novel properties like large direct energy gap (3.37 eV), high melting point (2248 K) high refractive index, and large exit on binding energy of (60 meV) at room temperature. This semiconductor also exhibit several favorable properties like good transparency, high electron mobility, wide band gap, strong room temperature luminescence, high thermal conductivity, antibacterial and UV protection [Taunk, P. B. et al, 2015], [Vanaja, A. and K. S. Rao, 2016].

ZnO has attracted much interest as one of the multifunction inorganic nanoparticles due to its unique combination of superior physical, chemical, biological, electrical, optical, long-term environmental stability, biocompatibility, low cost and non-toxic properties. Therefore, nano-ZnO can potentially be applied to gas sensors, photo catalyst for degradation of waste water pollutants, catalysts, semiconductors, varistors, piezoelectric devices, field emission displays,

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ultraviolet (UV) photodiodes, surface acaustic wave (SAW) devices, UV shielding materials, rubber, medical and dental materials, pigments and coating, ceramic, concrete, antibacterial and bactericide and composites [Nehal, S. A. et al, 2015].

Many techniques are nature used to synthesize ZnO nanoparticles via precipitation method, spray pyrolysis method, microemulation method, hydrothermal method and sol-gel method [Kulkarni, S. S. & D. S. Mahendra, 2015]. The process parameters for the preparation of the ZnO nanoparticle, by CBD include deposition temperature, deposition time, concentration ratio of the reaction solution, types of chelating compounds and additives, pH values, and so on [Wen-Yao, H., 2012]. Chemical bath deposition is one of the useful solution methods for the preparation of such as semiconductors from aqueous solution, with advantage such as low processing temperature, allowing growth variety of substrates and easy adaptation to large area processing at low fabrication cost [Wallace, I. et al, 2015]. The adopted ZnO nanoparticles CBD approach, requiring low temperature treatments, is suitable for integration will colloidal lithography [Kathalingam, A. et al, 2010].

In the present work, ZnO *nanoparticles powder was prepared by chemical* reaction method. The structural powder was characterized by XRD and SEM. The optical property was investigated by UV-Vis spectrophotometer.

Experimental Procedure

Zinc sulphate, ethylenediamine and sodium hydroxide were used as the starting materials for the preparation of the ZnO nanoparticles by chemical reaction method. Deionized (DI) water (80 mL) was added to the solid materials of zinc sulphate (0.02 M) and ethylenediamine (1 mM), to prepare the reaction solution. After the solution was prepared, it was stirred with magnetic stirrer to maintain at temperature (50 ˚C - 55 ˚C). Then, NaOH (0.045 M) was added to the homogeneous solution and the pH value of the solution was controlled the range of 13. Next, the obtained solution was heating in oven at 50 ˚C for 8 h. And then, the homogenous powder was annealed in furnace at 500 ˚C for 1 h. The annealed powder was grinding with mortar and pestle for 3 h to obtain ZnO nanoparticles. The parameter of annealed ZnO nanoparticles was determined from the X-ray diffraction (XRD) measurement using (Multiflex - 700) with CuK_{α} (λ = 1.54056 Å) radiation operation at 40 kV and 40 mA. Scanning Electron Microscopy (SEM) was conducted with a JEOL, Japan JSM-(5610 LV) operated at 15 kV to determine the morphological changes as a function of annealing temperature. The optical absorption spectrum was recorded in the range of 300-800 nm using UV-Vis spectrophotometer (UV-1800, Shimadzu, Japan). Synthesis and characterization flow chart of ZnO nanoparticles at 500 ˚C were shown in Figure 1.

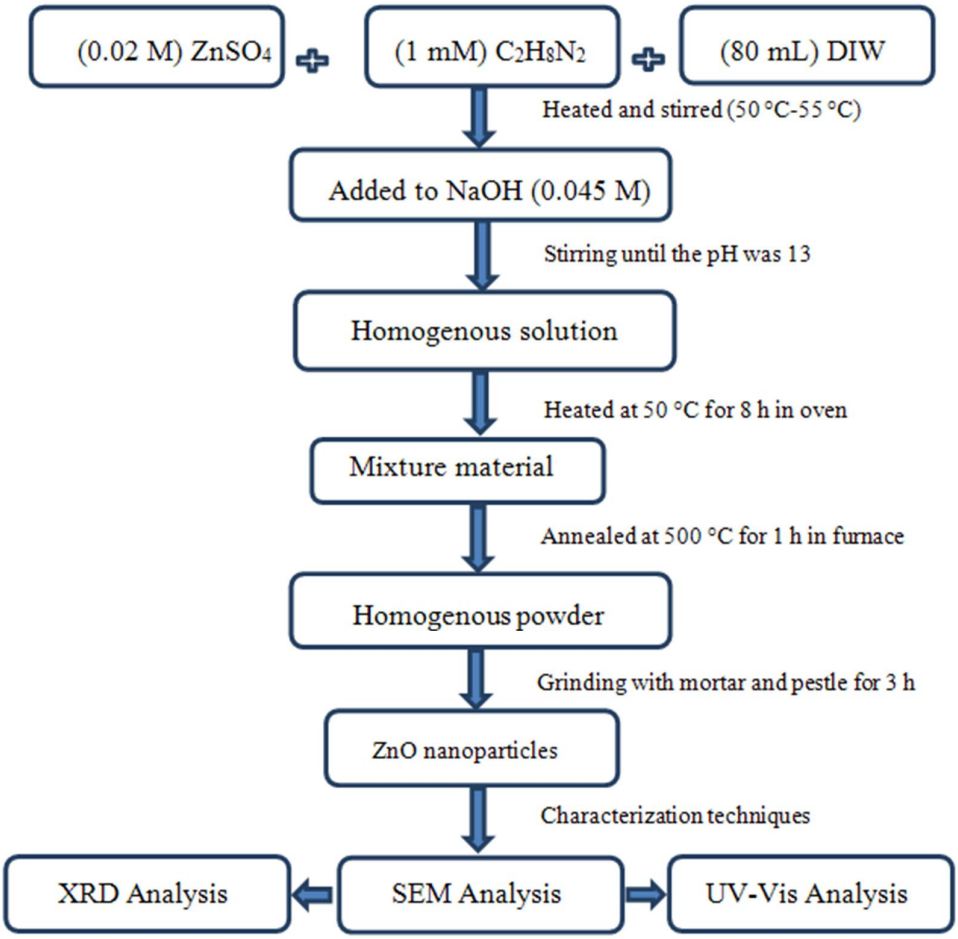

Figure 1 Synthesis and characterization flow chart of ZnO nanoparticles at 500 ˚C

Results and Discussion

XRD Analysis

The X-ray diffraction data was recorded by using CuK_α radiation (1.54056 Å). The intensity data was collected over a 2θ range of 10-70. X-ray diffraction studied confirm that the synthesized materials was ZnO with wurtzite phase and all the diffraction peaks agreed with the reported JCPDS (Joint Committee on Powder Diffraction Standards) data and no characteristics peaks were observed other than ZnO. The average crystallite size D was determined from the XRD line broadening measurement using Scherrer equation.

$$D = \frac{0.9\,\lambda}{\beta \cos\theta}$$

where, λ is the wavelength (CuK_α), β is the full width at half maximum (FWHM) of the ZnO and θ is the diffraction angle.

Figure 2 showed XRD diffraction pattern of ZnO nanoparticles. The detected peaks corresponded with those of hexagonal phase zinc oxide were found at the lattice plains of (100),

(002), (101), (102), (110), (103), (200), (112), (201) in the 2 theta values: 31.58˚, 34.223˚, 36.034˚, 47.350˚, 56.371˚, 62.661˚, 66.173˚, 67.701˚, 68.901˚ respectively. All diffraction peaks of sample corresponded to the characteristics hexagonal wurtzite structure of zinc oxide nanoparticles (a = 3.2676 Å and c = 5.2266 Å). The diffraction peaks related to the impurities were not observed in the XRD pattern, confirming the high purity of the synthesized powder. Diffraction angles, crystallite sizes and lattice parameters data of ZnO nanoparticles powder was summarized in Table (1-3).

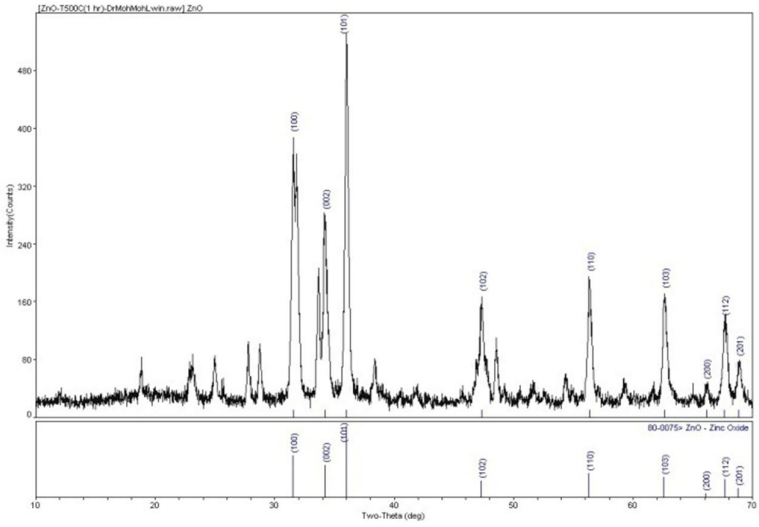

Figure 2 XRD pattern of ZnO nanoparticles at 500 ˚C

Table 1 Diffraction angles of all identified peaks for ZnO nanoparticles at 500˚C

No	Peaks	2θ (deg)	
		Observed values	Standard values
1	(100)	31.580	31.537
2	(002)	34.223	34.210
3	(101)	36.034	36.021
4	(102)	47.350	47.303
5	(110)	56.371	56.327
6	(103)	62.661	62.611
7	(200)	66.173	66.091
8	(112)	67.701	67.674
9	(201)	68.901	68.800

Table 2 Crystallite sizes of ZnO nanoparticles at 500˚C

No	Peak	FWHM (deg)	Crystallite size (nm)
1	(100)	0.496	14.9233
2	(002)	0.441	29.7784
3	(101)	0.302	17.2563
4	(102)	0.468	26.0518
5	(110)	0.370	17.0916
6	(103)	0.398	21.5713
7	(200)	0.321	33.2406
8	(112)	0.419	32.3068
9	(201)	0.338	82.3077
Average crystallite size			30.5031

Table 3 Lattice parameters of ZnO nanoparticles at 500˚C

No	**Peak**	**Lattice parameter (Å)**		
		a	**c**	**c/a**
1	(101)	3.2687	5.2382	1.6025
2	(102)	3.2687	5.2170	1.5960
3	(103)	3.2687	5.2153	1.5955
Average c/a ratio				1.598

SEM Analysis

The grain size, shape and surface properties like morphology were investigated by Scanning Electron Microscopy (SEM). Figure 3 represents the SEM image of ZnO nanoparticles synthesized by CBD annealed at 500 ℃ for 1 h. The SEM image was observed with the magnification of 2 µm, the ZnO nanoparticles were uniform, crack free and they formed hexagonal shape. Few particles were granular in shape and aggregation.

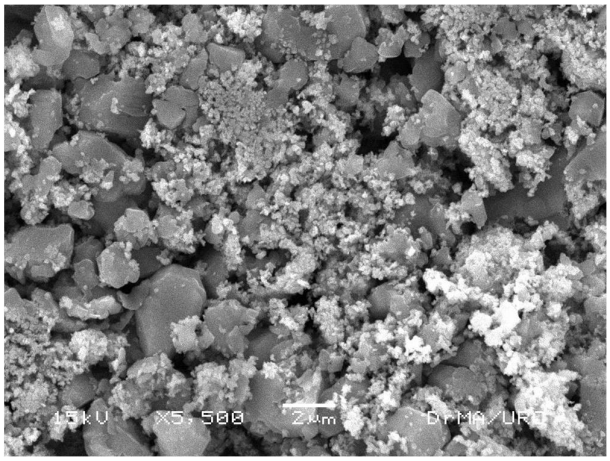

Figure 3 SEM image of ZnO nanoparticles at 500 ˚C

UV-Vis Analysis

UV absorption was related to the electronic transition from filled valence states to empty conduction states. Therefore, the optical band gap can be defined as the difference between the valence band and conduction band in momentum space. The optical band gap is dependent upon the particle shape, particle size and defect concentration in the crystal. The band gap energy can be determined by substituting the value of the cutoff wavelength in the following equation.

$$E_g = hc/\lambda$$

where, h = 6.63×10^{-34} Js, c = 3×10^{8} ms^{-1}, λ = cutoff wavelength (388 nm). The absorption coefficient (α) as a function of photon energy (hυ) can be expressed by the Tauc equation,

$$\alpha h\upsilon = B\,(h\upsilon - E_g)^n$$

where, hυ is the incident photon energy, B is the edge width parameter and n is an exponent that determines the type of electronic transition causing absorption, which is $^1/2$ or 2 for direct or indirect transition, respectively.

The UV-Vis absorption spectra and optical band gap of ZnO nanoparticles at 500˚C annealing temperature with different powder concentration were shown in Figure 4 & 5. A broad absorption peak was observed in each spectrum at 375.5 nm which is a characteristics band for the pure ZnO. No other peak was observed in the spectrum confirmed that the synthesized product was ZnO only. Interestingly, an obvious red shift in the absorption edges were observed for the product annealed at 500˚C. This red shift of the band gap energy was due to aggregation of nanocrystallites into larger grains as reported by various authors in different literatures. The direct energy band gap was calculated to be nearly equal to 3 eV and the large band gap clearly the wide transparency of the crystal.

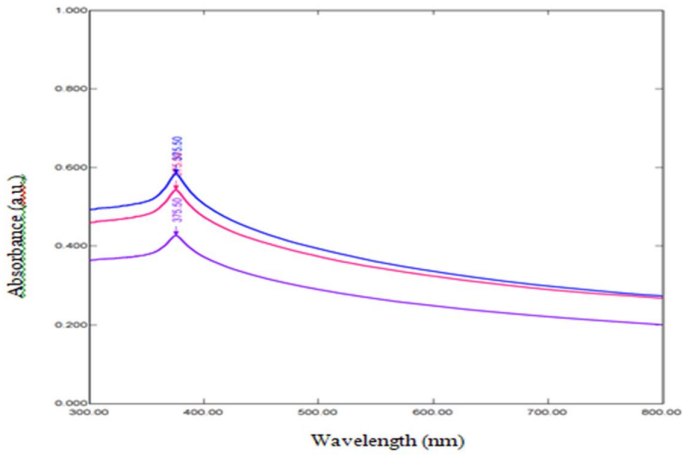

Figure 4 Absorption spectrum of ZnO nanoparticles with different powder concentration at 500 ˚C

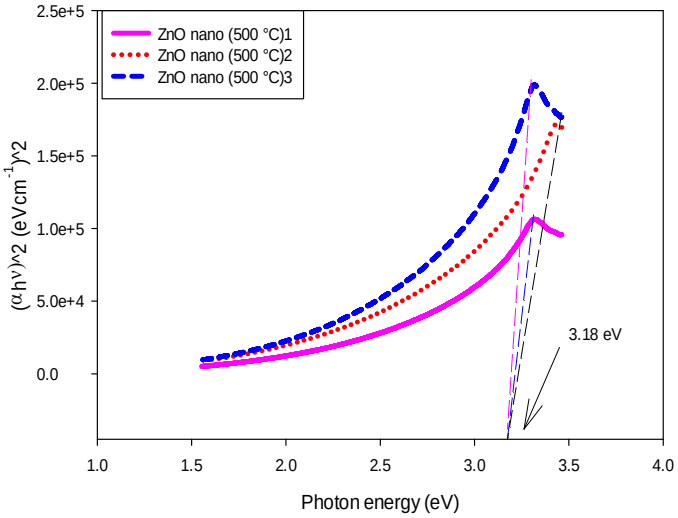

Figure 5 Optical band gap of ZnO nanoparticles with different powder concentration at 500˚C

Conclusion

ZnO powder has been successfully synthesized via direct precipitation route using wet chemical reaction method. XRD analysis confirmed the formation of polycrystalline nature of ZnO nanoparticles. The analysis of XRD pattern showed only the presence of pure wurtize crystal structure with average crystallite size of about 30.502 nm. The microstructural properties of ZnO nanoparticles were uniform in hexagonal shape. Few particles were granular nature of aggregation. A broad absorption peaks were observed in each spectrum at 375.5 nm. The band gap of the ZnO nanoparticles was calculated from the UV-Vis absorption, and it was stably observed spectra that there were red shifts in the absorption edges of different powder concentration compared with bulk material (3.3 eV). The energy band gap of sensitized nanoparticles was obtained to be approximately equal to 3 eV. ZnO is a wide band gap semiconductor having high optical transparency in visible and near ultraviolet range of spectrum. Therefore, it would be a promising candidate for solar cell applications.

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