

Effect of Reaction Time on Some characterization of ZnO Nanoparticles

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Manuscript Details

Available online on <http://www.irjse.in>
ISSN: 2322-0015

Editor: Dr. Arvind Chavhan

Cite this article as:

Shaikh RS and Ravangave LS. Effect of Reaction Time on Some characterization of ZnO Nanoparticles, *Int. Res. Journal of Science & Engineering*, January 2018, Special Issue A2 : 187-191.

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ABSTRACT

ZnO nanoparticles were synthesized using chemical sol-gel precipitation method using solution of zinc acetate and sodium hydroxide distilled water as starting materials. The stock solutions of Zinc acetate (0.5M) and Sodium hydroxide (0.5M) were used for synthesis of ZnO nanoparticles at different reaction time via, 2hours, 4hours, 6hours and 8hours. The synthesized samples were calcined at 100°C temperature for two hour. The four different samples were prepared by using reaction times 2h, 4h, 6h and 8h. Prepared ZnO particles were characterized by using XRD, FTIR, scanning electron microscopes (SEM), UV-visible spectrophotometer and Photoluminescence (PL). The XRD pattern exhibit wurtzite hexagonal crystal symmetry. The particle size estimated by using Debye-Scherrer formula was found varying with increasing reaction time. The characterizations, scanning electron microscopes (SEM) and FTIR show the significant structural and morphological changes of ZnO particles. The optical study carried out using UV-visible spectrophotometer and photoluminescence spectroscopy (PL) of the samples shows that absorption edge of UV-spectra are highly blue shifted. The Photoluminescence spectrum exhibits two emission peaks one at 389nm and excitation emission located at 524 nm. The green emission peak attributed to oxygen vacancy or interstitial related defects. The significant morphological changes of the ZnO nanoparticles were investigated from SEM images.

Keyword: ZnO Nanoparticles, XRD of ZnO, Uv-Visible spectra of ZnO.

INTRODUCTION

The objective of the present work is to synthesis of hexagonal wurtzite ZnO nanoparticle with different surface modification have a desirable properties and applications in biological physical chemical and medical fields. ZnO is an important compound semiconductor material due to its direct energy band gap (3.37eV) and large excitation binding energy (60meV) at room temperature. It has the unique physical and optical properties which can be used in a variety of application such as oxide coatings for solar cell, gas sensors, UV photodiode, optoelectronics, and chemical catalytic, photochemical and electric properties [1-3]. ZnO nanostructure has a great advantage to apply to a catalytic reaction process due to their large surface area and high catalytic activity [4]. ZnO semiconductor nanowires and nanorodes are attractive compounds for nano-metre scale electronic and photonic device application because of their unique chemical and physical properties [1-2]. A wide variety of nano-devices including ultraviolet photodetectors, sensor, p-n junction diode, schottky diode LED device have been fabricated using ZnO nanorodes and nanowires. ZnO has also been confirmed as a promising functional material for nanodevices such as field emitter and gas sensors [4-5]. Various methods have been described in earlier literature for the production of ZnO nanostructures such as. Hydrothermal, Chemical precipitation, Chemical Vapor, Co-precipitation, Anodization, Thermal decomposition, Spray pyrolysis, Electro-chemical deposition, Combustion method and Chemical Sol-gel precipitation method [6-16].

In the present paper ZnO nanoparticles have been prepared by using simple very economic chemical Sol-gel method. The growth of ZnO nanoparticles depends upon reaction time variation solution mixture. We study the effect of reaction time on structural, morphological, optical change of ZnO nanoparticles.

METHODOLOGY

Using chemical precipitation Sol-gel method, Zinc acetate and sodium hydroxide were used in the experiments. The entire chemical used were of analytical

reagent grade obtained from Merck (Mumbai) India and distil water is used for the preparation of solution. A solution of Zinc acetate (0.5M) and Sodium hydroxide (0.5M) were prepared. The solutions were individually stirred vigorously for two hours. The solutions of Zinc acetate and Sodium-hydroxide was then added slowly drop wise in a molar ratio of 1:2 under vigorous stirring and the stirring was continuous for two hours. The precipitate obtained was filtered and washed thoroughly with distil water. The precipitate was dry in an oven at 100°C for 2 hours. Other samples were prepared for reaction time 4h, 6h and 8hours and precipitates obtained were dried and ground to fine powder using agate mortar and pestle. The obtained powder was calcined at temperature 100°C, for 2 hours. The prepared samples were then characterized by various techniques such as XRD, SEM, FTIR, UV- Visible spectrophotometer and Photoluminescence spectrometer.

RESULTS AND DISCUSSION

XRD Study:

The XRD pattern of ZnO particles grown at different reaction time (2h, 4h, 6h and 8h) was shown in figure 1. The XRD pattern exhibit distinct peaks attributed to hexagonal structure of the ZnO particles. All the samples exhibit wurtzite structure with hexagonal symmetry. The pattern is well matched with standard JCPDS card Number [36-1455], the various peaks at different diffracting angles (2θ) correspond to different reflections from the planes (100), (002), (101), (102), (110), (103) and (200). Similar structure of hexagonal ZnO nano particles was investigated in earlier literature [7]. The high intensity and narrow line width indicate the good crystallinity [5] of ZnO NPS. The XRD pattern reveals that crystallites vary with reaction time of solution mixture. The crystallinities of ZnO structure was found increased on increasing from reaction time of solution mixture used in the preparation. The crystallinity is found increased for time 2h to 8h. The particle size calculated form Debye-Scherer relation (1) varies from 33nm to 42 nm.

$$D = \frac{0.94\lambda}{\beta \cos\theta} \quad (1)$$

Where D is the crystalline size (nm), λ is a wavelength of incident X - ray (nm) β is the full width at half maximum and θ is the diffraction angle. The XRD study concluded that ZnO nanoparticles are increased in size for time of reaction mixture 2h to 8h.

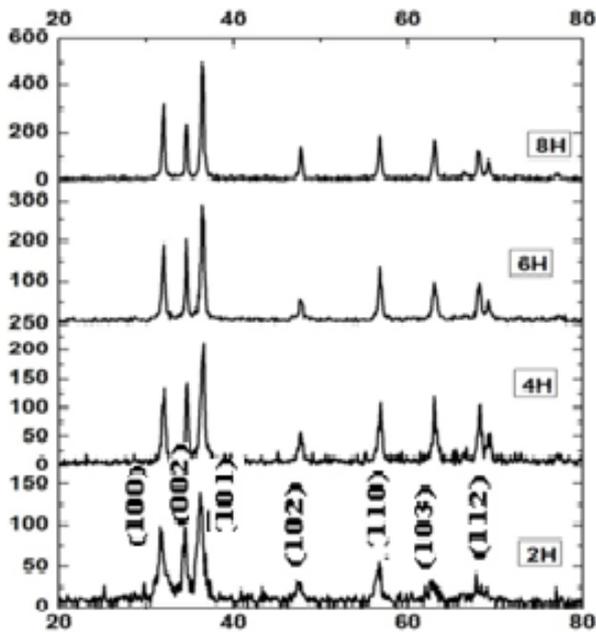


Fig. 1. XRD of ZnO Nanoparticles

ZnO sample with different reaction Time	ZnO crystalline size (D)nm
ZnO 2 H	33.32
ZnO 4H	35.57
ZnO 6H	38.40
ZnO 8H	42.63

FTIR Study:

Figure 2 shows the FTIR spectra of ZnO nanocrystals prepared at different time reaction of mixture. The FTIR is powerful tool for investigation of structure and functional groups. The FTIR spectra exhibit very low intense peak at 3482 cm^{-1} can be assigned to O-H stretching mode due to adsorption of atmospheric moisture. The peak at 1632 cm^{-1} is attributed to OH bending mode of water and at about 1410.14 cm^{-1} the -COO mode arises from the absorption of atmospheric CO_2 on the surface of the nanoparticles [7]. A strong band at 666.30 cm^{-1} is assigned to the stretching mode of Zn-H. The main absorption band at 496 cm^{-1} is due to Zn-O stretching vibrations. The O-H bending and -COO vibration bands decreasing in case of 2h to 8h ZnO NPs.

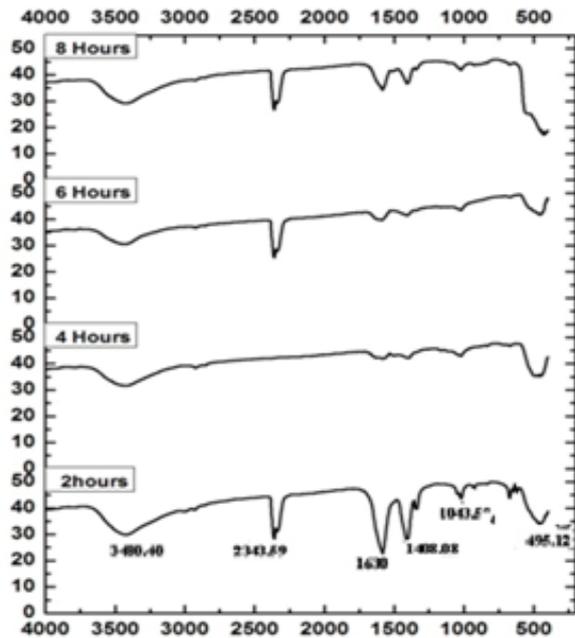


Fig. 2 FTIR Spectra of ZnO Nanoparticles

Frequency of Vibrational Band	Intensity	Vibrational Group
496.67	23.769	Zn-
1410.14	61.665	Zn-O
1632.25	51.237	ZnO-H
3482.83	47.281	OH

Surface Morphology:

The morphology of the nanostructures obtained for prepared ZNO NPS by using Scanning Electron Microscopy tool. The obtained SEM images are as shown in Figures 3. The SEM micrographs exhibit different morphology depending upon reaction time of solution mixture used in preparation of ZnO NPS. The ZnO nanoparticles prepared from reaction time 2h to 8h exhibit excellent nanorods. The different morphology of ZnO NPS leads to significant change in structural and optical properties.

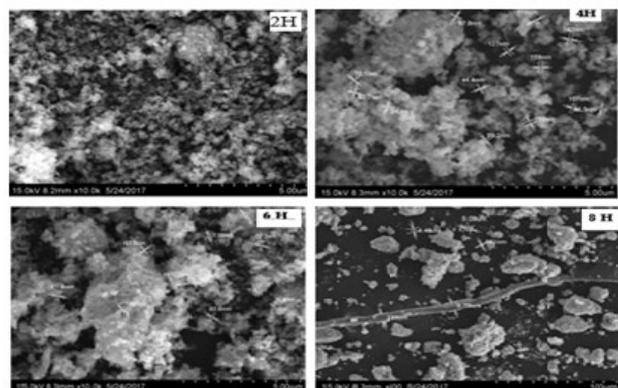


Fig. 3 SEM Images of of ZnO Samples

UV-Visible Absorption Study:

UV- visible absorption spectra of prepared ZnO nanoparticles is presented in figure 4. The optical absorption exhibits strong absorption peaks in UV region at 362 and 352 nm of the electromagnetic spectrum by the ZnO NPs prepared for reaction time 2h to 8h. The spectra show intense absorption at 192 to 216 nm wavelength. The samples 4H and 8H show higher blue shifting as compared to others. The band gap energies obtained from the spectra are tabulated in table 1 shows variation with reaction time. The ZnO particles Prepared form reaction mixture for reaction time 2h to 8h exhibit increasing trend of the band gap energy.

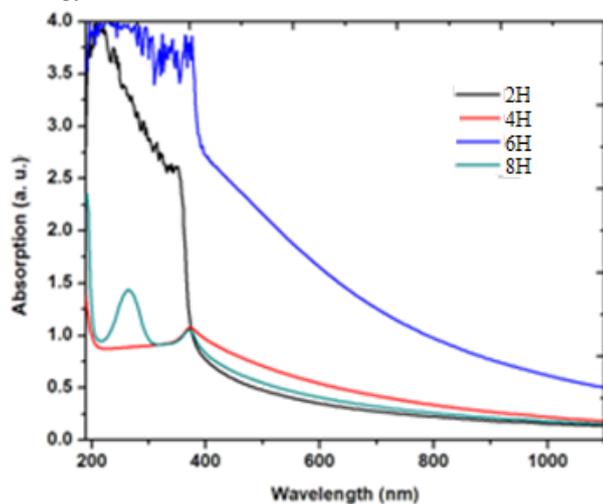


Fig. 4 UV- Visible Spectra of ZnO Particles

Table 3. Grain size and Band Gap		
Reaction Time	Grain Size (nm)	Band Gap Eg (eV)
2 hours	33	3.31
4 hours	35	3.33
6 hours	38	3.36
8 hours	42	3.42

PL photo luminance:

In PL spectra of the 8 hours reaction time samples was shown in figure 5. The presence of the peak at 372nm is corresponding to the excitation emission or UV emission in ZnO Nanoparticles. The peak in the spectral range 472nm to 562nm corresponding to the visible emission of ZnO nanoparticles. The peak 420nm corresponds to the blue band and the peak at 460nm corresponds to weak blue-green band, the peak at 442nm to 566nm is due to various defects such as interstitial Zn and the presence of the acceptor and donor states in the region between the valance and

conduction bands. The green band results due to the presence of slight oxygen vacancy defect in the ZnO nanoparticles.

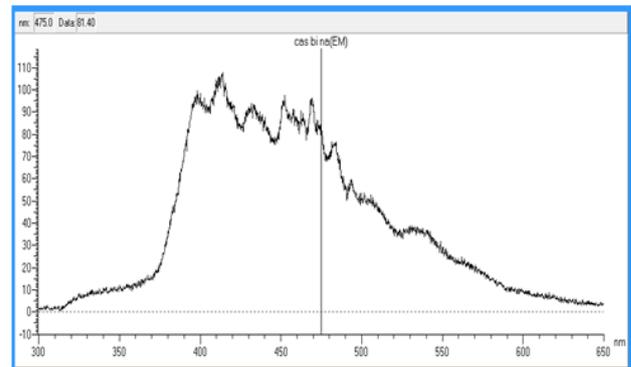


Fig. 5 PL spectra of ZnO particles prepared for reaction time 8H

CONCLUSION

In this study, ZnO nanorods of significant morphology were successfully prepared by Sol-Gel chemical precipitation method. The XRD pattern confirms the excellent hexagonal crystallinity of ZnO nanorods prepared from reaction mixture of time variation 2hours to 8hours. XRD spectra confirms the ZnO samples constitute nano size particles. The FTIR spectra exhibits strong absorption band at 496 cm^{-1} attributed to Zn-O stretching mode for time variation reaction 2h to 8h. FTIR study concludes that samples prepared were of Zinc oxide material in spite of the surface absorption of H_2O and CO_2 . The sharp optical absorption peak at 362 nm and 352 nm exhibited by ZnO (2h to 8h) nanoparticles in the UV region of electromagnetic spectrum with significant blue shifting. From the study of XRD, FTIR SEM and UV - Visible it is concluded that for presently employed Sol-gel preparation technique, good quality ZnO nanoparticles can be obtained from the reaction mixture at time variation reaction 2h to 8h.

Acknowledgement:

The authors are acknowledges to Director, Physics Research Centre, Shri Sant Gadge Maharaj College Loha. Maharashtra, India 431708 for providing research facilities.

Conflicts of interest: The authors stated that no conflicts of interest.

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