International Journal of Engineering Science Invention
ISSN (Online): 2319 – 6734, ISSN (Print): 2319 – 6726
www.ijesi.org ||Volume 6 Issue 11|| November 2017 || PP. 76-79

Influence of pH on Structure, Morphology and UV-Visible Spectra of ZnO nanorods.

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Abstract: *Zinc oxide (ZnO) nanoparticles are synthesized by using a simple chemical Sol-Gel chemical precipitation method. Zinc acetate, sodium hydroxide and distilled water as a starting materials. The stock solutions of Zinc acetate (0.75 M) and Sodium hydroxide (0.75) were used for synthesis of ZnO particles at different pH values (8, 9, 10, 11, and 12). Sol-Gel fabricated ZnO nanoparticles at different pH value were calcinated for two hours at 100 ^oC and characterized by using X-ray diffractometer, FTIR, Scanning Electron microscopy (SEM) and Uv-Visible spectrophotometer. The effect of pH on various characterizations was investigated. The XRD pattern exhibit wurtzite hexagonal crystal symmetry. The particle size estimated by using Debye-Scherrer relation was found varying between 25 to 37 nm on increasing pH of reaction mixture. The SEM micrographs show significant improvement in the surface morphology of ZnO rods with increasing pH values. UV –visible spectra exhibit considerable variation with pH values of reaction mixture used for preparation of ZnO NPs.*

Keywords*: ZnO nano particles; Sol-gel technique;, UV-visible spectroscopy.*

Date of Submission: 30-10-2017 Date of acceptance: 14-11-2017

I. INTRODUCTION

The objective of the present work is to fabricate the hexagonal wurtzite ZnO nano particles with different surface modifying morphology. Modification of surface morphology of NPS directly related to the marked changes in chemical and physical properties. The different morphologies of ZnO nanostructures including ZnO QM dots, Nanorods, nanowires, Nano Flowers, Nanoribbons, nanospindle, etc[(1-3]. The excellent different morphologies of hexagonal ZnO nanostructures having wide band gap are commonly important material used in electronics, lasers, Optoelectronics Piezoelectric, concrete, rubber, gas and biosensors, biomedicine and solar cells, antibacterial and anti fungal applications. These wide range of applications leads to fabricate ZnO nanostructures with modified surface morphology [4-6]. The properties of ZnO strongly depend on their dimensions and morphologies. ZnO nano size QD'S represents unique class of materials, generates novel properties that differ from those of their bulk form due to their small size and large surface to volume ratio [7]. The shape and size of NPs, depends on conditions of preparation, materials used in fabrication, temperature of calcinations etc, directly affect the UV- visible absorption [8].Many different preparation techniques are developed for synthesis of ZnO nanostructures, such as spray pyrolysis, microemulsion, hydrothermal process direct precipitation method, Sol-Gel chemical hydrolysis technique etc.To fabricate ZnO NP's with different modified morphologies we employed simple, low-cost room temperature, high yield sol-Gel chemical hydrolysis technique. The growth of ZnO NPs depends upon the technique parameter such as reaction time, molar concentration of surfactants and pH of the reaction mixture.
In present work we study the effect of pH, on structure, morphology and UV-visible absorption spectra.

II. EXPERIMENTAL METHODS:

The chemicals used for this work were of high purity analytical grade obtained from Merck (Mumbai).

Sol -Gel Precipitation Method for preparation of ZnO Nanoparticles:

A solution of Zinc acetate (0.75M) and sodium hydroxide (0.75M) was prepared. Above two solutions were individually stirred vigorously for two hours. With continuing stirring sodium hydroxide solution was added slowly drop wise in zinc acetate solution so that pH of the reaction mixture reaches to (8,9,10,11 and 12), and stirring was continued for 8 hours. The precipitate obtained was filtered and dried at room temperature for two day. Obtained precipitate of Zno particle was calcinated at 100 ^0C for two hours in oven to remove the unwanted by products.

The prepared sample was then characterized by various techniques such as X – ray diffraction (XRD), FTIR, UV-Visible absorption spectra and Scanning Electron Microscopy (SEM).

III. RESULTS AND DISCUSSION

1. XRD Study

The XRD pattern of ZnO particles grown at different pH values (8,9,10,11and12) of rection mixture was shown in figure 1. The XRD pattern exhibit distinct peaks attributed to hexagonal structure of the ZnO particles. All the samples exhibit wurtize structure with hexagonal symmetry. The pattern is well matched with standard JCPDS card No. (36-1451), these peaks at different diffracting angles (2θ) correspond to different reflections from the planes (100), (002), (101), (102), (110), (103) and (200) respectively. Similar structure of hexagonal ZnO nano particles was investigated in earlier literature [9]. The high intensity and narrow line width indicate the good crystallinity [10] of NPS. The XRD pattern reveals that crystallinity varies with pH values of reaction mixture. The crystallinity of ZnO structure was found increased on increasing from pH values of reaction mixture used in the preparation. The crysallinity is found decreased for pH values 12. The particle size calculated form Debye-Scherrer relation (1) vary from 25 to37 nm.

$$D=\frac{0.94\lambda}{\beta Cos\theta} \qquad (1)$$

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 high quality ZnO nanoparticles are obtained for pH value of reaction mixture 10 and 11.

2. FTIR Study

Figure 2 shows the FTIR spectra of ZnO nanocrystals prepared at different pH values of reaction mixture. The FTIR is powerful tool for investigation of structure. The FTIR spectra exhibit very low intense peak at 3480 cm^{-1} can be assigned to O-H stretching mode for the adsorbed atmospheric moisture. The peak at 1630 cm^{-1} is attributed to OH bending mode of water and at about 1408.08cm^{-1} the -COO mode arises from the absorption of atmospheric CO_2 on the surface of the nanoparticles [11]. A strong band at 667.30 cm^{-1} is assigned to the stretching mode of Zn-H. The main absorption band at 495cm^{-1} is due to Zn-O stretching vibrations. The O-H bending and –COO vibration bands decreasing in case of 10 and 11 pH ZnO NPs.

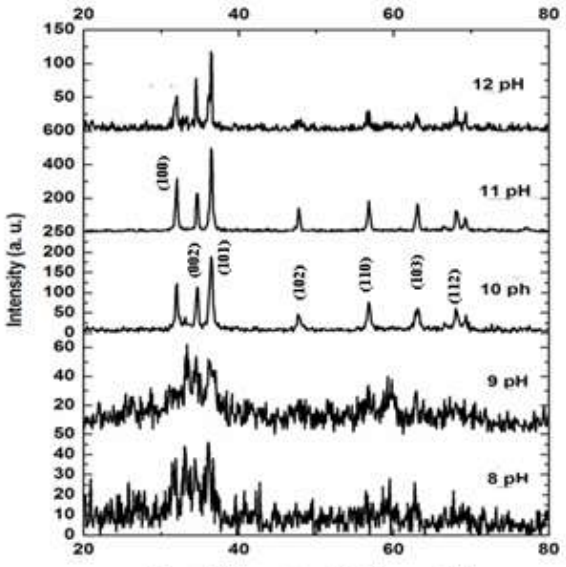

Figure 1. XRD spectra of ZnO Nano particles

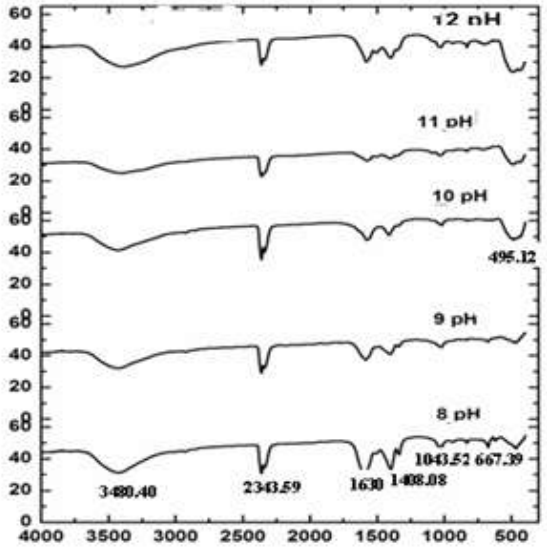

Figure 2. FTIR spectra of ZnO Nano particles

3. Surface Morphology

The morphology of the nanostructures obtained for prepared ZNO NPS by using Scanning Electon Miscroscopy tool. The obtained SEM images are as shown in Figures 3. The SEM micrographs exhibit different morphology depending upon pH of reaction mixture used in preparation of ZnO NPS. The ZnO nanoparticles prepared from reaction mixture 11 pH exhibit excellent nanorods. For pH value 10 the NPS exhibit small rod like crystals structure. All the micrographs on slight magnification exhibit nanorod structures. The different morphology of ZnO NPS leads to significant change in structural and optical properties.

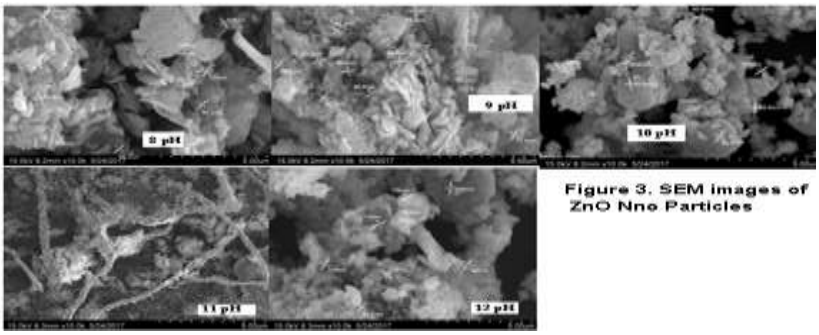

Figure 3. SEM images of ZnO Nno Particles

4. UV-Visible Absorption Study

UV- visible absorption spectra of prepared ZnO nanoparticles is presented in figure 4. The optical absorption exhibits strong absorption peak by the ZnO NPs prepared from reaction mixture of pH value 10 and 11 in the UV region at 372 and 358 nm respectively. The spectra show intense absorption at 190 to 210 nm wavelength. The 11 pH sample show higher blue shifting as compared to others. The band gap energies obtained from the spectra are tabulated in table 1 shows variation with pH values. The ZnO particles Prepared form reaction mixture of pH values10 and 11 exhibit 3.31 eV and 3.47 eV band gap.

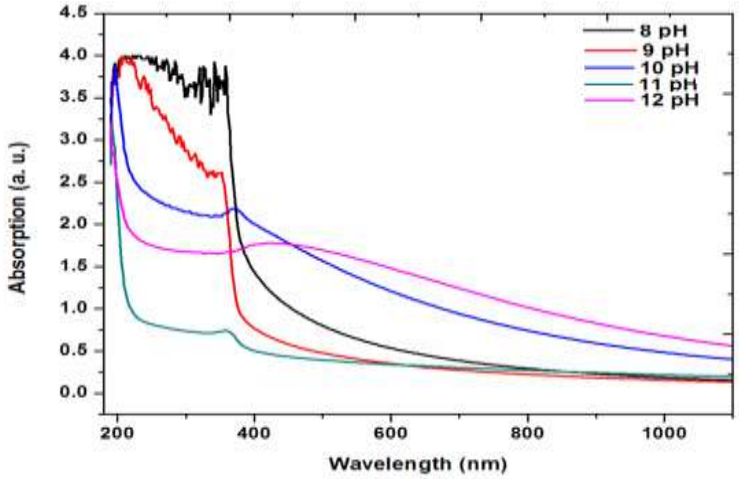

Figure4 . UV-Visible Spectra of ZnO Nno Particles

Table 1. Grain size and Band Gap.		
pH	**Grain Size (nm)**	**Band Gap Eg (eV)**
8	28	2.7
9	25	2.87
10	29	3.31
11	35	3.47
12	37	2.94

IV. CONCLUSION

In this study, ZnO nanorods of significant morphology were successfully fabricated by Sol-Gel chemical precipitation method. The XRD pattern confirmed the excellent hexagonal crystallinity of ZnO nanorods prepared from reaction mixture of pH values 10 and 11. The FTIR spectra exhibits strong absorption band at 495 cm^{-1} attributed to Zn-O stretching mode for pH values 10 and 11. The sharp optical absorption peak at 372 nm and 358 nm exhibited by ZnO(10 and 11 pH) 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 optimized pH value **11**.

ACKNOWLEDGEMENTS

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

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International Journal of Engineering Science Invention (IJESI) is UGC approved Journal with Sl. No. 3822, Journal no. 43302.

Ravangave L Influence of pH on Structure, Morphology and Uv-visible Spectra of ZnO nanorods.." International Journal of Engineering Science Invention(IJESI), vol. 6, no. 11, 2017, pp. 76-79.