Sensors & Transducers, Vol. 200, Issue 5, May 2016, pp. 45-49

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Sensors & Transducers

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Synthesis and Characterization of ZnO Nanoparticles Using Sol-gel Process

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Received: 15 April 2016 /Accepted: 16 May 2016 /Published: 31 May 2016

Abstract: In the Present work structural, morphological and compositional properties of ZnO nanopowders synthesized using Zinc nitrate and NaOH using sol-gel process were reported. The synthesized nanopowders were further analyzed using X-Ray Diffraction (XRD), Scanning electron microscopy (SEM) and Fourier Transform Infrared (FTIR) spectroscopic characterizations. Crystalline size and Lattice strain determined from XRD spectra. Morphology of Nanopowders viewed from SEM images observed at different magnifications. The presence of Functional groups analyzed from FTIR spectra. From the results it was very clear that particles synthesized using Zinc nitrate and NaOH plays a vital role on crystalline size, surface morphology of Nanopowders. Synthesized nanopowders can *be* utilized as building materials in fabrication of various optoelectronic devices including solar cells, LED's etc. due to its significant structural, morphological and optical properties. *Copyright © 2016 IFSA Publishing, S. L.*

Keywords: FTIR, Nanoparticles, SEM, XRD, Zinc oxide.

1. Introduction

Nanotechnology is an emerging field of science and technology and it has been applied to various fields right from medicine, textile and education to defense and manufacturing. The concept of miniaturizing devices to the ultimate atomic scale became dominant technological development for the last few years. Nanostructured materials are objects of intermediate size between microscopic and molecular structures. They include nanorods, nanowires, nanopores, nanosheets, nanoparticles etc. Nanoparticles considered as particular interest in applications of optoelectronic devices [1]. Among these nanomaterials, the metal oxide nanostructures have become of particular interest to scientists for the development of different optical, biochemical and biomedical nanodevices [2]. Metal oxide nanoparticles show fast electron transfer properties as they have high surface area to volume ratio, low toxicity, are environment-friendly, have chemical stability and biocompatibility [3]. They rapidly help in improving the performance of Nanomaterials. Among metal oxide Nanoparticles ZnO nanoparticles due to their wide energy band gap of 3.37 eV, biocompatibility, high electron mobility, fast electron transfer rate, environmental friendly, high melting point, these are used to fabricate sensitive and precise nanodevices based on nanomaterials for the application of sensing, optical absorption and

luminescence emission[4]. Researchers reported ZnO Nanoparticles could enhance light-trapping for solar energy technology and LED's [6], ZnO Nanostructures are considered as excellent material for fabrication of highly sensitive and selective gas sensors [7]. ZnO Nanoparticle dispersed PANI is a promising material for emissive layer in polymer light-emitting diodes [8]. ZnO nanoparticles coating with PVA is a good material for small-signal, visible blind, and wavelength selective UV detection [9].

As seen from literature various growth methods such as co-precipitation, ball milling, laser ablation, hydrothermal, Sono chemical, solid state reaction method were reported for synthesis of ZnO nanoparticles [10]. From literature it has also been observed that sol -gel method has several advantages because of low temperature (<100 °C) processing, cheap, environment- friendly etc [11]. Scientists synthesized ZnO Nanoparticles by sol-gel process.

In the present work ZnO Nanopowders were synthesized using Zinc nitrate and NaOH by Sol-gel method. These Nanopowders further characterized to determine the influence of NaOH concentration on structural, morphological, compositional and optical properties of ZnO Nanopowders.

2. Experimental Procedure

2.1. Chemical Required

Zinc Nitrate $Zn(NO_3)_2$, Sodium Hydroxide (NaOH), Ethanol, all chemicals utilized were of Analytical grade. All chemicals are used without further purification.

2.2. Experimental Procedure

The nanopowders were synthesized by using the following process. First 0.2 M aqueous solution of Zinc nitrate was prepared by dissolving zinc nitrate in 60 ml ethanol with continuous stirring using a magnetic stirrer for 2 hrs .Aqueous solution of sodium hydroxide was prepared in the similar way with continuous stirring with magnetic stirrer for 2 hrs. When the chemicals dissolved, the prepared aqueous sodium hydroxide solution is added to Zinc nitrate solution and resultant mixture so formed is kept under vigorously stirring for 3 hours till white precipitate *is* obtained within the solution [12]. The resultant precipitate *is* centrifuged and allowed to stay to digest for 24 hrs at room temperature. During this time, OH^- and NO_3^- ions were diffused through the medium and white gel-like precipitate of $Zn(OH)_2$ was formed. The remaining solution is centrifuged for 10 min and the precipitate was removed [13]. The obtained precipitate is kept in an oven around 70 ºC till the precipitate dries. During drying $Zn(OH)_2$ is completely converted into ZnO. In the final step the particle obtained was grinded to obtain powder.

2.3. Chracterization Techniques

2.3.1. X Ray Diffraction (XRD)

XRD patterns of the samples were recorded using powder X-Ray Diffractometer (XRD-SMART lab) - Rigaku, JAPAN using secondary monochromatic CuK_α radiation of wavelength λ = 0.1541 nm at 40 Kv/50mA in the scan range 2θ = 20 to 90º. Samples were supported on a glass slide. Structural properties including crystalline size, lattice strain were determined from XRD pattern.

2.3.2. Scanning Electron Microscopy (SEM)

SEM micrographs of Nanopowders were observed at different magnifications using Field Emission Scanning Electron Microscope (FESEM-SUPRA 55) - CARL ZEISS, GERMANY. A drop of nanoparticles dissolved in methanol was placed on copper grid .The micrographs gives information about morphology of nanopowders

2.3.3. Fourier Transform Infrared (FTIR) Spectra

FTIR spectra were obtained from FT-RAMAN Spectrophotometer 50-5000cm^{-1} (BRUKER RFS).Presence of functional groups resolved from spectra.

3. Results and Discussions

3.1. X-Ray Diffraction

Structural properties of nanopowders were analyzed from XRD. Fig. 1 represents XRD spectra of ZnO nanopowders. Various diffraction peaks were observed in the spectra of ZnO. Line broadening clearly represent presence of nanoparticles. In spectra diffraction peaks appeared at scattering angles (2θ) = 31.774º, 34.400º, 36.248º, 47.499º, 56.724º, 62.771º, 66.290º, 67.866º, 69.019º, 72.56º, 76.85º, 81.259º, 89.470º, 92.63º, 95.147º and 93.487º respectively. The XRD patterns of the samples reveal that all peaks correspond to the characteristic peaks of the hexagonal wurtzite structure of ZnO with space group P63mc and lattice parameters of a = b = = 0.3250 nm and c = 0.5207 nm according to the JCPDS database 36-1451. The crystalline size is calculated using Scherer's formula $D = 0.89\lambda/b \cos\theta$, where k is the X-ray wavelength (0.15406 nm), h is the Bragg diffraction angle, and b is the full width at half maximum (FWHM) of the peak diffraction is found to be 37.87 nm, No diffraction peaks from impurities and residues were detected, indicating that the synthesized products are pure ZnO nanoparticles [14]. From spectra it was also clear that synthesis procedure adopted in this work possess good control over the size of the nanoparticles.

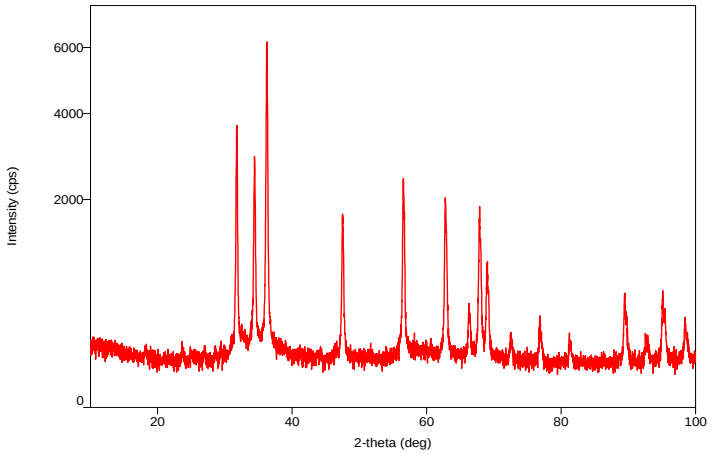

Fig. 1. XRD spectra of ZnO nanoparticles.

3.2. Scanning Electron Microscopy

Morphological properties of nanoparticles were analyzed from SEM micrographs [15]. Fig. 2 represent SEM spectra of ZnO Nanopowders observed at different magnifications .The images clearly represent formation of Nanoparticles. From images it was observed that, sample synthesized at NaOH represents the formation of monodispersed and spherical shaped nanoparticles [16]. Micrographs clearly show particles synthesized using NaOH represents zinc oxide is in pure form and particles are beautiful white colored spherical nanoparticles.

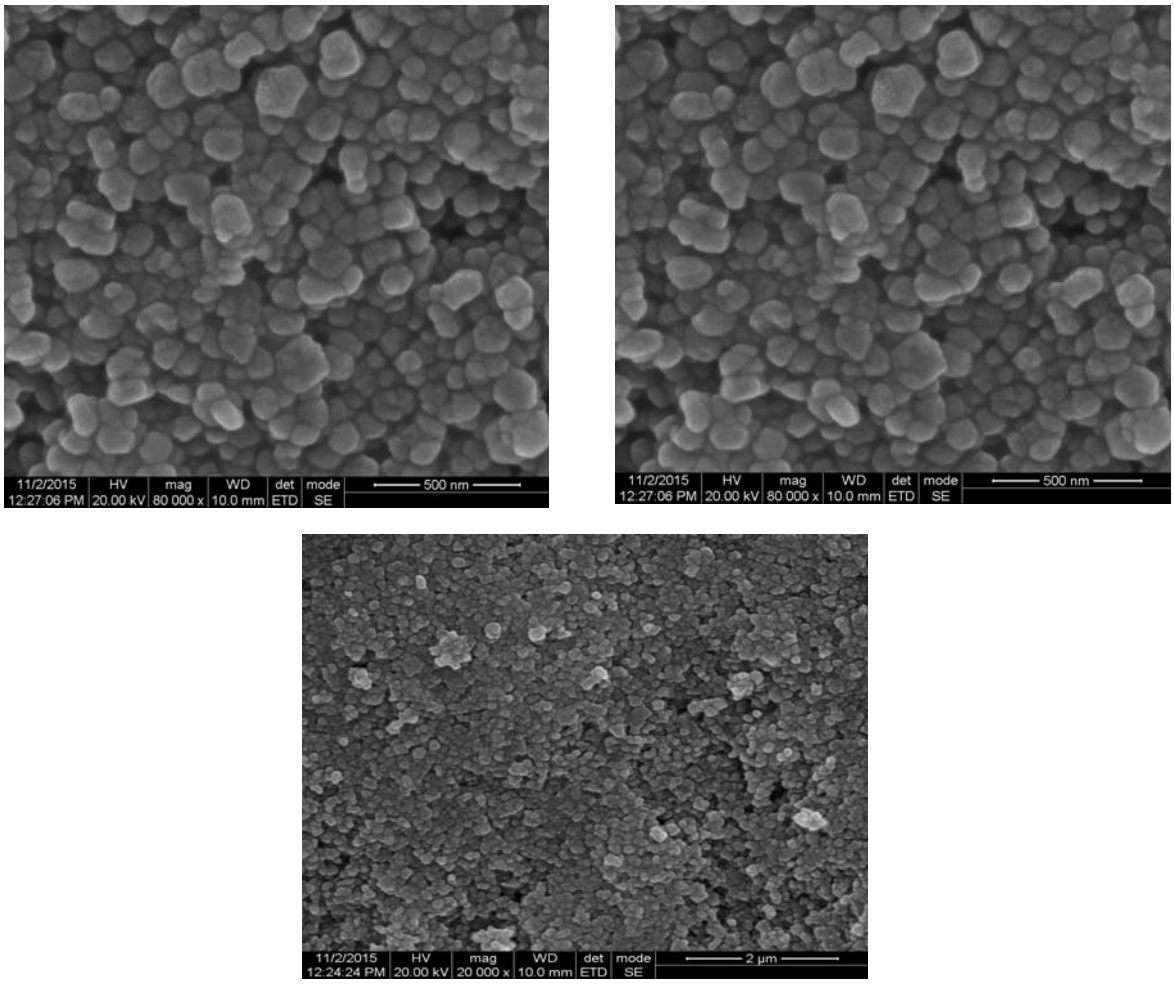

Fig. 2. SEM images of ZnO nanoparticles.

3.3. Fourier Transform Infrared Spectroscopy

Infrared spectroscopy is used to determine the presence of certain functional groups.The formation of wutzite structure was further confirmed from FTIR speatra. Fig. 3 represent FTIR spsectra of ZnO nanopowders recorded in the range 4500 - 500 cm^{-1}.

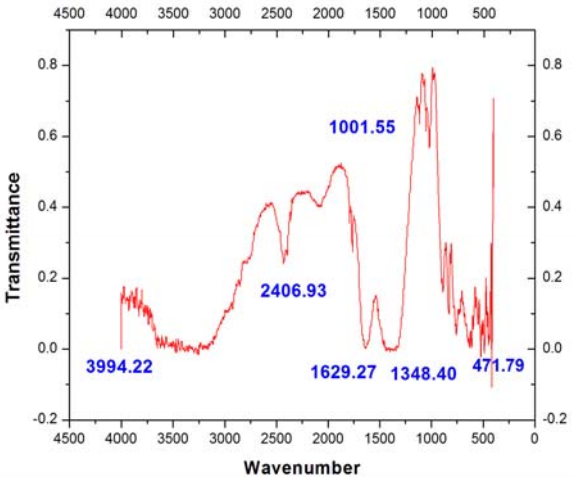

Fig. 3. FTIR spectra of ZnO nanoparticles.

Various bands were observed in the FTIR spectra. The position and number of absorption bands not only depend on crystal structure and chemical composition but also on crystal morphology.The broad band observed around 3500 cm^{-1} and 1600 cm^{-1} corresponds to O-H stretching vibration due to the absorbed water on the surface of the samples [17]. The absorption around 2500 cm^{-1} is because of the presence of CO_2 molecules in the environment. The peak around 2900 cm-1 is due to C-H band Band around 1600 cm^{-1} may be due to deformation vibration of H_2O molecule [18]. The carbonate stretches in samples are observed in samples at 1540 and 1480 cm^{-1}. The intense absorption peak at ~400 cm^{-1} is related to the stretching vibrations of Zn–O bond [19]. The results further confirm XRD results of the spectra.

4. Conclusions

In the present work ZnO nanopowders were synthesized using zinc nitrate and NaOH precursors using sol-gel method and powders were further characterized using XRD,SEM and UV-Vis optical absorption spectroscopic techniques. XRD confirms formation of wurtzite struture of ZnO with crystalline size of 37.87 nm. SEM confirms formation of nearly spherical images. The structural properties of nanopowders were further confirmed from the FTIR spectra.Various functional groups were predicted from FTIR spectra. Optical absorption reveals absence of absorption band in the visible region in all the samples. The results clearly specify fabrication method utilized in this work is simple, low cost. As synthesized nanopowders can be used in fabrication of optoelectronic devices etc due to its structural and optical properties.

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