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Synthesis and characterization of ZnO Nanoflowers

A. Anusiya[1], B. Jansi Rani[1], G. Ravi[1], R. Yuvakkumar[1 *]

[1]Nanomaterials Laboratory, Department of Physics, Alagappa University, Karaikudi, Tamil Nadu- 630 003, India.

Abstract- *ZnO nanoflowers was synthesized by facile hydrothermal route .The structural, morphological and optical properties of ZnO nanoflowers were investigated by X-ray diffractometer (XRD), Scanning Electron Microscopy (SEM), Raman Spectroscopy, Photoluminescence (PL) and Fourier transform infrared spectroscopy (FTIR). XRD pattern of the sample confirmed the hexagonal phase of ZnO nanoflower. SEM images clearly exaggerated the nanoflower morphology consists of functionalized nanosheets. The Raman spectra located at 435 and 479 cm^{-1} correspond to E_{2H} and interfacial surface phonon modes of hexagonal ZnO. PL results gave the information of oxygen defects present in the samples. The absorption band observed from FTIR at ~410 cm^{-1} confirmed the Zn-O (metal oxygen) vibration.*

Keywords- *ZnO nanoflowers; SEM; FTIR.*

I. INTRODUCTION

Zinc oxide (ZnO), a wide direct band-gap (3.37 ev) semiconductor with a large excitation binding energy of 60 meV is known to be one of the most important functional oxides with near UV emission, transparent conductivity and piezoelectricity [1-4]. ZnO is widely used in light emitting diode, photocatalytic performance and biological nanosensor because of its wide band gap [5]. Due to the high electron mobility, tunable magnetic properties and transparency, ZnO has become a key material for applications in potential areas including laser diodes, solar cells, gas sensors, dilute magnetic semiconductors, spintronics and optoelectronic devices [6-9]. Several techniques have been proposed to prepare doped ZnO including the sol-gel method, metal organic chemical vapor deposition, spray pyrolysis, RF magnetron sputtering, DC magnetron sputtering, medium- frequency magnetron sputtering, pulsed laser deposition, hydrothermal and co- precipitation method. In this work, ZnO nanoflowers prepared by hydrothermal route because this method was simple, eco-friendly and economically cheap to produce the nanoparticles when compared to other methods. Recently many researchers are working on ZnO nanostructures for various novel applications. Al-Gaashani and co- workers were investigated the development of microwave susceptors based on SiC composites and their application for a one-step synthesis of ZnO nanostructures [10]. Vishnukumar and colleagues were worked on recent advances and emerging opportunities in phytochemical synthesis of ZnO nanostructures [11]. Sze- Mun lam and labmates revealed the influence of PVP surfactant on the morphology and properties of ZnO micro/nanoflowers for dye mixtures and textile waste water degradation [12]. Jian-Ai Quek and group reported mechanistic investigation of visible light responsive Ag/ZnO micro/ nanoflowers for enhanced photocatalytic performance and antibacterial activity [13]. Sanjeevk.Sharma and his team were investigated synthesis and characterization of hybrid Ag-ZnO nanocomposite for the application of sensor selectivity [14]. In this work, we synthesized 3D ZnO hierarchical well functionalized nanoflowers by simple hydrothermal route and the physical properties have been explored by XRD, Raman, SEM, PL and FTIR studies.

II. MATERIALS AND METHODS

Zinc nitrate ($Zn(NO_3)_2.6H_2O$),Urea (CH_4N_2O), PVP, deionized water and ethanol were procured from Sigma Aldrich. All the chemicals were analytical grade and were used without further purification. To synthesis ZnO nanostructures, initially 0.1 M ($Zn(NO_3)_2.6H_2O$) + 0.1 g PVP were dissolved in 100 ml deionized water under stirring. Then 0.2 M of Urea solution was added drop wise into the precursor solution. The solutions were transferred into the Teflon lined stainless steel autoclave with 250 ml capacity and the temperature Maintained at 150^{o}C for 12 hours. After it cooled down to room temperature, it was then filtered and washed with distilled water and ethanol several times. The filtered particles were dried at 80^{o}C and grained using mortar with pestle. The white color powder was then annealed at 400^{o}C for 2 hours in muffle furnace. The nanoparticles were collected for further characterization such as X-ray powder diffraction (X'pert PRO analytical diffractometer) using CuKα as radiation (1.541 A˚) source was carried out for structural characterization. Imaging Spectrograph STR 500 nm focal length laser micro Raman spectrometer SEKI, Japan with resolution: 1/0.6 cm-1/pixel and Flat Field: 27 mm (W) × 14 mm (H) was used for Raman analysis. Photoluminescence (PL) spectrum was investigated using Varian Cary Eclipse Photoluminescence Spectrometer with Oxford low temperature LN2 77K setup. Infrared (IR) spectra were observed using Fourier transform infrared spectrophotometer using Thermo Nicolet 380 with resolution 0.5 cm^{-1} and

S/N ratio: 2000:1 ppm for 1 minute scan. The morphological studies were examined by Scanning Electron Microscope (ZEISS-V80).

III. RESULTS AND DISCUSSION

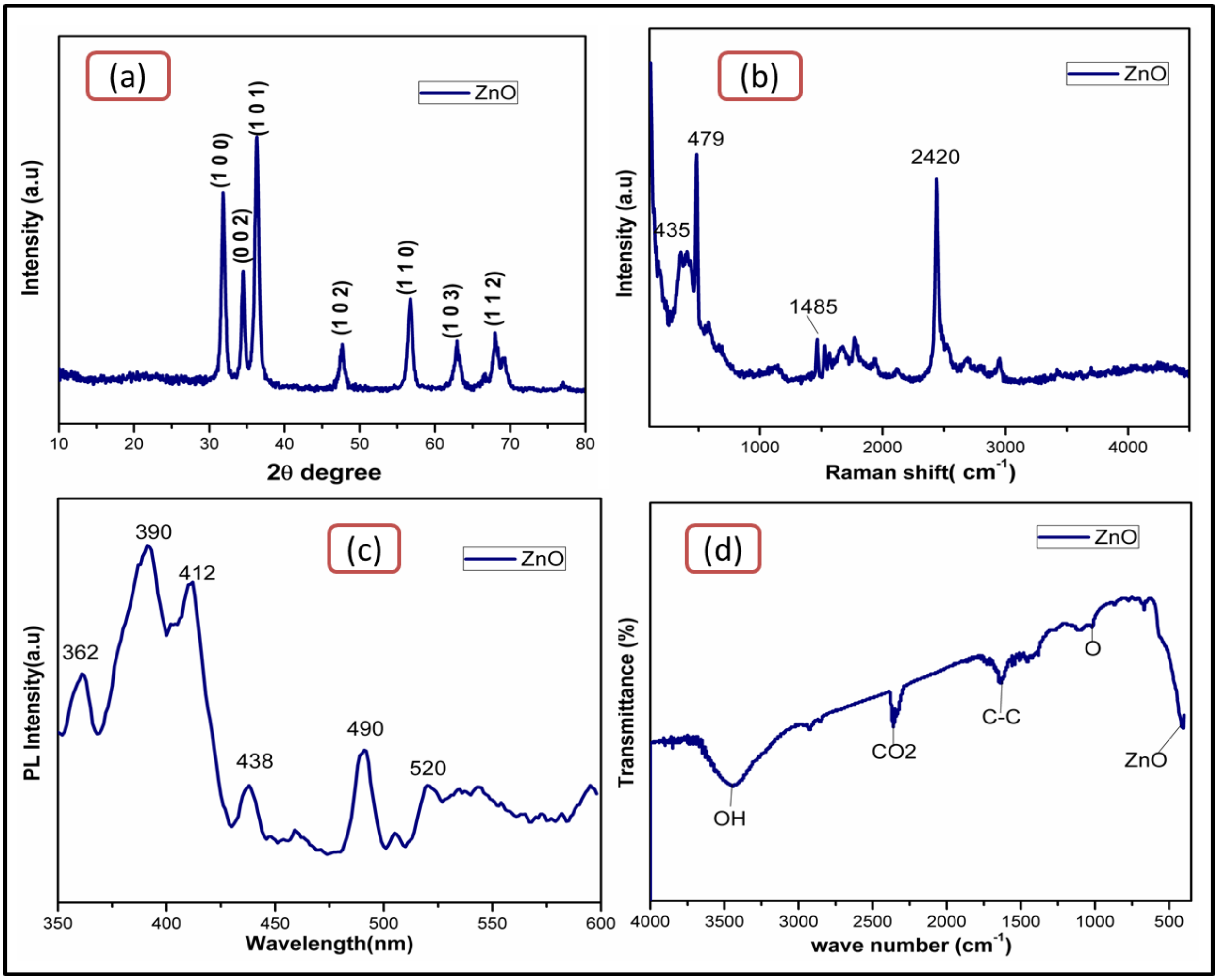

Figure 1(a). XRD, (b) RAMAN, (c) PL and (d) FTIR of ZnO nanostrucrures.

Figure 1 (a) shows the XRD spectra of ZnO nanostructures. The diffracttion peaks located at 2θ values of 31, 34, 36, 47, 56, 62 and 67^{o} corresponds to the crystal planes (100), (002), (101), (102), (110), (103) and (112) respectively which were completely coincided with the standard hexagonal ZnO of JCPDS card number 89-1397. The average crystallite size was calculated using Debye-Scherrer formula: $D = 0.9\lambda/\beta\cos\theta$ where λ is X-ray wavelength, β is full width at half maximum intensity, and θ is Bragg's angle. The average crystallite size of ZnO sample is 21.93 nm. Figure 1 (b) shows the Raman spectra of ZnO nanostructures. The Raman peaks in ZnO sample positioned at about 435, 479 and 1460 are assigned to E_{2H}, interfacial surface phonon modes and C-O symmetric stretching respectively [15-16]. Figure 1 (c) shows that the photoluminescence spectra of ZnO nanostructures at excitation wavelength 350 nm. All the samples exhibited strong emission in UV region with a band centered at 390 nm. The two high intense emission peaks located at 490 and 520 nm attributed to blue-green emissions were also reported by literature for ZnO nanostructures [17]. The weak emission at about 570 nm can be assigned to surface states or oxygen vacancies. Figure 1 (d) shows the FTIR spectra of ZnO. The FTIR spectra of ZnO nanostructures were recorded from 300 to 4000 cm^{-1}. The spectral bands at 460 cm^{-1} clearly shows the presence of ZnO approximately same to the reported literature [18-23]. The band at 1060 cm^{-1} is due to the oxygen stretching and bending frequency [19]. Stretching modes of C-C and C=O are observed at 1630 cm^{-1}. The peaks located at 2350 cm^{1} are due to the atmospheric CO_2. Band at 3440 cm^{-1} indicated the presence of OH group in the synthesized ZnO nanoflowers.

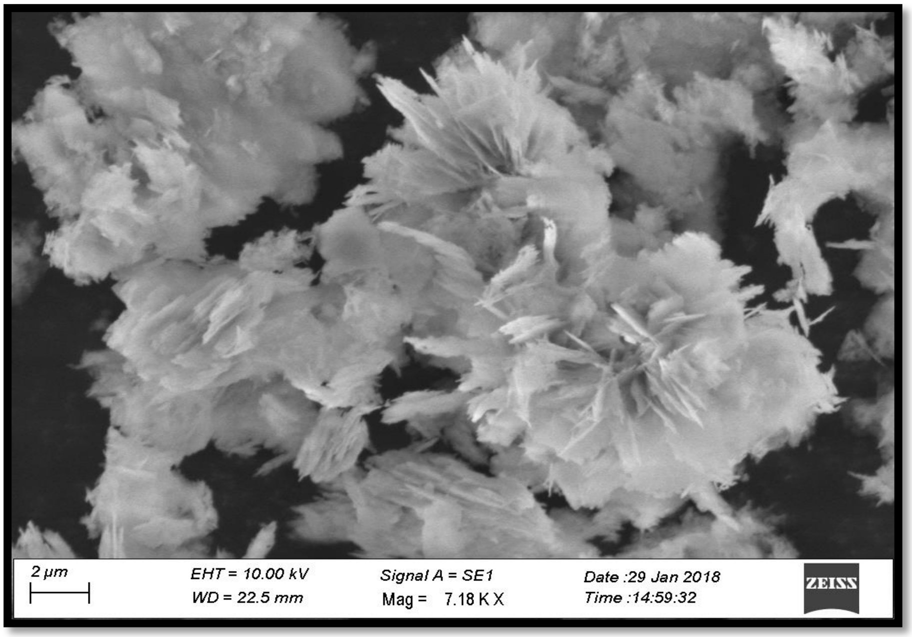

Figure 2. SEM image at 2 µm scale of ZnO nanostructures.

Fig.2 shows the SEM micrograph of ZnO hierarchical nanoflowers. The presented SEM result gives the surface morphological information of ZnO hierarchical nanoflowers. It clearly exaggerated the formation of hierarchical nanoflower consists of more funtionalized nanosheets together. The optimum pressure and temperature condition maintained during the synthesis protocol results well defined, uniformly distributed, strongly functionalized typical 3D ZnO hierarchical nanoflowers.

IV. CONCLUSIONS

3D hierarchical ZnO nanoflowers were synthesized by simple hydrothermal route successfully. Physical and optical properties of synthesized 3D ZnO nanoflowers were investigated by XRD, Raman, PL and FTIR. The morphological evaluation of synthesized 3D hierarchical ZnO nanoflowers consist of functionalized nanosheets surface was examined through SEM images. The controlled synthesis with optimum temperature and pressure condition favors to form 3D hierarchical ZnO nanoflowers for various renewable energy applications.

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