

Microwave assisted hydrothermally synthesized nanostructure zinc oxide reinforced polyaniline nanocomposites

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Received: 26 April 2011, Revised: 22 July 2011 and Accepted: 26 July 2011

ABSTRACT

ZnO in different nanostructures were synthesized by microwave assisted hydrothermal route. Different experimental conditions such as microwave irradiation power, exposure time have been investigated to reveal the process of formation of the ZnO nanostructures. It was revealed that the microwave exposure time plays a vital role in determining the diameter of the rods. The interaction of microwaves with the growth units of ZnO was systematically investigated to explain formation of different structural geometry of ZnO on nanoscale. ZnO nanostructures consisted of flower-like, sword-like, needle-like and rods-like structures were prepared by microwave assisted hydrothermal process at different conditions of microwave power and irradiation time. The ZnO nanostructures are in hexagonal phase. It is considered that microwave can interact with growth units of ZnO to generate active centers on the surface of ZnO nuclei so that needle-like ZnO rods are created on those sites, resulting in the formation of the flower-like ZnO nanostructures. Polyaniline - ZnO nanocomposites (PZ) in various weight % of nanostructure ZnO were synthesized by the chemical oxidation method in sulphuric acid medium using ammonium persulphate as oxidant at 276K. The synthesized polymer nanocomposites were characterized by XRD, FTIR and UV-VIS spectroscopy. Copyright © 2011 VBRI press.

Keywords: Conducting polymer; polyaniline; nanocomposites; zinc oxide; microwave assisted hydrothermal.



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papers in various reputed international journals.

Introduction

Intrinsically conducting polymers have aroused a great interest among the researchers due to high mobility of the charge carriers and as excellent hosts for the trapping of metals and semiconducting nanoparticles [1]. The dispersion of nanoscale inorganic fillers into an organic conducting polymer creates a new, economic way to obtain desired high performance conducting polymer nanocomposites. The influence of the nanoscale fillers on the structure and properties of polymer are the crucial clue to produce novel nanocomposites for multifunctional applications [2]. The inorganic fillers at nanoscale exhibit high surface to volume ratio and thus expected to modify drastically the electrical, optical, thermal and dielectric properties of polymer. Amongst the various conducting polymers, polyaniline (PANI) is a most studied polymer because of its relative ease in preparation, good environmental stability and tuneable conductivity [3]. PANI and its composites have a wide range of potential technological applications including storage batteries, electrochemical devices, light emitting diode, corrosion inhibitor and variety of chemical sensor and bio-sensors [4]. Various methods have been widely used for the synthesis of the nanocomposites of PANI with the inorganic nanoparticles such as TiO_2 , ZnO, CdS, Pd and Au. [5, 6]. Since the conducting polyaniline and inorganic semiconducting material ZnO in nanoform both are having a wide range of technological applications, we got motivated to make composite of PANI and ZnO and believed to get novel properties resulting from the molecular level interaction of these two dissimilar chemical components.

Among the nanocrystalline inorganic semiconductors, nanostructure ZnO has been currently attracted worldwide interest as a representative II–VI group compound semiconductor with a wide direct band gap (3.37 eV) and a large exciton binding energy (60 meV) and become excellent electronic and photonic material and exhibits a wide range of technological applications [7, 8]. Various chemical and physical methods have been reported for the preparation of the ZnO nanostructures with various sizes and morphologies, including chemical vapour deposition [9], physical vapour deposition [10], vapour-liquid solid assisted method [11], chemical solution deposition method [12], pulsed laser deposition [13], electrochemical deposition [14], hydrothermal synthesis [15], microwave assisted synthesis [16], spray pyrolysis techniques [17] and thermal evaporation [18] etc. Well defined ZnO nanostructure with different shapes have been successfully synthesized and reported in the literature, such as sharp needle-shaped tetrapods and coexistent tetrapods and nanowires [19, 20], nanorods [21], nanowhiskers [22], nanobelts [23], nanosheets [24], nanotriangles [25], nanotubes [26], nanoprism [27], nanonails [28] and nanosphere [29]. For commercial use of ZnO nanostructures, a simple and inexpensive synthesis process is required necessarily. Microwave synthesis is known for homogenous volumetric heating, and high reaction rate compared with other physical and chemical methods. For this purpose, we have synthesized ZnO nanorods by microwave assisted hydrothermal route. Chitosan graft with

styrene was successfully synthesized using microwave irradiation [30].

Polymer nanocomposites with this versatile inorganic ZnO exhibit some novel properties such as improved electrical conductivity, improved thermal stability etc. Chauhan and Yunus have prepared a novel organic/inorganic composite [31]. Byun and Im have studied the degradation kinetics of polyaniline-nylon composite film [32]. Fulsalba and his co-workers studied the thermal properties of polyaniline-molybdenum trisulfide composites [33]. Bhat and Vivekchand reported the synthesis of polyaniline-CdS/ZnO nanocrystal composites [34]. Yao-Yi Cheng and Shang-Chih Chou successfully synthesized ZnO nanorods on a PEDOT co-PANI copolymer film using an aqueous solution method [35]. Khan and Khalid synthesized nano-sized ZnO and PANI-ZnO composites [36]. I-V characteristics of the p-n junction between vertically aligned ZnO nanorods and polyaniline (PANI) thin film were investigated by Yinhu Li and Jian Gong [37]. This paper therefore, deals to explore the synthesis of nanostructure ZnO by simple microwave assisted hydrothermal process and PANI-ZnO nanocomposites by simple chemical oxidation polymerization and to study the nanostructure ZnO reinforced polyaniline composites using XRD, UV-VIS, FTIR, SEM and TEM analysis.

Experimental

Materials and methods

Aniline (99.5%) and hydrated zinc nitrate ($\text{Zn}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$, 99.5%) were procured from E. Merck. Aniline was distilled prior to use. All supplementary chemicals (ammonium persulfate, NaOH, HCl, acetone, ethanol) were of analytical grade and solutions were prepared with double distilled water. FTIR characterization was done using a Shimadzu FTIR-8101A spectrophotometer via making pellet with KBr at 8 ton pressure. The XRD measurement was performed on a Philips PW1710 automatic X-ray diffractometer using $\text{Cu-K}\alpha$ wavelength ($\lambda=1.54059 \text{ \AA}$). UV-visible study was carried out on a UV-1800 Shimadzu double beam spectrophotometer. SEM images were taken on JEOL JSM-6360 analytical scanning electron microscopes and the transmission electron microscopy (TEM) was carried out using a Hitachi H-7000 operated at 100 kV and 30 μA .

Synthesis of nanostructure zinc oxide

Nanostructure zinc oxide was prepared by microwave assisted hydrothermal routes. 0.5 g of $\text{Zn}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ was put into 100 ml deionised water under continuous stirring. After 10 min stirring, 10 ml of 2M NaOH aqueous solution was introduced drop wise into the solution under stirring, resulting in a white aqueous solution, which was then transferred into Teflon-line stainless steel autoclave. The autoclave was put into silica carbide chamber introduced in microwave irradiation system. Microwave is having a chamber of 360 mm \times 210 mm \times 430 mm dimensions with a 2.45 GHz frequency multimode source and maximum deliverable power output 700 W. We prepared the samples at different conditions of microwave power and irradiation

time. The reaction products in the form of a precipitate were filtered and washed with distilled water and ethanol to remove the ions possibly remaining in the final products. Finally the products were dried in hot air oven at 80°C. The final white products were collected for characterization and preparation of nanocomposites in polyaniline matrix.

Synthesis of polyaniline-ZnO nanocomposites

The polymerization of monomer (aniline) was initiated by the drop wise addition of oxidizing agent (ammonium persulphate) in acidified solution (HCl) prepared by using doubly distilled water under constant stirring at 0–4°C. During this stirring prepared zinc oxide (ZnO) in different weight percentage (5%, 10%, 15%, and 20%) with aniline was added for synthesizing the composites. The monomer to oxidizing agent ratio was kept at 1:1. After complete addition of the oxidizing agent the reaction mixture was kept under constant stirring for 4 hrs. Precipitated polymer was filtered and washed with distilled water and ethanol until the filtrate was colourless. Finally the polymer composite was dried in a vacuum oven at 80°C for 12 hrs. In this way four polymer composites PZ-1, PZ-2 PZ-3 and PZ-4 have been synthesized.

Results and discussion

Fig. 1 shows XRD pattern of nanostructured ZnO prepared on 240 W microwave irradiation powers, 25 min exposure time by microwave assisted hydrothermal method. The diffraction peaks of synthesized ZnO have been indexed to the hexagonally wurtzite structured ZnO with lattice constant, $a = 3.289$ and $c = 5.149$ Å, which were well matched with that in JCPDS, 36-1451. It can be clearly seen from the XRD patterns of ZnO that the high intensity and half width of the XRD pattern indicate that ZnO is crystallized well with particle size in nano-scale [6].

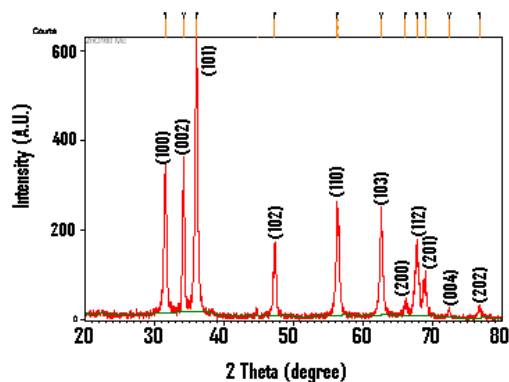


Fig. 1. XRD pattern of synthesized ZnO.

Fig. 2 shows the TEM image of the ZnO nanostructure. The rod-like structure having diameter in nano range can be observed from TEM. The rods may be a part of flower-like geometry of ZnO. The TEM depicts round shape nanoparticles having average size less than 100 nm. **Fig. 3** shows the SEM images of the ZnO nanostructures. The typical ZnO nanostructures consisted of the ZnO flower-like, sword-like, needle-like nanorods with 50 – 100 nm in width and several micrometers in length. **Fig. 4(a)** and **(b)** show the UV–vis absorption spectra of the as-prepared

ZnO nanostructure obtained at different microwave irradiation times and powers, respectively.

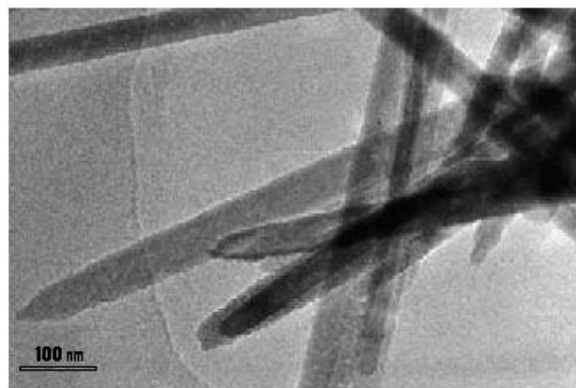


Fig. 2. TEM image of synthesized ZnO.

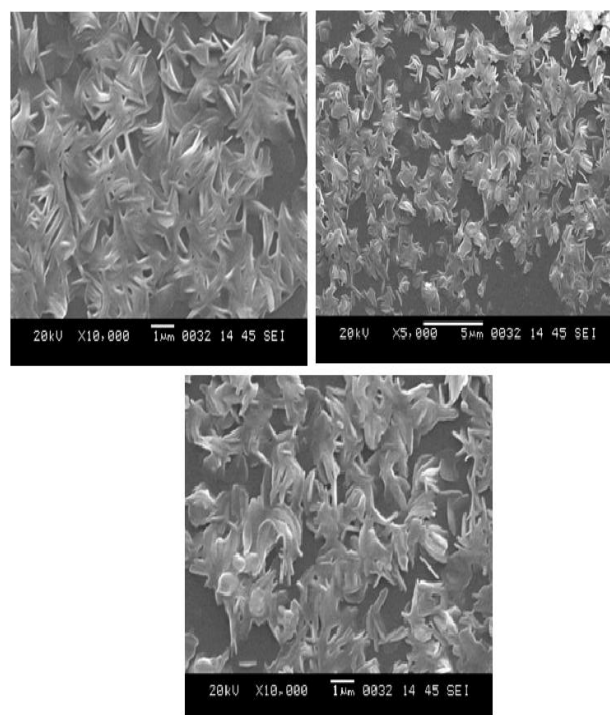


Fig. 3. SEM images of ZnO at different magnification.

From **Fig. 4**, it can be noticed that the absorption peaks are blue shifted by about 25–40nm as compared to the value (380 nm) expected for the bulk ZnO material [34]. This blue shift can be attributed to the quantum confinement effects within the nanorods. With increasing irradiation time, the peak gradually shifts from 335 to 355 nm. Longer irradiation time gives rise to an increase in the radial dimension of the rods. This could be attributed to an Ostwald ripening process. Such a process of widening of the rods is quite possible since there is no capping. The effect of irradiation power on the absorption spectra of the samples is shown in **fig. 4(b)**. Interestingly, it has only affected the total absorption, but does not alter the position of the absorption peak. Thus the size of ZnO nanorods was significantly influenced by microwave irradiation time but not by the power, i.e. the extent of growth is determined by the reaction time, but is independent of applied power in

the given range. It is considered that microwave can interact with growth units of ZnO to generate active centers on the surface of ZnO nuclei so that needle-like ZnO rods are created on those sites, resulting in the formation of the flower-like ZnO nanostructures.

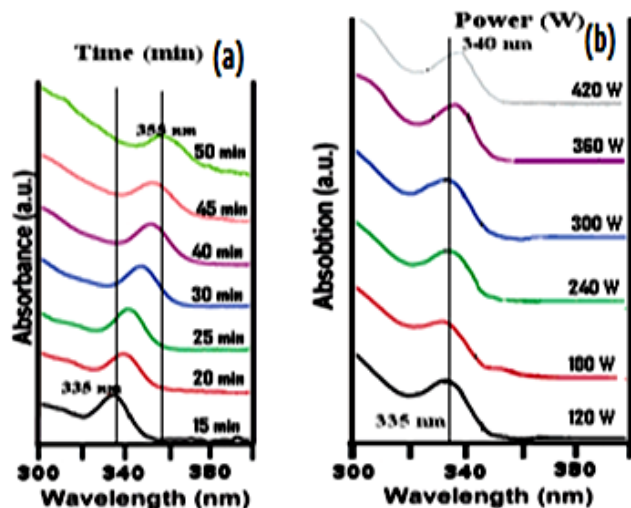


Fig. 4. UV-visible spectra of pure ZnO nanostructures synthesized (a) at different exposure time for microwave power 250 W and (b) at different power for microwave exposure time 25 min.

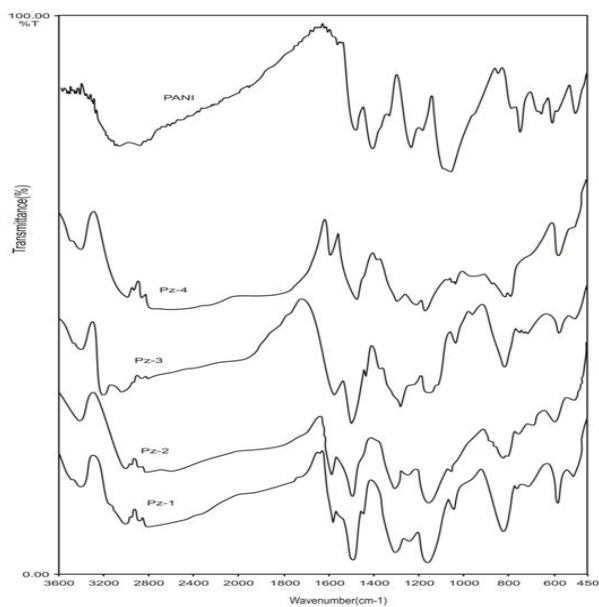


Fig. 5. FTIR spectra of pure PANI and nanocomposites.

FTIR spectra of pure PANI and the composites of PANI-ZnO in weight percentage 5%, 10%, 15%, 20% are shown in **fig. 5**. The spectra of nanocomposites exhibit several characteristics peaks at 3450, 1600, 1500, 1491, 1307, 1155, 826 cm^{-1} . These peaks were attributed to the N-H stretching of secondary amine, benzenoid ring, C-N stretching band vibrating of the dopant anion and paradisubstituted benzene ring respectively. The decrease in peak area with respect to polyaniline indicates the structural change in the polymer due to doping [38]. Since the characterisation peaks of polyaniline and ZnO are seen in composites PZ-1, PZ-2, PZ-3 and PZ-4 spectra, this indicates that the polyaniline/ZnO composites were successfully synthesized.

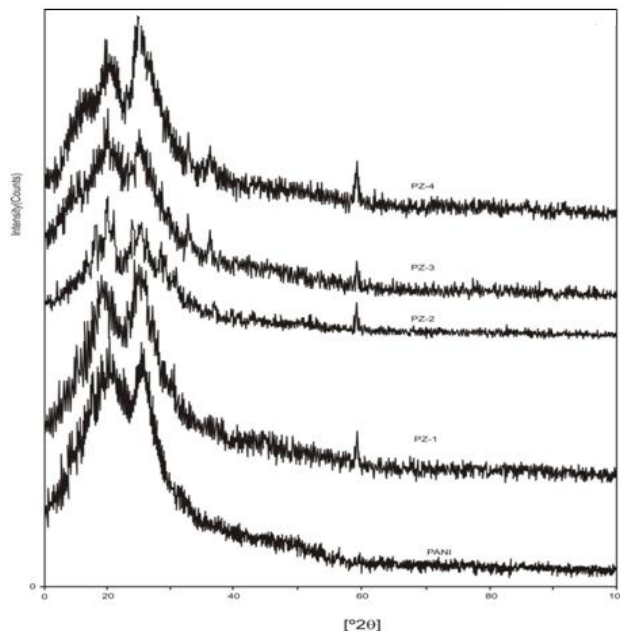


Fig. 6. XRD pattern of pure PANI and nanocomposites.

Fig. 6 shows the XRD of pure PANI and PANI-ZnO nanocomposites. It has been suggested by XRD study that PANI undergoes interfacial interaction with ZnO crystallites and loses its own morphology by its mixing with ZnO. PANI-ZnO nanocomposites show peaks of ZnO as well as polyaniline indicating that ZnO crystallites have been uniformly mixed within the polymer chain.

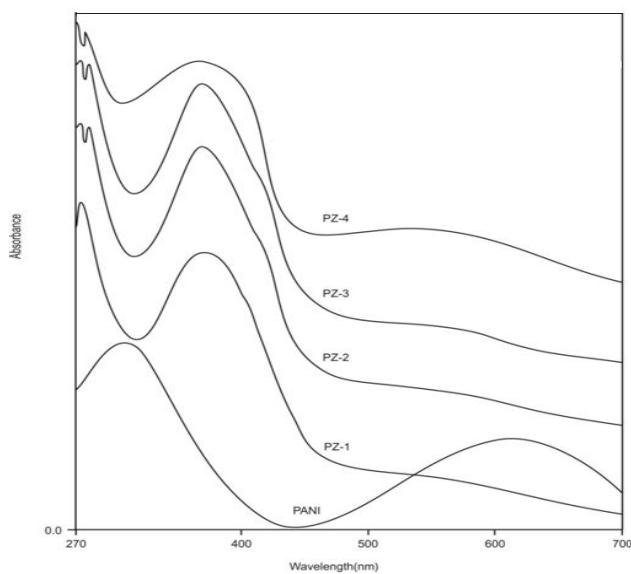


Fig. 7. UV-visible spectra of pure PANI and nanocomposites.

The UV-visible spectra of pure PANI and PANI-ZnO nanocomposites are shown in **Fig. 7**. Spectra of pure PANI shows two absorption bands around at 380 nm and 630 nm corresponds to $\pi-\pi^*$ transition in UV and bipolaronic band in visible. As the weight percentage of ZnO increases in PANI, a band around 380-390 nm corresponding to polaron- π^* transition is created in the band gap of polyaniline by

substitution of ZnO [39] may be responsible for better electrical conductivity of these nanocomposites.

Conclusion

Flower-like, sword-like, needle-like and rod-like ZnO nanostructures were prepared by microwave assisted hydrothermal process at different conditions of microwave power and irradiation time. The microwave exposure time plays a vital role in determining the diameter of the nanorods. The nanostructures are in hexagonal phase. XRD and FTIR of PANI-ZnO nanocomposites revealed that PANI undergoes interfacial interaction with ZnO crystallites which are embedded in polymer matrix. Polaronic band formation in the nanocomposites is responsible for good electrical conductivity as suggested from UV-VIS spectra.

Acknowledgements

The authors gratefully acknowledge UGC, New Delhi (India) for financial assistance provided to carry out this work through major research project (F No.33-7/2007 -SR).

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