

SYNTHESIS AND CHARACTERIZATION OF ZnO-PVA NANOPOLYMER

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Abstract : ZnO-PVA nanopolymer were successfully synthesized by chemical method at 60 °C. The prepared nanopolymer is characterized by structural and thermal techniques. From XRD and FT-IR techniques reveal structural related information and functional groups present in the prepared nanopolymer. TG-DTA exhibited the thermal decomposition of ZnO-PVA nanopolymer at different temperatures with relevant weight loss.

Keywords: Nanopolymers, XRD, Texture coefficient, ZnO-PVA and TG-DTA.

1. Introduction

Semiconductor oxide materials are of the great interest for the development of smart and functional devices and systems. The most studied compound in II-VI group semiconductors in metal oxide family is Zinc oxide (ZnO). Among all the II-VI compounds, ZnO is good photo-catalytic activity, high stability and non-toxicity chemically more stable. ZnO is a suitable semiconductor host for a variety of dopants on account of its wide energy band gap (3.37 eV at 300 K) and a large exciton binding energy (60 meV) [1]. ZnO is also investigated as an antibacterial agent in both micro scale and nanoscale formulations. It has been a promising material UV light emitting-diodes, nanosensors, flexible and lightweight optoelectronic devices, computerized gadgets and solar cells [2]

Polyvinyl alcohol (PVA) is a high chemical resistance, water soluble, transparent, biodegradable polymer and easily interacts with other organic and inorganic materials. It has many technological, biomedical and pharmaceutical applications. [3-5]. The PVA works as a stable host material for ZnO nanopolymers because the PVA has excellent distribution of ligand radicals on the side-chain with winding mesh structure. PVA can supply hydroxyl groups on the surface of nanofibers, which can also serve to adsorb heavy metal ions [6]. Solution based techniques are very powerful and low cost and can be regarded as promising synthetic means for the development of active materials. Technological point of view for the preparation of nanoparticles is a very skill full and challenging job. The chemical method is attracting increased interests because of its high purity, low temperature, high interfacial adhesion, uniformity, homogeneity, and nanostructural features of films. The aim of the work present is to synthesis and characterization of ZnO-PVA nanopolymers using simple and inexpensive chemical route and investigate their structural and thermal properties.

2 Experimental

2.1 Materials

All the chemicals used were of analytical reagent (AR) grade, Sodium hydroxide (NaOH), Zinc nitrate hexahydrate ($\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$) and Poly Vinyl Alcohol (CH_2CHOH) were used as starting materials and used without further purification. Double distilled water was used as solvent in the experiment.

2.2 Preparation of ZnO-PVA nanopolymer

ZnO nanopolymer was prepared by adding 25 mL of 0.1 M NaOH drop wise into 25 mL of 0.05 M zinc nitrate hexahydrate ($\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$) is warmed up to 60 °C stirred for 2 hours. Then after 1g of PVA is added to the above solution under continuous stirring of 4 hours until viscous transparent solution is obtained. The prepared solution is poured into Petri dish and left for 48 hours at room temperature. After solvent evaporation a thin film containing ZnO-PVA nanopolymer was obtained. The prepared polymer is ready for characterized by XRD, FT-IR and Thermal gravimetric analysis etc.

2.3 Characterization Techniques

In the present investigation X-ray diffraction pattern of prepared nanopolymer is recorded on Shimadzu XRD-6100 diffractometer with $\text{CuK}\alpha$ radiation (1.5406 Å). Shimadzu IR Affinity 1S Spectrophotometer in the region 650-4000 cm^{-1} using HATR accessories is used for the FT-IR spectrum. Thermal gravimetric analysis of nanopolymer is performed on Shimadzu DTG-60H recorded at 10 °C per min from room temperature to 600 °C.

3 Results and Discussion

3.1 XRD Studies

X-ray diffraction pattern of the ZnO-PVA nanopolymer is shown in Fig. 1. The diffraction peaks can be indexed to the cubic phase of ZnO which is well matched with standard JCPDS card No: 77-0191 and corresponding space group is $\text{Fm}\bar{3}\text{m}$ (225). The peak observed at $2\theta = 19.45^\circ$ indicate the PVA and corresponding d-spacing value is 4.56 Å [7]. The strong, sharp maximum intensity peak is observed at $2\theta = 43.89^\circ$ and other peaks are less intensities. The strong and sharp diffraction peak reveals the high crystallinity of the prepared ZnO-PVA nanopolymer. The average crystallite size (D) of ZnO-PVA nanopolymer can be calculated by using Debye Scherrer's formula [8].

$$D = K\lambda / \beta \cos\theta$$

where K is a constant (shape factor, about 0.9), λ is the wavelength of X-ray radiation (1.5406 Å), β is the full width at half maximum (FWHM) intensity of the diffraction line and θ is the diffraction angle. Based on FWHM intensity, the average crystallite size is around 87 nm. The lattice cell parameter of ZnO-PVA nanopolymer is $a = 4.227$ Å and the volume $V = 75.53$ (Å)³. The dislocation density (δ) can be calculated by by using this equation $\delta = 1/D^2$ [8] and estimated value is 0.0132×10^{16} lines/ m^2 and microstrain of the prepared nanopolymer can be calculated by using this equation $\varepsilon = \beta \cos\theta / 4$ [8] estimated value is 0.3979×10^{-3} . The X-ray diffraction patterns also reveal when the degree of orientation of different planes is not similar. In order to investigate the possibility of a preferred orientation, the Harris analysis was performed [9],

$$\text{TC}_{(hkl)} = \frac{\frac{I(hkl)}{I_o(hkl)}}{\left(\frac{1}{N}\right) \sum \left(\frac{I(hkl)}{I_o(hkl)}\right)}$$

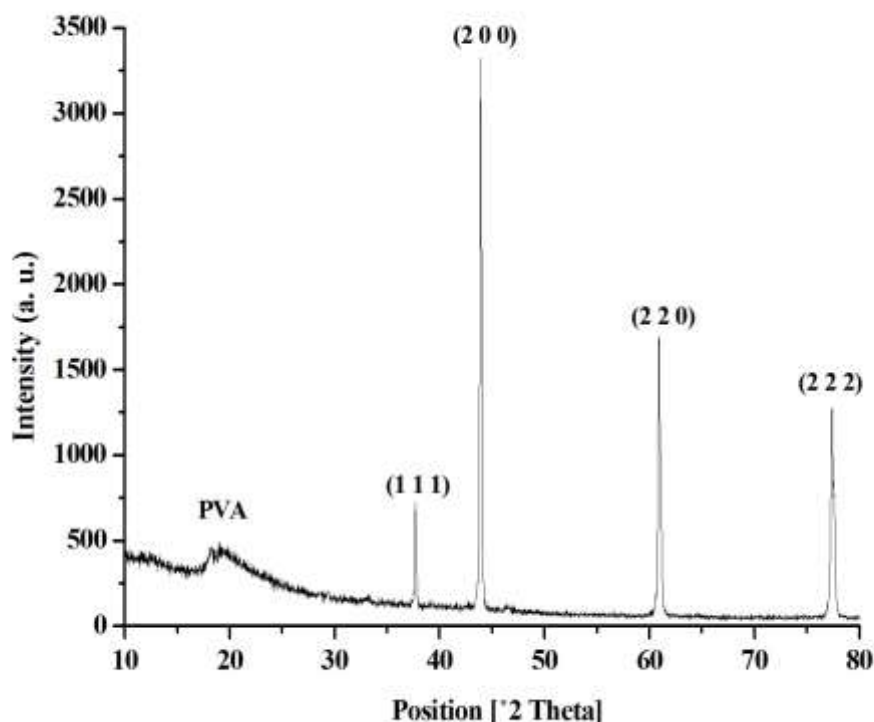


Fig. 1. X-ray diffraction pattern of ZnO-PVA nanopolymer

where $I(hkl)$ is the measured intensity, $I_0(hkl)$ is the standard intensity of the XRD reference of the randomly oriented grains and N is the number of diffraction peaks. The calculated value of texture coefficient is 1 indicate the well-crystalline nature of ZnO-PVA nanopolymer.

3.2 FT-IR studies

Fig. 2. shows typical FT-IR spectra of ZnO- PVA nanopolymer recorded in the range of $650\text{--}4000\text{ cm}^{-1}$. The band at 3316 cm^{-1} is due to --OH group in the polymer backbone [10]. The band at 2933 cm^{-1} is due to the CH_2 groups. The peak at 1713 cm^{-1} is due to the C=O vibrational bands. The observed band at 1090 cm^{-1} is assigned to the $\nu(\text{C--O})$ stretching vibration of the ether group [11]. The band at 940 cm^{-1} result from CH_2 rocking vibration and the band at 850 cm^{-1} is due to the C--C stretching vibrations of PVA [12].

3.3 TG-DTA studies

ZnO-PVA nanopolymer was performed at a heating rate of $10\text{ }^\circ\text{C min}^{-1}$ under nitrogen atmosphere, the possible gravimetric and thermal changes were investigated by TGA and DTA. Fig. 3. Shows the thermal decomposition process of ZnO-PVA nanopolymer. The TGA curve exhibits three apparent mass losses (91%), the first one between $33\text{ }^\circ\text{C}$ and $138\text{ }^\circ\text{C}$ is due to the loss of residual solvent, water and corresponding weight loss is 4 %. The second mass loss observed in the region $180\text{--}400\text{ }^\circ\text{C}$ is due to the decomposition of precursors of ZnO and the corresponding weight loss is 47% while the third mass loss in the range of $445\text{--}530\text{ }^\circ\text{C}$ is due to evaporation of organic chains belonging to PVA which is also evidenced by the exothermic peak of DTA and corresponding weight loss is 22%.

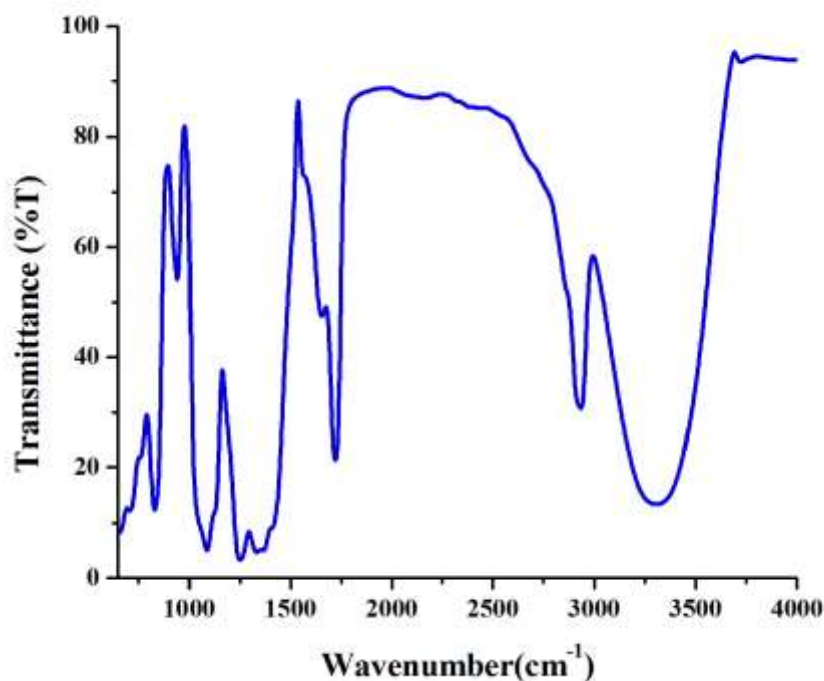


Fig. 2. FT-IR spectrum of ZnO-PVA nanopolymer.

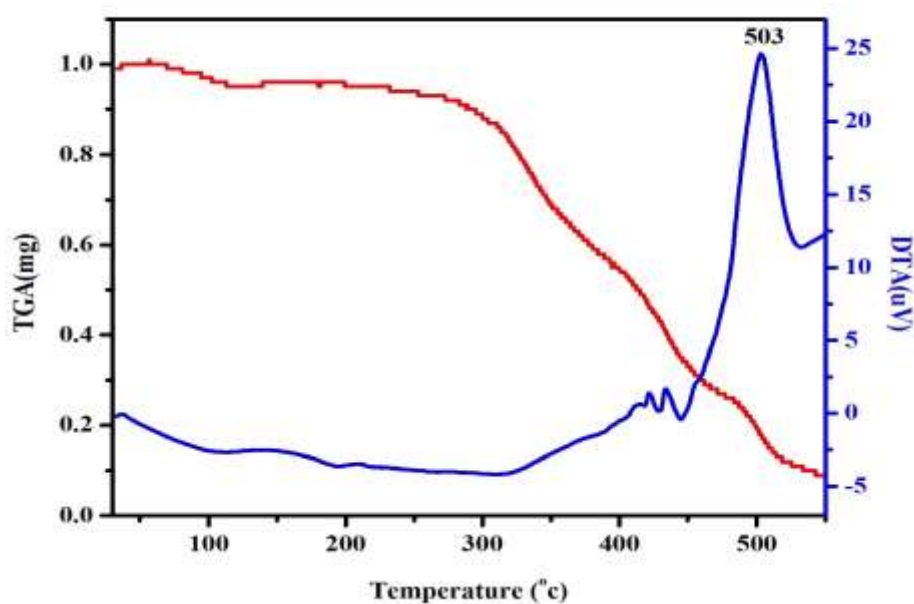


Fig. 3. TG-DTA of ZnO-PVA nanopolymer

4 Conclusion

ZnO-PVA nanopolymer was synthesized by chemical method. X-ray diffraction pattern confirms the prepared sample exhibits cubic structure and it is in the order of nanosize. The FTIR spectrum of PVA-ZnO nanopolymer exhibited different bands of OH stretching and C=O group which is attributed to interaction between ZnO-PVA nanopolymer matrix. TG-DTA analysis gives the thermal stability of the ZnO-PVA nanopolymer.

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