South Asian Journal of Engineering and Technology Research Article

Open Access Full Text Article 146(2024) 43- 48 DOI: 10.26524/sajet.2024.14.23

Synthesis and structure of Zinc oxide doped Polyindole Nanocomposites

L. Renuka[a,*], **B. Helina**[b]

[1]Research Scholar (Reg.No:18221282132009), Research Department of Physics, St. Xavier's College (Autonomous), Palayamkottai-627002. (Affiliated to Manonmaniam Sundaranar University, Tirunelveli-627012, Tamilnadu, India).

[2] Research Deparment of Physics, St.Xavier's College (Autonomous), Palayamkottai-627002. (Affiliated to Manonmaniam Sundaranar University, Tirunelveli-627012, Tamilnadu, India).

Corresponding author.
Correspondence: L. Renuka
E-mail: renuvisaka93@gmail.com

Article info
Received 18th July 2024 Received in revised form 20 August 2024
Accepted 22 Septemper 2024

Keywords
Polyindole (PIN), Zinc Oxide (ZnO), SEM, UV, XRD.

https://sajet.in/index.php/journal/article/view/293

Abstract
We present the synthesis and characterization of nano Zinc Oxide (ZnO), nano Polyindole (PIN), and nano PIN/ZnO composite in this work. Polyindole was produced by oxidative chemical polymerization using ferric chloride. Scanning electron microscopy (SEM) was used to analyze the structure and form of the PIN, PIN/ZnO nanocomposite and ZnO nanoparticle. The elemental analysis and chemical characterization were shown using the EDAX technology. The X-Ray Diffraction (XRD) technique was used to measure the crystallites' sizes and the degree of crystallinity. It is clear that the dopant improves conductivity since the UV-vis spectrum shows that the band gap of the doped PIN decreases.

1.INTRODUCTION

The customizable physical properties, excellent homogeneity, and flexible processability of polymeric nanocomposites embedded with inorganic nanoparticles have piqued attention characteristics like electric, mechanical, magnetic, and optical as well as electrical attributes. The physical and electrochemical characteristics of Composite Polymer Electrolytes (CPE) have been shown to be significantly influenced by the size of the dispersed particles [1-6]. In contrast to their atomic or bulk equivalents, transition metal oxides, such as copper oxide, iron oxide, nickel oxide, and zinc oxide nanoparticles, exhibit unique physicochemical characteristics and a high specific surface area.[7-11]. In the present work,PIN/ZnO was characterized by XRD, SEM, EDAX and UV.

2. EXPERIMENTAL

Materials Used

- Indole monomer (Sigma - Aldrich 99.5%)
- Ferric chloride (Merck 99%)

- Zinc Sulphate (Merck 99%)
- Sodium hydroxide (Merck 99%)
- Deionized water

2.1 SYNTHESIS OF POLYINDOLE

Indole (0.03M, monomer) was dissolved in distilled water (100ml) and Ferric chloride ($FeCl_3$ as oxidant, 0.06M) was added drop wise to the above solution under heating and stirring at 80°C. The colour of the reaction mixture was changed from colourless to dark green due to polymerization. After 3 hours the reaction mixture was filtered and washed with water. Polyindole was collected and dried in room temperature.

2.2 SYNTHESIS OF ZnO NANOPARTICLE

Sodium hydroxide (NaOH, 0.6M) was added slowly into the Zinc Sulphate ($ZnSo_4$ $6H_2O$, 0.3M) solution under stirring then refluxed for 2 hr, the black precipitate of Nickle hydroxide was filtered and washed with water to remove impurities. Dried ZnO nanoparticles were collected at 360°C muffle furnance.

2.3 SYNTHESIS OF ZnO DOPED POLYINDOLE NANOCOMPOSITE

Indole (0.03M, monomer) was dissolved in hot water (100ml) and Ferric Chloride ($FeCl_3$ as oxidant, 0.06M) was added drop wise to the above solution under heating and stirring. The zinc Oxide (2g) nanoparticles were added to the monomer solution and heating and stirring was continued. After 3 hour the PIN/ZnO nanocomposite was thoroughly washed with water. ZnO doped Polyindole nanocomposite was dried at room temperature.

3. CHARACTERIZATION

3.1 X-RAY DIFFRACTION

Figure 1(a), (b) & (c) displays the XRD patterns for the PIN, ZnO and Pin/ZnO nanocomposite. The Polyindole amorphous character may be clearly seen in Fig.1(a).[12] Based on the XRD pattern, a single phase of zinc oxide nanoparticles can be confirmed (Fig. 1(b)). The (100), (002), (101), (102), (110), (103), (112) and (201) crystal planes are accountable for the peaks at 2θ values 31.74°, 34.44°, 36.25°, 47.56°, 56.59°, 62.83° and 67.94° respectively. These peaks are nicely indexed to the ZnO standard card (JCPDS NO. 36-1451). The prepared ZnO nanoparticles particle size was measured at 39.98 nm using the Scherrer formula, which calculates the average particle size as $\tau=K\lambda/\beta \cos(\theta)$. The formula takes into account the shape factor, x-ray wavelength, line broadening at half the maximum intensity (FWHM) in radians (β) and Bragg angle (θ). It is evident that as the quantity of polymer in the polymer nanocomposite increases, the peak intensity of the ZnO decreases. This suggests that the domain of amorphous area has been significantly increased by the addition of ZnO to the polyindole matrix. The produced PIN/NiO nanoparticles have a particle size of 28.12 nm. When the concentration of doping increases, the diffraction peak intensity often drops significantly, indicating a loss of crystallinity as a result of lattice deformation.

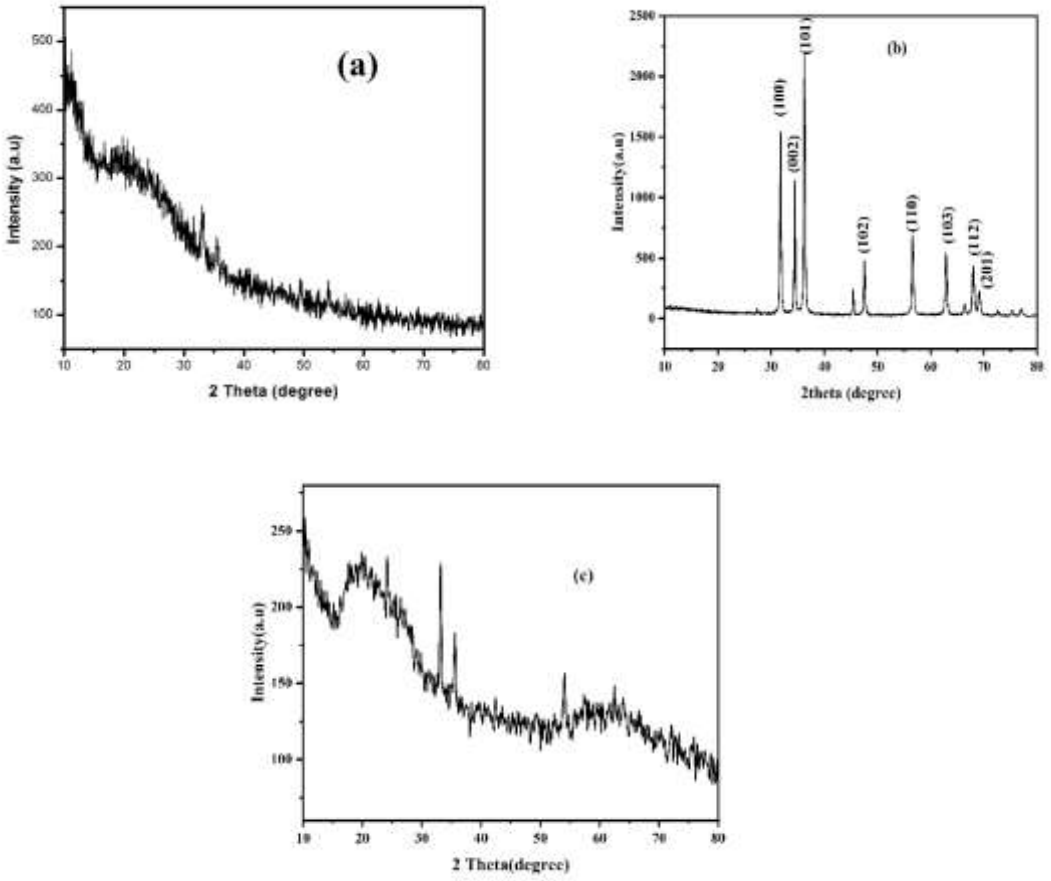

Fig 1. XRD of (a) PIN (b) ZnO & (c) PIN/ZnO

3.2 UV Analysis

Figure 2(a) shows the UV-Visible spectrum for PIN and PIN/ZnO. Absorption occurs when the energy contained in a photon is absorbed by an electron resulting in a transition to an excited state. The optical absorption studies are useful for the identification of band gap, impurity states, refractive index, extinction coefficient etc. Absorbance of PIN was at 237 nm. Similarly for PIN/ZnO there exists an absorbance peak at 367 nm. The band gap of PIN and PIN/ZnO were shown in the figures 2(a) and 2(b) respectively. The band gap is calculated by extrapolating the energy axis. The optical band gap for pure PIN and PIN/ZnO are shown in the following table:

Materials	**Bandgap(eV)**
PIN	4.45
PIN/ZnO	2.72

The table shows that when PIn is doped with ZnO, the band gap between the conduction and valence bands decreases.

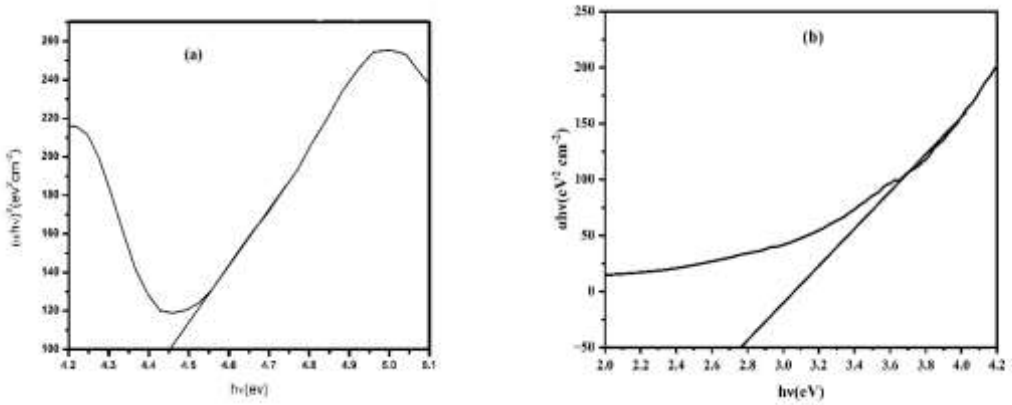

Fig 2. UV Tauc's Plot of (a) PIN & (b) PIN/ZnO

3.3 FIELD EMISSION SCANNING ELECTRON MICROSCOPY

FESEM was used for assessing the shape and dimensions of PIN, ZnO & PIN/ZnO; high and low resolution FESEM pictures are shown in figure 3. The polyindole that was synthesized reveals the production of irregularly shaped particles that range in size 1 micron & 500nm in fig 3 (a) & (b).. The high-resolution picture shows the existence of smooth-surfaced, spherical particles that have fused together to create chunk-like shape. Figure 3(e) & (f)displays pictures captured by a scanning electron microscope (SEM) of PIN/NiO nanocomposites, demonstrating their spherical shape and 1μm width.

3.4 ENERGY-DISPERSIVE X-RAY ANALYSIS (EDAX)

The EDAX analysis report for PIN/NiO is shown in Figure 3(c). The polyindole derived from EDAX is confirmed by the proportion of C, H, N, and O. To verify that the nickel oxide nanoparticles are incorporated into the PIN matrix, the EDAX analysis of the PIN/NiO is also carried out.

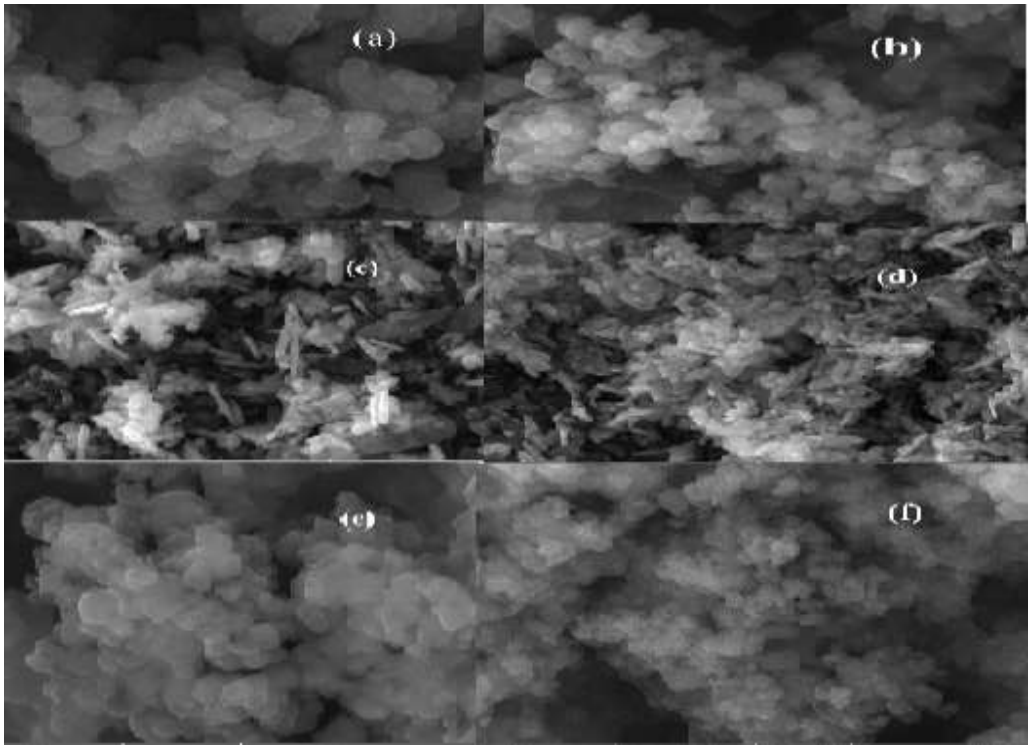

Fig 3.FESEM image of (a,b) PIN, (c,d) NiO & (e,f) PIN/NiO

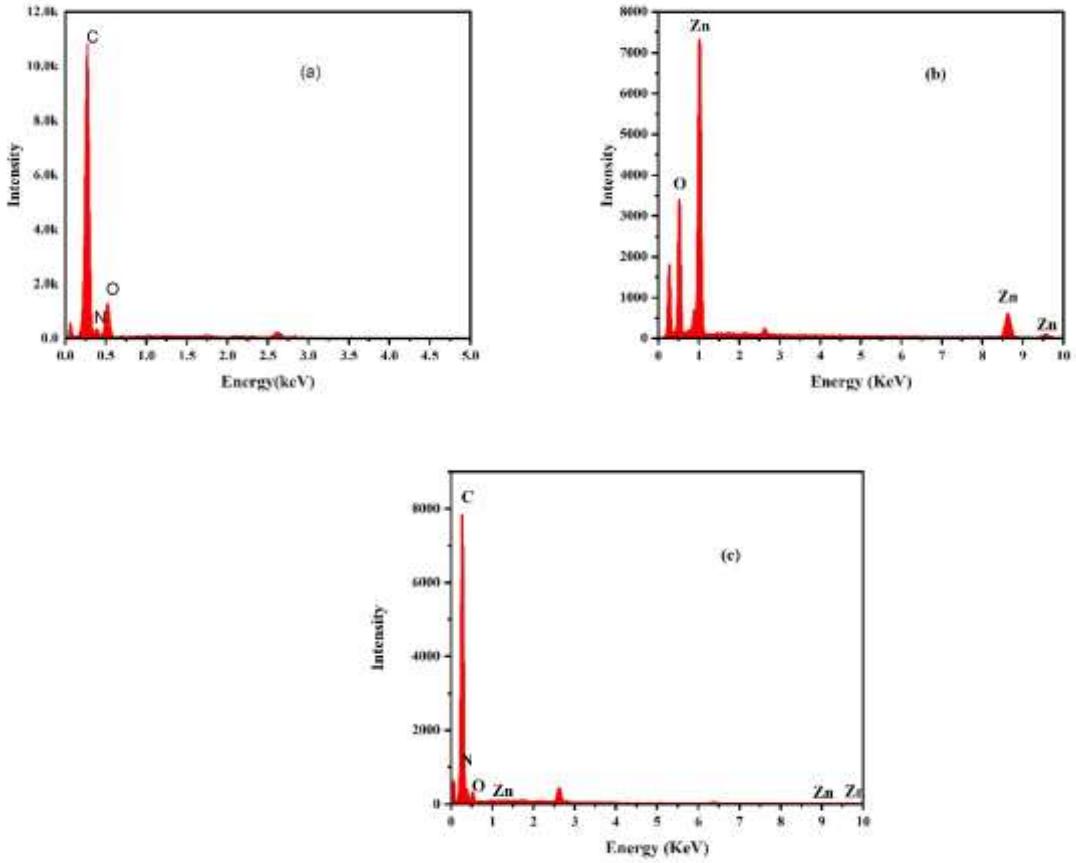

Fig 4. EDAX OF (a) PIN (b) ZnO &(c) PIN/ZnO

Conclusion

In the present study, nano Zinc oxide particles, polyindole and polyindole (PIN)/ZnO nanocomposite were prepared and characterised. The SEM images showed that the nanostructured nature of the prepared materials. The elemental composition in the materials is confirmed by EDAX method. The X-ray diffraction (XRD) technique has shown there is a significant change in the crystallite nature of in polyindole (PIn)/NiO nanocomposite. From UV-Vis analysis, the absorption spectra of PIN is around 237nm and have the optical band gap of 4.45 eV and for PIN/ZnO the absorbance and optical band gap are absorbed at 367 nm and 2.72 eV respectively.

Acknowledgement

Nill

Funding

No funding was received to carry out this study.

REFERENCE

1. Prakash, R.; Srivastava, R.C.; Pandey, P.C. Copper (II) ion sensor based on electropolymerised undoped conducting polymers. J. Solid State Electrochem. 2002, 6, 203–208.
2. Pawar, S.G.; Patil, S.L.; Chougule, M.A.; Mane, A.T.; Jundale, D.M.; Patil, V.B. Synthesis and characterization of polyaniline: TiO2 nanocomposites. Int. J. Polym. Mat. 2010, 59, 777–785.

3. Pan, W.; He, X.; Chen, Y. Preparation and characterization of poly (vinyl alcohol)=antimony-doped tin oxide nanocomposites. Int. J. Polym. Mater. 2011, 60, 223–232.
4. Hossein, E.; Hamid, R.K. Preparation and characterization of PAn-HPC=Fe3O4 and PAn-HPC=Fe2O3 nanocomposite using hydroxypropylcellulose as a steric stabilizer. Polym.-Plast. Technol. Eng. 2010, 49, 1591–1596.
5. Panaitescu, D.M.; Radovici, C.; Ghiurea, M.; Paven, H.; Iorga, M.D. Influence of rutile and anatase TiO2 nanoparticles on polyethylene properties. Polym.-Plast. Technol. Eng. 2011, 50, 196–202.
6. Bhat, A.H.; Banthia, A.K. Mechanical and dynamic analysis of poly (vinyl alcohol)-modified inorganic filler composite. Polym.-Plast. Technol. Eng. 2008, 47, 115–121.
7. Ganesan, R.; Nancy, A.P.; Paramasivam, T.; Boukos, N.; Vengidusamy, N.; Arumainathan, S. Synthesis and characterization of polyindole–NiO-based composite polymer electrolyte with LiClO4. Int. J. Polym. Mat. 2011 (In Press).
8. Guo, Z.; Liang, X.; Pereira, T.; Scaaro, R.; Hahn, T. CuO nanoparticle filled vinyl-ester resin nanocomposites. Fabrication, characterization and property analysis. Compos. Sci. Technol. 2007, 67, 2036–2044.
9. Li, S.; Xu, S.; He, L.; Xu, F.; Wang, Y.; Zhang, L. Photocatalytic degradation of polyethylene plastic with polypyrrole=TiO2 nanocomposite as photocatalyst. Polym.-Plast. Technol. Eng. 2010, 49, 400–406.
10. Seckin, T.; Kotepe, S.; Kvlcm, N.; Bahce, E.; Adguzel, I. Low dielectric constant polyimide-zirconium nanocomposites with improved thermal properties. Int. J. Polym. Mat. 2008, 57, 429–441.
11. Zikry, A.F. Dielectric behavior of silica=polyacrylamide nanocomposites. Int. J. Polym. Mat. 2008, 57, 383–395
12. G. Rajasudha , A. P. Nancy , Thangadurai Paramasivam , Nikos Boukos , V. Narayanan & A. Stephen (2011) Synthesis and Characterization of Polyindole–NiO-Based Composite Polymer Electrolyte with LiClO4 , International Journal of Polymeric Materials and Polymeric Biomaterials, 60:11, 877-892, DOI: 10.1080/00914037.2010.551367