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Synthesis and Characterization of PVA/PEO/In₂S₃ Polymer Nanocomposite Films and Study of their Optical, Electrical and Thermal Properties

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ABSTRACT

In this study, polymer nanocomposite films consisting of poly vinyl alcohol (PVA) and poly ethylene oxide (PEO) blend matrix dispersed with nanosize particles of Indium sulphide (In_2S_3) have been prepared by well known solution casting method. The structural, optical and thermal studies were performed using X-ray diffraction (XRD), Fourier transform infrared (FT-IR), Ultra violet- visible spectra (UV-Vis), Scanning electron microscope (SEM) and Thermogravimetric analysis (TGA). The structural properties of polymer nanocomposite films characterized by XRD measurements confirm the semi-crystalline structures of these materials. The IR absorption spectra indicate that the PVA/PEO undoped blend and doped with In_2S_3 are immiscible. The XRD and DSC showed that the incorporation of nano In_2S_3 into the polymeric system causes decrease in the crystallinity of samples. The thermal stability and mechanical strength of the blended samples increased with increase in In_2S_3 when compared to pure PVA/PEO blend films. The SEM analysis indicate that the change of the surface morphology of PVA/PEO/In_2S_3 nanoparticle system as phase segregation.

Graphical Abstract



UV-Visible spectra of $PVA/PEO/In_2S_3$ polymer nanocomposite films.

Keywords: Polyvinyl alcohol, polyethylene oxide, In₂S₃, PVA/PEO/In₂S₃ films.

INTRODUCTION

In the recent years, polymer nanocomposite materials have attracted the attention of many researchers due to their unique optical, electrical, thermal, mechanical, magnetic and dielectric properties. One of the advantages of nanofillers as polymer additives compared to conventional additives is that the loading requirements are quite low. Nanoparticles exhibit size related properties that differ significantly from bulk materials. The high surface reactivity and large surface to volume ratio of the nanoparticles make them superior over traditional fillers [1]. Polymer blending is one of the most important contemporary ways for the development of new polymeric materials and it is a useful technique for designing materials with a wide variety of properties [2]. Polymer metal nanoparticle composites are promising functional materials in several fields such as optical, electrical, thermal, mechanical and antimicrobial properties [3-7]. Many reports in the literature show attempts for the synthesis of metal nanoparticles based polymer nanocomposites with the possibility of variation in their optical and electrical properties for their application in high performance capacitors, conductive inks and other electronic components [8, 9]. The structural, optical, thermal and electrical properties of polymers can be suitably modified by the addition of dopant depending on their reactivity with the host matrix [10]. Nanocomposites play a significant role in tailoring the properties of polymeric materials to suit any technology [11, 12]. The incorporated nanoparticles inside the polymer matrix will also affect the properties of the host matrix.

PVA (Poly vinyl alcohol) is an important material with technological aspect as a water -soluble and non -toxic polymer which is extensively used in paper coating, textile sizing and flexible water soluble packaging [13, 14]. It is strong, durable and exhibits high dielectric strength, high mechanical strength and good storage capacity [15]. PEO (polyethylene oxide) is the most interesting base material because of its high chemical and thermal stability [16]. PEO is semi crystalline polymer, possessing both amorphous and crystalline phases at room temperature.

Among the synthetic hydrophilic polymers, poly(vinyl alcohol)(PVA) and poly (ethylene oxide)(PEO) have excellent flexible-type film forming ability when films are prepared by solution-casting method. In these polymeric films, macromolecules chains have intra and inter molecular hydrogen bonded connectivity through their functional groups. The aqueous solution cast PVA film exhibits high degree of optical transparency and therefore PVA is frequently used as binder in preparation of various composite materials suitable for optoelectronic and microelectronic devices [17-19]. The high degree of solvating power of PEO for alkali metal salts makes it the most suitable matrix in preparation of flexible-type solid polymer electrolytes (SPEs) [20-23]. Further, inherent non-toxicity, non-carcinogenicity, high biocompatibility and high degree of swelling in aqueous solutions established the PVA and PEO as potential materials in pharmaceutical industries [24-27].

In search of advanced polymeric materials, polymers blending became the most popular technique for designing of materials having a wide variety of useful properties over pristine polymers [28-32]. Therefore, the polymer blends have emerged as a vibrant field of research and development in the branches of chemistry, physics, materials science, pharmaceutical, engineering and technology. Most of the studies have revealed that the blends of PVA and PEO are essentially incompatible due to formation of weak inter-polymer chains interactions. Although, PEO and PVA in aqueous solution form the hydrogen bonded blend structures in presence of water molecules, but strength of such hetero-molecular interactions gradually diminishes during slow evaporation of water molecules from the solution when it is casted to form the film. Due to this fact, finally polymers phase separated PVA-PEO blend films were formed [33].

In this present work, our study is confined to the preparation of films of PVA/PEO/In₂S₃ by solution casting method and to study the fundamental physical properties of PVA/PEO blend films undoped and doped with different concentration of In_2S_3 nanoparticles. A systematic investigation on structure, morphology, thermal stability, miscibility and band tail property relationship of nano In_2S_3

polyblend system are discussed using different tools and techniques like XRD, UV-VIS, SEM, FT-IR and TGA techniques.

MATERIALS AND METHODS

Polyvinyl alcohol (molecular weight 125,000) was received from SD's lab-chem industry and polyethylene oxide (molecular weight 5,000,000) supplied by alfa aesar were used as basic polymeric composite materials. In_2S_3 nanopowder prepared by the electrochemical procedure in our research laboratory had been used. Deionized water was used for all experiments. All chemicals were used as received without further purification.

Preparation of PVA/PEO/In₂S₃ blend films: Nanocomposites of PVA/PEO dispersed with different concentrations of In_2S_3 nanoparticles were prepared by solution casting method. Equal quantity of PVA and PEO (50/50 Wt/Wt %) was dissolved in deionized water and then heated gently using a water bath to prevent thermal decomposition of the polymer. The solution was brought to the room temperature and stirred for about 6 hours until the polymer is completely dissolved and forms a clear viscous solution. Then resulting viscous solution casted onto plastic petri dish and kept in dry atmosphere at 70°C about 48 h. After drying the membrane was peeled from petri dish and kept in vacuum desicator until uses. The polymer nanocomposite films of various In_2S_3 concentrations were prepared by following the same procedure. In_2S_3 nanoparticles (0.25%, 0.5%, 0.75% and 1%) were added to the homogeneous PVA/PEO solution and the resulting polymer nanocomposite solution was stirred for about 6 h to get viscous solution. The solution was casted into plastic petri dishes and kept in dry atmosphere at 70°C for about 48 h. The films were peeled from the petri dishes and kept in vacuum desiccators.

Measurement techniques: The XRD difractograms of PVA/PEO films undoped and doped with different concentrations of In_2S_3 were recorded using Rigaku Miniflex II desktop X-ray difractometer equipped with Cu- Ka radiation ($\lambda = 0.1540$ nm). Ultraviolet-visible absorption spectra were measured in the wavelength range of 200-800nm using JASCO UV-VIS spectrophotometer. The SEM images of the samples were recorded on ESEM Quanta -200 FEI- Netherlands. FT-IR measurements were performed using JASCO, FT-IR in the spectral range of 4000-400 cm⁻¹. Thermogravimetric analysis (TGA) was used to characterize the decomposition and thermal stability of prepared samples by thermogravimetric analyzer (TGA) Q-50 instrument, USA). The loss of mass was recorded during heating from ambient temperature upto 700°C with rate of 10°C min⁻¹.

RESULTS AND DISCUSSION

X-ray Diffraction (XRD): X-ray diffraction pattern enables one to find out whether the material is crystalline or amorphous and thus useful in knowing the structure of the polymeric material. The figure 1 shows the X-ray diffraction patterns of pure PVA, PEO and PVA/PEO blend films undoped and doped with different concentrations of In_2S_3 nanoparticles at room temperature. The pure PVA exhibits only a broad and shallow diffraction peak around the 2 Θ value of 19.45°, while the pure PEO has two well defined reflection peaks at 19.20° and 23.47°. The major reflection peaks of In_2S_3 appear at 2 Θ values 21.40°, 30.59°, 35.49°, 45.72°, 51.04° and 60.68° [**34**]. The undoped PVA/PEO blend polymer sample show well defined peaks at 19.18 and 23.03 which are unique to the feature of PEO. This result reflects that PEO remain as separate phases with no significant mixing [**35**]. The XRD pattern of doped films shows two peaks at 19.18° and 23.38°, their intensities slightly change with increasing In_2S_3 concentration but it is still lower than undoped one.

Fourier Transform Infrared analysis (FT-IR): Figure 2 shows the IR spectrum for PVA/PEO blend films undoped and doped with different concentrations (0.25%, 0.5%, 0.75% and 1%) of In_2S_3 nanoparticles. In the blend sample spectrum, the intensities of the most absorption bands such as v(OH), v(CH) and v(COC) stretching vibrations are changed irregularly compared to their values in



Figure 1. X-ray diffraction spectra of (a) PVA, (b) PEO,(c) 50/50 (Wt/Wt %) PVA/PEO undoped blend sample and blend sample doped with (d) 0.25% (e) 0.5%, (f) 0.75% (g) 1% of In_2S_3 nanoparticles.

individual polymers, while their positions remains unaffected. Therefore intermolecular hydrogen bonding between PVA and PEO may not be significant at all [35]. It can be seen that there is an increase in the absorption intensities of bands at 1730, 1567, 1463, 953 and 843 cm⁻¹ with increase in

the concentration of In_2S_3 . The peak at 798 cm⁻¹ corresponds to the bonding of In-S [36].



Figure 2. FTIR spectra of (a) 50/50 (Wt/Wt %) PVA/PEO undoped blend sample and blend sample doped with (b) 0.25% (c) 0.5%, (d) 0.75% (e) 1% of In₂S₃ nanoparticles.

Ultraviolet-Visible spectroscopy: The UV-visible spectra of the polymer nanocomposite films were recorded at room temperature in the wavelength range 200-800 nm and it is shown in figure 3. The $PVA/PEO/In_2S_3$ polymer composite films showed absorption peak in UV region while no absorption peak in visible region. It is evident that the films exhibit very small transmittance in the UV region and very high transmittance in the visible region. Consequently these materials are considered as optically transparent in the visible region. Meanwhile the $PVA/PEO/In_2S_3$ films showed higher absorbance compared to the pure PVA/PEO film.

Scanning Electron Microscopy: The morphology of the samples were studied by FE-SEM. SEM is used to investigate fully the effect of In_2S_3 nanoparticles content and to examine the dispersion of nanocomposites particles in the polyblend polymer matrix. Figure 4 shows typical SEM images of PVA/PEO blend films undoped and doped with different concentrations of In_2S_3 nanoparticles. The SEM figure 4a for undoped PVA/PEO film is found to be harder, homogeneous and coherent. It is apparent that the addition of In_2S_3 nanoparticles in PVA/PEO blend films exhibits changes in the



surface morphology of the system (figures4b-4e). As the content of In_2S_3 increases upto 1%, the film surface becomes rough with some white spots (small particle aggregates) which indicates the segregation of In_2S_3 in the polymeric system. White spots on the backscattered images seem to be agglomerates of In_2S_3 particles, which increase with increasing the concentration of In_2S_3 . The figure 4c gives rise to spherulitic structure, which is due to the In_2S_3 segregated into the interlamellar regions of the blend. The figure 4d(i) and 4d(ii) represents clustered blossom and bird like structure respectively. Figure 4e shows crystalline type white spot.





Figure 4. SEM images of (a) 50/50 (Wt/Wt %) PVA/PEO undoped blend sample and blend sample doped with (b) 0.25% (c) 0.5%, (d) 0.75% (e) 1% of In_2S_3 nanoparticles.

Thermogravimetric Analysis (TGA): Thermogravimetric analysis is a process in which substance is decomposed in the presence of heat which causes bonds within the molecule to be broken. The sample weight decreases slowly as the reaction begins, then decomposes rapidly over comparatively narrow temperature range and finally levels off as the reactants becomes spent. The shape of TGA curves depends upon the kinetic parameters [35]. TGA and Dr TGA thermograms as a function of temperature in the range from 20°C to 700°C for undoped PVA/PEO and doped PVA/PEO with different concentrations of In_2S_3 nanoparticles are shown in figure 5. All the samples have four steps of decomposition. The table 1 represents the decomposition steps and percentage weight loss for PVA/PEO blend undoped and doped with different concentration of In₂S₃ The lower values of percentage weight loss in the first decomposition step which includes the melting point of PEO (2.83-5.42%). This may be due to the splitting or volatization of small molecules, and/ or the evaporation of moisture. The second decomposition region in the TG curves a wider temperature range including the melting point of PVA and has percentage weight loss (29.92-36.09%). The later process in the TG curves includes the main decomposition step and has more significant weight loss (38.57-46.82%). The total percentage weight loss decreases with the increase of the concentration of nano In₂S₃ in the polymer film.

In ₂ S ₃	Decomposition	Temperature (°C)		Weight loss (%)	
Wt %	region	Start	End	Partial	Total
0.0	1 st	49.10	137.63	4.116	97.046
	2^{nd}	230.38	362.12	36.09	
	3 rd	362.12	448.54	46.82	
	4^{th}	448.54	572.02	10.02	
0.25	1 st	27.10	132.36	5.42	94.33
	2^{nd}	231.43	363.17	35.58	
	3 rd	363.17	461.19	43.13	
	4^{th}	461.19	570.80	10.20	
0.5	1^{st}	26.24	139.74	4.021	91.262
	2^{nd}	219.84	357.90	36.19	
	3 rd	357.90	467.51	42.36	
	4^{th}	467.51	592.93	8.691	
0.75	1 st	38.25	143.95	3.051	85.00
	2^{nd}	210.35	360.01	33.48	
	3 rd	360.01	468.57	39.88	
	4^{th}	468.57	579.23	8.589	
1.0	1^{st}	31.69	127.09	2.831	81.084
	2^{nd}	213.51	356.85	29.92	
	3 rd	356.85	447.49	38.57	
	4^{th}	447.49	548.67	9.763	



Figure 5. TGA and Derivative TGA of (a) 50/50 (Wt/Wt %) PVA/PEO undoped blend sample and blend sample doped with (b) 0.25% (c) 0.5%, (d) 0.75% (e) 1% of In_2S_3 nanoparticles.

Frequency dependent conductivity measurements: The AC conductivity of PVA/PEO polymer films and PVA/PEO/In₂S₃ nanocomposite films are measured using conductivity bridge instrument at frequency of 50Hz and graphically represented by figure 6. The conductance of polymer PVA/PEO film enhanced with the addition of In_2S_3 nanoparticles. As the concentration of nanoparticles in the polymer matrix increases, the conductivity also increases since the concentration of nano fillers increases, the inter particles distance are less and availability of charge carriers responsible for conductivity are more. Further, as the frequency increases, conductivity also increases. This is because at higher frequencies charge carriers will have high mobility.





Figure 6. Frequency dependent conductivity plot of concentration v/s conductance at 50Hz.

APPLICATION

Photo-voltaic activity: The photo-voltaic property of PVA/PEO films undoped and doped with different amounts of In_2S_3 were studied by conductometer and potentiometer at dark, sunlight and UV light [37]. The experimental results (Figure 7 and 8) suggest that as concentration of dopant nanoparticles is increased, the conductance and potential also increased. The photo-voltaic property of PVA/PEO/In₂S₃ nanocomposite films is enhanced to an appreciable extent in presence of UV-light than the sunlight and dark conditions. It can be attributed due to the λ_{max} of In_2S_3 nanoparticles has undergone blue shift after embedding into PVA/PEO matrix, hence it is UV active.

Table 2. Conductivity and potential measurements for PVA/PEO/ In₂S₃ films

Property	Catalyst	Dark	sunlight	UV
Conductivity	PVA/PEO	1.02	1.51	2.01
measurements in µS	0.25% PVA/PEO/ In ₂ S ₃	1.03	2.20	2.82
	0.5% PVA/PEO/ In ₂ S ₃	2.01	2.32	3.02
	0.75% PVA/PEO/ In ₂ S ₃	2.03	2.55	4.91
	1% PVA/PEO/ In ₂ S ₃	3.05	3.71	6.05
Potential	PVA/PEO	0.021	0.030	0.060
measurements in V	0.25% PVA/PEO/ In ₂ S ₃	0.025	0.032	0.071
	0.5% PVA/PEO/ In ₂ S ₃	0.031	0.047	0.085
	0.75% PVA/PEO/ In ₂ S ₃	0.042	0.059	0.120
	1% PVA/PEO/ In ₂ S ₃	0.063	0.082	0.113



Figure 7. Concentration v/s conductance graphs at different concentration of PVA/PEO/In₂S₃ films under different experimental conditions (Dark, sunlight and UV light)



Figure 8. Concentration v/s potential graphs at different concentrations of PVA/PEO/In₂S₃ films under different experimental conditions (Dark, sunlight and UV light)

CONCLUSION

 $PVA/PEO/In_2S_3$ Polymer nanocomposite films were prepared by solution casting method. The SEM images showed that the film surfaces had characteristic structures pertaining to different concentration of dopant. Incorporation of nanoparticles into PVA/PEO matrix and the corresponding shift in the absorption bands were observed by XRD and IR techniques. UV-Vis spectroscopy reveals the blue shift in the absorption edge indicating an increase in the band gap energy upon doping. The conductivity and potential studies indicates enhancement of photovoltaic effect with increase in the amount of nano In_2S_3 .

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