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Research Article

PREPARATION, THERMAL AND OPTICAL PROPERTIES OF POLYETHYLENE METAL OXIDE THIN FILM

Shakeel Ahmad Khanday

Department of Physics, Degree College Boys Anantnag
Jammu & Kashmir (India).

Corresponding author:

Shakeel Ahmad Khanday,
Department of Physics,
Degree College Boys, Anantnag,
Jammu & Kashmir (India).

ABSTRACT:

Pure poly (ethylene oxide) (PEO) films and PEO-FeCl₃ films of various concentration were prepared and studied for thermal and optical properties. Various analytical techniques like XRD, EDX, SEM, TGA, and UV were used to examine the structural properties of PEO and PEO-FeCl₃ systems. X-ray scans (XRD) revealed the existence of some crystalline part in the PEO matrix. The concentration of FeCl₃ has a great effect on the crystallinity of PEO. Thermogravimetric analysis (TGA) data established that accumulation of FeCl₃ to PEO films enhanced the thermal stability of PEO.

INTRODUCTION:

The addition of metal chlorides to a polymer is also pursued because transparent films can be produced from them. The addition of conductive fillers/metal fillers can also improve the thermal behavior remarkably [1-3]. These transparent films can be used as optical filters, polarizers, total reflectors, narrow pass-bands, etc. Optical absorption spectrum is very important in terms of optical characterization of the materials. The absorption spectrum is usually studied by ultraviolet (UV) absorption method. UV absorption is generally thought to be associated with electron (or anion) transitions from the top of the valence band to the bottom of the conduction band. These transitions can be direct or indirect depending on wave vector. Davis and Shalliday⁵ reported that near the fundamental band edge both direct and indirect transitions occur and can be observed by plotting $(\alpha h\nu)^{1/2}$ and $(\alpha h\nu)^2$ as a function of energy ($h\nu$). Many studies on poly (ethylene oxide) (PEO)-salt composites have been reported; they are mainly based on electrical and thermal properties. Peng Su and co-workers reported that the addition of an appropriate amount of FeCl_3 [4] results in a beneficial effect on defect-free electrospun chitosan/PEO blend nanofibers. PEO is a unique polymer because it is soluble both in aqueous and organic solvent and is a useful polymer available in a variety of molecular weights. PEO forms yellow complexes with FeCl_3 , showing photochromic effect under UV irradiation. FeCl_3 is known to be one of the most efficient oxidants in several polymeric polymerizations (especially poly pyrrole) leaving chloride ion and hence making it conducting [5]. The Fe^{3+} ions are bound by the coordinating oxygen atoms of the PEO chain. This will suppress crystallization of PEO and ensure homogeneous distribution of FeCl_3 along the PEO. In the Fe^{3+} ions, d-electron shells are not filled as Fe^{3+} ions have d^5 configuration

MATERIAL AND METHODS:

PEO/ FeCl_3 films were prepared by dissolving PEO in water at 30°C in solution 1, while an appropriate amount of FeCl_3 was dissolved in water in solution 2 at the same temperature. Finally these 2 solutions were mixed together with continuous stirring for 24 h and then cast on a petri dish at room

temperature. After solvent evaporation thin films of the composites were separated from the petri dish and were kept in a desiccator

RESULT AND DISCUSSION:

Absorption spectra of the films were recorded in the UV/Vis wavelength region of 200-800 nm using a PerkinElmer UV/VIS spectrometer. A Rigaku (Japan) FX Geiger Series RAD-B system was used for X-ray diffraction measurements [6]. Polymeric samples were cut into pieces 2.5 cm² each and then put into the standard glass sample holder. Then the sample holder along with the sample was placed into the X-ray generating chamber under accelerating voltage of 350 kV and current of 20 mA. Scanning electron microscopy was done by Micro analyzer Model JSM 5910. The thin films were cut smoothly with a sharp blade. Special holders were made for SEM analysis to hold the film perpendicularly upright. The surfaces of the film were made conducting by coating with gold (Au).

The absorption coefficient (α) was calculated from absorbance (A), after correction for reflection, using the following relation:

$$I = I_0 \exp(-\alpha x) .$$

$$\text{Hence } \alpha = 2.303/x \log I_0/I = 2.303 \times A, (2)$$

Where (I_0) is the incident intensity and (I) is the transmitted intensity, while x is sample thickness.

These expressions can be applied to both direct and indirect transitions and are helpful in the determination of the band structure of the materials. When a direct band gap exists, the absorption coefficient has the following dependence on the incident photon:

$$\alpha(\omega)h = \beta(h\omega - E_{opt})^r \text{ for } h\omega > E_{opt} ,$$

$$\alpha(\omega)h = 0 \text{ for } h\omega < E_{opt},$$

where β is a constant equal to $(4\alpha_0 / nc\Delta E)$, α_0 is the extrapolated dc conductivity and ΔE is a measure of the extent of band tailing, and r is an exponent that can assume values of 1, 2, 3, 0.5, and 3/2. For allowed direct transitions r takes the value of 0.5 and in some materials quantum selection rules forbid direct transition; in such cases r will take the value of 3/2. The absorption study of PEO composite with

MnCl₂ was also carried out during the course of this investigation for comparison. It is noted that the addition of 15% wt MnCl₂ to PEO resin reduces the absorption of PEO at the UV region, especially at 260 nm, while it shows enhancement at 320 nm, although the variation is not as sharp as already been reported for 5% wt by Zihlif et al. Figure 1 shows the plot of absorption coefficient vs. wavelength for pure PEO and FeCl₃-PEO composites [7-9]. It was found that the addition of 5% wt FeCl₃ to PEO shows almost the same spectrum as pure PEO, but for higher concentration composites the trend is different. Absorbance is high at 260 nm and remains almost the same up to approx. 550 nm and then it decreases, which is opposite to the trend observed in the case of the PEO-MnCl₂ system reported by Zihlif et al [10]

Thermogravimetry analysis

Thermogravimetric analysis The major weight losses are shown in the temperature range of 250 to 500 °C for PEO with and without salt (FeCl₃). For pure PEO the initial weight loss starts at 250 °C. A decrease in sample weight was observed from 350 to 500 °C, indicating thermal stability for PEO up to 350 °C [11-13]. This weight loss can be due to burning of organic phase and the thermal dehydration of inorganic particles. No major weight change was observed after 500 °C. The total weight loss at 500 °C for composites was lower than that of pure PEO, indicating that inorganic particles dispersed into the PEO matrix. It is clear from the thermal decomposition of PEO-salt films shifts unsystematically for various filler compositions. For a composition of FeCl₃ (5% wt) there is a shift towards higher temperature than that of pure PEO, which means that there is enhanced thermal stability of intercalated polymer as also reported elsewhere. The absorbance vs. wavelength spectra also show that both pure and FeCl₃ (5% wt) composite behavior is the same, while for the rest of composites it is different [14]. This means that 5% wt addition of salt is not making any complex, while further increases in the weight of salt promote complex formation. The composites with complexes are less thermally stable. Moreover, 10% and FeCl₃ (15% wt) addition somewhat reduced the thermal stability.

CONCLUSION:

Pure PEO and PEO-FeCl₃ films were prepared by solvent cast method and the effects of FeCl₃ salt on the optical and thermal properties were studied by means of different spectroscopic techniques. The FeCl₃ influence on the degree of crystallinity of PEO was ascertained by X-ray scans. The adding of FeCl₃ to PEO films affected thermal stability in an unsystematic manner. It was also observed from TGA spectra that the weight loss for the PEO-FeCl₃ system was different from the weight loss values for pure PEO

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