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Synthesis and Structural Properties of Polystyrene Complexed with Cadmium Sulfide

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Abstract: ***Polystyrene (PS) complexed with Cadmium sulfide (CdS) are prepared by a solution cast technique. Several experimental techniques such as XRD, SEM, FTIR and Raman Spectroscopy are employed to characterize the sample. The powder X-ray diffraction (XRD) result indicates that the nanoparticles are crystallized in cubic phase. SEM, FTIR and Raman spectra of the sample are recorded and discussed briefly.***

Keywords: CdS/PS thin film, XRD, SEM, FTIR, Raman Spectroscopy

1. Introduction

Polymer nanocomposites represents a new alternative to conventionally filled polymers because of their nanometer sizes, filler dispersion nanocomposite exhibit markelly improved properties when compared to the unfilled polymers or their traditional composites [1].

Forming composite by filler is a very promising approach as it can enhance the conductivity as well as provide strength to the polymeric film. To study how the mechanism of enhancement of properties of polymer changes when filled by nanoparticles, we have selected insulating polymer that is Polystyrene as a basic polymer.

Polystyrene (PS) is one of the most interesting polymers due to their novel properties. It is readily available and presents good environmental stability, easy processing, light weight and low cost. [2]

Polystyrene is a thermoplastic substance, which is in solid (glassy) state at room temperature, but flows, if heated above its glass transition temperature (95^0C) (for molding of extrusion) and becomes solid again when it cools off. Pure solid Polystyrene is a colourless hard plastic with limited flexibility. It can be cast into moulds with fine detail. Polystyrene can be transparent or can be make to take on various colours.

With reference to above and taking into consideration the properties of PS also, to fulfill our motivation and for the detail study of structural, optical, thermal and in modifying dc conductivity an attempt has been made to synthesize CdS filled PS composite thin films with 1, 5, 10 and 15 weight percent of CdS concentration in PS.

2. Experimental

The unfilled polystyrene and CdS filled polystyrene thin films were prepared by dissolving PS resin (MW 100,000–400,000) in toluene at 100^0C under vigorous stirring for 1 hour. In PS, CdS was remaining in the dispersed phase. The composition of CdS in PS is 1, 5, 10 and 15 by weight percent. Before casting the mixture, solution of PS and CdS was stirred for two hours using magnetic stirrer so that the CdS nanopowder uniformly mixed with PS solution. The sample was obtained by casting the solution on a leveled glass substrate and evaporating the solvent at room temperature under normal atmospheric pressure.

The glass plate was kept over a pool of mercury for perfect leveling, so as to ensure uniform thickness. The whole assembly was placed in a closed chamber to protect from dust. The solution was allowed to evaporate at room temperature. The film on the glass substrate was kept for 12 hours at 313 K for out gassing and another 12 hours at room temperature to remove the traces of solvent. It was cut into small pieces of desired size, which were then washed with ethyl alcohol to remove surface impurities.

Thus in the present study the thickness measurement of all the thin film samples has been carried out occulometer in conjunction with microscope. The thickness was found to be of the order ~46.14 µm(± 3 µm) of all the samples and taken nearly constant throughout the entire work.

3. Result & Discussion

Structural Characterization [X-Ray Diffraction (XRD)]

Many materials especially polymers, have a substantial volume fraction without crystalline order. Though these regions are often termed amorphous, they frequently have specific local disorder. Structural information of composites is very important in order to understand the interchain interaction that could affect the electrical conductivity of the polymer composite. The X-ray diffraction is a powerful tool to determine the structure and crystallization of polymer matrices. The effect of CdS in PS was analyzed through XRD techniques.

Figure 1 shows the XRD pattern of unfilled PS. Concerning the commercially-available PS, two large humps centered at $2\theta=10^0$ and 20^0 are the characteristics of its amorphous nature [3]. The pattern is characterized by the appearance of halos. Halos show the amorphous phase is dominant in the compound [4].

From XRD diffractrogram, it is concluded that the amorphous nature of the unfilled PS thin film. Our result is in good agreement with [4, 5, 6].

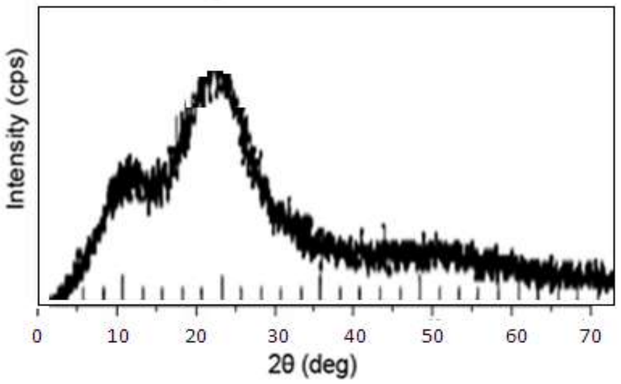

Figure 1: X-ray diffractogram of unfilled Polystyrene thin film

Figures 1(a, b, c and d) are the diffractograms for 1, 5, 10 and 15 weight percent of CdS filled PS composite thin films respectively. The typical XRD result indicates the amorphous structure of polystyrene and confirms the existence of CdS in PS composite thin films. For all the composite thin films, a broad peak represents amorphous nature. Peak position for all filled samples is observed at $2\theta=$ 19.03^0, 19^0, 18.7^0 and 18.33^0 for 1, 5, 10 and 15 weight percent PS thin films respectively. From diffractograms it is observed that as percentage of CdS in PS increases peak shifted towards the lower angle, along with increase in peak width where as peak height decreases. This type of findings might be due to the small size of the CdS particles [8].

Apart from broad peak, two small hump like structure is observed in all diffractograms in the region $\theta = 25^0$-45^0 which analogous to the presence of CdS in PS, which are clearly observed in CdSdiffractogram [explained in Chapter 5: Section 5.3.1.]

Polymers form structures that are often partially crystalline and partially amorphous [9].

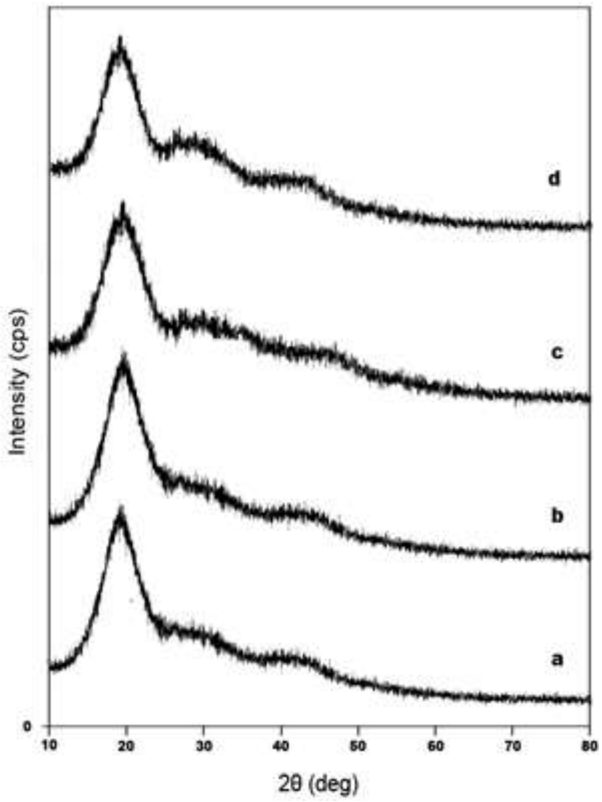

Figure 1: X-ray diffraction pattern for (a)1, (b)5, (c)10 and (d)15 weight percent of CdS filled PS samples respectively.

Scanning Electron Microscopy (SEM)

Surface morphology was observed using SEM image presented in Figure 2. All SEM images (for 1, 5, 10 and 15 weight percent CdS filled PS thin films) show that the film is almost homogenous, without any pinholes or cracks and covered the substrate well. It shows the unevenly dispersion of CdS nanoparticles in PS.

Figure 2 (a) (1 weight percent sample)CdS filled PS shows that the average 6 nm sized CdS are rarely dispersed form in PS microspheres. As weight percentage of CdS in PS increases (Figure 2 (b, c and d) presence of number of CdS particle increases and also the agglomeration is clearly observed.

Thus SEM of all the samples revealed the successful incorporation of CdS nanoparticles in PS matrix. The SEM images for lower loading percentage of CdS were similar to one another but as the CdS proportion reached towards highest value, the morphology of CdS can be easily observed in host matrix.

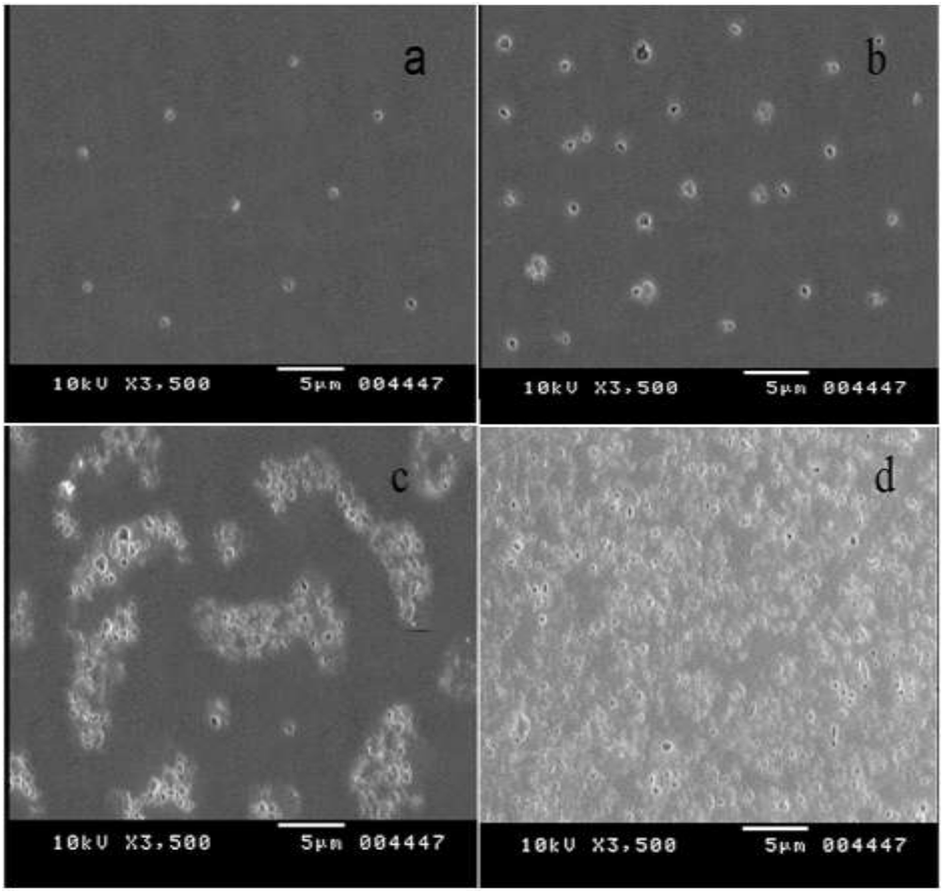

Figure 2: SEM pattern for (a)CdS-1%/PS(b) CdS-5%/PS(c)CdS-10%/PS and (d)CdS-15%/PSsamples

Fourier Transform Infrared Spectroscopy (FTIR)

FTIR spectra of unfilled PS thin film is shown in Figure 3 and its corresponding frequency correlations are enclosed in Table 3 (a).

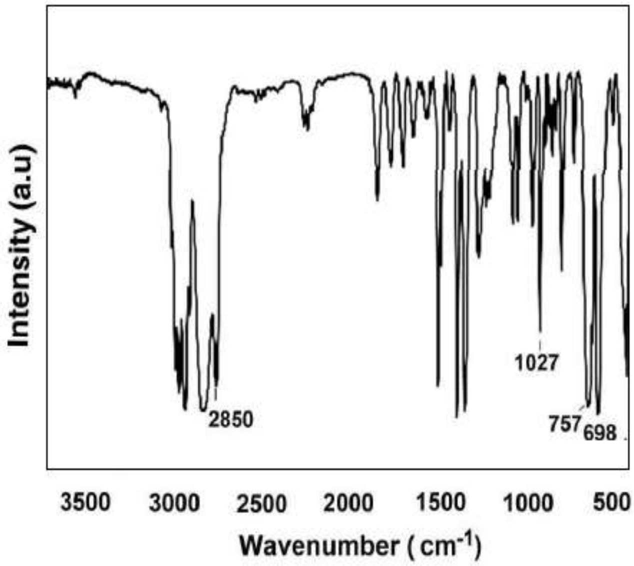

Figure 3: FTIR spectra for unfilled PS thin film

It shows 5 peaks over the 2800 cm^{-1} - 3100 cm^{-1}range, attributed to C-H streaching vibrations in the main chain and in aromatic rings, and the peaks at 1601 cm^{-1}, 1492 cm^{-1}, 1451 cm^{-1}, 1027 cm^{-1}, 757 cm^{-1}and 698 cm^{-1}are due to deformation and skeletal vibrations of C-H in PS [10,4]. It should be noted that the FTIR spectra of unfilled PS do not show the clear peak of carbonyl group.

Table 3 (a): Frequency correlation table for unfilled PS

S.N.	Frequency (cm^{-1})	Correlation
1	1601	C-H stretching
2	1492	C-H stretching
3	1451	C-H stretching
4	1027	C-H stretching
5	757	C-H stretching
6	698	C-H stretching

Figure 3(a and b) represents the FTIR transmission spectra of (a) 1 and (b)15 weight percentage of CdS filled PS composite thin films respectively, in the range 500 to 400 cm^{-1}and frequency correlation obtained from various stretching and bending vibrations are enclosed in Table 3 (b) and (c) respectively. The absorption peak values of 5 and 10 weight percentage CdS added PS composite thin films is nearly same as that of 1 and 15 weight percentage of CdS added PS composite thin films hence avoided in order to save space.

Figure 3 (a and b) shows the same spectral bands as that of in Figure 3 with slight variation in the peak value that might due to variation in CdS concentration in PS.The peaks at 2922 cm^{-1}, 2851 cm^{-1} and 1452 cm^{-1}, due to the aliphatic backbone of the polystyrene macromolecule associated with C-H stretching [11].C=N stretching vibration gives intense peak at 2372 cm^{-1}.

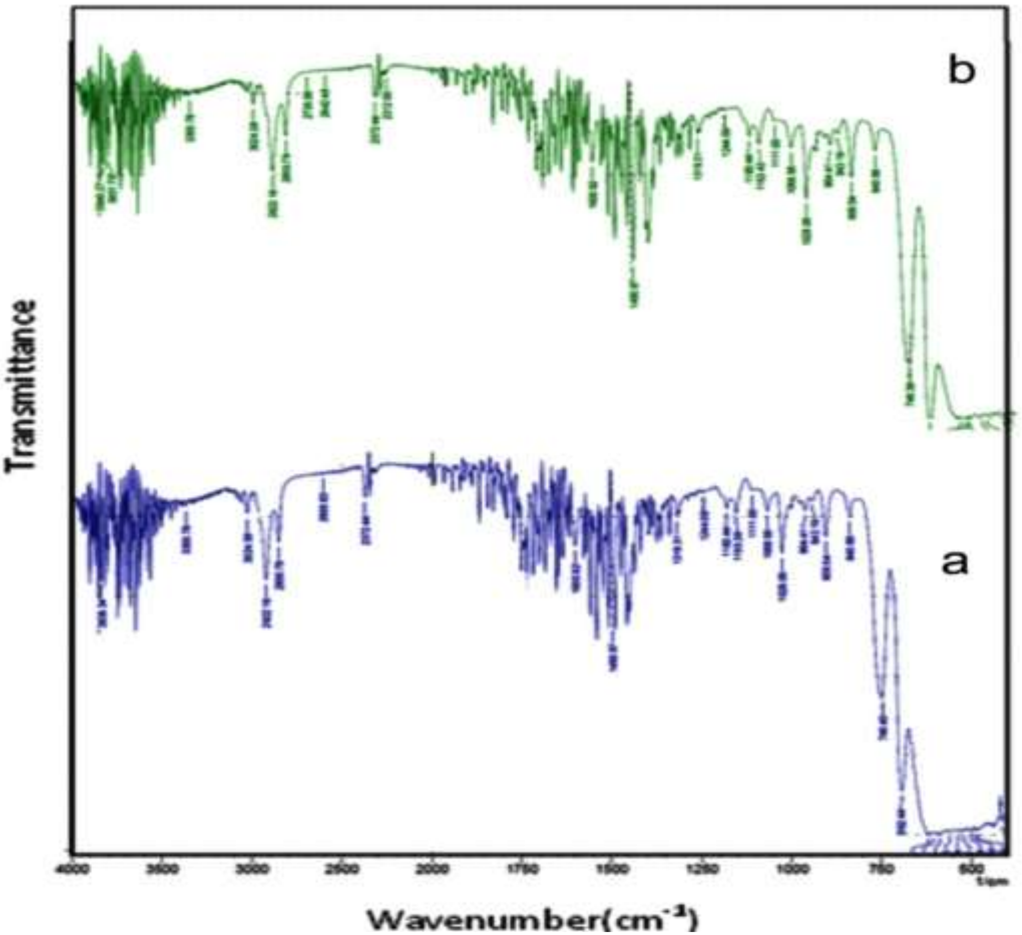

Figure 3: FTIR spectra for (a) CdS-1%/PS (b) CdS-15%/PSsamples

Peaks due to C-H bending are observed at about 906 cm^{-1} and 692 cm^{-1}. C=C asymmetric stretching occurring at 746 cm^{-1}. The peak at 1028 cm^{-1} is assigned to O-H bending on the surface ofPS. The weak peaks at 1155 cm^{-1}and 1111 cm^{-1} due to C=C bending [12]. As CdS percentage increases, the characteristic peaks of CdS appear in the case of the composite thin film.

Table 3 (b): Frequency correlation table for CdS-1%/PS sample

S.N.	Frequency (cm^{-1})	Correlation
1	2922	C-H stretching
2	2851	C-H stretching
3	2372	C≡C stretching
4	1452	C-H stretching
5	1155	C=C Asymmetric Bending
6	1111	C=C Asymmetric Bending
7	1028	O-H Bending
8	906	C-H Bending
9	746	C=C Asymmetric Stretching
10	692	C-H Bending

Functional groups of unfilled PS are present in Figures 3 (a) and (b) and our result is in good agreement with 13, 4,5 and 6. The presence of all the entire vibrational peak frequencies confirms [Figure 3 (a) and (b)] the existence of filler CdS in PS composite samples. It also shows the reduction in the intensity of peak frequencies. The reduction in the intensity of peak frequencies indicates the morphological changes [15].

As on comparison of FTIR spectra of 1, 5, 10 and 15 weight percentage CdS filled PS composite thin films, the reduction in intensity of peak frequencies is more prominent in the thin film samples of high weight percentage. Thus structural changes are more advanced in thin films of 15 weight percentage which may be due to increasing weight percentage of filler CdS in the composite sample.

From above explanation it is concluded that, the appearance of new peaks along with changes in the existing peaks in the FTIR spectra directly indicates the complexation of CdS with PS and are consistent with result of XRD.

Table 3 (c): Frequency correlation table for CdS-15%/PS sample

S.N.	Frequency (cm^{-1})	Correlation
1	2922.16	C-H stretching
2	2850.79	C-H stretching
3	2372.44	C≡C stretching
4	1490.97	C-H stretching
5	1153.43	C=C Asymmetric Bending
6	1111	C=C Asymmetric Bending
7	1028.06	O-H Bending
8	906.54	C-H Bending
9	748.38	C=C Asymmetric Stretching
10	692.44	C-H Bending

Raman Spectroscopy

The Raman spectra of the prepared samples of CdS filled PS thin films for 1 and 15 weight percent of CdS was recorded and are shown in Figure 4 in the range 100-1000 cm^{-1}. The Raman spectra of all composite thin films were taken but in order to save space, only for lower and higher percentage that is for 1 and 15 weight percentage are shown here however Raman spectra of 5 and 10 weight percentage of composite thin films and also unfilled PS thin film are avoided.

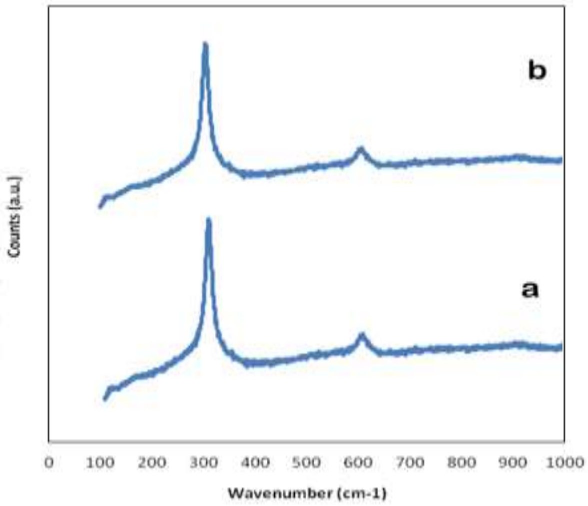

Figure 4: Raman spectrum for (a) CdS-1%/PS and (b) CdS-15%/PS samples

From the Figure 4 (a) the Raman shift at 304.93 cm^{-1} corresponds to the longitudinal optical phonons(LO) mode of CdS [16] Very little wave number shift is observed for 15 weight percentage CdS filled PS film. The other peaks are from the vibrations of the organic molecules. The Raman spectra of nanoparticles of CdS clearly show first and second order longitudinal optical phonon Raman peaks at 304.93, 606.79 cm^{-1}, 3rd one is hump like structure at 917cm^{-1}.

From Raman spectroscopy, it is observed that a CdS filled PS composite thin film of 1 and 15 weight percentages was formed. It also confirms the stability of composite thin film.

4. Conclusion

We have successfully prepared a new PS composite thin film using CdS as filler by solution casting technique. The complex formation in CdS/PS composite system has been confirmed from the XRD results. SEM of all the samples revealed the successful incorporation of CdS nanoparticles in PS matrix.FTIR confirms that the prepared sample is PS/CdS thin film. In Raman Spectroscopy asymmetry is the Raman line shape indicated the effect of phonon confinement.

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