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$CdCl_2$ doped PVA-PVP Blends investigated using Doppler Broadening Spectroscopy and complementary techniques

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Abstract *Films of Cadmium chloride ($CdCl_2$) doped Poly(vinyl alcohol) (PVA) - Poly(vinyl pyrrolidine) (PVP) blend, in the doping range varying from 0.5 wt% up to 5.4wt%, were studied using Doppler Broadening Spectroscopy (DBS).Using the line shape parameters of DBS, the micro-structural changes in the blend films caused by doping were studied. DSC spectra on the doped samples support the interpretation of results obtained using XRD and DBS. It is noted that the degree of crystallinity of PVA-PVP blends increases from 6% up to 23% on increasing the $CdCl_2$ content from 0.5wt% up to 3.3 wt%. This is correlated with the lineshape parameters of DBAR.*

Introduction

Doppler Broadening Annihilation Radiation (DBAR) of Positron Annihilation Spectroscopy (PAS) is an efficient tool to understand the interaction of low momentum electrons and high momentum electrons of the material by extracting the line shape parameters (S- and W-parameters) from DBAR spectrum. Poly(vinyl alcohol) (PVA) and Poly(vinyl pyrrolidine) (PVP) have gained the attention of researchers due to their compatibility after blending. The modified band structure of PVA-PVP doped with metal salts has been studied by various groups. The DBAR techniques have been also studied for PVA-PVP blend doped with $CoCl_2$ and MPDMAPP [1, 2].

Experimental

Semicrystalline, PVA (1,40,000 molecular weight), amorphous PVP (50,000 molecular weight) and $CdCl_2$ were purchased from HiMedia Laboratories Pvt. Ltd, Mumbai. $CdCl_2$ doped PVA-PVP blend films are prepared by solution casting method. Aqueous solution of blends prepared by taking PVA and PVP in ratio of 50:50 (proportion by weight) and dissolving in double distilled water and standard solution is prepared by dissolving $CdCl_2$ in double distilled water. Different volumes of standard solution was added to aqueous solutions of PVA-PVP blends to get samples of dopant range from 0.5wt% up to 5.4wt%. An air cooled, temperature controlled oven was used to get the films from the solutions, which had maintained just above the room temperature (at 35 $^{\circ}$C). The doped films were peeled off from glass substrate, carefully labeled and stored.

The DBAR spectrum have recorded using HPGe detector having beryllium window, by placing the positron source sandwiched between the assemblies of $CdCl_2$ doped PVA-PVP blend films. The acquired spectra were analyzed to yield the line-shape parameters [3]. X-ray diffraction pattern (Intensity Vs 2θ) (see Fig.2) was obtained over scattering angle (2θ) varying from 10° up to 80°, using Cu K_{α} radiation of wavelength 1.5406Å and analyzed in order to understand the nature of samples at different concentration of $CdCl_2$. In order to understand the thermal properties of $CdCl_2$ doped PVA-PVP blend films, Differential Scanning Calorimetry (DSC) curves are recorded using TA instruments DSC Q20 V24.10 Build 122. Samples taken for DSC study are of weight 1-3 mg and, heated from room temperature 50 $^{\circ}$C up to 400 $^{\circ}$C in nitrogen atmosphere (see Fig.3).

Results and Discussion

DBAR Study

The S-parameter is defined as ratio of area under the 511 keV peak normalized to the total area, and W-parameter is defined as normalized area under the wings region of the peak [4]. Obtained values revealed the nature of PVA-PVP blends at a different level of $CdCl_2$. In Fig.1, decrease in S-parameter from 0.5% up to 1.8% and it increases at 3.3% and above; indicates the

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annihilation of positron with the low momentum electrons in the $CdCl_2$ doped PVA-PVP blends. This reveals that crystalline and amorphousness of material with incorporation of different concentration $CdCl_2$ in PVA-PVP blend, this is reflecting in XRD and DSC studies too. The W-parameter shows reverse trend as that of S-parameter.

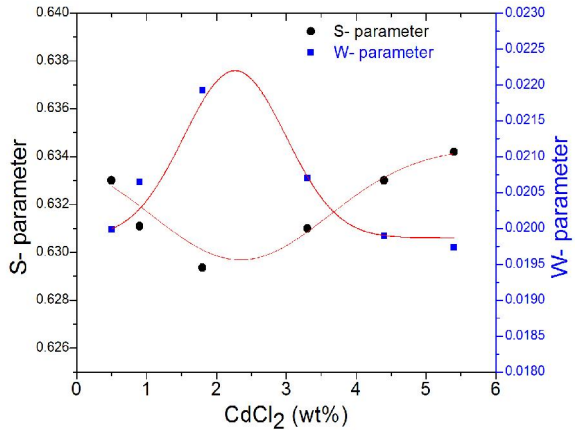

Fig. 1: Variation of S- and W-parameters at different level of $CdCl_2$

XRD studies

In the X-ray diffraction pattern, seen in Fig. 2 shows the more than one sharp peak, indicates that more than one cryslalline regions in $CdCl_2$ doped PVA-PVP blend films. The broad diffraction peak is seen at $2\theta=20^{\circ}$ reveal the semicrystalline nature of the film. This corresponds to (110) reflections (JCPD file No. 41-1049). And the intensity of this peak increased initial dopant levels and decreased at 5.4%, reflects the enhancement in amorphousness due to interaction between ions of dopant (Cd^{2+} and Cl^{-}) with polymer matrix.

DSC Analysis

The DSC scans confirmed the multi-crystalline domains of different thickness. In the dopant levels, 0.5%, 0.9% and 1.8% just resolved additional endotherms with followed by one dominating sharp endotherm (T_m mentioned in Fig. 2) indicates the presence of different thickness crystallite domains. These multi crystalline domains are PVA+Cd^{2+}/Cl^{-} and PVP+Cd^{2+} /Cl^{-}.

Conclusions

The Crystalline and amorphous nature of PVA-PVP blends at different levels of $CdCl_2$ have been studied by DBAR, XRD and DSC studies. And this modified nature is due to inter/intra molecular interactions of functional groups of PVA and PVP with $CdCl_2$.

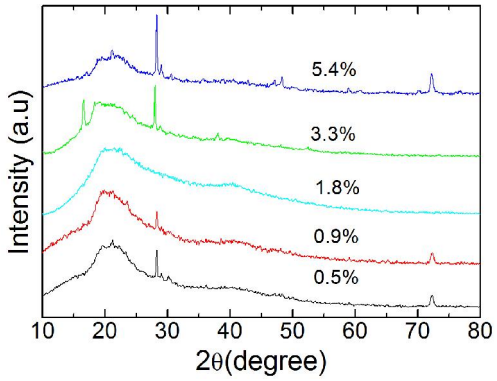

Fig. 2: X-Ray diffracted pattern at different level of $CdCl_2$

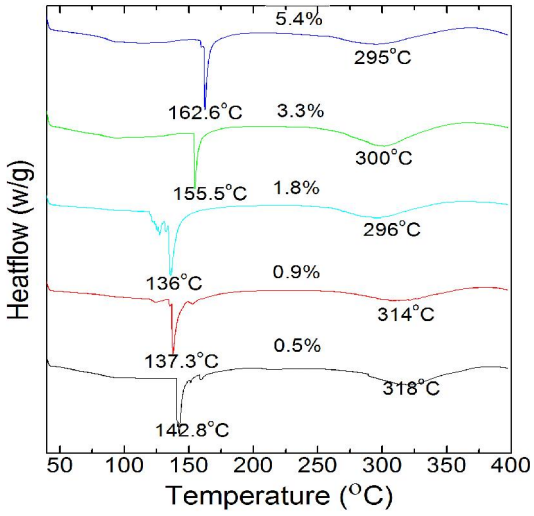

Fig. 2: DSC scans at different level of $CdCl_2$.

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