Original Article

Effect of cesium bromide on the structural, optical, thermal and electrical properties of polyvinyl alcohol and polyethylene oxide

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The molecular structures of polyvinyl alcohol and polyethylene oxide doped with various concentrations of CsBr were investigated by FT-IR, UV/Vis. and XRD. The surface of the prepared films was studied by SEM. Thermal and electrical properties were studied via TGA, DSC and DC electrical conductivity to use as a sensor in electronic devices. X-ray diffraction displays the semicrystalline nature of PVA/PEO, characterized by two broad bands at $2\theta \approx 19$ and 23\textdegree, which decreased by increasing cesium bromide. FT-IR spectra revealed that there is a change in intensities of some bands compared with the spectrum of the pure blend. This indicates that interaction and complexation between PVA/PEO polymer blend and the filler took place in amorphous regions. UV/Vis. spectroscopy revealed that the values of the optical energy gap are decreased with increasing the CsBr concentration, which decreased from 5.90 to 4.90 eV for direct transition and decreased from 4.72 to 3.51 eV for indirect transition. TGA curves depicted that the thermal stability of samples was changed. DSC analysis showed a single glass transition temperature ($T_g = 60^\circ$C), which confirms the miscibility of the prepared films. The DC graph showed that the conductivity of the samples had been increased with increasing the fillers content and the activation energy was decreased from 1.03 to 0.36 eV. SEM displays transparent, soft and a uniform surface for polyvinyl alcohol and polyethylene oxide while after adding CsBr there is a semi- tori/ granules randomly distributed on the surface.

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1. Introduction

Polymer study remains a growing field in which blends play an essential role due to their relatively simple preparation. A blending of polymer materials is the newest method for improving a new polymeric matrix and a beneficial tool for creating materials with a wide variety of properties [1]. Polymer properties are improved by combining two or more polymers to be used in specific applications. [2]. The melt blending and solvent casting are the appropriate way for fabricating polymer blends [3].

Polyvinyl alcohol (PVA) can be classified as amorphous or semicrystalline according to the internal structure. Semicrystalline PVA can be synthesized via the creation of both amorphous and crystalline areas [4]. Furthermore, PVA has a carbon chain backbone with hydroxyl groups. These OH-groups act as a source of hydrogen bonding, which assists in the formation of polymer blends.

Polyethylene oxide (PEO) is a semicrystalline polymer possessing both amorphous and crystalline phases at room temperature [5]. PEO is characterized by high viscosity, high chemical and thermal stability, good water solubility, non-ionic and heat forming [6,7]. PEO is described by their flocculent thickening sustained release lubrication dispersing fiber and water retention [8–10]. PEO is a simple matrix copolymer having enteric linkages, whereas PVA is a polymer containing OH groups, which acts as a source of hydrogen bond and connect with PEO to create the polymer blend PVA/PEO [11]. We used the ratio 50/50 wt.% of PVA/PEO polymer blend in the present study to give good miscibility as in previous work [2]. Our current research has shown that metals halides enhanced the physical properties and the structure of polymer blend [12,13].

Transition metal halides (TMH) or metallic salt possess a significant influence on the physical properties of filling polymeric matrix [14]. The improvement of optical and electrical properties of the blend can be accomplished via the addition of some filler to the host polymer matrix. PVA/PEO with appropriate metal halide can get complexes which are convenient for such applications, for example, electronic and optical devices [15,16].

The current work focuses on the role of cesium ion in improving and modifying the optical, structural, electrical and thermal properties of PVA/PEO polymer blend filled with various amounts of cesium bromide.

2. Experimental

2.1. Materials

PVA was supplied by Merck-Germany with $M_w \approx 75,000$ gm mol$^{-1}$ and also PEO was supplied by ACROS, New-Jersey, U.S.A. with $M_w \approx 60,000$ gm mol$^{-1}$. PVA and PEO were utilized as a fundamental material. CsBr was obtained from Sigma Aldrich Germany.

Equimass of Polyvinyl alcohol and Polyethylene oxide (50/50 wt. %) was dissolved in DDW (double distilled water) and the solution was stirred for about 8 h at room temperature to complete dissolution until a suitable viscous solution is achieved. The obtained solution of PVA/PEO was doped with various concentrations of CsBr (S = 0.5, 1, 2, 4, 10 and 20 wt. %). The PVA/PEO/CsBr solution was dissolved again in DDW with continuous stirring at the same conditions. The viscous solution was poured onto cleaned glass Petri-dishes and put in an oven for 4 days to ensure removal of the solvent traces. The prepared samples were peeled from Petri-dishes. The films were cut into suitable pieces for measurements.

2.2. Measurements

FTIR analysis was accomplished for all prepared samples via single-beam Fourier Transform-Infrared (Nicolet iS10- U.S.A.) in the wavenumber range from $4 \times 10^3$ to $4 \times 10^2$ cm$^{-1}$. XRD analysis was achieved by PAN’alytical X Pert PRO’ XRD system using Cu K$_\alpha$ radiation (where $\lambda = 1.541\AA$, the tube worked at voltage 30 kV and 20 range from 5$^\circ$ to 50$^\circ$. The peak locations [20] in XRD analysis was utilized to detect the various crystalline phases in the polymer blend PVA/PEO and filled films. UV/Vis. spectroscopy was measured in a spectrophotometer (JASCO 630, Japan) where ($\lambda$) range from [190–1100] nm, to investigate the variation in the structure of the prepared films and their optical properties due to the addition of CsBr. TGA curves for the prepared films were measured in an equipment (NETZSCH- STA 409’CD-Germany) in the temperature range from 30 to 600 $^\circ$C at a heating rate 5$^\circ$ degree per min. DSC analysis was carried out using a calorimeter (SETARAM’ labsy TG/DSC. 16) at temperature from 30 to 600 $^\circ$C and the rate of heating was 5$^\circ$ per min. Electrical conductivity was achieved by auto-ranging multi-meter (METRISO’ 5000 A, USA). The surface of the samples was studied using scanning electron microscopy (SEM) in an equipment (Quanta FEG/250), working at 30 kV accelerating voltage.

3. Results and discussion

3.1. FT-IR

FT-IR analysis for the obtained samples was utilized to establish the interaction between polyethylene oxide and polyvinyl alcohol filled with cesium bromide, which causes particular modifications in the functional groups of PVA/PEO polymer blend. Fig. 1 shows the FTIR spectra of polyethylene oxide, polyvinyl alcohol and PEO/PVA composite.

The main vibrational band of polyethylene oxide and polyvinyl alcohol were detected. The assignment of essential bands of FTIR spectra presented in this study are summarized and listed in Table 1. A strong broad band at about 3336 cm$^{-1}$ is attributed to asymmetric stretching of OH group of polyvinyl alcohol. The band at about 2902 cm$^{-1}$ is assigned to C–H symmetric stretching. The band between 3000–3500 cm$^{-1}$ of PVA is missing after the blend. This band is attributed to asymmetric stretching of –OH group of PVA. So, it is the evidence that the PVA has the hydrogen bond with the PEO. The bands corresponding to CH$_3$ scissoring and CH$_2$ asymmetric bending appear at about 1464 cm$^{-1}$ and 1345 cm$^{-1}$, respectively. The band corresponding to CH$_3$ symmetric twisting occurs at about 1274 cm$^{-1}$, a sharp band at about 1114 cm$^{-1}$ is attributed
was shifted from 955 to 952 cm\(^{-1}\). This confirms the interactions and complexation between PVA/PEO and CsBr.

### 3.2. XRD

Fig. 3 depicts the XRD patterns of PVA/PEO virgin polymer and the virgin doped with various amounts of CsBr. The X-ray diffraction scan of unfilled PEO/PVA shows two diffraction peaks at 2\(\theta\) = 19\(^{\circ}\) and 23\(^{\circ}\) [21], which indicates a semicrystalline nature of the blend system. The intensities of the two peaks were reduced with increment CsBr contents implying a decrease in the degree of crystallinity consequently increasing the conductivity. It is clear that, the incorporation of CsBr into the polymeric matrix causes an increase in the amorphous regions of the samples. Relative area under an amorphous peak centered at 2\(\theta\) = 19\(^{\circ}\) (as an example) are taken as a measurement of the degree of crystallinity and the values were listed in Table 2. Also, the last samples (S5) show new peaks nearly at 20.6, 29.1, 36.0, 41.9, and 47.0\(^{\circ}\), which occur after the addition of cesium bromide. These peaks may correspond to the face center cubic (FCC) structure of cesium bromide attributed to [h k l] Miller index [111], [110], [111], [200] and [210]. Their positions were in agreement with (JPCDS data no. 7647-17-8). From all previous results, it can be noted that the interaction between polymer blend and the filler causes

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**Table 1 – The bands position and the assignment of the main vibrations band of PVA and PEO.**

<table>
<thead>
<tr>
<th>Wavelength (cm(^{-1}))</th>
<th>Band assignments</th>
</tr>
</thead>
<tbody>
<tr>
<td>3336</td>
<td>OH stretching.</td>
</tr>
<tr>
<td>2902</td>
<td>CH(_2) asymmetric stretching.</td>
</tr>
<tr>
<td>1464</td>
<td>CH(_2) scissoring</td>
</tr>
<tr>
<td>1345</td>
<td>CH(_2) asymmetric bending</td>
</tr>
<tr>
<td>1274</td>
<td>CH(_3) symmetric twisting</td>
</tr>
<tr>
<td>1114</td>
<td>C—O—C stretching</td>
</tr>
<tr>
<td>955</td>
<td>Out-of-plane rings C—H bending</td>
</tr>
<tr>
<td>844</td>
<td>C—C stretching vibrations</td>
</tr>
</tbody>
</table>

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**Table 2 – The values of the energy gap for indirect and direct transition (\(E_g^d\) and \(E_g^a\)) for the prepared samples and its activation energy (\(E_a\)).**

<table>
<thead>
<tr>
<th>Area under the peak 2(\theta) = 19(^{\circ})</th>
<th>(E_a) (eV)</th>
<th>(E_g^a) (eV)</th>
<th>(E_g^d) (eV)</th>
<th>Sample</th>
</tr>
</thead>
<tbody>
<tr>
<td>332.12</td>
<td>1.03</td>
<td>4.72</td>
<td>5.90</td>
<td>S0</td>
</tr>
<tr>
<td>972.01</td>
<td>0.93</td>
<td>4.55</td>
<td>5.60</td>
<td>S1</td>
</tr>
<tr>
<td>792.37</td>
<td>0.87</td>
<td>4.52</td>
<td>5.55</td>
<td>S2</td>
</tr>
<tr>
<td>582.17</td>
<td>0.75</td>
<td>4.50</td>
<td>5.50</td>
<td>S3</td>
</tr>
<tr>
<td>385.59</td>
<td>0.64</td>
<td>4.43</td>
<td>5.43</td>
<td>S4</td>
</tr>
<tr>
<td>253.64</td>
<td>0.53</td>
<td>3.85</td>
<td>4.75</td>
<td>S5</td>
</tr>
<tr>
<td>105.07</td>
<td>0.36</td>
<td>3.51</td>
<td>4.90</td>
<td>S6</td>
</tr>
</tbody>
</table>
3.3. UV/vis. optical studies

The UV/vis. spectroscopy of the PVA/PEO samples filled with various concentrations of CsBr in the wavelengths range from 190 to 1100 nm is displayed in Fig. 4. The figure shows an absorption edge at 200 nm for all samples. The position of the absorption edge was shifted towards longer wavelength (red shift) by increment of CsBr concentrations. These shifts show the creation of intermolecular hydrogen bonding among Cs\(^+\) with the hydroxyl group, which is in agreement with FTIR result [22,23]. The appearance of absorption edges may be attributed to n → π\(^*\) (R-band) electronic transitions. Also, in all films, there is no absorption peak on the longer wavelength (visible region), which indicates that the samples are transparent.

3.4. A calculation of energy gap (E\(_g\))

The band structures of a polymeric material can be understood by investigating its optical absorptions curves. The fundamental absorptions display an unexpected augmentation, known as absorption edge as in Fig. 4, which can be used to calculate the energy gap (E\(_g\) = h c /\(\lambda\)). The energy gap can be calculated by the following equation [24]:

\[
(\alpha h\nu) = C(h\nu - E_g)^{1/2} \text{for } h\nu > E_g
\]

(a) PVA/PEO blend
(b) PVA/PEO with 20 wt.% CsBr.
Where $h\nu$ is photon energy, $E_g$ is the energy gap, $C$ is constants and $\alpha$ is the absorption coefficient. The value of $\tau$ determines the type of electronic transition, which takes the value 2 for direct transition and 1/2 for the indirect transition. The value of $(\omega)$ can be determined by the following formula [25]:

$$\alpha(\omega) = 2.303 \times A/d$$  \hspace{1cm} (2)$$

where $d$ is the thickness of the prepared films and $A$ is the absorbance. Shalliday and Davis [26] recommended that at the fundamental band gap, both direct and indirect transition arise and can be determined by the plotting of $(\omega h\nu)^{1/2}$ and $(\omega h\nu)^{\tau}$ respectively, against the energy of incident photon [$h\nu$]. Fig. 5(a, b) displays the plot of $(\omega h\nu)^{1/2}$ against [$h\nu$] and Fig. 6(a, b) shows the plot of $(\omega h\nu)^{\tau}$ against [$h\nu$], which is used to calculate the energy gap [Eg]. Figs. 5 and 6 illustrate the width and nature of the band gaps for the selected samples. The values of the band gaps were calculated by extrapolating the linear portion of the plots and recorded in Table 2. It can be observed that the values of the direct and indirect band gap were decreased with increasing cesium bromide content. The reduction in the energy gap was due to the defects in the polymer composite as well as the increment in the degrees of disorder. This defect creates a localized state in energy levels. These overlaps are responsible for reducing in Eg with increasing in cesium bromide contents in the polymer blend PVA/PEO [27]. All the previous results confirmed the XRD studies.

### 3.5 DSC analysis

The thermal properties of PVA/PEO polymer blend were studied by differential scanning calorimetry (DSC) to evaluate how the thermal transition of the polymer blend was influenced by the various amounts of cesium bromide [28] as showed in Fig. 7. All films were heated in the range 30–500°C with a heating rate of 10 degrees centigrade per minute. The glass transition temperature ($T_g$), melting point ($T_m$) and decomposition temperature ($T_d$) are listed in Table 3. Pure PVA/PEO curve displays a small endothermic peak at 60°C corresponding to $T_g$ [29]. The existence of a single $T_g$ shows the miscibility of the polymer composite [30]. The endothermic peak at 245 and 404°C was attributed to ($T_m$) and ($T_d$), respectively, are observed for Pure PVA /PEO curve. The positions of $T_g$, $T_m$, and $T_d$ for the prepared materials were slightly shifted toward higher temperatures, which suggests that the CsBr acts as enhancer the thermal properties of the PVA/PEO blend. These results confirm that the filler contributed in the enhancement of the thermal stability, which signifies the creation of intermolecular interaction between cesium bromide and polymer blend. The presence of such interactions was established by the results of X-ray diffraction and FTIR finding.

### 3.6 Thermogravimetric analysis (TGA)

Fig. 8 interpreted thermogravimetric analysis of residual mass as a function of temperature (T °C) for PVA/PEO composites and blend prior and after the addition of CsBr. All samples were heated in the range of 30–500°C with a heating rate of 10 degrees centigrade per minute. The initial weight loss (in the first region) for all films arises at 30–230°C because of the moisture evaporation and the second loss appears at 230–280°C. After that the prepared films were stable up to 280–380°C. In The third region, the weight loss for all samples appears at 400–440°C. From Fig. 8, it is clear that the thermal decomposition of all films was shifted towards higher temperature compared with PVA/PEO blend, which signifies the improvement of the thermal stability of the polymer matrix.

#### Table 3 - Glass transition ($T_g$), melting point ($T_m$) and thermal decomposition ($T_d$) temperature for all prepared films.

<table>
<thead>
<tr>
<th>CsBr wt.%</th>
<th>$T_g$ (°C)</th>
<th>$T_m$ (°C)</th>
<th>$T_d$ (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.0</td>
<td>60.0</td>
<td>245</td>
<td>404</td>
</tr>
<tr>
<td>0.5</td>
<td>68.56</td>
<td>250</td>
<td>419</td>
</tr>
<tr>
<td>1.0</td>
<td>68.60</td>
<td>252</td>
<td>421</td>
</tr>
<tr>
<td>2.0</td>
<td>69.59</td>
<td>254</td>
<td>422</td>
</tr>
<tr>
<td>4.0</td>
<td>69.69</td>
<td>255</td>
<td>423</td>
</tr>
<tr>
<td>10.0</td>
<td>63.64</td>
<td>249</td>
<td>425</td>
</tr>
<tr>
<td>20.0</td>
<td>71.03</td>
<td>257</td>
<td>427</td>
</tr>
</tbody>
</table>
This improvement may be attributed to the structural decomposition of the PVA/PEO blend. From Fig. 8, all films are stable up to about 445 °C, which can be understood as a result of the chemical interaction between cesium bromide and PVA/PEO blend. Then the addition of CsBr to PVA/PEO blend improves its thermal stability. These results indicate that CsBr behaves as an effective thermal barrier due to its large aspect ratio and thereby hinders the degradation of PEO/PVA. Table 4 listed the ratio weight loss at various decomposition temperatures of the films taken from the thermogravimetric analysis thermogram.

### Table 4 – The percentage weight loss at various decomposition temperatures of PVA/PEO blend and their complex was taken from the (TGA) thermogram.

<table>
<thead>
<tr>
<th>CsBr Wt. %</th>
<th>T (°C)</th>
<th>D60</th>
<th>D65</th>
<th>D70</th>
<th>D80</th>
<th>D90</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.0</td>
<td></td>
<td>406</td>
<td>396</td>
<td>325</td>
<td>265</td>
<td>253</td>
</tr>
<tr>
<td>0.5</td>
<td></td>
<td>410</td>
<td>405</td>
<td>396</td>
<td>277</td>
<td>257</td>
</tr>
<tr>
<td>1.0</td>
<td></td>
<td>414</td>
<td>411</td>
<td>401</td>
<td>308</td>
<td>261</td>
</tr>
<tr>
<td>2.0</td>
<td></td>
<td>419</td>
<td>416</td>
<td>404</td>
<td>323</td>
<td>265</td>
</tr>
<tr>
<td>4.0</td>
<td></td>
<td>422</td>
<td>419</td>
<td>409</td>
<td>392</td>
<td>269</td>
</tr>
<tr>
<td>10</td>
<td></td>
<td>426</td>
<td>421</td>
<td>411</td>
<td>396</td>
<td>271</td>
</tr>
<tr>
<td>20</td>
<td></td>
<td>433</td>
<td>425</td>
<td>415</td>
<td>400</td>
<td>282</td>
</tr>
</tbody>
</table>

Fig. 9 – Dependence of (log $\sigma$) on [1000/T [K-1]] for PVA/PEO doped with different concentrations of CsBr.

3.7. DC electrical measurements

Fig. 9 depicts the dependence of log ($\sigma$) on the absolute temperature for different concentrations of the CsBr in the polymer blend. From this figure, the values of ($\sigma$) increase with increasing temperature for all the prepared samples. The conductivity behaviour did not display any abrupt jump with temperatures. This shows the semicrystalline nature of the prepared materials [31], which is confirmed by an X-ray diffraction study. The increment in ($\sigma$) with T (K) can be linked to the reduction in the viscosity and thus to the augmented chain flexibility, which is in agreement with the free volume theory [32,33]. The determination of activation energy and charge conduction behaviour was investigated by the following formula:

$$\sigma = \sigma_0 \exp(-\Delta E_a/KT)$$

(3)

Where K is the Boltzmann constant = $1.38 \times 10^{-23}$ J/K, $\sigma_0$ is an exponential factor, $\Delta E_a$ is the activation energy and T is the absolute temperature. The values of activation energy for the prepared samples were computed and listed in Table 2. The electrical conductivity increased with increasing CsBr concentrations while the values of activation energy decreased with increasing CsBr amounts. The conductivity was improved, which suggests that the CsBr can be used as a good filler to enhance the electrical conductivity of PVA/PEO blend.

3.8. A determination of hoping distance ($R_o$)

The mechanisms of DC conduction can be understood via Kuivalainen model [34]. The electrical conductivity ($\sigma$) can be expressed by the following formula:

$$\sigma = \frac{A e^2 \gamma(T) T^2}{K T} \frac{\zeta}{R_0} \exp\left(\frac{-2B R_0}{\gamma}ight)$$

(4)

where $A=0.45$, $B=1.39$, $\gamma_p$ and $\gamma_{bp}$ are the concentration of polaron and bipolaron, respectively, $R_0$ is the typical separation between impurities, $\zeta = \left(\frac{\gamma_p}{\gamma_{bp}}\right)^{1/2}$ is the average decay length of the polaron and bipolaron wave function, $\zeta_1$ and $\zeta_\perp$ are the decay lengths parallel and perpendicular to the polymer matrix. The transition rate can be expressed by the following formula:

$$\gamma(T) = 1.2 \times 10^{17} (T/300 K)^{11}$$

(5)

The parameter $\zeta_\perp = 0.22$ nm while $\zeta_1 = 1.06$ nm, which are dependent on the inter-chain resonance energy and the inter-chain distance. The values of Ro are calculated via Eqs. (4) and (5).

Fig. 10 – The variation of hoping distance with T(K) for PVA/PEO/CsBr polymer composite.
Fig. 10 shows that the values of the hoping distance [Ro] present a linear decrease with an increase in the temperature for all prepared films. This reveals that the concentrations of thermally activated polaron and/or bipolarons increment gradually as the temperature augmented. The values of the hoping distance ranging from 8.01 to 1.83 nm are shown in Fig. 10, which suggests that the conduction mechanism for the prepared films is an intrachain one-dimensional hopping type [35]. Fig. 11 depicts that the values of hoping distance decrease with increase CsBr concentrations that means the CsBr is a good filler to enhance the electrical conductivity of PVA/PEO.

3.9. Scanning Electron Microscopy (SEM)

Scanning electron microscopy (SEM) has been used to study the influence of the dispersion of CsBr particles on the surface of prepared films. Fig. 12 displays the SEM of PVA/PEO doped with different concentrations of CsBr. The morphology of pure polymer PVA/PEO is transparent, smooth and brittle, which is displayed to be in a uniform surface. After the addi-
tion of CsBr with different concentrations, the images have uniform surfaces; however, it is semi-tori with various sizes. These semi-tori occur as white spots in all filled samples. The observed white or bright spots are distributed uniformly on the back-scattered images and appear to be agglomerate of the Cs particles which increment with augmented the CsBr content. There are spherulites in SEM image, which confirm the PEO crystallite domains and are increment with increasing cesium content. The degree of roughness of the samples is increment with increasing cesium content. This shows segregation of the CsBr in the polymer chain, which confirmed the complexation and interaction between PVA/PEO pure blend and CsBr. In the last two samples S5 and S6, it was noticed an increase in the bright spots numbers and that is considered as a big proof that the saturation state was reached. So, there is no need for preparing of samples with higher concentrations.

4. Conclusion

FT-IR spectra of PVA/PEO polymer blend filled with different concentrations of CsBr shows a relative reaction between the blend and CsBr. The intensities of some vibrational bands are change after the addition of CsBr. This suggests that there are interaction and complexation between PVA/PEO and CsBr. XRD scans revealed a semicrystalline nature of polymer composite, which is characterized by two broad bands at 2θ =19 and 23° and showed an increase in the amorphous regions with increasing CsBr. The values of the energy gap were reduced with a continuous increase in the filling level of CsBr due to charge transfer complexes (CTCs) between the functional groups of PVA/PEO and cesium’s atoms. DSC and TGA revealed that the incorporation of CsBr to the PVA/PEO polymer blend causes an increase in thermal stability. The conductivity has been enhanced as compared with the PVA/PEO blend. The hoping distance (Rn) was decreased from 8.01 to 1.83 nm. SEM shows uniform surfaces; however, there are semi-tori with various sizes. These semi-tori occur as bright spots in all filled samples with different degrees of roughness. The interactions and complexation between the PVA/PEO and CsBr were confirmed by XRD, FTIR, UV/Vis., DSC and SEM studies.

Conflict of interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

REFERENCES


