

Spectroscopic investigations of Dy³⁺ doped PVA/PVP blend polymer films

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Abstract:

The polymeric blend Polyvinyl alcohol (PVA)/Polyvinyl pyrrolidone (PVP) and various concentrations of Dy³⁺ ions doped PVA/PVP polymer films were prepared using a solution cast process. The prepared polymer films spectroscopic properties were studied through X-ray diffraction (XRD), Fourier transforms infrared (FTIR), Ultraviolet-Visible-Near Infrared (UV-Vis-NIR) analysis. XRD confirmed the semi-crystalline nature of blend polymer films. Structural details and ion-polymer interaction mechanisms were studied through FTIR spectra. From the energy band positions of the absorption spectra, bonding parameters (β & δ) were determined to identify the ionic/ covalent nature of the prepared polymer films. Mott's transitions evaluated the optical absorption edge and bandgap. The absorption edge was found to be 5.1 eV for pure PVA/PVP systems and 3.8 eV for PVA/PVP with Dy³⁺ incorporated. The refractive index of the samples was also measured and found to increase from 2.4 to 2.63 as doping increased to the highest level.

Keywords: PVA/PVP: Dy³⁺ blend polymer films, XRD, FTIR and UV-Vis-NIR.

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I. Introduction

Today, researchers are interested in the luminescent properties of crystalline and amorphous materials doped with rare-earth ions (RE) because they are applied to optoelectronic components and sensors such as lasers light-emitting diodes (LEDs), amplifiers, fiberglass, color screens, etc. [1-7]. Furthermore, the rare-earth ions doped polymer blended materials encourage photonic materials for various applications in the applied spectroscopy field. Polymer blending is an essential method for developing new polymeric material for multifunctional device applications and low fabrication costs than the currently available materials.

Polyvinyl alcohol (PVA) is a semi-crystalline, water-soluble, chemical resistant, high mechanical strength, and non-toxic synthetic polymer. PVA has lots of textiles, paper, synthetic fibres, and polymer industries [8]. In addition, PVA shows interesting physical properties because OH groups are liable for hydrogen bond structure with other polymers [9].

The second polymer chosen for a polymer blend with PVA is polyvinyl pyrrolidone (PVP). It was well-known that PVP has more rapidly ionic mobility than other polymers with a low scattering loss, making it an ideal polymer for blending. Furthermore, due to the carbonyl group (C=O) in the side chains of PVP, it presents different complexes with a selection of inorganic salts in their additional enhancement as luminescent materials allow those with applications in optoelectronic industry and signal transmission [10-12].

A white light-emitting diode (W-LED) plays a vital role in solid-state lighting designs owing to its properties such as long lifetime, low electrical consumption, brightness, high efficiency, and Eco-friendly [13]. Among RE ions, Dy³⁺ ions (4f⁹) are suitable for white light applications without any other RE [14].

II. Experimental

II.1. Samples preparation and measurements

The solution Cast technique was used to make blended polymer films. Polyvinyl alcohol (PVA) (CH₂CH(OH))_n [average molecular weight 1,25,000 99%], Polyvinyl pyrrolidone (PVP) [average molecular weight 13,00,000 99%] and Dysprosium nitrate hydrate (DyN₃O₉·xH₂O) [average molecular weight 348.51 g/mol 99.9%] were used as precursor materials. PVA/PVP (0.5/0.5 wt%) was added to distilled water and allowed to evaporate with continuous stirring at room temperature (RT). A quantity of Dysprosium nitrate (0.0, 0.1, 0.2, 0.3, 0.4 & 0.5 wt %) was added to a polymer blend solution with stirring continuously for 1 day to get a homogeneous solution. Then, the solution was cast onto Petri dishes and gradually evaporated for three days at RT. After drying, the dried composite polymer films were removed from the Petri dishes and stored in vacuum desiccators to prevent moisture absorption until use. The thickness of the equipped films is 0.01 cm.

X-ray diffraction (XRD) pattern was obtained using Shimadzu model XRD-6000 using CuK_α radiation ($\lambda = 1.5406\text{\AA}$), the tube functioned at 30 kV, the Bragg's angle (2θ) in the region from 10° to 60° . The single-beam Jasco FT-IR 430 spectrometer has been used to obtain FT-IR transmittance spectra for synthesized formulations in the 500 to 4000 cm^{-1} range. The absorption spectra were obtained using spectrophotometer PerkinElmer Lambda 900 in the wavelength region $200 - 2000\text{ nm}$. From the absorption spectra, optical parameters were calculated. Emission spectra and their lifetime analysis were reported with Edinburg instrument FLS 980 equipped with a Xenon arc lamp.

III. Results and discussion

3.1. X-ray diffraction (XRD) analysis

Fig.1 shows the XRD pattern of the pure PVA/PVP and PVA/PVP: $x\text{ wt\% Dy}^{3+}$ ($x = 0.0, 0.1, 0.2, 0.3, 0.4$ & 0.5 wt\%) blend polymer films made a recording in the region $10^\circ \leq \theta \leq 60^\circ$. The XRD pattern shows the characteristic peaks at 15° and 22° , indicating its semi-crystalline nature and corresponding to the (200) reflection plane, which was assigned to previous literature work (JCPDS card No. 65-2870) [15-16]. In this pattern, PVA/PVP peak intensity gradually decreases as Dy³⁺ ion concentration increases from 0.1 to 0.5 wt%, correspond to the incorporation in the PVA/PVP blend polymer film. The peak intensity is strong for the lower concentration compared to a higher concentration of Dy³⁺ ion. From obtained data expose that lower Dy³⁺ ion concentration film becomes more crystalline. In contrast, increasing Dy³⁺ ion concentration reduces the intermolecular interaction between the blend polymer chains, reflecting a reduction of the degree of crystallinity.

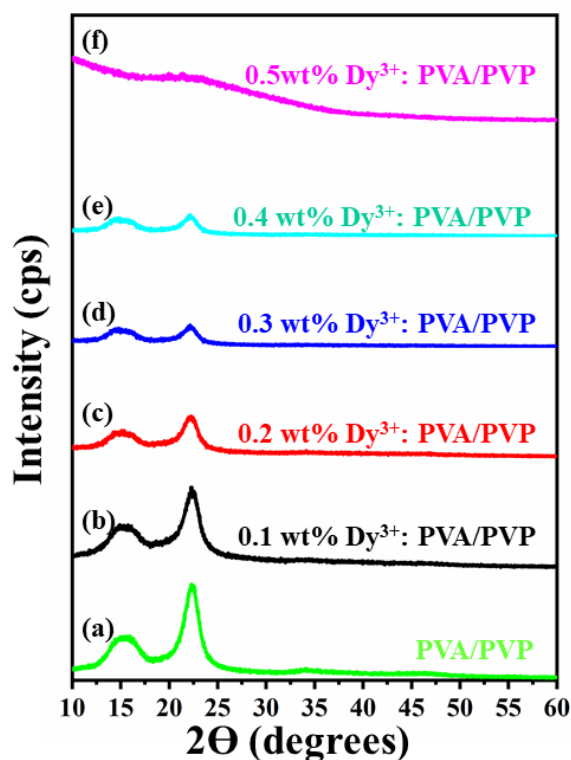


Fig.1 XRD pattern of (a) pure PVA/PVP (b-f) 0.1 to 0.5 wt% Dy³⁺: PVA/PVP blend polymer

3.2. Functional group analysis

The influence of Dy³⁺ ion on the polymer structure is studied using FT-IR spectroscopy to find interactions among various concentrations of PVA/PVP polymer films. The FT-IR transmission spectra of pure PVA, pure PVP, and PVA/PVP (0.5/0.5) blends without and with different constituents of Dy³⁺ are presented in Fig. 2 (a) & (b) in the range of 500 to 4000 cm^{-1} at room temperature. Table 1 lists all the prepared polymer films FT-IR transmission bands and their assignments.

Fig. 2 (a) presents the FT-IR spectra of pure PVA, broadband centered at 3294 cm^{-1} relating to the OH symmetric stretching vibration [17]. The bands were noticed at 2904 cm^{-1} and 1072 cm^{-1} and ascribed to the C-H stretching, C-O stretching vibrations to in-plane and out plane bending vibrations [18]. The bands at 1708 cm^{-1} and 1267 cm^{-1} have been ascribed to C=C vibration and CH₂ bending [19]. A weak band was noticed at 1409 cm^{-1} , consequent to the C-H wagging vibration and C-O stretching of acetyl groups [20]. For pure PVP, the bands at 3228 cm^{-1} and 2937 cm^{-1} correspond to the OH and CH stretching vibrations [21]. The vibrational

bands noticed at 1641 cm⁻¹, 1444 cm⁻¹, 1277 cm⁻¹, 924 cm⁻¹, and 827 cm⁻¹ correspond to C=O stretching, C-H bending, C-N stretching, C-C stretching, and CH₂ rocking of PVP [22].

Fig. 2 (b) represents the FT-IR spectra of Pure PVA/PVP polymer blend without and with various concentrations of Dy³⁺. The bands of pure PVA, pure PVP get shifted in the pure PVA/PVP blended polymer with a frequency shift. The bands at 3294 cm⁻¹ and 2920 cm⁻¹ are related to the O-H stretching and CH₂ asymmetric PVA/PVP blend polymer [23]. The vibrational bands at 1533 cm⁻¹ which is corresponding to pyridine ring C=N of PVP [24]. The bands corresponding to CH₂ twisting (out of plane) and C-H bending vibrations occur at 1300 cm⁻¹ and 1057 cm⁻¹ [25]. Upon adding Dy³⁺ to PVA/PVP blend polymers, FT-IR spectra show shifts in some bands and changes in the intensities of other bands compared with PVA/PVP polymer film. This designates the complex structure between PVA/PVP (0.5/0.5) polymer blend and Dy³⁺ ions.

Table 1 Assignments of the IR characterization bands for pure PVA, pure PVP, and Pure PVA/PVP blend

Pure PVA		Pure PVP		Pure PVA/PVP	
Wavenumber (cm ⁻¹)	Assignment	Wavenumber (cm ⁻¹)	Assignment	Wavenumber (cm ⁻¹)	Assignment
3294	-OH stretching	3228	-OH stretching	3294	-OH stretching
2904	C-H stretching	2937	-CH stretching	2920	CH ₂ asymmetric
1708	C=C stretching	1641	C=O stretching	1533	C=N
1409	CH ₂ wagging	1444	C-H bending	1300	CH ₂ twisting
1267	CH ₂ bending	1277	C-N stretching	1057	CH bending
1072	C-O	924	C-C stretching
....	827	C-C ring

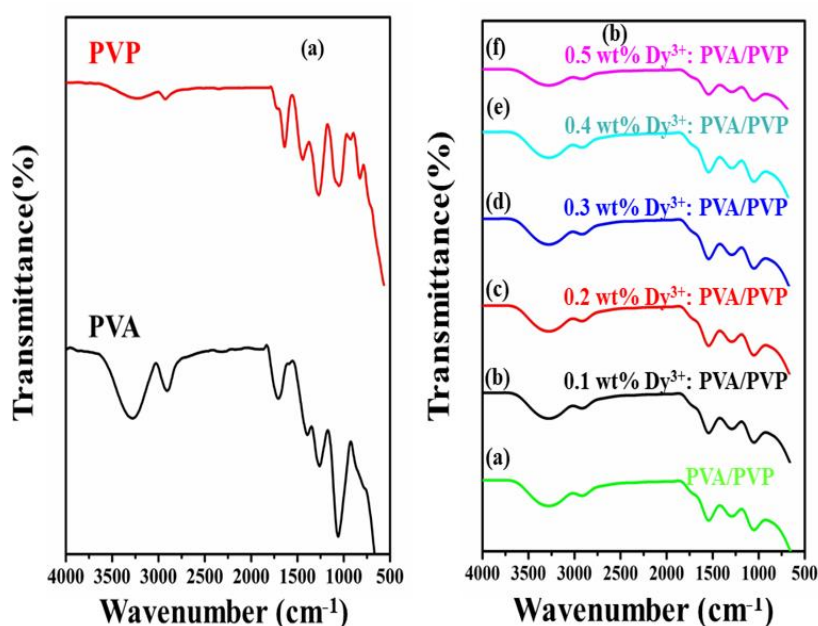


Fig. 2 FT-IR spectra of (a) pure PVA & PVP (b) PVA/PVP and 0.1 to 0.5 wt% Dy³⁺: PVA/PVP blend polymer film

III.3. UV-Vis spectroscopy analysis and bonding parameters

Pure PVA/PVP absorbance spectra region 200-800 nm were displayed in Fig.3. The spectra show absorption band at 220 nm attributed to $n \rightarrow \pi^*$ transition is due to the semi-crystalline nature of the sample [26], which confirms XRD results. In other terms, another absorption band was noticed at 278 nm was assigned to $\pi \rightarrow \pi^*$, which comes from unsaturated bonds [C=O and/or C=C], primarily C=O, which confirms from FTIR results and notices at 1641 cm^{-1} and 1708 cm^{-1} .

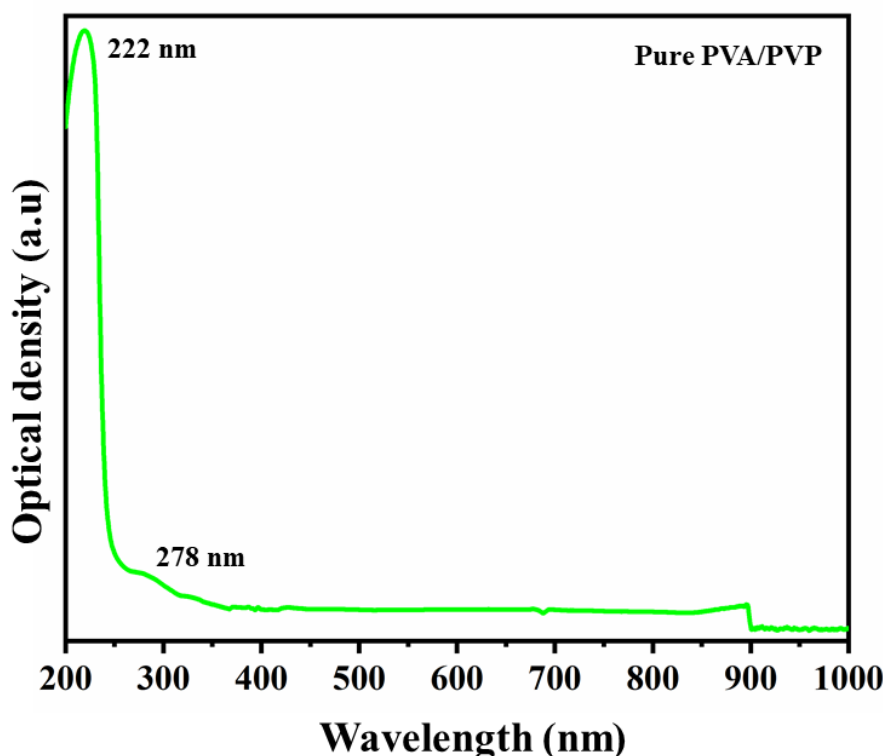


Fig. 3 Absorption spectra of pure PVA/PVP blend polymer films

Absorption spectra were measured in the UV-Vis (300-510) and NIR (600-1800) for PVA/PVP: $x\text{Dy}^{3+}$ ($x = 0.1, 0.2, 0.3, 0.4$ & 0.5 wt %) along with their assignments of optical absorption transitions and shown in Fig.4 (a) & (b) respectively. From Fig. 4 (a), four optical transitions were noticed at 324 nm (${}^6\text{H}_{15/2} \rightarrow {}^6\text{P}_{3/2}$), 349 nm (${}^6\text{H}_{15/2} \rightarrow ({}^4\text{M}, {}^4\text{I})_{15/2}$), 364 nm (${}^6\text{H}_{15/2} \rightarrow {}^4\text{I}_{11/2}$), and 448 nm (${}^6\text{H}_{15/2} \rightarrow {}^4\text{I}_{17/2}$) and Fig. 4 (b) shows five transitions at 755 nm (${}^6\text{H}_{15/2} \rightarrow {}^6\text{F}_{3/2}$), 805 nm (${}^6\text{H}_{15/2} \rightarrow {}^6\text{F}_{5/2}$), 907 nm (${}^6\text{H}_{15/2} \rightarrow {}^6\text{F}_{7/2}$), 1097 nm (${}^6\text{H}_{15/2} \rightarrow {}^6\text{H}_{7/2}$) and 1293 nm (${}^6\text{H}_{15/2} \rightarrow {}^6\text{F}_{11/2}$) respectively. The same absorption levels of Dy^{3+} in PVA and PVP polymer films were reported by Sivaiah and Seeta Rama Raju [27]. For all samples, the optical intensity increases when the concentration of Dy^{3+} ions in the PVA/PVP blend polymer film increases. The nephelauxetic ratio (β) and bonding parameter (δ) were determined from the noticed energy level positions to describe the bonding nature of the Dy^{3+} ligand band in the PVA/PVP blend polymer films [28].

The nephelauxetic ratio is defined as the ratio between the wavenumber (cm^{-1}) of absorption transitions of the Dy^{3+} ion in PVA/PVP under study (ν_c) and the related aqua-ion transition (ν_a) given by

$$\beta = \frac{\nu_c}{\nu_a} \quad (1)$$

From the average values of β termed as $\bar{\beta}$, the bonding parameter (δ) values were estimated with the following expression

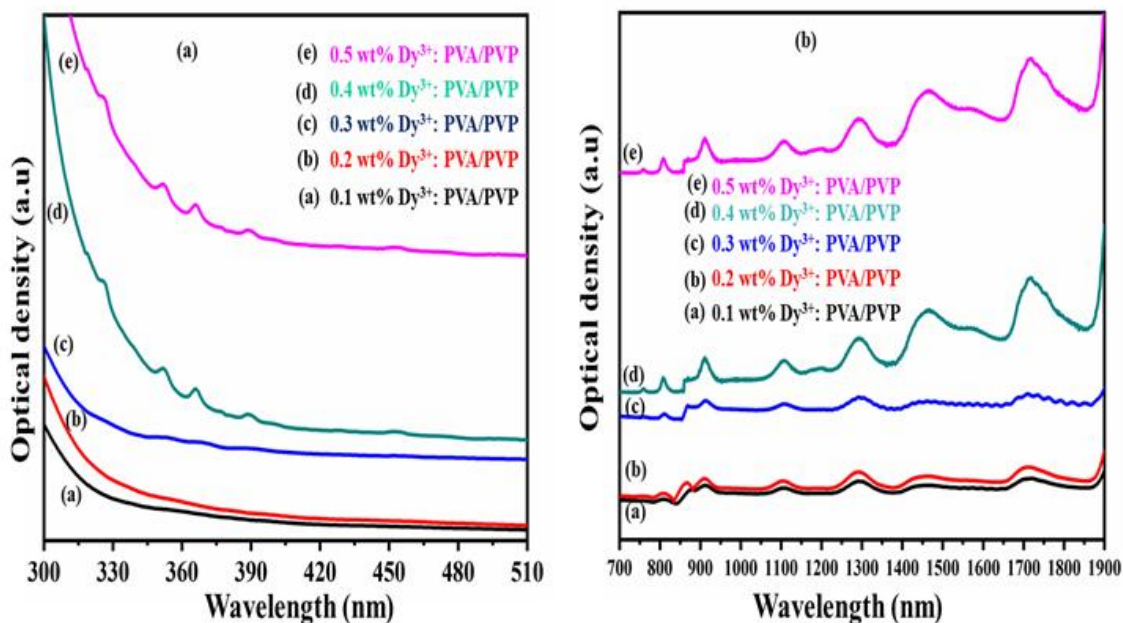
$$\delta = \frac{(1-\bar{\beta})}{\bar{\beta}} \times 100 \quad (2)$$

The noticed band positions (cm^{-1}) and bonding parameters (β, δ) values of the PVA/PVP: $x\text{Dy}^{3+}$ polymer films were listed in Table.2. Depending on the positive or negative sign of the value, the bonding of Dy^{3+} ions with their surrounding ligands might be ionic or covalent. The prepared polymer films have negative values in this study, indicating that they are ionic.

Table 2 Absorption peak positions and energies along with their assignments of Dy³⁺: PVA/PVP blend polymer films

Transition from ⁶ H _{15/2}	Peak position λ (nm)	Energy (cm ⁻¹)		□ = v _c /v _a
		Crystal (v _c)	Aqua (v _a)	
⁶ P _{3/2}	324	30864	30803	1
(⁴ M, ⁴ I) _{15/2}	349	28653	29244	0.98
⁴ I _{11/2}	364	27472	28152	0.97
⁴ I _{15/2}	448	22321	22293	1
⁶ F _{3/2}	755	13245	13212	1
⁶ F _{5/2}	805	12422	12432	0.99
⁶ F _{7/2}	907	11025	11025	1
⁶ H _{7/2}	1097	9115	9115	1
⁶ F _{11/2}	1293	7733	7730	1

□ = 0.99, δ = 1

Fig. 4 Optical absorption spectra of 0.1 to 0.5 wt% Dy³⁺: PVA/PVP blend polymer films in (a) Uv-Visible and (b) NIR region

III.3.1. Optical bandgap

From the absorption spectrum, an optical bandgap was determined for crystalline and amorphous materials. The absorption coefficient (α) was estimated using the following relation to determine the bandgap of the prepared blend polymer films.

$$\alpha = 2.303 \frac{A}{d} \quad (3)$$

where A denotes absorbance and d denotes film thickness.

Table 3 Absorption edge, indirect bandgap, and refractive index of pure PVA/PVP and Dy³⁺: PVA/PVP blend polymer films

Sample	Absorption edge (eV)	Indirect bandgap energy (eV)	Refractive index (n)
PVA/PVP	5.1	5	2.4
0.1 wt% Dy ³⁺ : PVA/PVP	4.9	3.9	2.56
0.2 wt% Dy ³⁺ : PVA/PVP	4.8	3.8	2.6
0.3 wt% Dy ³⁺ : PVA/PVP	4.45	3.75	2.6
0.4 wt% Dy ³⁺ : PVA/PVP	4.2	3.7	2.6
0.5 wt% Dy ³⁺ : PVA/PVP	3.8	3.6	2.63

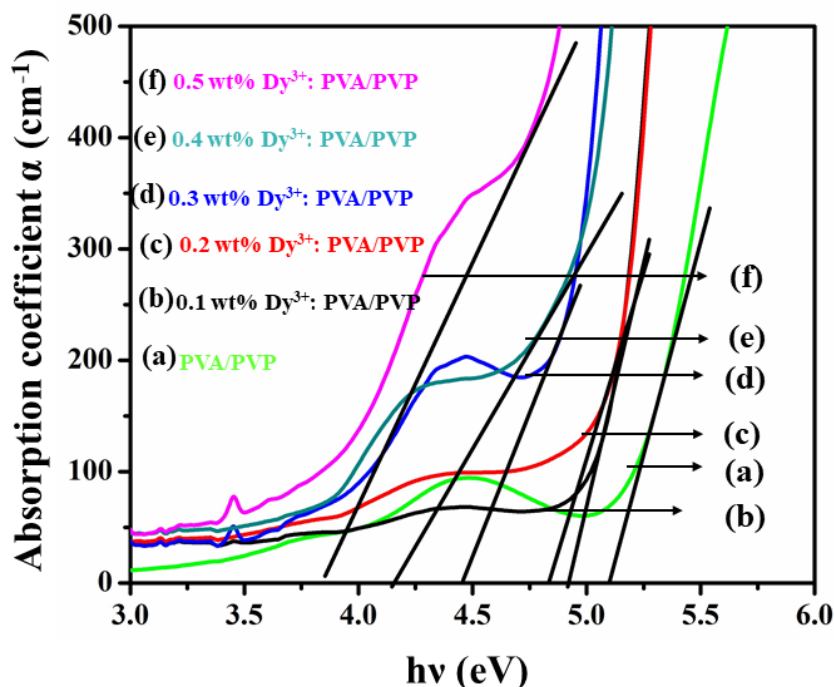
Fig. 5 The absorption coefficient α versus photon energy $h\nu$ of (a) pure PVA/PVP (b-f) 0.1 to 0.5 wt% Dy³⁺: PVA/PVP films.

Fig.5 presents the absorption coefficient and photon energy dependency for host and Dy³⁺ doped polymer films. From this figure, it was noticed that a strong relationship between increasing absorption coefficient and the Dy³⁺ ion concentration. The value of the absorption coefficient increases as the concentration of Dy³⁺ ions increase [29-30]. Extrapolating the linear component to zero absorption coefficients yielded the fundamental absorption edge. The obtained absorption edge values were presented in Table.3. It is clear, the absorption edge values slightly decreased with increasing Dy³⁺ ion in PVA/PVP blend polymer film. While there is no difference between the absorption edges of pure PVA/PVP and PVA/PVP: xDy³⁺ (x= 0.1, 0.2, 0.3,

0.4 & 0.5 wt %), this small change is ascribed to Dy³⁺ ions complication in the PVA/PVP chain. In determining the optical bandgap, the optical absorption edge plays a crucial role. The optical band gap was calculated with the common mathematical formula and listed in Table.3.

$$\alpha = \alpha_0 (h\nu - E_g^{\text{opt}})^n / h\nu \quad (4)$$

where $n = \frac{1}{2}$ for indirect bandgap, α is the absorption coefficient, and E_g^{opt} is optical bandgap energy.

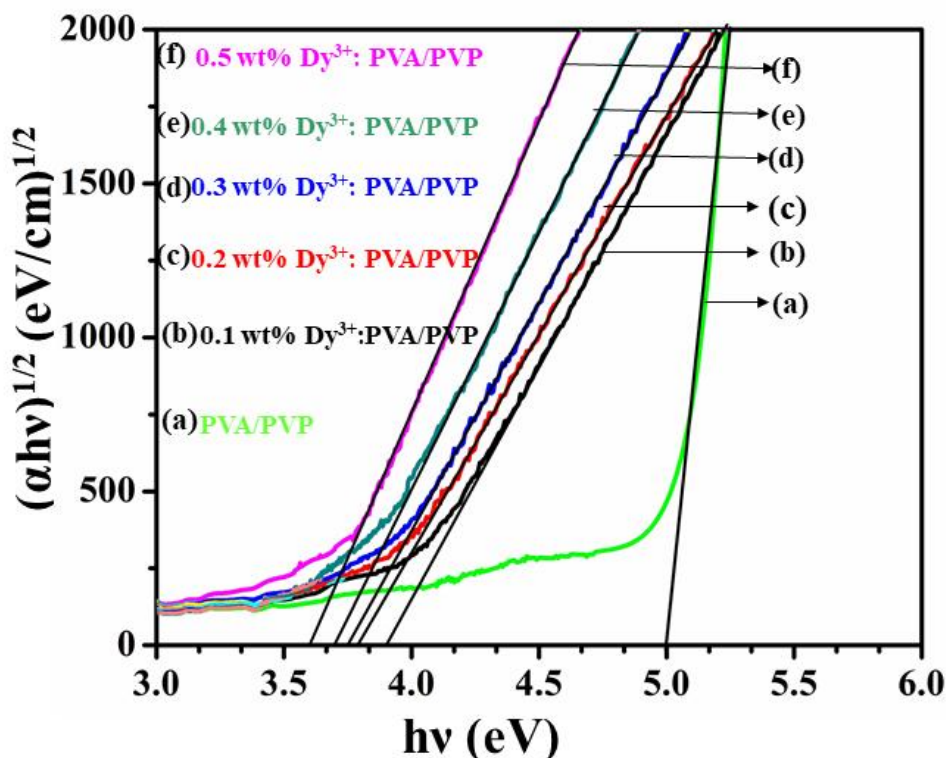


Fig. 6 Tauc Plots of $(\alpha h\nu)^{1/2}$ with $(h\nu)$ for (a) pure PVA/PVP (b-f) 0.1 to 0.5 wt% PVA/PVP blend polymer films.

Fig.6 presents Tauc's plots $(\alpha h\nu)^{1/2}$ vs $h\nu$ for pure PVA/PVP and Dy³⁺ polymer films. The indirect energy bandgap values decreased with increasing concentration from 0.1 to 0.5 wt% of Dy³⁺ ion. It is well known that the optical bandgap depends on the strength bonding. This deviation in the bandgap of the PVA/PVP system with Dy³⁺ ion concentration can also be predictable based on varying in average bond energy of amorphous solid as a function of composition [31].

III.3.2. Refractive index

The energy bandgap values may be used to estimate the refractive index (n) as shown in Table.3 using the following relation [32].

$$n = \sqrt{\frac{12.417}{\sqrt{E_g - 0.365}}} \quad (5)$$

Table .3 shows a slight variation in the refractive index values for PVA/PVP and PVA/PVP: xDy³⁺. As previously stated, the results can be attributed to the free-carrier effect, which produces a rise in refraction and, as a result, a rise in the refractive index. This result is essential in planar optical waveguide fabrication. Total internal reflection is used to transfer light in the middle layer. This does not happen till the intermediate layers refractive index is more significant than adjacent layers. A correlation is noticed between the refractive index and bandgap; as the refractive index increases by raising the Dy³⁺ ion concentration, the bandgap reduces. It is also seen that a significant difference between the values of the bandgap and refractive index.

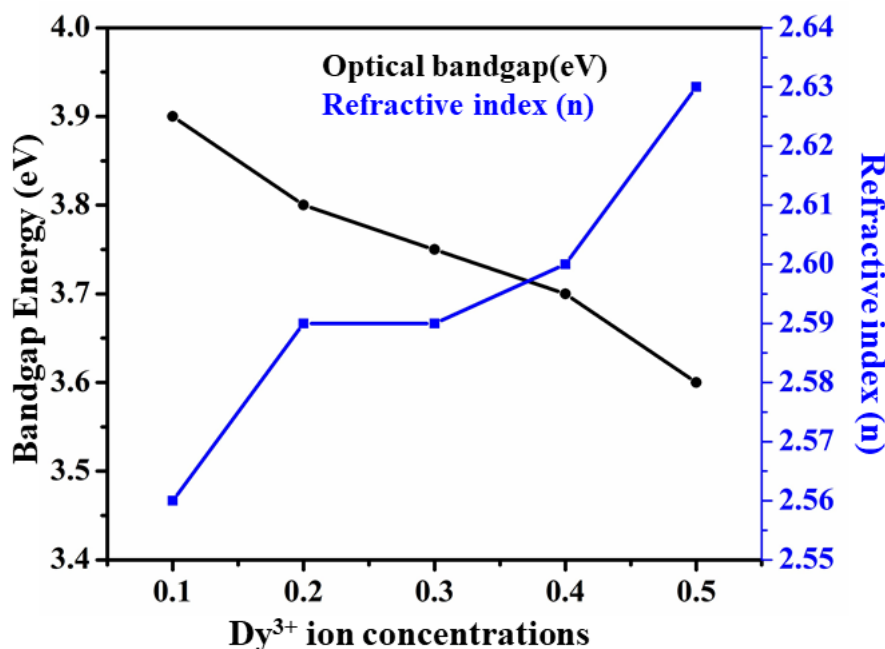


Fig.7 represents the variation of the bandgap and refractive index of PVA/PVP: xDy³⁺ blend polymer films as a function of Dy³⁺ ion concentration.

IV. Conclusions

Using the solution cast process, various Dy³⁺ ions doped PVA/PVP blended polymer films were prepared. XRD confirmed the semi-crystalline nature of the synthesized polymer films. FTIR spectra disclosed that Dy³⁺ ion has a drastic effect on structural properties of PVA/PVP blend polymer films. UV-Vis-NIR absorption spectra showed a shift in optical bandgap with increasing Dy³⁺ ion concentration. XRD, FT-IR, and UV-Vis confirmed the formation of intermolecular interactions and complexation between the PVA/PVP and the Dy³⁺. The optical absorption edge and optical band gaps (indirect) decreased as the concentration of Dy³⁺ increased.

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