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# Structural and optical characterization of PVA:KMnO<sub>4</sub> based solid polymer electrolyte

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## ABSTRACT

Solid polymer electrolyte films of polyvinyl alcohol (PVA) doped with a different weight percent of potassium permanganate (KMnO<sub>4</sub>) were prepared by standard solution cast method. XRD and FTIR techniques were performed for structural study. Complex formation between the PVA polymer and KMnO<sub>4</sub> salt was confirmed by Fourier transform infrared (FTIR) spectroscopy. The description of crystalline nature of the solid polymer electrolyte films has been confirmed by XRD analysis. The UV-Visible absorption spectra were analyzed in terms of absorption formula for non-crystalline materials. The fundamental optical parameters such as optical band gap energy, refractive index, optical conductivity, and dielectric constants have been investigated and showed a clear dependence on the KMnO<sub>4</sub> concentration. The observed value of optical band gap energy for pure PVA is about 6.27 eV and decreases to a value 3.12 eV for the film sample formed with 4 wt% KMnO<sub>4</sub> salt. The calculated values of refractive index and the dielectric constants of the polymer electrolyte films increase with increasing KMnO<sub>4</sub> content.

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## Introduction

Over the last few decades, solid polymer electrolytes (SPEs) based on optical materials have received a considerable attention of scientists, owing to their wide range of applications in high energy rechargeable batteries, fuel cells, supercapacitors, photo-electrochemical and electrochromic displays [1,2]. Understanding the chemistry and physics of SPE is significant because these materials are important for modern and advanced technological applications [3]. The addition of small amount of dopant materials can significantly promote the optical, electrical and thermal properties of polymeric materials, which enables the development of new composite materials with high quality for device industry [4,5].

The most important aspect of improving characteristics is the transition of polymeric materials from insulating to conductive behavior by adding ionic substances [6]. Polyvinyl alcohol (PVA) based polymer electrolyte, in particular, have attracted enormous attention in view of their satisfactory performance and biodegradability [7]. PVA is a potential semi-crystalline polar polymer having an excellent charge storage capacity, high dielectric strength, good mechanical stability, and it has dopant-dependent optical and electrical properties [8–10]. PVA has carbon chain backbone with

hydroxyl (O–H) groups that can be a source of hydrogen bonding which assists the formation of polymer complexes [11] and makes them as an excellent host material for SPEs.

Consideration of the optical absorption behavior of SPEs. The shape and shift of the onset absorption spectra is an exceptionally valuable method for grasp the basic mechanism of the optical transitions in polymeric materials, as well as giving information about the energy band structure [12]. The UV-Visible absorption spectroscopy is an essential tool to investigate the effect of salt concentration on the microstructure properties of the host polymer [13].

Although much attentions have been devoted to the optical properties of SPE films based on PVA incorporating with several metal salts [14–18], but, no reports can be seen in the literature about the optical properties of PVA:KMnO<sub>4</sub> based polymer electrolytes. Potassium permanganate is a strong oxidant inorganic chemical compound, with the characteristic of safe, inexpensive, water-soluble, non-toxic, and commonly available [19]. KMnO<sub>4</sub> was considered as an environmentally friendly material due it is widely used for water disinfection, toxic matter oxidation, as well as to kill algae in wastewater [20]. The main goal of the present work is to investigate the structural and optical properties of PVA:KMnO<sub>4</sub> based solid polymer electrolytes. From the absorption spectra of SPE films, sufficient information about the band structure of the samples can be obtained. The FTIR and XRD study have

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been carried out to reveal the effect of the added salt on the structure of the polymer host material.

## Experimental work

The potassium permanganate ( $\text{KMnO}_4$ ) with purity 99%, provided by Sigma-Aldrich, and polyvinyl alcohol (PVA) with low molecular weight, 98–99% hydrolyzed, obtained from Alfa Aesar, were used to prepare polymer electrolyte films by casting from aqueous solution. The chemicals were used directly without any purification. Films of pure PVA and its SPEs with different weight percent of  $\text{KMnO}_4$  were prepared by solution casting technique. Polyvinyl alcohol powder and different concentration of potassium permanganate (0%, 1%, 2%, 3%, and 4% by weight) were mixed separately and dissolved in distilled water. The obtained solutions were coded as SPE-0, SPE-1, SPE-2, SPE-3, and SPE-4, respectively. The solutions have been thoroughly stirred using a magnetic stirrer at 90 °C for two hours until the homogeneous viscous molten state were obtained. The solution was chilled to room temperature, and then poured into a glass Petri dish (diameter 80 mm), and the solvent was allowed to evaporate slowly at ambient temperature, under atmospheric pressure for a two weeks. Transparent and flexible SPE films of thickness around 0.42 mm have been obtained.

The Fourier transform infrared (FTIR) spectra of pure and  $\text{KMnO}_4$  doped PVA polymer films have been achieved by a Perkin Elmer FTIR spectrometer (Frontier spectrometer) in the wavelength range of 4000–400  $\text{cm}^{-1}$ , to investigate any possible interactions between ions of the salt and functional groups of the polymer. The XRD patterns of the prepared films were recorded with (X'PERT-PRO) X-ray diffractometer with  $\text{CuK}\alpha$  radiation source in the  $2\theta$  range (10°–70°), to report the information about the crystal structure of the product SPE films. Double Beam UV-Visible spectrophotometer (PerkinElmer, Lambda 25), was used to collect the optical absorbance spectra of the PVA: $\text{KMnO}_4$  solid polymer electrolyte films at room temperature in the wavelength range (190–800) nm.

## Results and discussion

### FTIR analysis

The FTIR spectroscopy analysis was conducted to investigate the complex formation between the PVA polymer and  $\text{KMnO}_4$  salt at the molecular level. The recorded FTIR spectra of pure PVA and its complexes with 4 wt% of  $\text{KMnO}_4$  are shown in Fig. 1. The main bands of pure PVA sample were observed at 3212–3446  $\text{cm}^{-1}$  which were assigned to the intramolecular and intermolecular hydroxyl groups (O–H stretching vibration band) [21]. The other

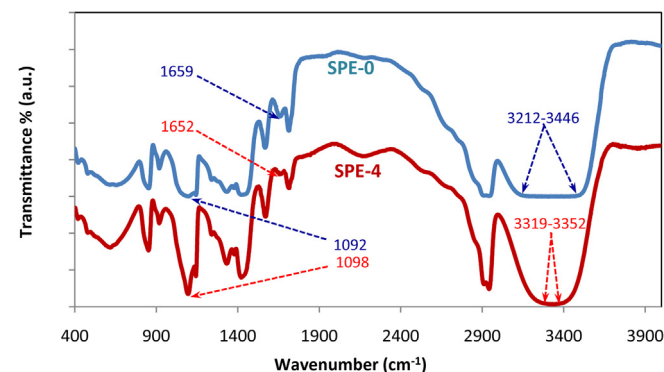


Fig. 1. FTIR spectra of pure PVA and its SPE doped with 4 wt% of  $\text{KMnO}_4$ .

absorption peaks observed in the pure PVA spectrum, are 2942  $\text{cm}^{-1}$ , 1713  $\text{cm}^{-1}$ , 1659  $\text{cm}^{-1}$ , 1427  $\text{cm}^{-1}$ , 1377  $\text{cm}^{-1}$ , 1092  $\text{cm}^{-1}$  and 852  $\text{cm}^{-1}$ . These peaks correspond to the  $\text{CH}_2$  asymmetric stretching, C=C stretching, C=O stretching, C–H bending, C–H wagging, C–O–C stretching of acetyl groups, and C–H rocking vibrations, respectively [22–24]. The positions of these peaks are found to be shifted in PVA: $\text{KMnO}_4$  polymer electrolyte sample. The strong characteristic peak at 3212–3446  $\text{cm}^{-1}$  for hydroxyl group is shifted to 3319–3352  $\text{cm}^{-1}$  in the salt-doped PVA sample, indicating the reduction of hydroxyl bands in the complexes sample [25]. The absorption bands at 1659  $\text{cm}^{-1}$  and 1092  $\text{cm}^{-1}$  in pure PVA are slightly displaced to 1652  $\text{cm}^{-1}$  and 1098  $\text{cm}^{-1}$  in the SPE-4 sample, respectively. This reveals the interaction of  $\text{KMnO}_4$  with PVA matrix. The shifting and the change in intensity of the peaks are clear indications the formation of charge transfer complex between  $\text{KMnO}_4$  salt and the PVA molecules. A similar effect has also been reported in the literature for different solid polymer electrolyte systems [26–28].

### XRD analysis

The X-ray diffraction pattern of pure PVA and PVA complex with different weight percent of  $\text{KMnO}_4$  are presented in Fig. 2. The broad diffraction peak at  $2\theta = 19.25^\circ$  of SPE-0 sample indicating the semicrystalline nature of PVA [29]. This crystalline nature arises from the intermolecular interaction between the PVA backbone chains through hydrogen bond [30]. The significant reduction in the relative intensity of the characteristic peak of PVA in the SPE samples, clearly indicates that the crystalline fraction decreases as a result of  $\text{KMnO}_4$  addition [31–33].

The crystallinity of the films was estimated from the ratio of the integrated intensity of peaks associated with crystalline reflections to the total integrated area of the spectrum, using peak separation Software Fityk 0.9.8, and according to equation [34,35]:

$$X_C = A_C \times 100/A_T \quad (1)$$

where  $X_C$  is the crystalline fraction,  $A_C$  represents the crystalline area, and  $A_T$  represents the total crystalline and amorphous area. The Eq. (1) assumes a linear relationship between intensity of XRD pattern and crystalline fractions in the samples. The calculated value of the degree of crystallinity, as well as the center and full width at half maximum (FWHM) of fitted XRD pattern are listed in Table 1. It is clear from Table 1, that the crystalline fraction decreases significantly. The noticeable change in crystallinity from 16.07% for pure PVA to 9.21% for PVA: $\text{KMnO}_4$  (96:4) reveals a good reactivity between the host polymer and the added salt. The value of crystalline fraction of pure PVA achieved in this work is in good agreement with the value predicted by Kulshrestha and Gupta [36].

The mechanism of ionic transport in SPEs is still not completely understood, but the increase of amorphous nature of polymer electrolyte samples responsible for the enhancing in conductivity as will be shown later.

### Band gap study

The most direct and maybe the simplest method for probing the band structure of polymeric materials is to measure the absorption spectra, which can be used to estimate the optical band gap energy and to determine the types of electronic transition [37,38]. The absorption coefficient as a function of frequency  $\alpha(\nu)$  can be defined as the relative decrease rate in light intensity, which can be calculated from the absorbance  $A$  data, using the Beer Lambert's formula [39]:

$$\alpha(\nu) = 2.303 \frac{A}{d} \quad (2)$$

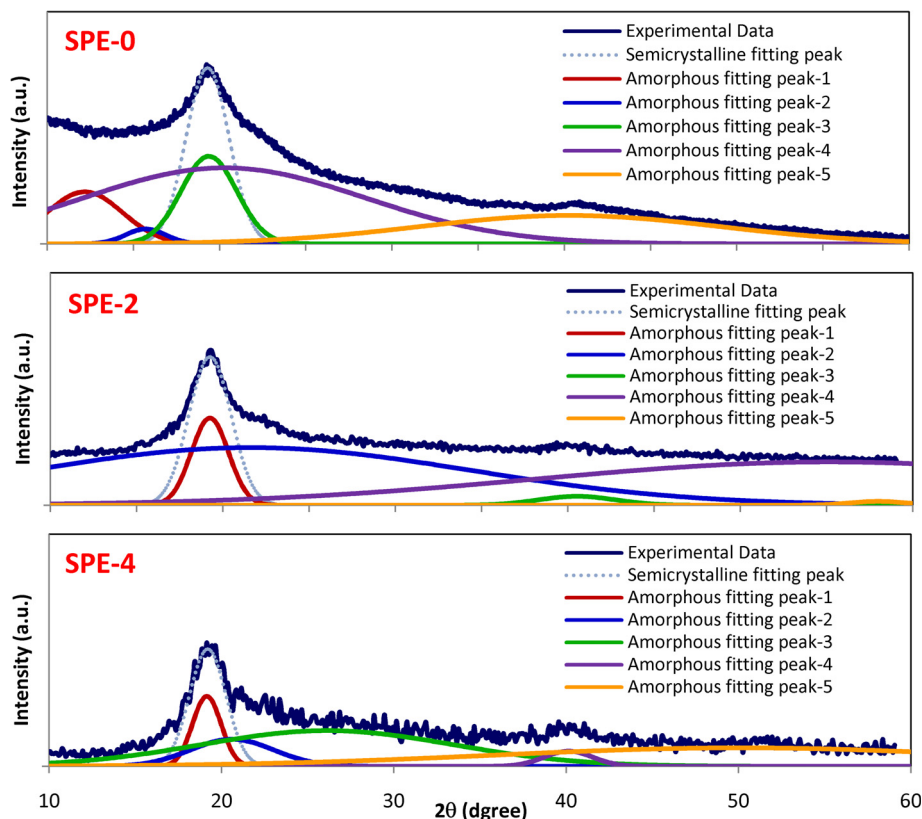


Fig. 2. Deconvoluted XRD patterns of SPE-0, SPE-2, and SPE-4 polymer electrolyte samples.

**Table 1**  
Center of the peaks, FWHM, and degree of crystallinity of PVA/KMnO<sub>4</sub> solid polymer electrolyte films.

Samples	Center of the peak (2θ)	FWHM of the peak	X <sub>C</sub> (%)
SPE-0	19.31	2.94	15.97
SPE-2	19.25	2.90	12.16
SPE-4	19.21	2.65	9.81

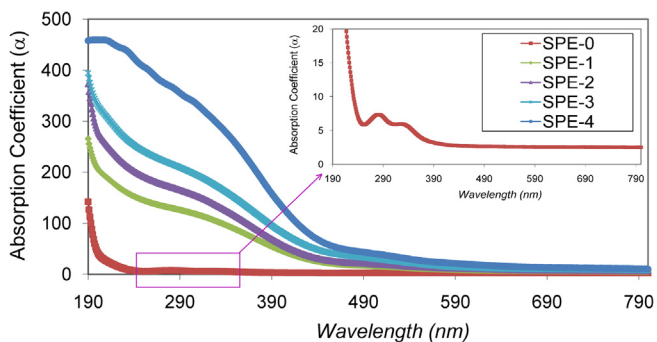


Fig. 3. The absorption coefficient spectra for PVA:KMnO<sub>4</sub> solid polymer electrolyte films.

where *d* is the sample thickness. Fig. 3 illustrate the optical absorption coefficient  $\alpha(\nu)$  for pure PVA and its complexes. From the inset of Fig. 3, it is obvious that pure PVA exhibits two absorption bands at 281 and 340 nm. These peaks are assigned to  $\pi \rightarrow \pi^*$  and  $n \rightarrow \pi^*$  inter band transitions, respectively [13,40,41]. It can be observed from the spectra of SPE samples the absorption onset shift towards the higher wavelength (red-shift phenomena) upon increasing KMnO<sub>4</sub> concentration. Form the shifting of absorption coefficient

towards the higher wavelength upon the increase of salt concentration a reduction of optical band gap can be expected.

At high absorption coefficient  $\alpha(\nu)$  levels, the value of  $\alpha(\nu)$  for non-crystalline materials are related to the incident photon energy ( $h\nu$ ) according to the formula given by Mott and Davis [42]:

$$\alpha(\nu) = \beta \frac{(h\nu - E_g)^\gamma}{h\nu} \tag{3}$$

where  $\beta$  is a constant depending on the specimen structure,  $E_g$  is the optical band gap energy,  $h$  is Planck's constant, and the exponent  $\gamma$  is an index determines the type of electronic transition responsible for absorption and can take values 1/2, 3/2 for direct and 2, 3 for indirect transitions [43,44]. Earlier works confirmed that PVA exhibits the direct band transition between the localized highest occupied molecular orbital (HOMO) and lowest unoccupied molecular orbital (LUMO) band edges [10,45]. Fig. 4 shows graphs of  $(\alpha h\nu)^2$  versus photon energy ( $h\nu$ ), the straight lines obtained with

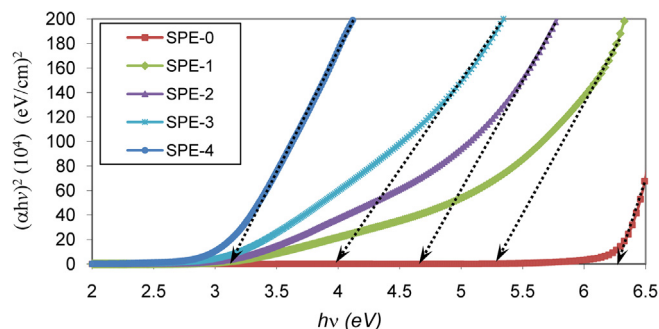


Fig. 4.  $(\alpha h\nu)^2$  versus photon energy ( $h\nu$ ) for PVA:KMnO<sub>4</sub> solid polymer electrolyte films.

$\gamma = 1/2$  indicating the electron transition is direct allowed in k-space. As illustrated in Fig. 4, the intercept of the extrapolation of the linear portion of these curves to zero absorption on  $h\nu$  axis, gives the value of optical band gap energy  $E_g$ . The determined values of optical band gap energy for all samples are presented in Table 2. From Table 2 it is clear that the values of  $E_g$ , reduced significantly from 6.27 eV for pure PVA to 3.12 eV for PVA complexed 4 wt%  $\text{KMnO}_4$ . It was reported that the modification in electronic structure may affect the optical properties of SPEs [40]. The decrease in  $E_g$  values is a consequence of the generation of new energy levels (traps) between the HOMO and LOMO, due to the formation of the disorder in the SPE films. This leads to an increased density of the localized states in the mobility band gap of the PVA matrix [46]. The XRD results (Fig. 2) strongly support the formation of disorder in the SPE films. To realize the formation of defect in PVA host polymer, the width of the tail of localized states within the forbidden band gap energy were calculated from the exponential region of the UV-Visible absorption spectra. At lower absorption coefficient  $\alpha(\nu)$  level, the values of  $\alpha(\nu)$  is described by the Urbach formula [47]:

$$\alpha(\nu) = \alpha_0 \exp\left(\frac{h\nu}{E_u}\right) \quad (4)$$

where  $\alpha_0$  is a constant and  $E_u$  is the Urbach tail interpreted as the width of the tails of localized states in the forbidden band gap [39]. From Eq. (4) it is obvious that the plot of  $\ln(\alpha)$  versus  $h\nu$  must be linear. Fig. 5 presents the Urbach plots for pure and doped PVA films. The  $E_u$  values are calculated from the reciprocal of the slope of the linear part of  $\ln(\alpha)$  versus  $h\nu$ . It is evident (see Table 2) that the energy tails values increases from 0.2672 eV for pure PVA to 1.2837 eV for 4 wt%  $\text{KMnO}_4$  composite. The  $E_u$  value for pure PVA is well agreement with the value reported by earlier workers [17,48]. The smallest value of energy tails for PVA can be ascribed to semi-crystalline nature of PVA. This is supported by XRD results.

It is well known that the value of  $E_u$  represent the disorder content and irregularities between HOMO and HOMO in polymers. Thus, the increase in  $E_u$  for doped samples suggests the increase of amorphous portion. The addition of salts to polymers may produces many trapped state, which reduces the values of optical band gap energy of the SPE films.

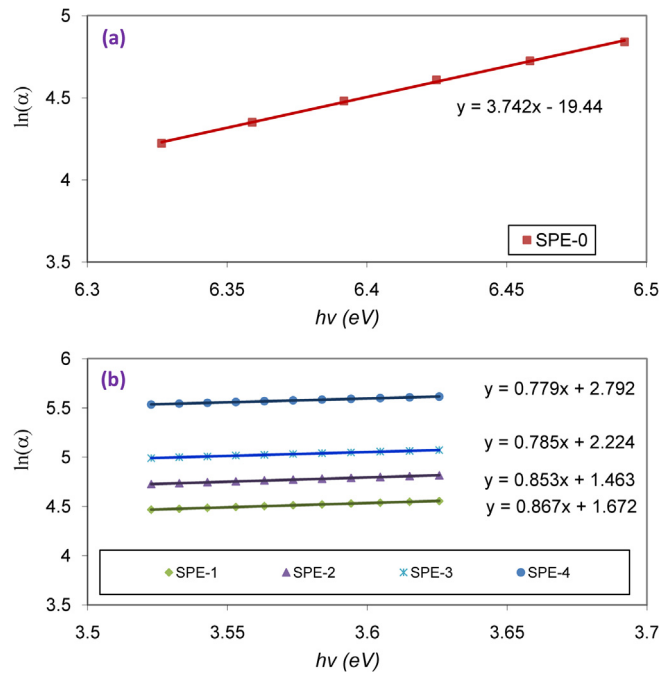
The increase of energy tails  $E_u$  as the concentration of  $\text{KMnO}_4$  increase is consistent with the drastic decrease of the optical band gap energy  $E_g$ . In general, the sum ( $E_g + E_u$ ) represents the mobility band gap energy; Table 2 contains values of the mobility gap for investigated SPE samples. The decrements in the mobility band gap energy values can be explicated by the fact that increasing in the  $\text{KMnO}_4$  content could lead to the formation of ionic complexes, disorder, and imperfections in the structure of the host polymer, leading to create new localized states of various depths in the forbidden band gap energy. This usually contributes to the decrease in the optical band gap energy [49].

### Refractive index study

To understand the polarizability of the present SPE samples, and hence its possible application in optoelectronic devices, the refractive index  $n$  must be studied. The fundamental optical characteris-

**Table 2**  
Optical energy results for PVA/ $\text{KMnO}_4$  solid polymer electrolyte films.

Samples	$E_g$ (eV)	$E_u$ (eV)	$E_g + E_u$ (eV)
SPE-0	6.27	0.2672	6.5372
SPE-1	5.28	1.1534	6.4334
SPE-2	4.66	1.1723	5.8323
SPE-3	3.98	1.2739	5.2539
SPE-4	3.12	1.2837	4.4037



**Fig. 5.** Urbach plot of  $\ln(\alpha)$  against photon energy ( $h\nu$ ) for: (a) pure PVA, (b) PVA:  $\text{KMnO}_4$  solid polymer electrolyte films.

tics of the films are the refractive index and dielectric constant. The values of refractive index can be obtained from the reflection coefficient  $R$  and extinction coefficient  $k$  data using the Fresnel formulae [50]:

$$n = \left( \frac{4R}{(R-1)^2 - k^2} \right)^{1/2} - \frac{R+1}{R-1} \quad (5)$$

The extinction coefficient  $k$  is related to the optical absorption coefficient and the incident wavelength according to [51]:

$$k = \frac{\alpha\lambda}{4\pi} \quad (6)$$

Fig. 6(a, b) shows the variation of the refractive index ( $n$ ) and the extinction coefficient ( $k$ ) versus incident photon energy for all samples. It is clear that the value of refractive index increases with increasing both  $\text{KMnO}_4$  content and incident photon energy. The observed increase in the refractive index due to incorporation of salts in different SPE systems have been reported in the literature [17,52].

The increasing trend of refractive index  $n$  upon  $\text{KMnO}_4$  addition can be understood in view of the intermolecular hydrogen bonding between  $\text{K}^+$  ions and the adjacent OH groups of PVA polymer chains and thus the polymer electrolyte films are dense, and hence higher refractive indices can be achieved, according to the well-known Clausius-Mossotti relation [53].

The increase of the extinction coefficient  $k$  with increasing  $\text{KMnO}_4$  content and incident photon energy can be ascribed to the variation of the absorption coefficient  $\alpha$  since extinction coefficient  $k$  is directly proportional to  $\alpha$ .

### Optical conductivity analysis

The optical conductivity  $\sigma$  for pure PVA and  $\text{KMnO}_4$  doped PVA samples was calculated using the absorption coefficient  $\alpha$ , and the refractive index  $n$  data using the following relation [7],

$$\sigma = \frac{\alpha n c}{4\pi} \quad (7)$$

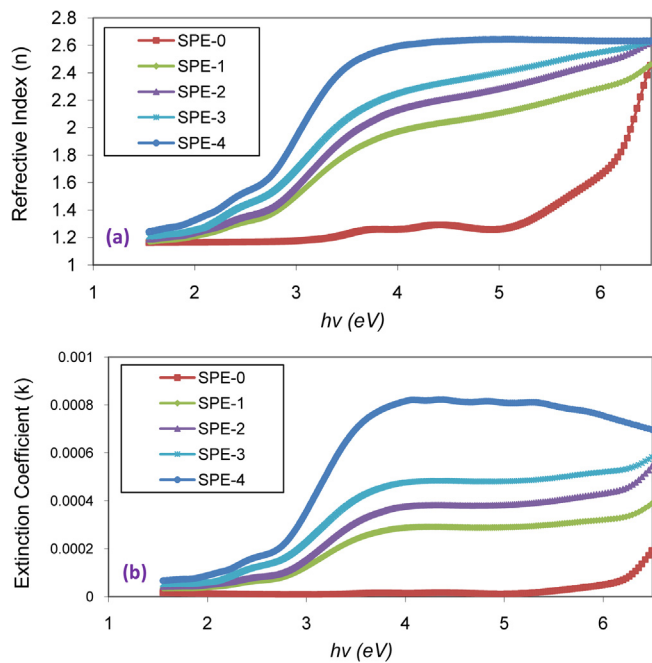


Fig. 6. The variation of (a) the refractive index; (b) the extinction coefficient, versus incident photon energy.

where  $c$  is the velocity of light in the space. Fig. 7 shows the variation of optical conductivity  $\sigma$  versus photon energy. It is clear that the optical conductivity of PVA polymer increased upon the addition of  $\text{KMnO}_4$  salt. This can be interpreted on the basis of formation of charge transfer complex between  $\text{KMnO}_4$  salt and the PVA molecules. The FTIR results discussed in Section “Experimental work” strongly supported this opinion [10,47]. Thus, the increase of optical conductivity of SPE films upon the addition of  $\text{KMnO}_4$  salt can be attributed to an increase of charge carrier concentration in the system. While, the increased optical conductivity at high photon energies is due to the high absorbance of SPE films in the ultra violet region [37].

Dielectric analysis

The optical dielectric constant  $\epsilon_r$  of the PVA: $\text{KMnO}_4$  solid polymer electrolyte films are calculated from the values of refractive index  $n$  and extinction coefficient  $k$  according to the relations [54,55]:

$$\epsilon_r = n^2 - k^2 = \epsilon_\infty - \frac{e^2}{4\pi^2 c^2 \epsilon_0} \frac{N}{m^*} \lambda^2 \tag{8}$$

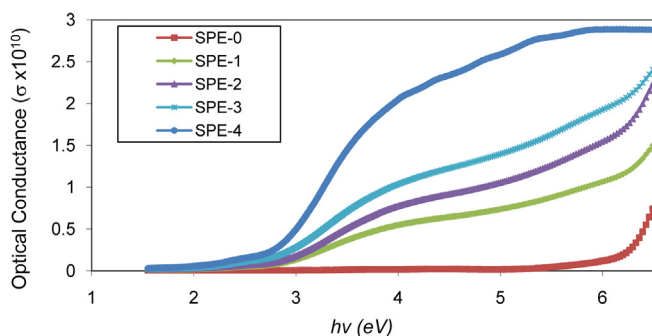


Fig. 7. The optical conductivity  $\sigma$  versus incident photon energy.

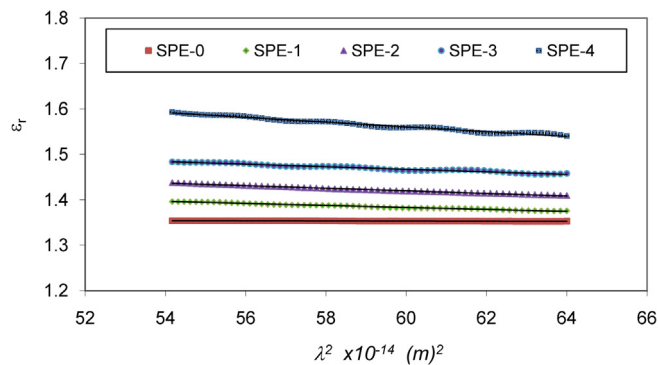


Fig. 8. Dielectric constant as a function of  $\lambda^2$ .

Table 3 Values of  $\epsilon_\infty$ , and  $N/m^*$  for PVA/ $\text{KMnO}_4$  solid polymer electrolyte films.

Samples	$N/m^* (\text{m}^{-3} \text{kg}^{-1}) \times 10^{55}$	$\epsilon_\infty$
SPE-0	1.9263	1.362
SPE-1	27.216	1.516
SPE-2	34.825	1.589
SPE-3	34.641	1.636
SPE-4	64.255	1.874

where  $e$  is the electronic charge,  $\epsilon_0$  is the free space dielectric constant,  $\epsilon_\infty$  is the residual dielectric constant, and  $N/m^*$  is the ratio of carrier concentration to the effective mass. Fig. 8 shows the relation between dielectric constant  $\epsilon_r$  and  $\lambda^2$  for the investigated SPE films.

The values of  $\epsilon_\infty$ , and  $N/m^*$  can be estimated from intercept at  $\epsilon_r$  axis, and the slope of the linear fitting. The calculated values are given in Table 3. It is evident that values of  $\epsilon_\infty$  and  $N/m^*$  for SPE samples increase with increasing salt content. This reveals that with increasing concentration of  $\text{KMnO}_4$  free ionic carriers increase.

The results of the present work show that, all optical parameters significantly affected by  $\text{KMnO}_4$  salt. From the band gap study, it is appeared that the band gap of PVA can be tuned moderately. The observed dispersion in refractive index spectra of the doped samples is crucial for optoelectronics application. It is well reported that optoelectronic devices require controlled and tunable band gap energy materials [48].

Conclusion

In this work, solid polymer electrolyte films of PVA: $\text{KMnO}_4$  have been prepared by the casting technique. Polymer salt complex formation is confirmed using FTIR. The XRD analysis reveals the reduction in crystallinity of PVA: $\text{KMnO}_4$  complexes that produce high conductivity. Optical quantities such as absorption coefficient, optical energy gap, energy tails, refractive index, optical conductivity, and dielectric constants were determined from the absorbance of UV-Visible spectra analysis. From the optical results obtained it was found that the direct optical band gap energy extensively decreased with increasing potassium permanganate concentration. The increase of optical conductivity of polymer electrolyte upon the addition of  $\text{KMnO}_4$  salt is attributed to an increase of charge carrier concentration. The increase in refractive index and dielectric constant in the doped samples is related to added salt. The large absorption coefficient and the compositional dependence of the optical parameters make this system suitable for optoelectronic devices application.

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