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Influence of Fast Neutron Irradiation on the Optical Properties of Poly(Vinyl Alcohol)/ Hydroxypropyl Cellulose Blends

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Authors' contributions

This work was carried out in collaboration between both authors. Both authors read and approved the final manuscript.

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ABSTRACT

Poly(vinyl alcohol) (PVA)/hydroxypropyl cellulose (HPC) (100/0, 80/20, 50/50, 20/80 and 0/100 wt/wt%) blend films are prepared by solution casting method and subjected to fast neutrons irradiation for fluence 1×10^7 n/cm². The effects of different HPC concentrations on the optical properties such as: near infrared, absorbance and transmittance spectra in the region 250–2500 nm of the prepared films are studied before and after irradiation with fast neutrons. The absorption coefficient and the extinction coefficient for the prepared films have been calculated. The transmittance spectra are used for the determination of the optical tristimulus transmittance values. The obtained results illustrate that the increase in the concentration of HPC with PVA and/or irradiation with fast neutrons changed the chemical bonds and hence changed the molecular configuration of PVA indicating that there are charge transfers complexes arose in the polymer blend matrices.

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Keywords: Poly(vinyl alcohol); hydroxypropyl cellulose; near infrared; absorption and extinction coefficients; tristimulus transmittance values; fast neutrons irradiation.

1. INTRODUCTION

Materials with improved characteristics are produced by blending two or more polymers in order to combine their properties for certain purposes which cannot be achieved by one of them alone. Blending, natural polymer with synthetic polymer seems to be an interesting way of polymeric composites. Blending is an especially important process for developing industrial applications of polymeric materials [1] as well as the study of blends properties, are of importance to find further applications of the resulting blend materials for biomedical and pharmaceutical devices [2].

Poly(vinyl alcohol) is a semicrystalline, synthetic water-soluble polymer and has very important applications due to the role of hydroxyl groups and hydrogen bonds [3]. These hydrogen bonds assist in the formation of polymer blends. PVA is used in surgical devices, sutures, hybrid islet transplantation, implantation, blend membrane [4], in synthetic cartilage in reconstructive joint surgery [5], as a new type of soft contact lens [6], as sheets to make bags for premeasured soap, for washing machines, or to make longer bags used in hospitals [7]. PVA was selected as the hydrogel component based on its favorable water-soluble, desirable physicochemical properties and its biocompatibility [8].

Hydroxypropyl cellulose belongs to the group of cellulose ethers soluble in water as well as in polar organic solvents. HPC can be used for production of time controlled delivery systems and is also used as a topical ophthalmic protectant and lubricant [6]. In pharmaceuticals HPC used as a disintegrants and a binder for the wet granulation method of making tablets [9,10,11].

Poly(vinyl alcohol) can be blended with hydroxypropyl cellulose and hydroxypropyl methyl cellulose [12]. Here, hydrogen bonding interaction is an important aspect of miscibility since intermolecular interactions regulate the compatibility among the component polymer molecules [13].

In the present work, thin transparent films of poly(vinyl alcohol)/hydroxypropyl cellulose (PVA/HPC) blends (100/0, 80/20, 50/50, 20/80 and 0/100 wt/wt%) are prepared. The effects of different weight percents of HPC on the optical properties by near-infrared; absorbance and transmittance in the spectral region 250–2500 nm of the prepared films are studied before and after irradiation with fast neutrons of fluence 1×10^7 n/cm². Variations in the group coordination in the near-infrared region are followed. The study has been extended to calculate the absorption and extinction coefficients for the investigated films.

2. EXPERIMENTAL DETAILS

2.1 Materials and Sample Preparation

Poly(vinyl alcohol) in the form of granules with MW 125 kg/mole is supplied from El-Nasr Company, Cairo, Egypt and hydroxypropyl cellulose (HPC; Pharmacoat 606) with MW 95 kg/mole is supplied from Shin Etsu Chemical Co., Tokyo, Japan.

Thin transparent films of poly(vinyl alcohol)/hydroxypropyl cellulose (PVA/HPC) blends are prepared by using solution-cast technique [11,14,15] which depends on the dissolution, separately, the weighted amounts of the PVA and HPC in double distilled water. Complete dissolution is obtained using a magnetic stirrer in a 50°C water bath. To prepare thin films of the homopolymers (PVA and HPC) and their blends (PVA/HPC) with different weight percentages (100/0, 80/20, 50/50, 20/80 and 0/100 wt/wt%), the solutions are mixed together at 50°C with a magnetic stirrer. Thin films were cast onto stainless steel Petri dishes of appropriate 0.01 cm thickness and 10 cm diameter. The prepared films are kept at room temperature (about 25°C) for 7 days until the solvent completely evaporated and then kept in desiccators containing fused calcium chloride to avoid moisture. The samples are measured at room temperature as solid slabs of dimensions 1 x 4 cm.

The prepared thin films of the blends are irradiated at room temperature with fission neutrons with mean energy of approximately 4.2 MeV from Americium-Beryllium (²⁴¹Am-Be) neutron source of activity 5 Ci (185 GBq) and with emission rate 0.87 x 10⁷ n/s at the Physics Department, Faculty of Science, Al-Azhar University, Cairo, Egypt. During irradiation the samples are fixed in positions that neutrons are incident approximately normal. The films are exposed to fast neutrons fluence 1 x 10⁷ n/cm². The fast neutron fluence is measured using a calibrated TLD-700 Thermoluminescence detector at the location of the sample.

2.2 UV/VIS/NIR Spectroscopic Measurements

The optical absorbance and transmittance in the ultraviolet, visible and near-infrared regions from 250 to 2500 nm for PVA/HPC blends before and after irradiation with fast neutrons are carried out using a Shimadzu (UV/VIS/NIR) Double Beam Spectrophotometer with standard illuminant C (1174.83), has a serial number B44360512, Model V-530 and band width 2.0 nm covers the range 200-2500 nm with accuracy ±0.05%.

The absorption coefficient (α) of the present materials strongly depends on optical transmission, reflection and thickness of the film and is calculated using the relation [16]:

$$\alpha = (1/d) \ln [(1-R)^2/T] \quad (1)$$

Where T is the transmittance and d is the thickness of the sample in cm (the reflectance, R is neglected in this calculation). Then,

$$\alpha = (1/d) \ln [1/T] \quad (2)$$

The extinction coefficient (K) is important parameters characterizing photonic materials and can be calculated using the relation [16]:

$$K = \alpha\lambda/4\pi \quad (3)$$

Where λ is the wavelength and α is the absorption coefficient.

The tristimulus transmittance values (x_t , y_t and z_t) of the prepared blends in the visible spectrum range 400-700 nm are calculated from the transmittance spectra using the CIE recommendations [17,18].

3. RESULTS AND DISCUSSION

3.1 Near-Infrared (NIR) Spectral Analyses

The absorbance spectra and the assignments of the most important bands in the near-infrared (NIR) region 900-2500 nm for PVA/HPC blends before and after irradiation with fast neutrons fluence $1 \times 10^7 \text{ n/cm}^2$ are shown in Fig. 1. Table 1 illustrates the variation of the peak positions as well as the bond vibration and chemical structure for the unirradiated PVA/HPC blends.

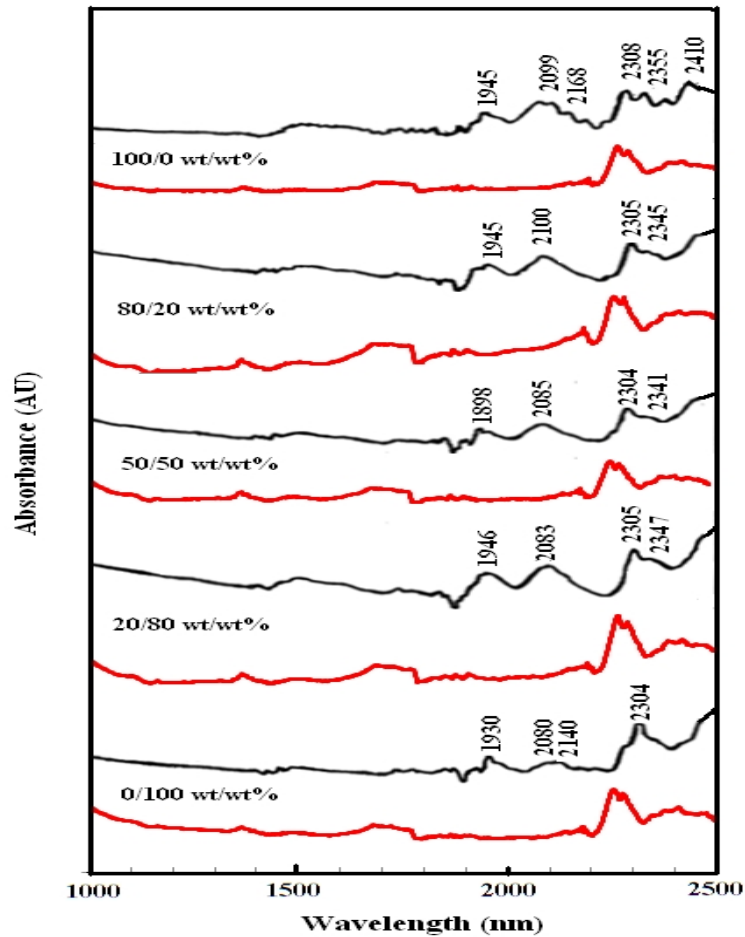


Fig. 1. NIR spectra of PVA/HPC blends unirradiated (black solid lines) and irradiated with fast neutrons fluence $1 \times 10^7 \text{ n/cm}^2$ (red solid lines)

It is noticed from the figure and the table that [19]:

- The bands at 2410 nm for pure PVA and at 2472 nm for pure HPC are disappeared in all doped concentrations.
- The band at 2352 nm appeared only in pure PVA and then shifted towards lower wavelength (2345 nm) with increasing the HPC concentration.

- Disappearance of band at 2200 nm for pure PVA and at 2197 nm for pure HPC.
- Band at 1945 nm shifted toward lower wavelengths by increasing HPC concentration up to 50 wt% (at about 1930 nm) as the pure HPC sample.
- The band at 1898 nm for pure HPC shifted toward higher wavelengths by doping HPC with PVA.
- The band at 1780 nm for pure PVA disappeared by doping HPC in all doped concentrations.
- The band at 1764 nm for pure HPC shifted toward lower values of wavelengths by adding HPC with different concentrations and appeared at around 1752 nm.
- The bands at 1724 nm for pure PVA and at 1732 nm for pure HPC disappeared and observed at about 1713 nm by doping PVA with different concentrations of HPC.

Table 1. Positions and chemical assignments of the most NIR absorption bands of the unirradiated PVA/HPC blends

Wavelength (nm)					Assignment	Chemical structure
PVA/HPC blends (wt/wt%)						
100/0	80/20	50/50	20/80	0/100		
2410	—	—	—	2472	C-H stretching + C-C stretching	= CH group
2355	—	—	—	—	C-H deformation-second overtone	HC=CHCH ₂
—	2345	2341	2347	—	CH ₂ sym. Str. + CH ₂ deformation	
2308	2305	2304	2305	2304	C-H stretching + C-H deformation	CH ₂ or CH ₃
2208	—	—	—	2197	C-H stretching + C = O stretching	- CHO
2168	—	2134	2130	2140	=C-H stretching + C=O stretching	- CHO
2099	2100	2085	2083	2080	O-H stretching + O-H deformation	ROH
1945	1945	—	1946	1930	O-H stretching + O-H deformation	H ₂ O
—	1906	1898	1871	1898	C=O str. Secondary overtone	-(O ₂ H)
1815	1830	1820	1818	1820	O-H stretching + 2(C-O) stretching	Cellulose
1780	—	—	—	—	C-H stretching first overtone	Cellulose
1752	—	1751	1751	1764	C-H stretching first overtone	CH ₂
1724	1713	1714	1713	1732	C- H stretching first overtone	CH ₃ or CH ₂

Moreover, it is observed from Fig. 1 that clear variations in the peak positions, and peak areas as well as disappearance of some bands of the PVA/HPC blends when compared with that shown for the homopolymers samples (PVA and HPC) in one hand and with the unirradiated blends on the other hand which noticed that strong local interaction between HPC and other groups to different chains in PVA will take place at the expense of the intermolecular interaction between these chains. This in turn can be directly correlated with

variations in the mechanical behavior of the polymer [20,21]. Furthermore, high energy radiation, such as fast neutrons fluencies are expected to make large chemical and physical changes in the polymers either by direct damage or by reaction with any oxygen present [22]. The variation in the intensities of some bands can be attributed to the radiation induced changes in the intermolecular bonds of PVA chains caused by fast neutrons fluencies which indicate that cross-linking of the chains may occur [23,24]. The changes in the intensities and positions of some bands by fast neutron fluencies may be attributed to change occurred in the concentrated of some active groups or bands, so that the structural modification of irradiated samples can be detected from their spectra [25].

3.2 UV/VIS Analyses

The study of optical properties in the UV/VIS regions is useful as analytical technique for two reasons. Firstly, it can be used to identify some functional groups in molecules and secondly, it can be used to help in a better understanding of the optical material constants [26,27].

Fig. 2 shows the UV/VIS absorbance spectra of PVA/HPC blends in the wavelength range 250-700 nm before (a) and after (b) fast neutrons irradiation with fluence $1 \times 10^7 \text{ n/cm}^2$. It is clear from the figure that, sharp drops in the absorbance values are detected in the UV region (250-360 nm). From Fig. 2a, the observed absorption shoulder centered around 270 nm may be attributed to $\pi \rightarrow \pi^*$ which comes from unsaturated bonds, mainly; carbonyl groups [28] (C=O and/or C=C). Also, it is clear that, the absorbance spectra are closed to each other in the visible region (400-700 nm) and the absorbance values for homopolymers (PVA and HPC) are lower than those for blend compositions through the wavelength range. Besides, the absorbance spectrum for the blend sample of composition 50/50 wt/wt% PVA/HPC is the highest one. From Figs. 2a and b, it is noticed that the absorption values of the blend samples increase with increasing the concentration of HPC as well as by irradiation with fast neutrons which may be attributed to the fact that either increasing the concentration of HPC and/or irradiation with fast neutrons increases the absorbancy of the sample which may be due to that there is a change in the molecular configuration which leads to the formation of new color centers [14,15,29]. Furthermore, the obtained variations in the blend samples may be due to some sort of interaction between the color centers created by the interaction of radiation with both PVA and HPC matrices.

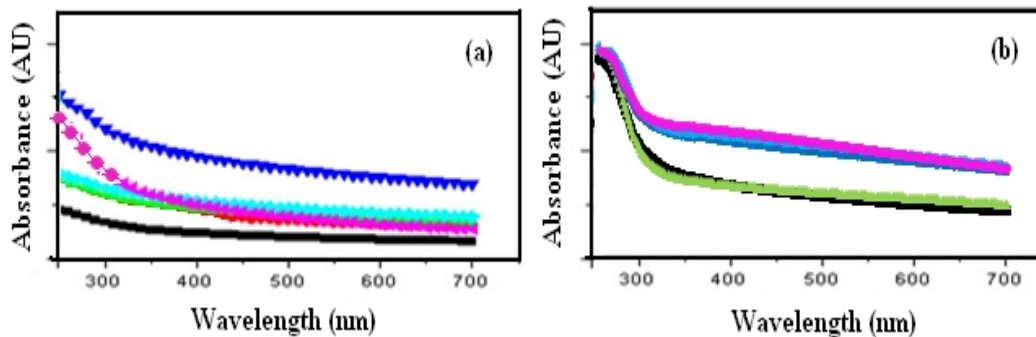


Fig. 2. The absorption spectra of PVA/HPC blend samples before (a) and after (b) irradiation with fast neutrons fluence $1 \times 10^7 \text{ n/cm}^2$: (■) 100/0, (▲) 80/20, (▼) 50/50, (◆) 20/80 and (●) 0/100 (wt/wt%)

3.3 Optical Absorption Measurements

Fig. 3 shows the relation between the absorption coefficient (α) as a function of wavelength in the UV range 250-400 nm and in the visible range 400-700 nm for PVA/HPC blends before (a, c) and after (b, d) irradiation with fast neutrons fluence 1×10^7 n/cm², respectively.

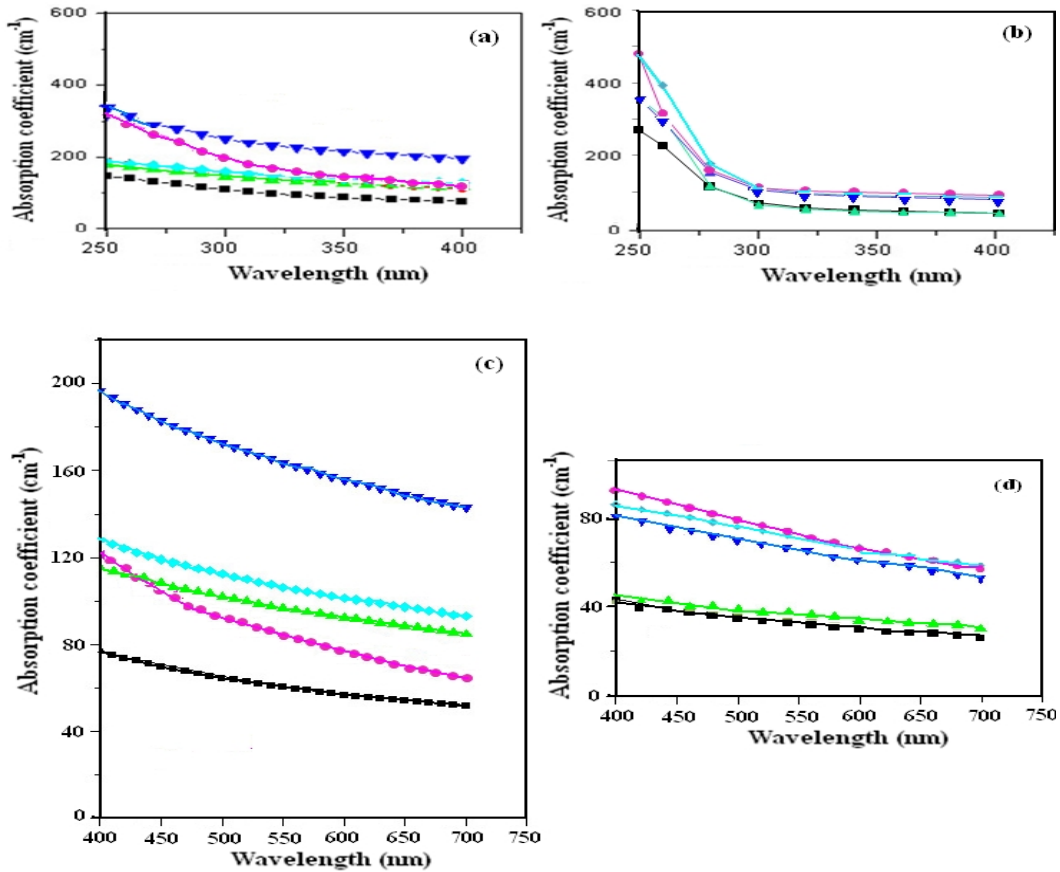


Fig. 3. The absorption coefficient (α) of PVA/HPC blended samples as a function of wavelengths in the UV range 250-400 nm and in the visible range 400-700 nm before (a,c) and after (b,d) irradiation with fast neutrons fluence 1×10^7 n/cm², respectively: (■) 100/0, (▲) 80/20, (▼) 50/50, (◆) 20/80 and (●) 0/100 (wt/wt%)

It is clear from Figs. 3a and c for the unirradiated PVA/HPC blends that the absorption coefficient values increase gradually with increasing HPC concentration. The increase in α with the increase in the HPC concentration up to 50 wt%, which is the highest one, may be attributed to the change of the molecular configuration which leads to the formation of new color centers [14,15]. From Fig. 3b, it is noticed for the irradiated PVA/HPC blends in the UV region that, the absorption coefficient (α) decreases with increasing wavelength and exhibits a steep down near the absorption edge and a straight line relationship is observed in the high α -region which may be caused by the transition of electrons from the valence band to the conduction band [29] and also may be due to the variation in the internal fields associated with structure disorder in the system [30].

In addition, it is clear from Fig. 3b and d that the values of the absorption coefficient (α) for PVA/HPC blends irradiated by fast neutrons are higher than that for pure PVA which may reflect the induced changes in the number of available final states according to the blend composition. Also, the variation in the values of the absorption coefficient with fast neutrons may be attributed to change of the molecular configuration which leads to the formation of new color centers and also due to the formation of highly active free radicals which recombine to form molecular species [14,15,31].

3.4 Extinction Coefficient

The extinction coefficient (K) describes the properties of the material to light of a given wavelength and indicates the amount of absorption loss when the electromagnetic (EM) wave propagates through the material, i.e. represents the damping of an EM wave inside the material. Fig. 4 shows the variation in the extinction coefficient (K) with wavelength in the range 250-700 nm of PVA/HPC blends before (a) and after (b) fast neutrons irradiation with fluence $1 \times 10^7 \text{ n/cm}^2$. It is clear from the figure that similar behaviors for all samples are observed and the values of K are found to be small in the order 10^{-4} throughout the studied wavelength range which indicates that the samples under investigation are considered to be insulating materials at room temperature [32]. In addition, the behavior of the extinction coefficient is preserved for all samples near the absorption edge. Furthermore, it is also clear from Fig. 4a that, the blend sample PVA/HPC 50/50 wt/wt% indicates highest values of K through the whole range of wavelength. Moreover, the values of K for PVA/HPC blends are higher than that of the PVA and HPC homopolymers values.

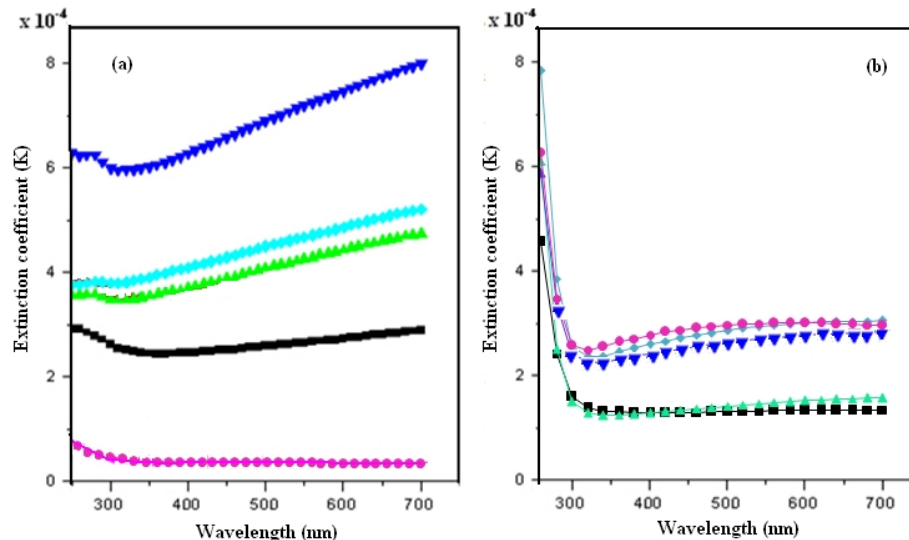


Fig. 4. Variation in the extinction coefficient (K) as a function of wavelength (λ) of PVA/HPC blend samples before (a) and after (b) irradiation with fast neutrons fluence $1 \times 10^7 \text{ n/cm}^2$: (■) 100/0, (▲) 80/20, (▼) 50/50, (◆) 20/80 and (●) 0/100 (wt/wt%)

From Fig. 4b, it is observed that sharp drops in the extinction coefficient values are detected in the UV region. The HPC homopolymer sample indicates highest values of K through the visible range of wavelength after irradiation with fast neutrons. In addition, the values of K of the PVA/HPC blends decrease after irradiation with fast neutrons in comparison with the

unirradiated one (except pure PHC sample) and are between the homopolymers values through the whole range of wavelength. These variations in the blend samples may be due to some sort of interaction between the color centers created by the interaction of radiation with both PVA and HPC matrices. In addition, the decrease in the extinction coefficient with an increase in wavelength shows that the fraction of light lost due to scattering.

3.5 Optical Transmittance Values

The tristimulus transmittance values (x_t , y_t and z_t) for individual polymers and their blends are calculated from the transmittance data (figures are not shown for simplicity) for CIE standard illuminant C. Fig. 5 shows the variation of the tristimulus transmittance (y_t) with wavelength (λ) in the range 400-700 nm for PVA/HPC blends before (a) and after (b) fast neutrons irradiation with fluence 1×10^7 n/cm². It is clear that the behaviours of y_t for the samples are similar and nearly have the same peak position at about 560 nm as that of the most polymers. Fig. 5a indicates that y_t at the peak position (y_{max}) decreases gradually with increasing HPC concentration until 50 wt% followed by an increase with increasing HPC up to 100 wt%. On other hand, it is clear from Fig. 5b that for pure PVA sample, the tristimulus transmittance values (y_t) decrease with neutrons fluence by about 9.1% in comparison with the unirradiated one, while for homopolymer HPC, significant decrease by about 15.6% in the tristimulus transmittance values for the irradiated sample in comparison with the unirradiated one is detected. These changes may be due to the cross-linking occurred during photodegradation; results from the oxidation of free radicals in the polymer material. Due to this process, change in the color of the polymeric material is occurred [33]. Also, from the figure, it is noticed that the tristimulus transmittance values (y_t) and the broadening of the bands are changed with neutrons fluence without changing in their positions either with increasing the concentration of or irradiated with fast neutrons fluence of 1×10^7 n/cm². The observed behaviours of the blended samples are nearly similar to that obtained for the homopolymer samples (PVA and HPC) which agree well and confirm the obtained results of the near-infrared analyses. This may be attributed to that there is a change in the molecular configuration which indicates to the formation of new color centers.

Table 2 illustrates the values of x_r , y_r and z_r at the peak positions for PVA/HPC unirradiated and irradiated blended samples and their percentage changes. It is observed from the table that, the values of x_t , y_t and z_t change remarkably with increasing the concentration of HPC for both the unirradiated and irradiated blended. On other hand, variations in the values x_t , y_t and z_t are detected of the irradiated PVA/HPC blended samples in comparison with the unirradiated samples.

From the data obtained, it is observed that the tristimulus transmittance values indicate that irradiation with fast neutrons may lead rupture in the bonds and formation of free radicals and finally cross-linking. Also, it may be presumed that the effect of fast neutrons radiation on the macromolecules of the blended samples is the destruction of the chemical bonds and linkages as well as creation of highly energetic electrons and ions [34]. These highly energetic species migrate in the plastic network causing further damages to the adjacent macromolecules through tracks. The highly active groups may recombine again at random leading to cross-links. The presence of oxygen enhances such recombination through its interaction with these active macromolecules. Therefore, the highly energetic ions and electrons thus formed during irradiation may be trapped somewhere in the plastic network forming color centers whose intensities will increase with the increase of the fast neutrons fluence.

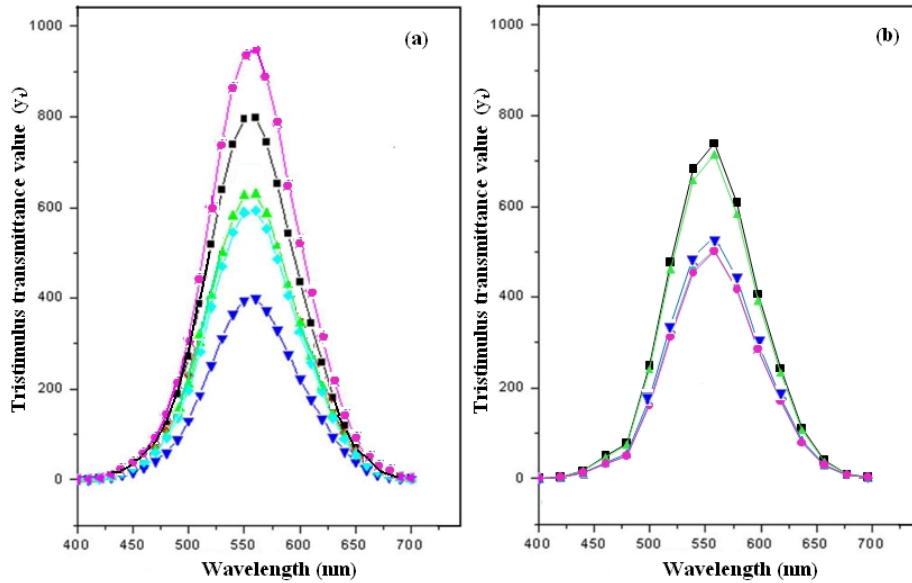


Fig. 5. Variation of the tristimulus transmittance value (y_t) with wavelength for PVA/HPC blend samples before (a) and after (b) irradiation with fast neutrons fluence 1×10^7 n/cm²: (■) 100/0, (▲) 80/20, (▼) 50/50, (◆) 20/80 and (●) 0/100 (wt/wt%)

Table 2. The tristimulus transmittance values (x_r , y_r and z_r) of PVA/HPC blends before and after irradiation with fast neutrons fluence 1×10^7 n/cm² calculated from transmittance data and their percentage changes

PVA/HPC blend (wt/wt%)	x_r		y_t		z_t			
	$\lambda = 445$ nm	$\lambda = 595$ nm	$\lambda = 560$ nm	$\lambda = 450$ nm				
100/0	314	728	800	1600				
80/20	257	628	683	1328				
50/50	242	585	625	1228				
20/80	142	371	400	728				
0/100	221	550	583	1142				
After irradiation	x_r	$(\Delta x_t)\%$	x_r	$(\Delta x_t)\%$	y_t	$(\Delta y_t)\%$	z_t	$(\Delta z_t)\%$
	$\lambda = 440$ nm		$\lambda = 600$ nm		$\lambda = 560$ nm		$\lambda = 445$ nm	
100/0	274	-12.7	674	-7.4	727	-9.1	1372	-14.3
80/20	270	+5.1	647	+3.0	701	+2.6	1352	+1.8
50/50	190	-21.5	493	-15.7	524	-16.2	955	-22.2
20/80	179	+26.1	468	+26.1	496	+24.0	899	+23.5
0/100	171	-22.6	469	-14.7	492	-15.6	857	-24.9

The +ve and -ve signs indicate that the values of x_r , y_r and z_r increases or decreases in comparison with the unirradiated values

4. CONCLUSION

It can be concluded that the changes occurred due to either increase HPC weight percent and/or fast neutrons irradiation may reflect the actual changes in the macromolecular structure of the polymer network. In addition, the obtained results are of great important for

the improvement of the optical properties of PVA. Hence, PVA/HPC blends look desirable and promising for many applications.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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