



Study of the Thermal and Structural Properties of PVA/HPC Blends Due to Fast Neutrons Irradiation

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Authors' contributions

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

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ABSTRACT

Blends of PVA/HPC are prepared using solution casting method and are irradiated with fast neutron irradiation of fluence 1.15×10^7 n/cm² to use as biomaterials according to their broad importance in practical and medical applications. Thermal properties and structural configuration of the blends are studied to characterize and reveal the miscibility map of such blend system. The obtained results of the thermal analyses [differential scanning calorimetry, DSC, and thermal gravimetric analysis, TGA] show variations in the first order thermodynamic transition (T_{th}), the melting temperature, shape and area of thermal peaks which are attributed to the different degrees of crystallinity and the existence of interactions between PVA and HPC molecules. The X-ray diffraction patterns (XRD) show broadening and sharpening of peaks at different HPC concentrations with PVA and irradiation with fast neutrons. The results obtained indicate changes in the crystallinity/amorphosity ratio, and explains the possibility of miscibility existence between the amorphous components of the two polymers PVA and HPC.

Keywords: PVA/HPC blends, differential scanning calorimeter; thermal gravimetric analysis; X-ray diffraction; fast neutrons irradiation.

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1. INTRODUCTION

Blending is an especially important process for developing industrial applications of polymeric materials [1]. 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) (PVA) can be blended with hydroxypropyl cellulose (HPC) and hydroxypropyl methyl cellulose (HPMC) [3]. Here, hydrogen-bonding interaction is an important aspect of miscibility since intermolecular interactions regulate the compatibility among the component polymer molecules [4].

Poly(vinyl alcohol) is a water-soluble polyhydroxy polymer, one of the few linear, non-halogenated aliphatic polymers. PVA has a two dimensional hydrogen-bonded network sheet structure. The physical and chemical properties of PVA depend largely on its method of preparation. PVA is considered as a good host material due to good thermostability, chemical resistance and film forming ability. PVA is an important material in view of its large scale industrial and medical applications [3,5-7].

Hydroxypropyl cellulose is soluble in water as well as in polar organic solvents (makes it possible to combine aqueous and non-aqueous conservation methods) [8]. HPC belongs to the group of cellulose ethers, which has been used already for a year by paper of conservators as glue and sizing material. HPC can be used for production of time-controlled delivery systems and is used as a topical ophthalmic protectant and lubricant [9]. In pharmaceuticals, HPC is used as a disintegrants and a binder for the wet granulation method of making tablets [10,11].

Physical or chemical cross-linking is achieved using functional cross-linking agents or by ionizing radiation [12]. Ionizing radiation such as: gamma or electron-beam produces two types of free radicals on the chains of poly(vinyl alcohol) [13]. Furthermore, chemically cross-linked PVA hydrogel has been gaining increasing attention in medical applications [14].

In the present work, blends of PVA/HPC of different compositions (100/0, 80/20, 50/50, 20/80, and 0/100 wt/wt%) are prepared in the form of thin films and are subjected to fast neutron irradiation of fluence 1.15×10^7 n/cm². Thermal properties as well as structural configuration of the blends are studied using

thermal analyses [differential scanning calorimetry and thermal gravimetric analysis] and X-ray diffraction (XRD) to characterize and reveal the miscibility map and the structural properties of such blend system.

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 casting method [11,15,16]. This casting method depends on the dissolution, by weighing, separately, amounts of 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 ratio (100/0, 80/20, 50/50, 20/80 and 0/100 wt/wt%), the solutions are mixed together at 50°C using magnetic stirrer. Then the mixed solutions were cast onto stainless steel Petri dishes to form thin films of appropriate 0.01 cm in thickness and 10 cm in 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 prepared films of the blends are irradiated at room temperature with fission neutrons with mean energy of 4.2 MeV from Americium-Beryllium (²⁴¹Am-Be) neutron source of activity 5 Ci (185 GBq) and with emission rate 0.87×10^7 n/s. During irradiation the samples are fixed in positions that neutrons are incident normal. The films are exposed to fast neutrons fluence of 1.15×10^7 n/cm². The fast neutron fluence is measured using a calibrated TLD-700 Thermoluminescence detector at the location of the sample.

2.2 Thermal and Structural Analyses

The thermal transition behaviour of the prepared blends is determined by Differential Scanning Calorimeter model Shimadzu DSC-50 (Kyoto,

Japan) from 20 to 500°C. A heating rate of 10°C/minute is used under nitrogen atmosphere and at a flow rate of 20 mL/minute. The dry sample weight was about 2.14 mg.

Thermal behaviour of the prepared blends is examined by Thermogravimetric Analyzer model Shimadzu TGA-50H (Kyoto, Japan) from 20 to 600°C. A heating rate of 10°C/minute is used under nitrogen atmosphere and at a flow rate of 20 mL/minute. The dry sample weight was about 2.41 mg. The standard uncertainty of the sample mass measurement is $\pm 1\%$. The instrument is calibrated using calcium oxalate, which is supplied along the instrument.

The X-ray diffraction (XRD) measurements of the prepared blends are recorded with a Scintag Irc X-Ray Diffractometer (USA) equipped with Ni-filtered CuK_α radiation ($\lambda = 0.15418$ nm) operated at 45 kV and 40 mA. The angle interval for data measurement is recorded within the range $3\text{-}50^\circ$ 2θ at a speed rate of 2 degrees/minute. The crystallinity index (CrI) which gives the time-save empirical measure of relative crystallinity is calculated for the unirradiated and irradiated blends using the relation [17]:

$$\text{CrI} = [(I_f - I_s)/I_f] \times 100 \quad (1)$$

Where I_f is the peak intensity of the fundamental band and I_s is the peak intensity of the secondary band.

3. RESULTS AND DISCUSSION

3.1 Differential Scanning Calorimetry

Differential scanning calorimetry (DSC) is an accurate technique used to determine transition temperatures and to observe their changes such as first order thermodynamic transition, glass transitions, melting cross-linking reactions, purity and rate of decomposition [18].

DSC curves for PVA/HPC blended samples unirradiated and irradiated with fast neutrons of fluence 1.15×10^7 n/cm² in the range of temperature from 20 up to 500°C are shown in Fig. 1. The transition temperature and the heat of fusion (ΔH , J/g) values associated with each transition obtained through analyses of DSC-curves for the blends are summarized in Table 1.

From the figure and the table, the observed transitions can be assigned as follows: The

unirradiated pure PVA sample (black curve - 100/0 wt/wt%) displays two endothermic peaks. The first broad peak represents the first order thermodynamic transition (T_{th}) at about 88°C which is assigned as a thermal effect due to moisture evaporation from the sample with an enthalpy 103.90 J/g [15,19,20]. The irradiated PVA sample with fast neutrons (red curve in the figure) shows four endothermic peaks. The first broad peak at about 36°C is assigned as a thermal effect due to moisture evaporation from the sample with an enthalpy 3.85 J/g. On other hand, the DSC curve for the unirradiated pure HPC (black curve - 0/100 wt/wt%) shows a first broad peak represents the first order thermodynamic transition broad peak (T_{th}) at about 84°C associated with heat of fusion 32.62 J/g which is in agreement with that previously reported in the literature [15]. The irradiated HPC sample (red curve in the figure) shows three endothermic peaks. The first broad peak is detected at about 50°C associated with heat of fusion 127.49 J/g.

In addition, from Fig. 1 and Table 1, it is clear that, the unirradiated DSC thermograms of all unirradiated PVA/HPC blends (black curves) show one single broad peak of which its transition temperature increases with increasing HPC content up to 50 wt%. The obtained temperature values of these peaks are found to be in the range of those of the homopolymers (PVA and HPC) which may indicate the miscibility of the blend systems. The broadening of the peaks depends on the OH content declined. In addition, the temperature widths for the blends are almost identical to that of pure PVA and HPC components, which supports single-phase behaviour in the blends. On other hand, similar behaviours for the irradiated PVA/HPC blends are observed.

The curves in Fig. 1 and the data in Table 1 indicate that the melting points (T_m) at the second step for the unirradiated and irradiated blend samples are lower than the value of unirradiated and irradiated PVA samples. Also, it is found that the decomposition endothermic peak of the irradiated blends became sharp and less in depth than that of the unirradiated blends and beside this shift towards higher temperatures. Furthermore, two other decomposition endotherms (third and fourth steps) are appeared after irradiate the PVA/HPC blends with fast neutrons fluence 1.15×10^7 n/cm². These endothermic peaks are located at about 323 and 448°C for pure PVA and at about

345 and 484°C for pure HPC. The temperature values of these two peaks are shifted toward higher temperatures by increasing HPC concentration.

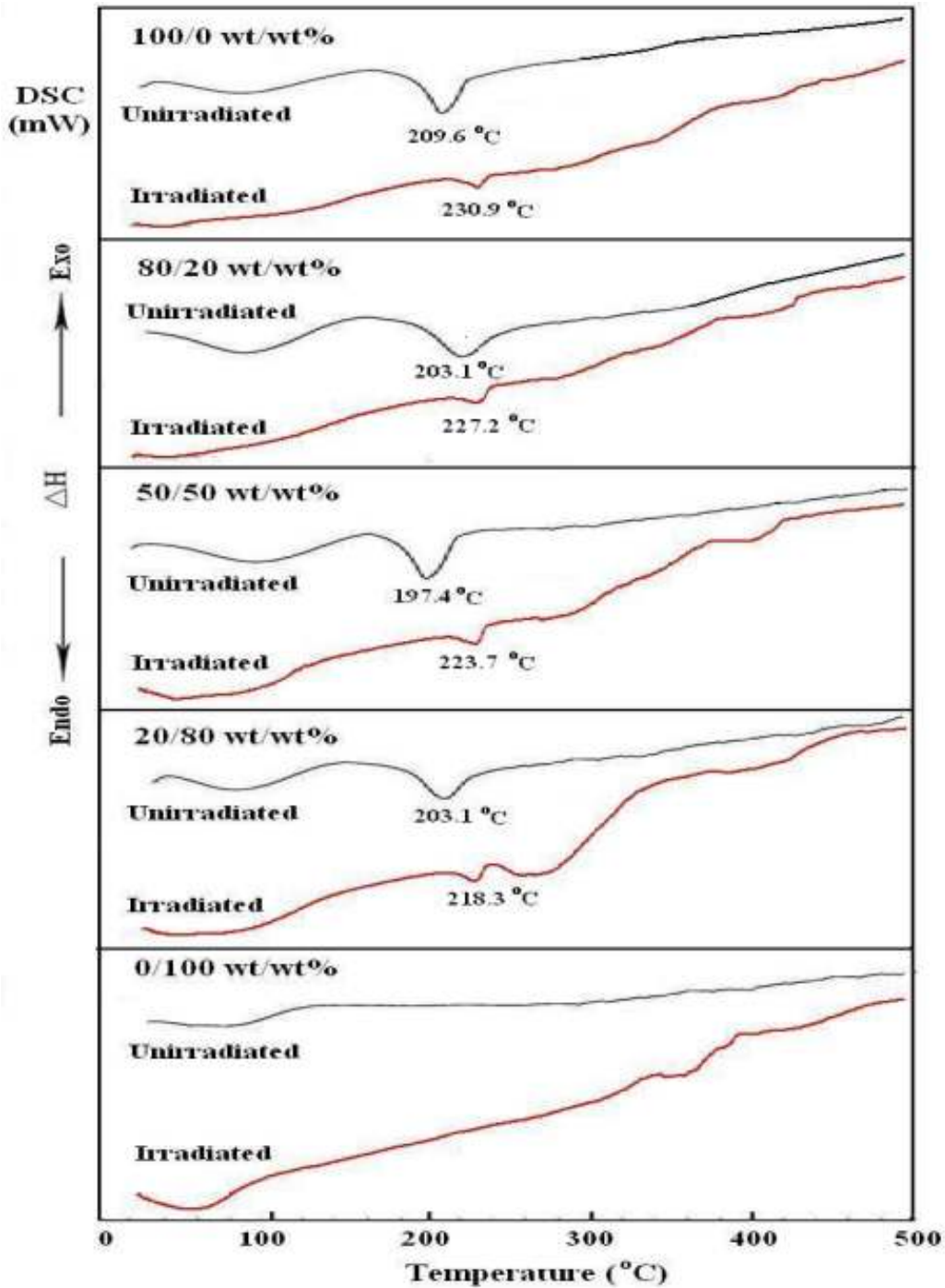


Fig. 1. DSC curves of PVA/HPC blends unirradiated (black solid curves) and irradiated with fast neutrons fluence 1.15×10^7 n/cm² (red solid curves)

Table 1. Values of transition temperatures and associated heat of fusion for PVA/HPC blends before and after irradiation with fast neutrons fluence 1.15×10^7 n/cm²

PVA/HPC blend (wt/wt %)	First order thermodynamic transition region (First step)		At melting transition region					
			Second step		Third step		Fourth step	
	T _{th} (°C)	ΔH (J/g)	T _m (°C)	ΔH (J/g)	T _m (°C)	ΔH (J/g)	T _m (°C)	ΔH (J/g)
Before irradiation								
100/0	88.1	-103.90	209.6	-67.38	Not detected		Not detected	
80/20	93.9	-143.05	203.1	-50.40	Not detected		Not detected	
50/50	95.4	-117.99	197.5	-68.30	Not detected		Not detected	
20/80	75.1	-116.31	203.1	-54.80	Not detected		Not detected	
0/100	83.9	-32.62	Not detected		Not detected		Not detected	
After irradiation								
100/0	36.1	-3.85	230.9	-11.06	322.9	-14.50	447.5	-10.77
80/20	Not detected		227.2	-15.12	270.6	-10.65	477.3	+18.84
					399.4	+11.53		
50/50	39.7	-136.77	223.2	-17.16	271.0	-31.69	461.9	-71.57
					376.4	-20.19		
20/80	41.6	-157.61	218.3	-11.81	259.9	-134.34	387.2	-35.10
							476.6	-52.34
0/100	50.3	-127.49	Not detected		345.2	-18.97	407.1	+8.26
							415.9	-7.12
							483.7	-52.94

The value of the heat of fusion (ΔH) which is represented by the area under the endothermic peaks of decomposition steps and rate has been found to be very effective to evaluate fire hazards. After irradiation the blends, for the second decomposition step, the blended sample 50/50 wt/wt% has the highest heat release value while the blended sample 20/80 wt/wt% has the lowest one. For the third decomposition step, the blended sample 20/80 wt/wt% has the highest heat release value while the lowest one is that for the blended sample 80/20 wt/wt%. For the fourth step, the blend 50/50 wt/wt% has the highest heat release value.

The decomposition temperatures of any polymer depend on its molecular weight and its purity; also, it is affected by its morphology. These decomposition temperatures depend on the crystallinity of the polymer - the highest the crystallinity has the higher decomposition peak [21]. The obtained changes in heat of fusion (J/g) and increase in the melting temperature points (T_m) suggested that the crystallinity and perfection of crystal structure are reduced with increasing of cross-linking due to irradiation. The variations in shape and area are attributed to the different degrees of crystallinity found in the samples with different HPC concentrations [22,23]. As well known by previous reported works [15], a change in the crystalline structure may result from

polymer-polymer interactions (PVA-HPC) in the amorphous phase. Therefore, disorder in the crystals is created, changing the enthalpy of the phase after irradiation with fast neutrons [24,25].

3.2 Thermogravimetric Analyses

The thermogravimetric (TGA) curves for unirradiated and irradiated PVA/HPC blends in the range of temperature from 20 up to 600 °C are shown in Fig. 2. Table 2a represents the deviation of the % weight loss for the unirradiated and irradiated PVA/HPC blends for the decomposition regions as well as the total weight loss. From the figure and the table, it is noticed that, the unirradiated homopolymers PVA and HPC (black curves) show 8.988 and 5.691% weight loss at the first decomposition stage, respectively, beginning at about 20°C, followed by thermal stability at the second decomposition stage. More significant % weight loss by 51.159 and 80.474 for PVA and HPC starting above 205 and 220°C, respectively, are detected. In addition, it is noticed that, the recorded thermogravimetric plots for all unirradiated blends (black curves in Fig. 2) show two degradation regions. On the other hand, for the irradiated blends with fast neutrons (red curves in the figure), four weight loss steps are detected which suggest the coexistence of more than one degradation process.

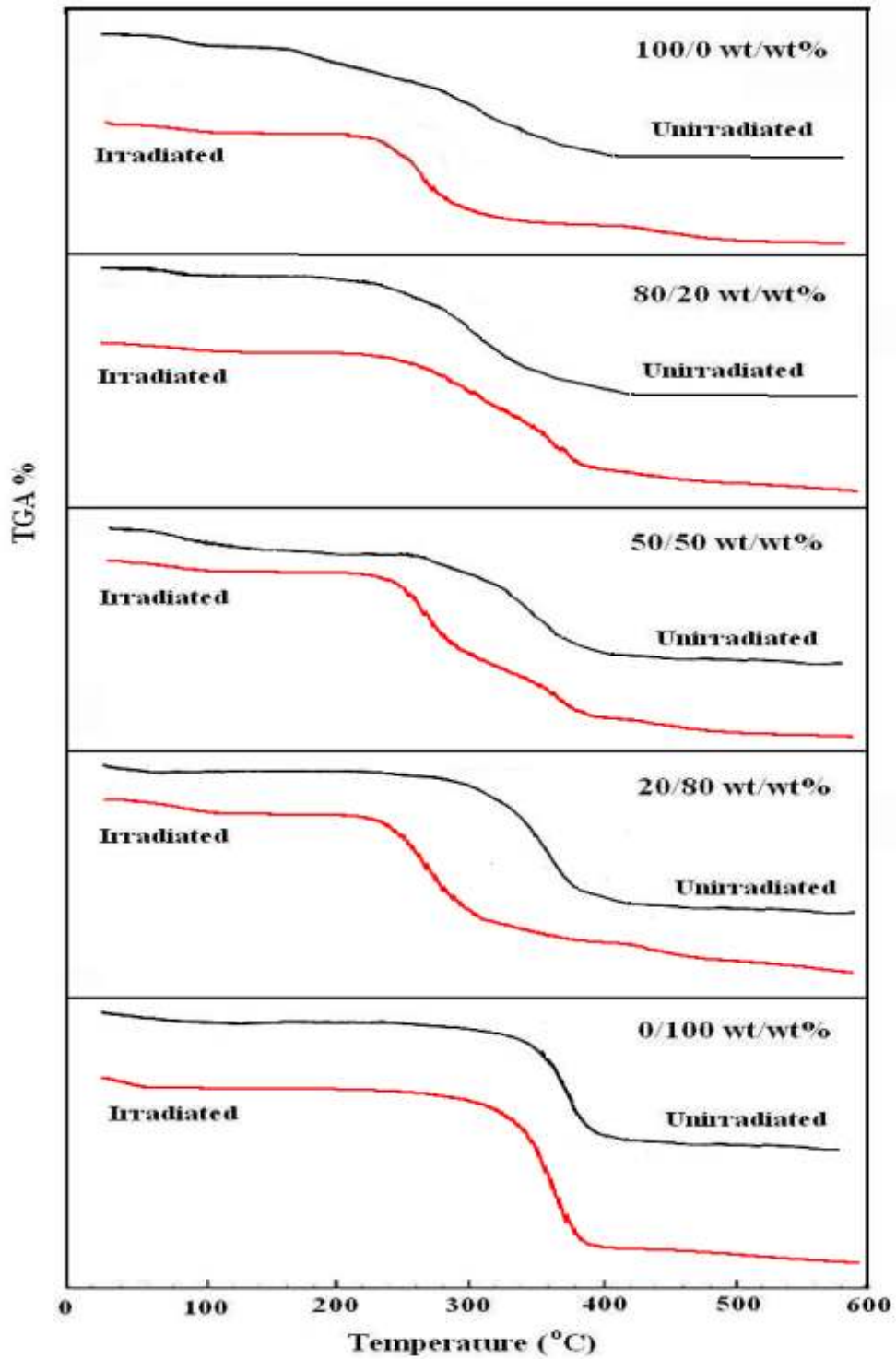


Fig. 2. TGA curves of PVA/HPC blends unirradiated (black solid curves) and irradiated with fast neutrons fluence $1.15 \times 10^7 \text{ n/cm}^2$ (red solid curves)

The lower values of % weight loss in the first decomposition stage affirm the presence of a thermal process due to moisture evaporation from samples. In addition, it may be due to splitting or volatilization of small molecules and/or monomers in which weight loss varies between 2.5-12.2% for unirradiated and 3.5-7.5% for the irradiated PVA/HPC blends, and begins at near 30°C. This lower value of weight loss enables one to suggest that the transitions observed in the corresponding temperature range of the earlier DSC spectra may explain an existence of physical transition [26].

The next process in TGA curves covers a wider temperature range (180-600°C), which includes the melting points, as physical transition and the degradation temperatures. The second and third loss peaks starting at about 220 and 300°C, respectively (Fig. 2 and Table 2a) for the unirradiated and irradiated pure PVA samples are due to crystal cleavage and may be attributed to melting and degradation of different

morphological components forming a high complex structure of PVA. It is characterized by the presence of the typical melting endotherm with a single second peak at about 304 and 260°C for unirradiated and irradiated PVA samples, respectively. These steps correspond to the weight loss due to the decomposition of the PVA structure and coincides with the temperature range over which a number of defined pyrolysis reactions takes place in PVA. The thermal stability of the samples can be determined by these initial temperatures of the thermal decomposition stages, which is in agreement with that previously reported by Chiu [27].

Furthermore, it is noticed from Table 2a that, the second and third steps mid-point temperatures of the irradiated PVA/HPC blends are higher than that of the pure PVA. By following the % weight loss occurred in the second step, the lowest % weight loss is occurred for blend 50/50 wt/wt%.

Table 2. TGA data for PVA/HPC blends before and after irradiation with fast neutrons fluence 1.15×10^7 n/cm²

(a)

PVA/HPC blend (wt/wt %)	At First order thermodynamic transition region (First step)		At melting transition region					
			Second step		Third step		Fourth step	
	% weight loss	Mid point (°C)	% weight loss	Mid point (°C)	% weight loss	Mid point (°C)	% weight loss	Mid point (°C)
Before irradiation								
100/0	8.988	70.9	51.159	303.6	Not detected		Not detected	
80/20	7.305	79.9	65.145	303.9	Not detected		Not detected	
50/50	12.209	65.5	47.095	341.8	Not detected		Not detected	
20/80	2.547	47.3	80.611	356.6	Not detected		Not detected	
0/100	5.691	50.9	80.474	360.2	Not detected		Not detected	
After irradiation								
100/0	3.467	81.7	34.718	259.9	Not detected		5.998	433.3
80/20	4.135	74.5	Not detected		44.771	322.57	6.366	451.9
50/50	4.318	73.2	33.067	262.1	20.626	354.00	6.063	449.6
20/80	7.516	74.3	65.942	265.5	Not detected		9.380	436.1
0/100	5.413	42.2	Not detected		82.214	348.92	6.402	503.2

(b)

PVA/HPC blend (wt/wt %)	Before irradiation		After irradiation	
	Total %weight loss	Residue %	Total %weight loss	Residue %
100/0	60.147	39.853	44.183	55.817
80/20	72.450	27.550	55.272	44.728
50/50	59.304	40.696	64.074	35.926
20/80	83.158	16.842	82.838	17.162
0/100	86.165	13.835	94.029	5.971

A fourth decomposition step is detected when the blends are irradiated to fast neutrons of fluence 1.15×10^7 n/cm². It is clear from Fig. 2 (red curves) and Table 2a that, for irradiated PVA/HPC blends, the decomposition of 80/20 and 50/50 wt/wt% blends occur at about 452 and 450°C, respectively, while that for 20/80 wt/wt% blend, the decomposition occurs at about 436°C and shows the highest % weight loss at this stage.

From Table 2b, at the end of the thermal decompositions of unirradiated and irradiated PVA/HPC blends. It is indicated that, the total % weight loss for both unirradiated and irradiated 20/80 wt/wt% blend are the highest total % weight losses. This means that, they have the lowest char residue in comparison with the other blends; [Char weight = $W_1 - W_2$; where W_1 is the total weight of the blend sample at the starting thermal process and W_2 is the total weight loss of the blend sample at the end process]; and also indicate that the thermal stability of this blend is enhanced and it is more resistant to fire hazards.

In addition to the above mentioned, the lower temperature loss (i.e., the first stage) may have corresponded to the breaking of the ester linkages, and the other stages may have corresponded to the degradation of the whole polymer. The second, third and fourth losses are the most important both in the rate of weight loss and in the total weight loss. If PVA is heated above 120°C water is eliminated to give conjugated double bonds, and it may give a formation of ether cross-links; i.e., on heating PVA above the decomposition temperature, the polymer starts a rapid chain-stripping elimination of H₂O [28].

3.3 X-Ray Diffraction Analyses

In the present work, X-ray diffraction is used to check the crystalline formation of PVA/HPC blends before and after irradiation with fast neutrons. XRD patterns of unirradiated and irradiated the homopolymers PVA and HPC and their blends in different weight percent ratios are shown in Fig. 3.

The appearance of sharp reflections and diffuse scattering, observed from the XRD of unirradiated pure PVA (100/0 wt/wt%), is characteristic of crystalline and amorphous phases of conventional semi-crystalline polymers. Moreover, several distinct crystalline peaks at $2\theta = 11.21, 12.69, 15.28, 19.45, 20.65, 23.52, 25.19, 28.15$ and 30.74° , assigned to a mixture of (110), (101), (10 $\bar{1}$), (200), (201) and

(20 $\bar{1}$) are observed which agree with that previously reported in the literatures [26,29,30]. The pattern of unirradiated pure HPC (0/100 wt/wt% in Fig. 3) shows amorphous features characterized by two halos centered at $2\theta = 16.356^\circ$ and 13.518° . It is also clear from the figure that, the XRD patterns of unirradiated PVA/HPC blends exhibited the characteristics of pure PVA but with less intensity for the crystalline peaks. Thus, one can say that the semi-crystalline structure of PVA is decreased upon mixing with different concentrations of HPC. In addition, some crystalline peaks are no longer detectable ($2\theta = 15.28, 20.65$ and 23.52°) since they are of vanishingly small intensity in the XRD patterns of homopolymers. This is attributed to weak reflections from the ordered structure.

It is also clear for the irradiated PVA and HPC and their blends that, the peaks observed from the unirradiated patterns started diminishing which means that fast neutron irradiation can cause structural variation in the polymeric network of PVA/HPC blends and are observed to coincide with that present in the XRD pattern of the irradiated pure HPC sample. This confirms the presence of HPC crystallites with the polymeric matrix [15]. By following all of the peaks intensities at the different 2θ values, changes in the intensity of the XRD peaks of the irradiated blends changed continuously with irradiation.

For the semi-crystalline amorphous blends, the non-crystallizing component could strongly modify the crystallization behavior of crystallizing component. The phase morphology of these systems of blends is governed by the compatibility of the amorphous phase and nature of the crystalline phase [31]. For such blends, miscibility between the amorphous components of both homopolymers PVA and HPC is possible.

Crystallinity is defined as the weight fraction of the crystalline portion of a polymer. XRD is used to measure the crystallinity in the polymers. The XRD patterns (Fig. 3) and Eq. (1) is using to calculate the crystallinity index (CrI) for both unirradiated and irradiated PVA/HPC blends. Two distinguish bands centered at $2\theta = 18.8-19.6^\circ$ as a fundamental band and at $2\theta = 28.1-28.4^\circ$ as a secondary one for the unirradiated PVA/HPC blends, and at $2\theta = 20.0-23.0^\circ$ as a fundamental band and at $2\theta = 42.0-43.5^\circ$ as a secondary one for the irradiated PVA/HPC blends, are chosen. Table 3 illustrates the variation of the calculated crystallinity index (CrI) for unirradiated and irradiated PVA/HPC blends.

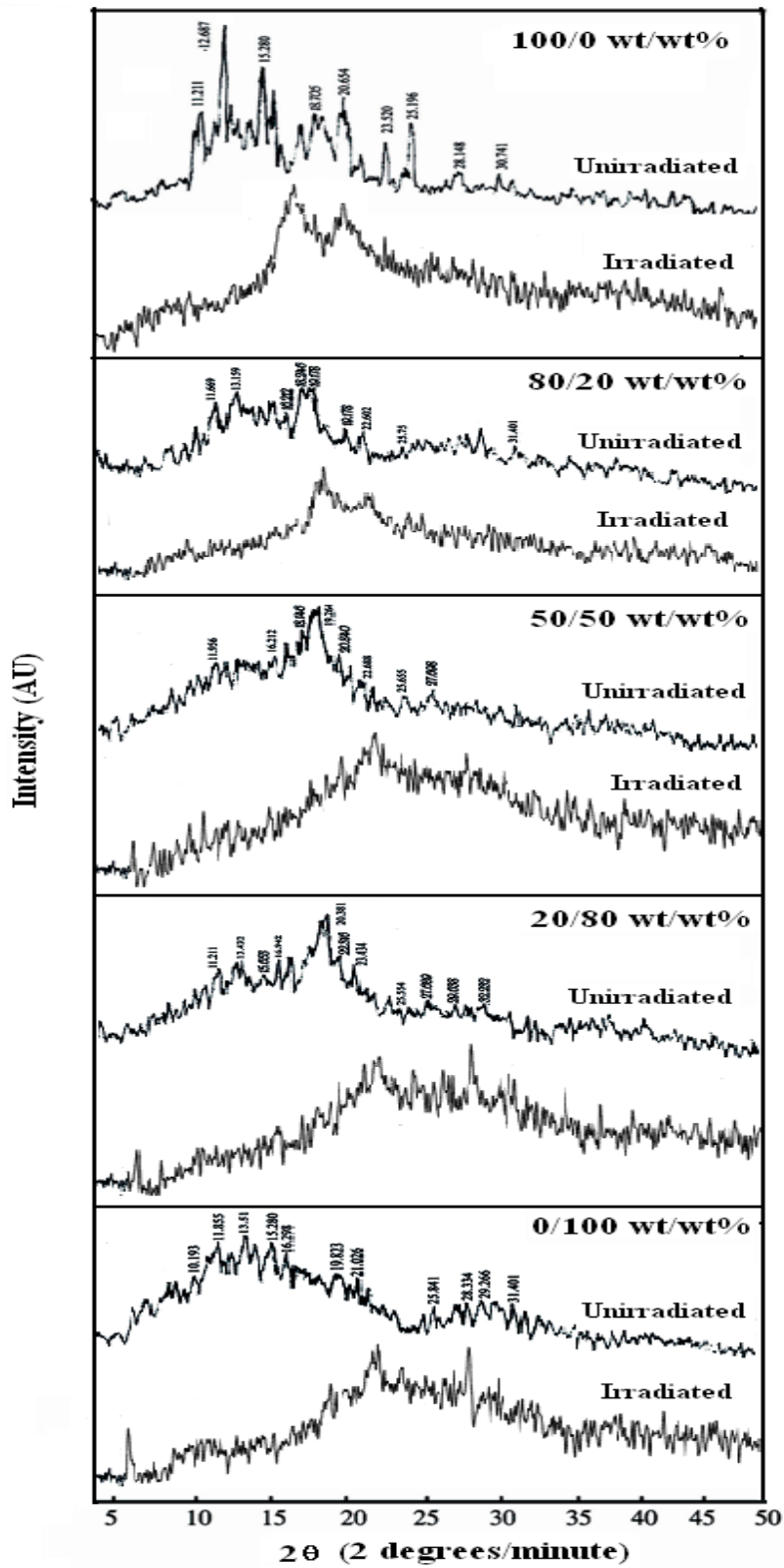


Fig. 3. Variation in X-ray diffraction patterns of PVA/HPC blends unirradiated and irradiated with fast neutrons fluence 1.15×10^7 n/cm²

Table 3. Variations in the crystallinity index (CrI) and their percentage changes for PVA/HPC blends unirradiated and irradiated with fast neutrons fluence 1.15×10^7 n/cm²

Blend sample PVA/HPC (wt/wt %)	Before irradiation		After irradiation	
	Crystallinity index (CrI)	$\Delta(\text{CrI})\%$ * According to HPC concentration	Crystallinity index (CrI)	$\Delta(\text{CrI})\%$ ** According to fast neutron irradiation
100/0	47.17	-	35.55	-24.63
80/20	49.98	5.96	36.75	-26.47
50/50	36.91	-21.75	30.20	-18.18
20/80	31.60	-33.01	22.97	-27.31
0/100	-	-	-	-

$$* \Delta(\text{CrI})\% = \frac{(\text{CrI})_{\text{blended PVA}} - (\text{CrI})_{\text{pure PVA}}}{(\text{CrI})_{\text{pure PVA}}} \times 100$$

$$** \Delta(\text{CrI})\% = \frac{(\text{CrI})_{\text{Unirradiated}} - (\text{CrI})_{\text{Irradiated}}}{(\text{CrI})_{\text{Unirradiated}}} \times 100$$

It is clear from the table that, the calculated values of crystallinity index (CrI) of PVA/HPC blends samples decrease either by increasing HPC concentration and/or by irradiation with fast neutrons reaches its minimum value for the blend sample 20/80 wt/wt%. This decrease in CrI may indicate that there was a decrease in crystalline regions, i.e., amorphosity dominated and implies changes in the structural regularity of the main chains of the polymeric molecules on doping.

4. CONCLUSION

The obtained results indicate that structural changes occurred in PVA matrix as HPC diffused. The decrease in crystallinity index has been proposed as one or another aspect of the complicated molecular and crystalline structure induced when irradiating with fast neutrons the PVA/HPC blends. This may be attributed to variations in the internal mechanisms that occurred by the induced effect of HPC and fast neutrons irradiation on the structure of the blends (PVA/HPC). As a result, variations in the macromolecular and micromolecular structure of the PVA network are produced.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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