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Nitrogen ion beam radiation induced modification on physico-chemical properties of poly (vinyl chloride)/ethylene vinyl acetate blends

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ABSTRACT

Poly (vinyl chloride) (PVC), ethylene vinyl acetate (EVA), and PVC/EVA (50/50 wt%) samples were synthesized using casting technique. The as-prepared samples were irradiated with various fluencies of nitrogen ion beam, using a broad beam cold cathode ion source. The structural, optical and thermal properties of pristine and irradiated samples were investigated using FTIR, XRD, UV-VIS, and TGA analytical techniques. FTIR and XRD investigation showed several changes in the crystal structure, intensity and position of the characteristic peaks for the investigated samples. The optical band gap energies for both PVC and EVA samples is being to decrease with increasing ion beam fluence, whereas 50/50 wt% PVC/EA blend sample exhibits an increment behavior as a result of increasing ion beam fluence. The thermal stability of ion beam bombarded samples decreased for PVC and EVA samples than that for pristine samples, whereas a noticeable increase in the stability of the 50/50 wt% PVC/EVA blend samples. It is clear that thermal decomposition is affected by nitrogen ion beam fluencies owing to the extent of crosslinking and degradation processes. The results showed that nitrogen ion beam bombardment significantly affect the physico-chemical properties of PVC, EVA, and PVC/EVA samples.

INTRODUCTION

Surface modification of polymers and polymer blends is considered to be of a growing interest. There are various applied techniques which were proposed to modify the desired physical and chemical properties. In this respect, an effective process for adjusting and modifying polymers and polymer blends is the using of irradiation treatments such as gamma rays, X-ray, electron beam irradiation, and ion beam bombardment. In addition, irradiation treatments were employed to advance the physical, chemical, optical, electrical, thermal and mechanical properties. Also, ion beam

irradiation is well recognized for modification of polymers and polymer blends which is greatly dependent on various parameters for example, ion species, ion beam fluence, ion beam energy, and target material. Ion beam technique is one of the most popular systems as it is very flexible technique to modify or induce special properties for a large number of materials, including polymers, polymer blends, inorganic compounds, metals, and semiconductors, [1-9].

Engineering plastic materials such as polymer blends are accepted for applications due to excellent thermal, mechanical, and optical properties. The most important

commercially PVC polymer which is highly used all over the world due to its easy processing, good mechanical, excellent thermal, higher chemical resistance and fire retardant properties, [10-15]. On the other hand, ethyl vinyl acetate (EVA) has better flexibility, toughness, lower chemical resistance, the ability to accept high filler loadings without huge change in its properties, and the ease of crosslinking [16-19].

The aim of the present work is to develop PVC, EVA and PVC/EVA (50/50 wt%) samples in a wide scale of applications and to study the influence of nitrogen ion beam irradiation on the structural, optical and thermal properties.

2. EXPERIMENTAL

2.1. Materials

PVC powder was obtained from Sigma-Aldrich (St. Louis, USA). EVA granular, (18 wt% of vinyl acetate content, VA %) was purchased from LOFINA Brazil. Tetrahydrofuran (THF) 99.9 % was purchased from Merck, Germany.

2.2. Preparation of PVC/EVA blend samples

PVC/EVA blend samples with various weight ratios of PVC and EVA polymers were prepared by solution casting technique, as shown in Table 1. PVC and EVA samples were separately dissolved in 50 ml of THF. PVC/EA blend sample was prepared by adding EVA solution to PVC one with 50/50 wt% of each. The obtained solutions were stirred for 1-2 hours at room temperature to get a homogenous solution. The as-prepared solutions were then casted onto glass plates and then allowed to evaporate and dried at room temperature. The as-prepared thin film, (0.2 mm), was carefully taken out.

Table (1): Formulation of PVC/EVA blend samples

Polymer	Weight ratio (wt%)		
PVC	100	50	0.0
EVA	0.0	50	100

2.3. Nitrogen ions beam irradiation.

A cold grid cathode Penning ion source was used to irradiate the as-prepared samples (0.2 mm thickness) with various fluencies of nitrogen ion beam. Figure 1 shows a schematic of the cold cathode ion source used for the irradiation process, installed at radiation physics department, National Center for Radiation Research and

Technology, (NCRRT), Egyptian Atomic Energy Authority, (EAEA), [20]. The ion source consists of stainless-steel anode of 30 mm inner diameter, 60 mm length, and two stainless-steel disc cathodes of 50 mm outer diameter and 2 mm thickness; The inner diameter of one cathode disc grid of 18 mm diameter. The anode and cathode were separated by pure Teflon insulator flange of 8 mm thickness. The anode is connected by positive power supply and the two cathodes are connected by earth. The ion current is measured on Faraday cup is placed on 5cm from the extractor electrode. The irradiation conditions are 3 KeV current energy, fixed ion current density $120 \mu\text{A}/\text{cm}^2$ and ion beam fluencies are 6×10^{17} , 12×10^{17} and 18×10^{17} ions. cm^{-2} . The investigated samples are fixed at 5 cm distance downstream from the ion beam exit aperture.

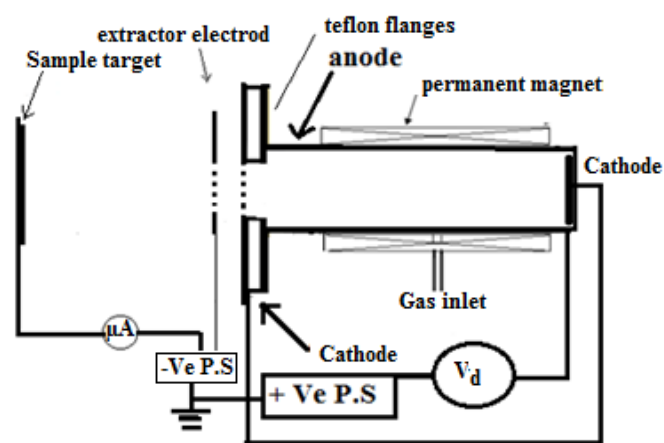


Fig. (1): Schematic diagram of a cold grid cathode Penning ion source [20]

2.4. Characterization

2.4.1. X-ray diffraction (XRD) measurements

XRD patterns of unirradiated and irradiated samples with nitrogen ion beam at various ion beam fluencies were recorded using a Shimadzu XRD 6000 X-ray diffractometer (made in Japan). XRD patterns were carried out at a scan rate of 2°min^{-1} on the diffractometer with $\text{CuK}\alpha$ radiation source with a wavelength of 0.1546 nm, a generator with voltage and current of 40 kV and 40 mA, respectively.

2.4.2. Fourier transform infrared (FTIR) spectroscopy

FTIR spectra of unirradiated and irradiated samples were measured at room temperature using FTIR spectrometer equipped with attenuated total reflectance (ATR) unit, (Vertex 70 Bruker Optics, Germany).

2.4.3. Ultraviolet/visible spectrophotometer (UV/VIS)

A UV/VIS absorption spectrophotometer (Unicam spectrometer, made in England) was used to measure the optical properties of the samples under investigation in the wavelength range of 200-800 nm.

2.4.4. Thermogravimetric analysis (TGA)

Thermogravimetric analysis (TGA) was conducted by a Shimadzu-50 (made in Japan). 10 mg of unirradiated and irradiated samples were used for TGA investigation from room temperature up to 600 °C at heating rate of 10 °C/minute in a nitrogen atmosphere with constant rate of about 50 ml/minute to prevent thermal oxidation process for the investigated samples. The instrument's reproducibility for temperature and mass was 1 °C and 0.2%, respectively.

3. RESULTS AND DISCUSSIONS

When an energetic nitrogen ion beam strikes a polymeric material, it loses its energy and induces different effects in the material. The effects of nitrogen ion beam bombardment on the optical, structural and physico-chemical properties of PVC, EVA, and PVC/EVA (50/50 wt%) samples were investigated.

3.1. XRD studies

X-ray diffraction patterns of unirradiated and irradiated PVC, EVA, and PVC/EVA samples before

and after being exposed to various fluencies of nitrogen ion beam are shown in figure 2. One main crystalline peak can be seen at about 21.48 degrees in the spectrums of both pristine and irradiated PVC samples, (see figure 2a). The crystalline phases of PVC matrix also gave rise to other low, intense peaks at about 19.76, 19.14, 23.6, and 18.06 degrees. These results confirm the semi-crystalline nature of PVC matrix with dominating amorphous content. It is clear from XRD patterns for both PVC and EVA that the main peak's intensity decreases without changing in its position. In general, the gradual decrease in the crystalline peak intensity as a result of increasing ion beam fluence denotes to a decrease in the amount of crystalline phase. This behavior indicates that the crystalline structure of ion beam bombarded samples has been destroyed due to main chain scission reaction throughout polymeric materials. These changes cause a disorder structure through ion beam irradiated samples, which increase the amorphous region as will show in FTIR measurement.

The peak intensity of irradiated PVC/EVA, (50/50 wt%) blend samples increases as ion beam fluence increases, (see Figure 2b), indicating a gradual increase in polymer crystallinity. This may be due to ion beam radiation-induced crosslinking reaction within molecular polymer chains or by the creation of single or multiple helices.

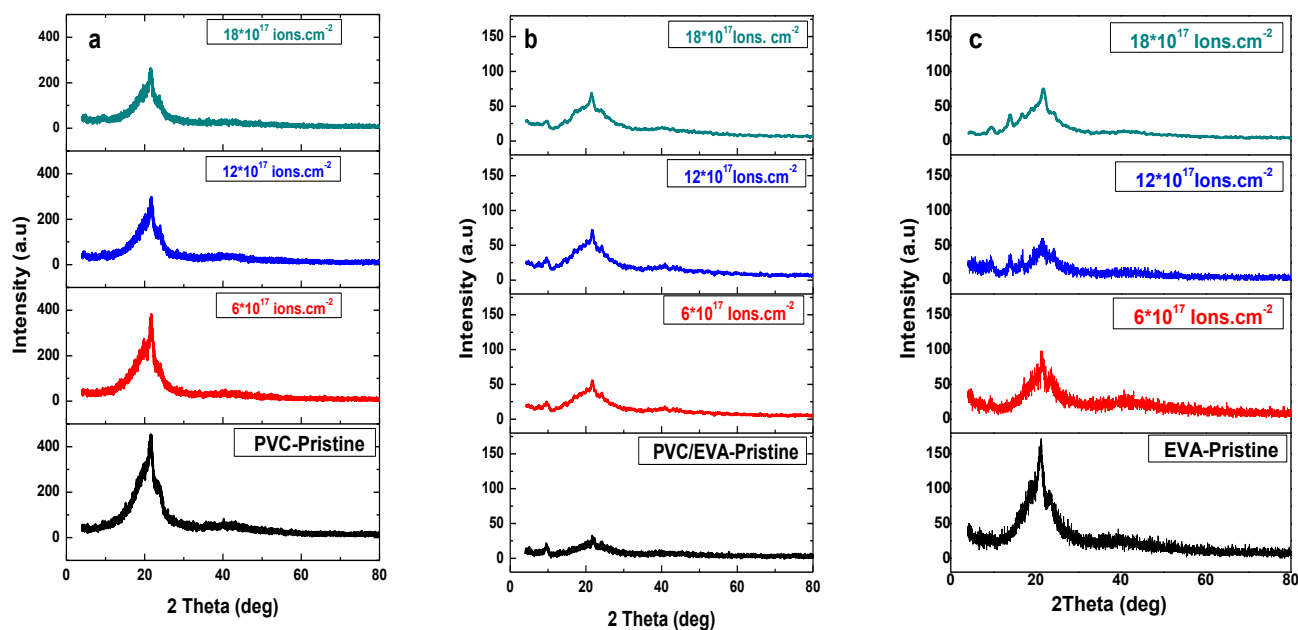


Fig. (2): XRD patterns of pristine and ion beam bombarded, (a) PVC, (b) PVC/EVA (50/50 wt%) and (c) EVA samples with various nitrogen ion beam fluencies

3.2. FTIR Studies

The structural changes in PVC, PVC/EVA (50/50 wt%) and EVA samples induced by various fluencies of nitrogen ion beam were recorded using FTIR spectrometry. The induced structural changes due to ion beam bombardment was estimated from the relative decrease and/or increase in the intensity of the characteristic bands associated with the functional groups present in the investigated samples. Upon ion beam bombardment, the breaking of chemical bonds, and the recombination of molecular fragments to form new molecular bonds yielding new chemical bonds. Figure 3(a-c) illustrates FTIR spectra of pristine and ion beam irradiated PVC, EVA, and PVC/EVA (50/50 wt%) samples, in the wave number range 400-4000 cm^{-1} . FTIR spectrum of PVC sample is shown in Figure 3a, where a characteristic peak appears around 671 cm^{-1} indicating the C-Cl stretching vibration which is correlated to the sensitive crystalline nature of PVC polymer chain. The band at 2979 cm^{-1} of C-H (CH-Cl) stretching and asymmetric stretching vibrations of C-H for CH_2 at 2910 cm^{-1} . The FTIR band around 1400 cm^{-1} is correlated to aliphatic C-H bending vibration. The peak at 1250 cm^{-1} can be assigned to the bending vibration of C-H near Cl. The C-C stretching vibration of PVC backbone chain occurs in the range 1000-1100 cm^{-1} [21,22]. On the other hand, a typical absorption bands of EVA sample exhibit strong absorbance band at about 2850 and 2920 cm^{-1} that are associated with the C-H asymmetric stretching vibration, whereas the band at 1734 cm^{-1} is attributed to the stretching vibration of carbonyl ester of carboxyl group [23], (see Figure 3b). In addition, the observed FTIR band at about 1247 cm^{-1} , (assigned to C-H), is attributed to the bending vibration of C-H near Cl, and at 667 cm^{-1} , (assigned to C-Cl bond). The -C=O peak at 1738 cm^{-1} is less intense than that in pure EVA, (see Figure 3c). The spectrum of PVC/EVA blend sample shows all the characteristic peaks with less intense than that for pure PVC and pure EVA and the observed peaks were

shifted to lower wavenumber. In general, Figure 3 displays the peak intensity reduction for irradiated samples as compared to pristine ones. This observation can be attributed to the breaking of chemical bonds throughout molecular chains, and the formation of radiation-induced free radicals due to the escape of the hydrogen and other volatile gases. In addition, one can conclude that the change and/or shift in the characteristic FTIR bands after the sample was exposed to various fluencies of nitrogen ion beam can be attributed to the chain degradation reaction, [23].

3.3. Ultraviolet visible (UV/VIS) studies

UV/VIS spectra in the range of 200-800 nm is used to determine the effect of nitrogen ion beam irradiation on the optical properties of PVC, PVC/EVA (50/50 wt%), and EVA samples. UV/VIS spectra of pristine as well as irradiated samples with various fluencies of nitrogen ion beam are shown in Figure 4. In the current work, the UV/VIS spectra show a remarkable change in the absorption edge with increasing ion beam fluence. The change in the absorption edge is largely dependent on both chemical structure of the investigated samples and ion beam fluence. This behavior can be attributed to the chemical changes that lead to absorption bands more or less defined primarily in the UV/VIS range. The observed UV/VIS changes could be interpreted by ion beam radiation-induced formation of extensive systems of conjugated double bonds. Ion beam irradiation of polymeric samples causes bond breaking and formation of new bond, leading to a combination of degradation and crosslinking reaction, with formation of unsaturated products [24-26]. Clearly, the absorbance spectrum of both pristine PVC and EVA samples gradually increases with increasing ion beam fluence, this is due to chain degradation reaction. On the other hand, PVC/EVA blend samples exhibit a decreasing behavior, this is due to chain crosslinking reaction.

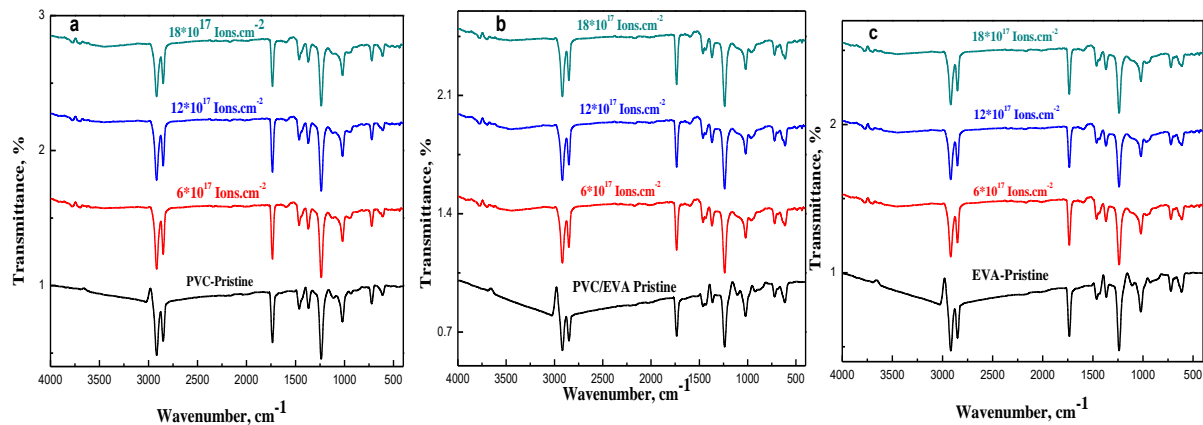


Fig. (3): FTIR spectra of pristine and ion beam bombarded, (a) PVC, (b) PVC/EVA (50/50 wt%) and (c) EVA samples with various nitrogen ion beam fluencies

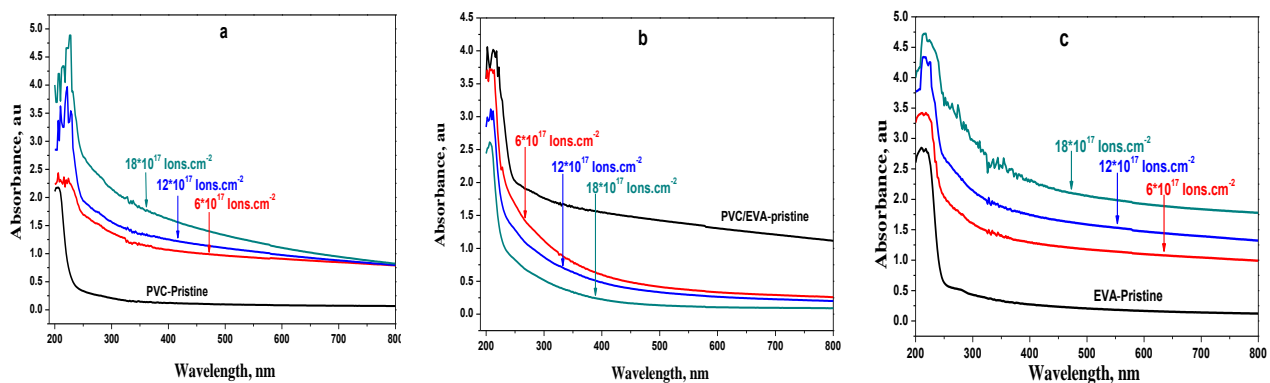


Fig. (4): UV/VIS absorption spectra of pristine and ion beam bombarded, (a) PVC (b) PVC/EVA (50/50 wt%) and (c) EVA samples with various nitrogen ion beam fluencies

Optical band gap energy determination

The optical band gap energy, (E_g , eV) is estimated using the following equation, [27]:

$$\alpha h\nu = A (h\nu - E_g)^n \quad (1)$$

where A (cm^{-1}) is the band tailing constant depending on the sample structure, α is the absorption coefficient, E_g is the optical band gap energy (eV), h is plank's constant (J.sec), ν is the frequency of radiation (sec^{-1}), and $h\nu$ is the energy of the incident photon energy (eV). In this respect, n represents an index with the values: $1/2$, $3/2$, 2 or 3 , which is dependent on the type of transition responsible for the absorption. The present polymeric samples (semi-crystalline materials) obeys the rule of indirect transition as well as direct transition. The value of α is calculated by Beer–Lambert's equation [28].

$$\alpha = \frac{2.303 A(\lambda)}{t} \quad (2)$$

The direct optical band gap energy can be estimated from the linear plots of $(\alpha h\nu)^2$ versus $h\nu$ as illustrated in figure 5 for (a) PVC, (b) PVC/EVA and (c) EVA samples, respectively at various ion beam fluencies. The dependence of E_g as a function of ion beam fluence is summarized in Table 2. The optical band gap energy for direct transition is being to be of decrement behavior as a result of increasing ion beam fluence as in PVC and EVA samples. The optical band gap energy decreases with increasing ion beam fluence and this can be attributed to the increase of structural disorder for the investigated samples. On the other hand, the increase in optical band gap energy for PVC/EVA blend samples with increasing ion beam fluence may be attributed to an increase in the structural crosslinking reaction throughout polymeric chains or by the formation of single or multiple helices, consistent with XRD results.

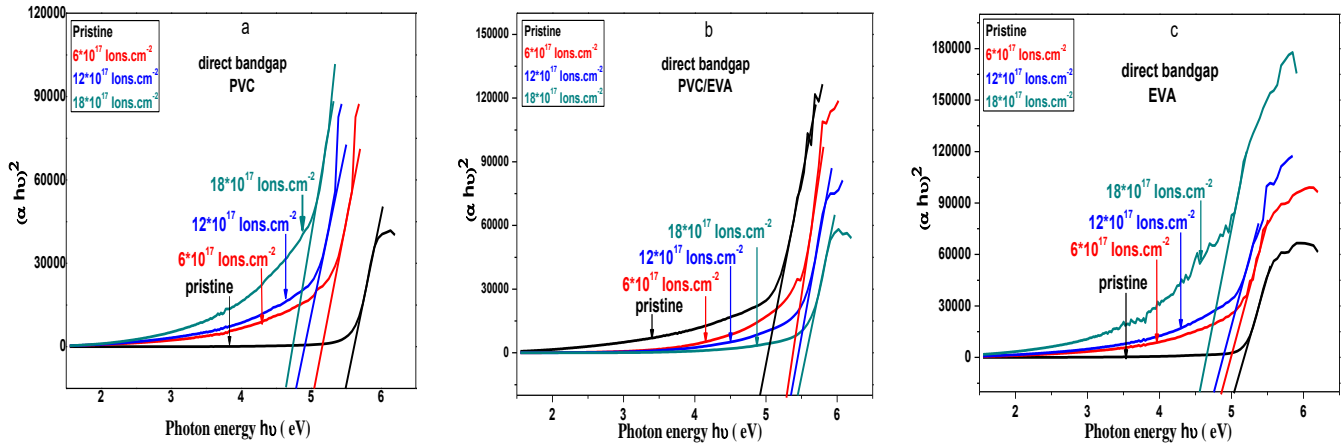


Fig. (5): The direct band gap energy of pristine and ion beam bombarded, (a) PVC, (b) PVC/EVA (50/50 wt%) and (c) EVA samples with various nitrogen ion beam fluencies

Table (2): The values of direct optical band gap energy for PVC, PVC/EVA, (50/50 wt%) and EVA samples at various nitrogen ion beam fluencies

Sample	PVC	PVC/EVA 50/50 wt%	EVA
Ion beam fluencies, $\text{ion}\cdot\text{cm}^{-2}$	E_g (eV)	E_g (eV)	E_g (eV)
pristine	5.50	4.93	5.03
6×10^{17}	5.08	5.30	4.87
12×10^{17}	4.80	5.36	4.75
18×10^{17}	4.64	5.47	4.55

3.4. Thermogravimetric analysis (TGA)

TGA analysis and their derivative (dw/dt , % s^{-1}) of pristine and irradiated PVC, PVC/EVA, (50/50 wt%) and EVA samples with various nitrogen ion beam fluencies are shown in figure 6-7, respectively. The TGA curve of thermal analysis for PVC sample is presented in Figure 6a. It can be clearly seen that PVC matrix have two main thermal decomposition stages in the range of about 300-400 °C within two maximum peaks, namely T_{s1} at about 300-400 °C (peak maximum at 360-370°C.) and T_{s2} at about 425-530 °C, (peak maximum at 475-486 °C). Firstly, prior to 260°C no or less noticeable weight loss is observed. This illustrates that PVC matrix having no water content either absorbed water in its crystal structure.

In addition, the first decomposition step can be attributed to the volatilization of C-Cl bond leading to the evolution of HCl molecules. Also, the second decomposition step is considered as the main decomposition stage due to C-C dissociation reaction throughout PVC matrix [29,30]. On the other hand, the thermal decomposition of EVA matrix proceeds in two decomposition stages in the range 250-400°C, (peak maximum at 320-330°C) and 440-520°C, (peak maximum at 460-490 °C). The first step can be attributed to the loss of acetic acid followed by the degradation of the remaining partially unsaturated polyethylene chain structure throughout the second stage (see figure 6c). In addition, figure 6b represents the thermal decomposition of 50/50 wt% PVC/EVA blend sample occurs in two degradation steps in the range 294-323 °C, (peak maximum at 290-320 °C) and 485-488 °C, (peak maximum at 470-490 °C).

It is revealed that as ion beam fluence increases, the decrease in the thermal stability of both PVC and EVA samples can be attributed to the dominance chain degradation processes over crosslinking. On the other hand, the thermal stability of PVC/EVA (50/50 wt%) blend sample increases upon increasing fluence of nitrogen ion beam bombardment more than pristine sample. This observation can be attributed to the permanent crosslinking process and this is consistent with XRD and UV/VIS results. It is also revealed that the decomposition of the investigated samples shows three decomposition steps with various decomposition rates.

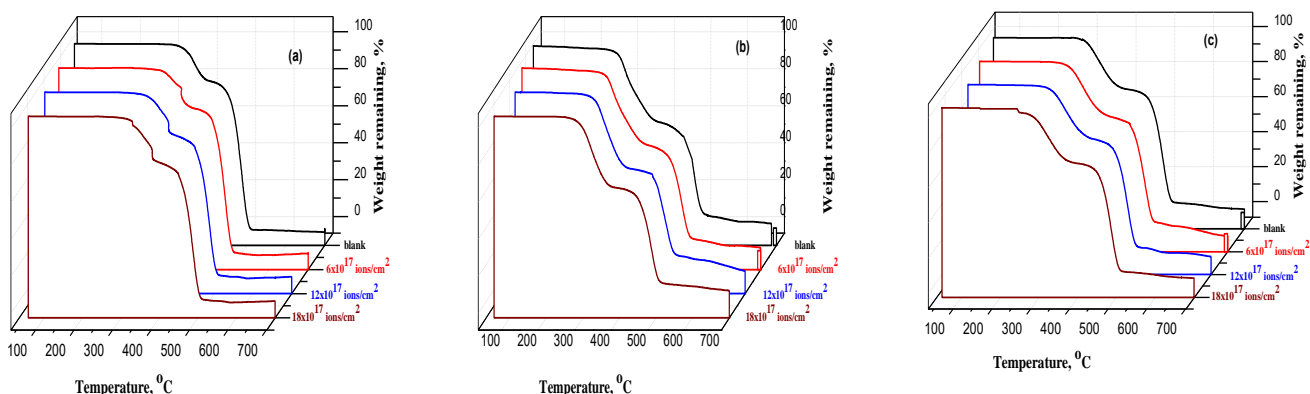


Fig. (6): TGA of pristine and ion beam bombarded, (a) PVC, (b) PVC/EVA (50/50 wt%) and (c) EVA samples with various nitrogen ion beam fluencies

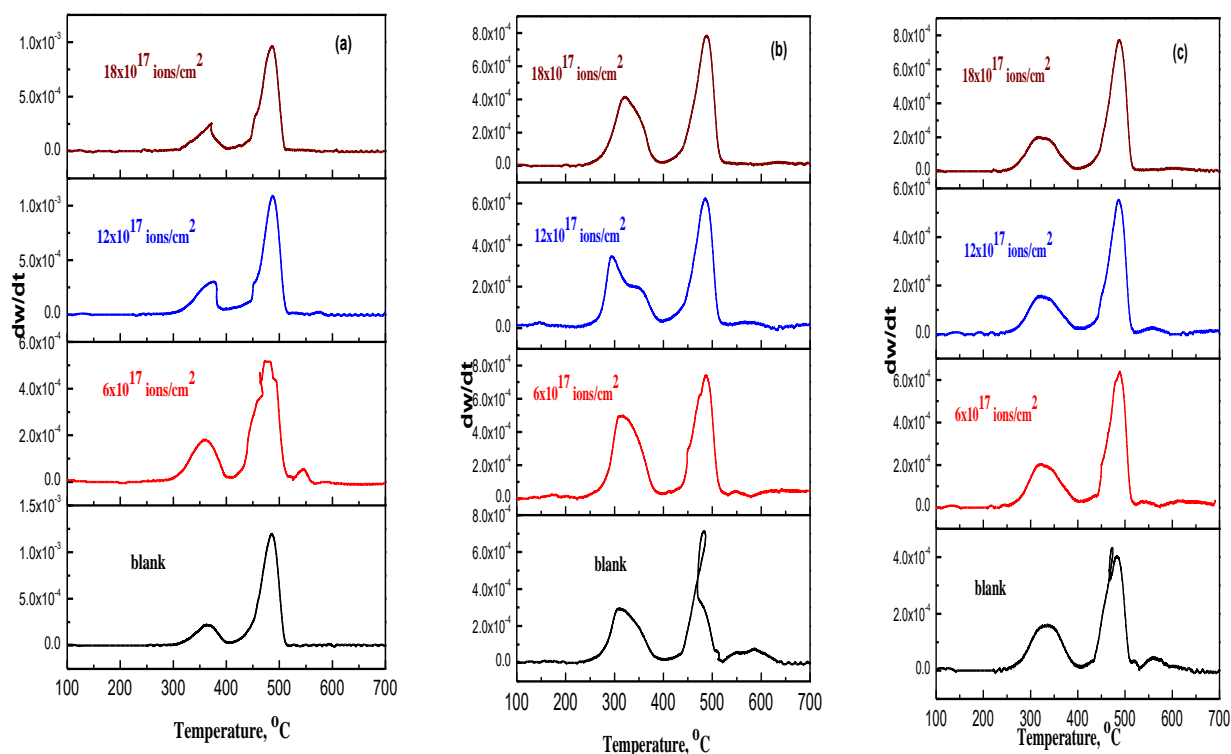


Fig. (7): dw/dt as a function of temperature for pristine and irradiated a) PVC, b) PVC/EVA (50/50wt%) and c) EVA at different N-ion beam fluencies.

Table 3 represents the characteristic temperatures and the corresponding weight loss % for pristine and ion beam irradiated samples. From Table 3 it can be seen that the thermal stability of the

investigated samples increases with increasing nitrogen ion beam fluencies, where EVA and PVC/EVA samples exhibit more thermal stability than that PVC sample.

Table (3): The characteristic temperature data of pristine and ion beam irradiated PVC, 50/50 wt% PVC/EVA and EVA at different temperatures

Ions/cm ²	PVC				PVC/EVA, 50/50 wt%				EVA			
	T _i °C	T _{s1} °C	T _{s2} °C	T _r °C	T _i °C	T _{s1} °C	T _{s2} °C	T _r °C	T _i °C	T _{s1} °C	T _{s2} °C	T _r °C
blank	284	362	486	627	98	312	468	603	253	332	483	652
6 x 10 ¹⁷	264	361	478	557	129	317	486	604	223	327	485	666
12x 10 ¹⁷	261	372	486	575	166	294	485	628	196	321	486	681
18 x 10 ¹⁷	261	369	485	579	242	323	488	667	150	326	488	682

CONCLUSION

In the present work, PVC, PVC/EVA, and EVA samples were conducted by the solution casting method.

According to XRD pattern, the intensity of crystalline peaks gradually decreases for PVC and EVA films as the nitrogen ion beam fluence increases, demonstrating that the crystalline structure has been destroyed due to the main chain scission reaction. While for PVC/EVA samples, the increases in intensity of crystalline structure may be due to crosslinking of polymer chains or by the formation of single or multiple helices.

The optical band gap energy of the investigated samples decreases gradually with the increase of ion beam fluence for PVC and EVA samples, while increase in case of PVC/EVA blend sample. These results confirm improving the optical properties of PVC and EVA films after nitrogen ion beam bombardment and render the samples to be more convenient for optoelectronic device applications.

The thermal stability of ion beam irradiated samples show a decrease in thermal stability upon ion beam bombardment for PVC and EVA, while PVC/EVA blend samples exhibited an increment behavior in the thermal stability properties. It is clear that the thermal decomposition is affected by nitrogen ion beam fluence owing to the crosslinking and degradation processes. The thermal stability of irradiated samples exhibited an increment in PVC/EVA blend sample leading to more convenient for thermal applications.

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