

Physical Properties of Electron Beam Irradiated Poly(vinyl butyral) Composites with Carbamate, Imidazole, and Tetrazolium Dye

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ABSTRACT: Films of poly(vinyl butyral) composites with triphenyl tetrazolium chloride dye (TTC) and the antioxidant nickel dibutyl dithiocarbamate (NBC) or 2-mercaptobenzimidazole (MBI) were prepared by the solution casting method using butyl alcohol as a solvent. The effect of various doses of electron beam irradiation (50–200 kGy) on color response, thermal, and mechanical properties were investigated. The color measurements showed that the films of the different composites possessed a high sensitivity to electron beam irradiation, in which the nearly colorless films were changed to deep red color, which can be easily detected by visual observations. In addition, the change in color depends on irradiation dose and the contents of the TTC dye. Moreover, the presence of the antioxidants NBC or MBI has no effect on the development of color. However, PVB/TTC and PVB/TTC/MBI composites showed high regular change in color as a function of irradiation dose. The thermogravimetric

analysis used to study the thermal stability indicated that PVB/TTC composites either before or after electron beam irradiation are thermally more stable than neat PVB polymer. The presence of the antioxidants NBC or MBI offered protection to PVB/TTC composites against decomposition or oxidative degradation resulted from irradiation. Blending unirradiated PVB with TTC dye, mixture of TTC and NBC, and mixture of TTC and MBI reduced the tensile strength by 4, 20, and 17% upon blending, respectively. However, the reduction in tensile strength of the entire composite films (~7% based on the initial value) upon exposure to a dose of 50 kGy is acceptable for practical applications. © 2006 Wiley Periodicals, Inc. *J Appl Polym Sci* 101: 4358–4365, 2006

Key words: PVB; dye; antioxidants; EB irradiation; color response; thermal; mechanical properties

INTRODUCTION

Exposure of polymers to high-energy radiation may bring about different types of reactions, leading to formation of chemical bonds between the polymer chains (crosslinking) or breaking the bonds to end with chain scission (degradation).¹ Several theories have been proposed to explain why chain scission predominates in some polymers, while crosslinking in others.^{2–4} It has been reported that poly(vinyl butyral) (PVB) is designated as crosslinking type of polymer, which enables the utilization of such polymer in applications that necessitates the stability for long periods after irradiation.⁵ Pacansky and Waltman studied the effect of the exposure of the PVB to a 175-kV electron beam accelerator under nitrogen atmosphere by infrared spectroscopy.⁶ The study revealed the formation and growth of carbonyl band attributed to the decomposition of the PVB and cleavage of the cyclic

acetal structure. The gel permeation chromatography analysis showed that the $G(S)$ for chain scission and that $G(X)$ for the crosslinking of PVB equals 0.6 scission/100 eV and 0.35 crosslinks/100 eV, respectively. In addition, engineering stress–strain measurements of the PVB films indicate a decrease in tensile strength and ductility as a function of irradiation dose. Irradiation of PVB in air induces the oxidation of polymer and the production of carbonyl groups. It was reported that oxidation processes predominate when PVB thin film were irradiated with absorbed electrons. Other workers reported that when PVB films were irradiated with accelerated electrons, oxidation process predominate.^{7,8}

Solutions of tetrazolium salt are known to form colored formazane via chemical and radiolytical reduction, which can be utilized for radiation dosimetry.⁹ In this regard, films were prepared from solution of PVB containing triphenyl tetrazolium chloride dye (TTC) and exposed to UV radiation. These films showed a response to the main UV spectral region range 400–180 nm; however, the maximum sensitivity was in the region 280–180 nm. Also, PVB/TTC films

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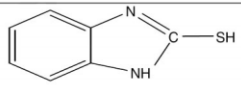
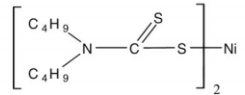
were utilized as dosimeter for high dose γ -radiation range up to 350 kGy.¹⁰

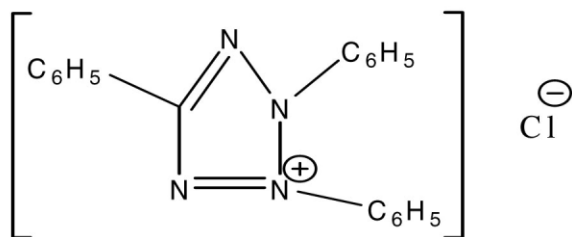
The present work was undertaken to investigate the effect of electron beam irradiation on color sensitivity, thermal, and mechanical properties of PVB films containing TTC dye and the antioxidants nickel dibutyl dithiocarbamate (NBC) or 2-mercaptobenzimidazole (MBI).

EXPERIMENTAL

Materials

The homopolymer PVB, a laboratory-grade, was purchased from Biolofrom BM18, Wacker, USA, and had an average molecular weight of 36,000. A laboratory grade *n*-butyl alcohol was used as a solvent for PVB and was supplied by Aldrich Chemical, USA. 2, 3, 5-triphenyl tetrazolium chloride dye (chemical structure shown below) used throughout this study was supplied by Aldrich, USA, and was used without further purification. Two types of antioxidants were used throughout this work. The chemical structure, commercial name, and producer of the antioxidants are shown below:

Commercial name	Chemical structure	Company
Nonflex (MBI)	 2-Mercaptobenzimidazole	Seiko Chem. Co. Ltd Japan
Antigen (NBC)	 Nickel di butyl di thiocarbamate	Sumitomo Chem. Co., Ltd. Japan



2, 3, 5-triphenyl tetrazolium chloride

Preparation of PVB composites

Films of neat PVB were prepared by the solution casting technique, in which 1 g of PVB was dissolved in 25 mL of *n*-butyl alcohol with continuous stirring

for 3 h at 80°C and the stirring was continued for 24 h at room temperature. The PVB solution was then poured onto a polyester sheet and left to dry in dark at room temperature. The obtained films were stored in dark at ambient temperature. The BVB composites were prepared by dissolving different concentrations of TTC dye or antioxidants (NBC and MBI) in the PVB solutions with continuous stirring at room temperature for about 3 h and cast into films as described earlier.

Electron beam irradiation

Electron beam irradiation was carried out in the 1.5 MeV, 25 kW electron accelerator facility of the National Center for Radiation Research and Technology (NCRRT) in which the required doses were obtained by adjusting the speed of the conveyer system and electron beam parameters.

Color measurements

A microcolor unit attached to a data station manufactured by Brano Lange (Germany) was used for color strength measurements. The L^* , a^* , b^* intercepts used in this system are based on the CIE color triangle (Commission International De E'claire units x , y , and z). In this system, the L^* value specifies the dark-white axis, a^* the green-red axis, and b^* the blue-yellow axis. The L^* , a^* , b^* intercepts of neat PVB films were measured and taken as a reference. The color difference (ΔE^*) of the PVB composites either before or after electron beam irradiation was calculated according to the following equation, in which the recorded value of the different color interceptions is the average of five measurements in different positions along the sample.

$$\Delta E^* = \{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2\}^{1/2}$$

Thermogravimetric analysis

The thermogravimetric analysis (TGA) studies were carried out on a Shimadzu-30 (TGA-30) instrument (kayto, Japan) at a heating rate (10°C/min) under flowing nitrogen atmosphere (20 mL/min.) from room temperature to 500°C.

Tensile mechanical properties

An Instron universal machine (Model 1195, High Wycombe, UK) was used utilizing a stretching speed of 10 mm/min. The films were cut in dumbbell shape and tested for tensile strength and elongation at break at room temperature, according to ASTM D 638 specifications.

TABLE I
Color Intercepts and Intensity of Films of Pure PVB and PVB Composites with Different Contents of the TTC Dye Before and After Electron Beam Irradiation to Various Doses

PVB composites (wt %)	Irradiation dose (kGy)	Color intercepts			Color intensity (ΔE^*)
		L^*	a^*	b^*	
Neat PVB	0.0	93.8	-0.1	-11.8	-
	0.0	93.0	-1.2	-7.2	4.8
	50	70.8	37.1	7.2	47.7
PVB/TTC (1.5)	100	66.7	43.2	14.6	57.5
	150	67.7	40.9	13.6	54.9
	200	60.9	35.6	13.2	54.6
	0.0	91.1	-2.7	-3.5	9.1
	50	44.7	62.0	44.7	95.8
PVB/TTC (3.5)	100	41.7	60.9	53.0	101.6
	150	37.7	59.0	54.7	105.2
	200	37.6	56.2	57.9	105.8
	0.0	93.7	1.0	-3.5	8.4
	50	68.1	44.3	16.2	55.7
PVB/TTC (4.5)	100	64.4	50.2	19.7	63.5
	150	61.7	52.5	22.0	67.6
	200	61.5	53.0	22.6	68.3

RESULTS AND DISCUSSION

Color response to electron beam irradiation

The color intercepts (L^* , a^* , and b^*) and intensity (ΔE^*) of films of PVB composites with different contents of the TTC dye before and after exposure to various doses of electron beam irradiation are shown in Table I. It can be seen that the unirradiated PVB/TTC films possessed higher proportions of whiteness (L^*), indicating that the TTC dye is nearly colorless before

electron beam irradiation. The PVB composites with 3.5 wt % of the TTC dye displayed lower whiteness compared to the PVB composite films with 1.5 and 4.5 wt %. However, the color intensity (ΔE^*) of unirradiated PVB/TTC films was found to increase with increasing the content of the TTC dye.

After exposure to electron beam irradiation, all the color parameters were totally changed. The green ($-a^*$) and blue ($-b^*$) color components of the unirradiated films were changed to the red ($+a^*$) and yellow ($+b^*$) color components. Also, the color intensity (ΔE^*) was greatly increased and accompanied with a significant drop in the whiteness component (L^*). At any of the TTC dye content, ΔE^* was found to substantially increase with increasing irradiation dose. However, the increase of ΔE^* in the case of PVB/TTC (3.5 wt %) is pronounced than in the case of the PVB composites with 1.5 or 4.5 wt % of the TTC dye. Also, the yellow color component ($+b^*$) was found to increase with electron beam irradiation dose. The color intensity ΔE^* was suddenly increased at a dose of 50 kGy and then tends to slightly increase with increasing irradiation dose up to 200 kGy.

Table II shows the different color parameters for PVB composites films with a constant content of the TTC dye (3.5 wt %) and various contents of the NBC antioxidant before and after electron beam irradiation to various doses. It should be mentioned that the L^* , a^* , and b^* values of the unirradiated PVB/TTC composite were taken as a reference in calculating the color intensity (ΔE^*) for the PVB/TTC/NBC or PVB/TTC/MBI composites. Before irradiation, it can be seen that the increase of the contents of NBC caused a

TABLE II
Color Intercepts and Intensity of PVB Composites with a Constant Ratio of the TTC Dye of 3.5 wt % and Various Ratios of the Antioxidant NBC Before and After Electron Beam Irradiation to Various Doses

PVB composites (wt %)	Irradiation dose (kGy)	Color intercepts			Color intensity (ΔE^*)
		L^*	a^*	b^*	
PVB/TTC	0.0	91.1	-2.7	-3.5	-
	Unirradiated	91.8	2.1	-3.7	0.94
	50	50.8	60.9	28.9	82.0
	100	44.4	58.9	32.1	85.1
	150	41.7	56.7	28.9	83.8
PVB/TTC (3.5)/NBC (0.5)	200	40.0	54.5	26.3	82.3
	0.0	88.0	-1.3	7.3	5.1
	50	41.8	57.2	41.3	89.6
	100	39.9	58.5	42.3	92.0
	150	40.7	59.9	52.6	100.2
PVB/TTC (3.5)/NBC (1)	200	28.4	44.7	42.5	91.1
	0.0	86.9	-0.2	-12.1	9.9
	50	47.0	58.5	32.2	83.5
	100	43.2	56.3	30.2	82.7
	150	40.6	53.7	26.0	80.5
PVB/TTC (3.5)/NBC (2)	200	38.8	51.1	24.0	79.9

TABLE III
Color Intercepts and Intensity of PVB Composites with a Constant Ratio of the TTC Dye of 3.5 wt % and Various Ratios of the Antioxidant MBI Before and After Electron Beam Irradiation to Various Doses

PVB composites (wt %)	Irradiation dose (kGy)	Color intercepts			Color intensity (ΔE^*)
		L^*	a^*	b^*	
PVB/TTC	0.0	91.1	-2.7	-3.5	-
	0.0	88.4	-2.6	-2.2	3.0
	50	51.1	54.4	23.0	74.6
	100	45.3	54.7	28.8	80.2
	150	44.0	58.3	31.3	84.6
PVB/TTC(3.5)/MBI (0.5)	200	42.0	52.4	27.4	80.0
	0.0	85.4	-2.0	-5.7	6.1
	50	42.2	61.2	42.3	92.6
	100	38.3	61.1	54.2	100.9
	150	36.8	60.2	44.2	98.1
PVB/TTC (3.5)/MBI (1)	200	32.9	57.7	40.0	94.5
	0.0	83.7	-1.7	-6.3	8.0
	50	45.3	60.3	26.8	85.6
	100	43.9	58.2	29.9	84.0
	150	42.5	56.7	30.5	83.9
PVB/TTC (3.5)/MBI (2)	200	41.1	55.5	26.6	82.4

substantial reduction in the green color component ($-a^*$) and an increase in the blue color component ($-b^*$). Also, the whiteness of the unirradiated PVB/TTC/NBC films (L^*) was found to decrease with increasing the contents of the NBC antioxidant. It seems that the presence of the NBC antioxidant has no effect on the color response to electron beam irradiation, in which similar trends to the PVB/TTC composites can be observed.

Table III shows the different color parameters of PVB composite films with a constant content of the TTC dye (3.5 wt %) and various contents of the MBI antioxidant, before and after electron beam irradiation to various doses. It can be seen that the change in the different color component of PVB/TTC/MBI largely depends on the contents of MBI and electron beam dose as seen in the case of PVB/TTC/NBC composites.

The regular change in the color component (a^*) and color intensity (ΔE^*) in terms of ratios between the values obtained at different electron beam dose ratios was investigated as shown in Figure 1. These calculations were performed for the composites containing 3.5 wt % of TTC and 1wt % of NBC or MBI antioxidants. It can be seen that the PVB/TTC and PVB/TTC/MBI composites showed high regularity, in which the ratios a^* (50 kGy)/ a^* (100 kGy) and a^* (100 kGy)/ a^* (150 kGy), etc., increased gradually with irradiation dose. Similar trend can be observed regarding the ratio of (ΔE^*). On the other hand, the PVB/TTC/NBC composites showed irregular trends in both a^* and ΔE^* with respect to dose ratios. Similar regularity in color change with increasing irradiation dose was seen in the case of 2 wt % of the MBI

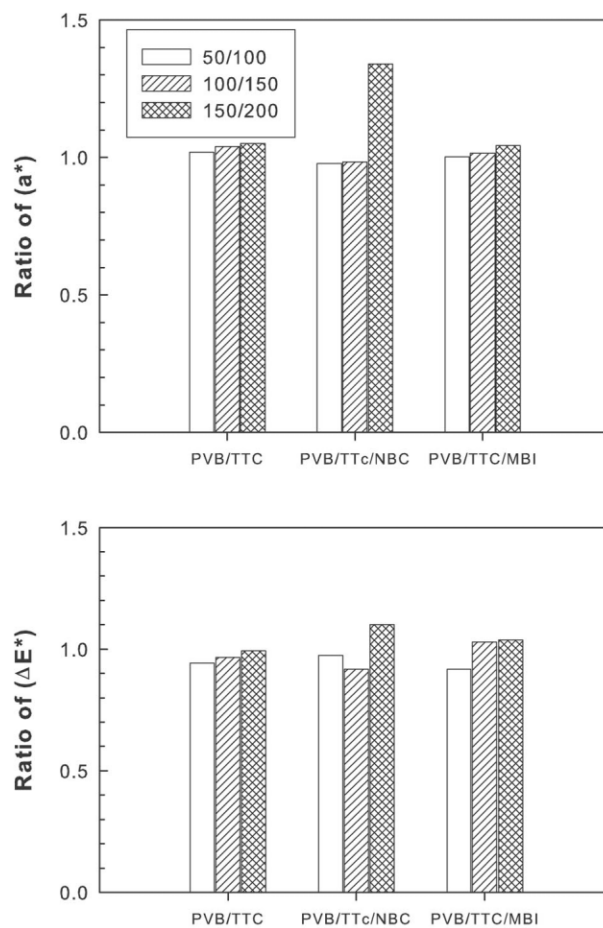


Figure 1 Ratios of the red color component (a^*) and color intensity (ΔE^*) corresponding to different irradiation doses ratios for different PVB composites.

antioxidant. This property may be used in producing suitable films for radiation dosimetry measurements. The asymmetric (discrepancy) in color change with irradiation dose was observed in the case of using 0.5 wt % of the MBI antioxidant.

On the basis of the data on Tables I–III, few conclusions may be made. First, the unirradiated PVB/TTC films are nearly colorless; however, they showed significant color sensitivity towards electron beam irradiation even with lower doses. The use of higher contents of the TTC dye above 3.5 wt % does not make any difference of the color intensity ΔE^* , whether before or after electron beam irradiation. The sensitivity in color change towards electron beam irradiation appeared clearly in the change in green and blue color components for the unirradiated PVB/TTC films to red and yellow color components. This change in color intensity is easy to detect by visual observation if these films were used for monitoring electron beam irradiation up to doses of 50 kGy. Second, the protection of PVB bulk polymer against oxidation degradation that may occur due to electron beam irradiation or heat, however, is of great importance. The results of color measurements showed clearly that the presence of antioxidants does not affect the color sensitivity of PVB/TTC composites towards electron beam irradiation. In addition, the use 1 or 2 wt % of the NBC or MBI antioxidants seems to enhance the development of color. Third, regular change in the red color component and the color intensity (ΔE^*) showed that the presence of NBC antioxidant affect the change in color as a function of electron beam dose.

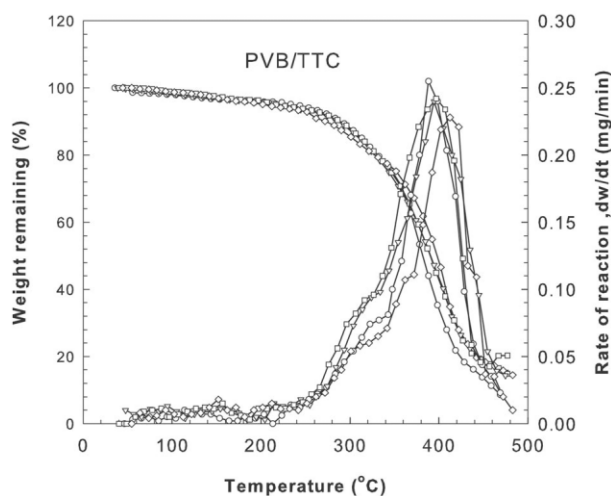


Figure 2 TGA thermograms and the corresponding derivatives of the TGA curves (rate of decomposition reaction (dw/dt)) for pure PVB and PVB composites with a constant ratio of the TTC dye (3.5 wt %): (●) unirradiated PVB, (▼) unirradiated PVB/TTC (3.5 wt %), (■) PVB/TTC (50 kGy), and (◆) PVB/TTC (200 kGy) of electron beam irradiation.

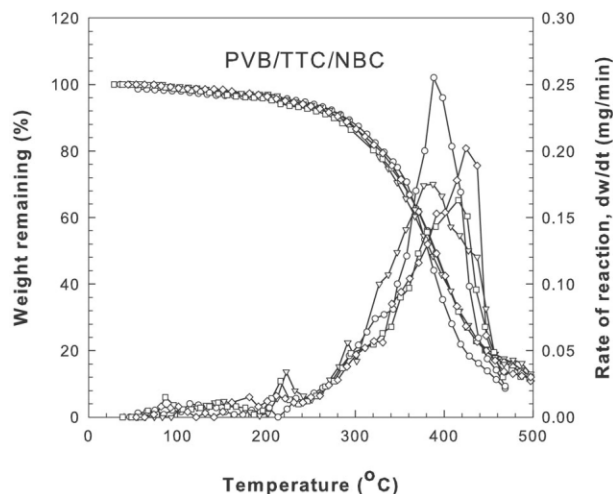


Figure 3 TGA thermograms and the corresponding derivatives of the TGA curves (rate of decomposition reaction (dw/dt)) for pure PVB and PVB composites with constant ratios of the TTC dye (3.5 wt %) and the antioxidant NBC (2 wt %): (●) unirradiated PVB, (▼) unirradiated PVB/TTC/NBC, (■) PVB/TTC/NBC (50 kGy), and (◆) PVB/TTC/NBC (200 kGy) of electron beam irradiation.

Thermal decomposition behavior

The average complete dissociation energy of PVB and TTC dye based on the reported dissociation energies of the different covalent bonds forming the polymer molecules was calculated to be 385.6 and 414.5 kJ/mol.¹¹ This finding may lead to the conclusion that the compounding of the TTC dye with PVB will eventually results in a composite with a relatively higher thermal stability than neat PVB bulk polymer. This theoretical calculation is for unirradiated composites; however, the effect of electron beam irradiation might change this sequence. Thermogravimetric analysis (TGA) was used to investigate experimentally the thermal decomposition behavior of the different PVB composites with the TTC dye and the antioxidant NBC or MBI before and after electron beam irradiation. Figures 2–4 show the TGA thermograms and the corresponding derivatives of the TGA curves (i.e., rate of decomposition reaction, dw/dt) for unirradiated pure PVB and PVB composites with a constant ratio of the TTC dye 3.5 wt %, and NBC or MBI antioxidants (2 wt %), before and after electron beam irradiation to the doses 50 and 200 kGy. As shown in these curves, the rate of decomposition reaction displayed similar trends; however, the temperatures (T_{max}) of the maximum value of the rate of decomposition reaction (dw/dt) differ from one material to another. Table III summarizes the T_{max} for the different PVB composites before and after electron beam irradiation to different doses.

On the basis of Figures 2–4 and Table IV, few conclusions can be made. (1) PVB thermally degraded in

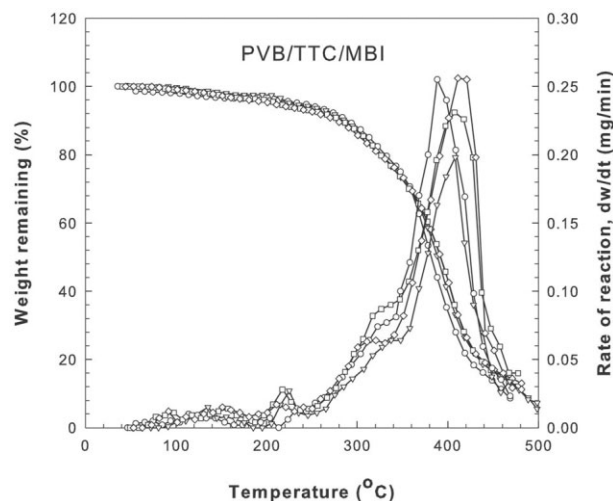


Figure 4 TGA thermograms and the corresponding derivatives of the TGA curves (rate of decomposition reaction (dw/dt) for pure PVB and PVB composites with constant ratios of the TTC dye (3.5 wt %) and the antioxidant MBI (2 wt %): (●) unirradiated PVB, (▼) unirradiated PVB/TTC/MBI, (■) PVB/TTC/MBI (50 kGy), and (◆) PVB/TTC/MBI (200 kGy) of electron beam irradiation.

nitrogen atmosphere with the evolution of mixture of gases.¹² The experimental results give supports to these properties as shown in Table IV. (2) As expected, the PVB/TTC composites before or after irradiation are thermally more stable than pure PVB as shown from the initial TGA thermograms or the obtained T_{\max} values. However, the electron beam irradiated pure PVB film to 50 kGy displayed higher thermal stability than the unirradiated composite or the one irradiated at 200 kGy. This may be explained on the bases that the change in structure and color (as described earlier) of the TTC dye had occurred due to irradiation. (3) The effect of NBC and MBI antioxidants on the thermal decomposition behavior of PVB/TTC particularly at high doses is very clear. The PVB/TTC/NBC or PVB/TTC/MBI composites possessed higher thermal stability than that of PVB/TTC composite based on the T_{\max} and the TGA thermograms. However, PVB/TTC/MBI is thermally more stable than PVB/TTC/NBC. This trends can be attributed to the higher dissociation energy of the C=N bond with

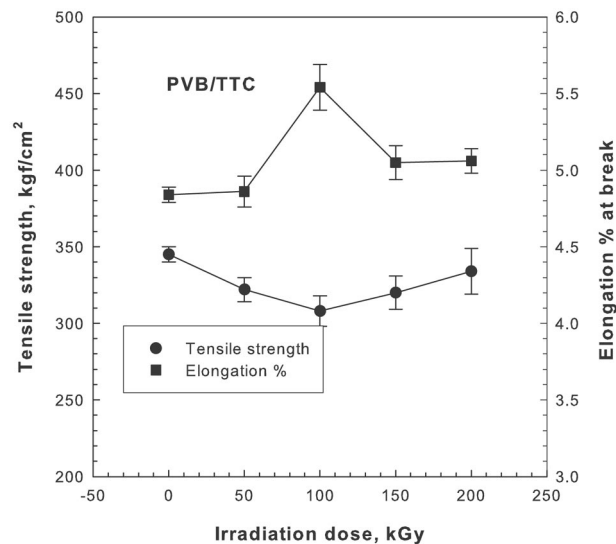


Figure 5 Tensile mechanical properties of PVB composites with a constant ratio of the TTC dye of 3.5 wt % before and after electron beam irradiation to various doses.

respect to the C=S forming the molecules of MBI and NBC, respectively, and hence the protection against thermal degradation. However, the presence of antioxidants NBC or MBI seems to compensate the decomposition of the TTC dye and the oxidative degradation that may occur during electron beam irradiation and heating.

Tensile mechanical properties

The initial stress-strain curves of neat PVA or its composites with the TTC dye and the NBC or MBI antioxidants, before or after electron beam irradiation, showed the behavior of brittle polymers with no yielding properties (not shown). Also, the tensile strength at the break point of unirradiated neat PVB polymer was 378 ± 12 kgf/cm², while the elongation was $2\% \pm 0.23\%$. The electron beam irradiation to doses 50 and 200 kGy results in a reduction in tensile strength to 360 and 248 kgf/cm², respectively, (not shown).

Figure 5 shows the tensile mechanical properties of PVB/TTC composites with a constant content of the TTC dye of 3.5 wt % before and after electron beam

TABLE IV
Temperatures (T_{\max} °C) of the Maximum Value of the Rate of Decomposition Reaction for Different PVB Composites

Irradiation dose (kGy)	T_{\max} (°C)			
	Pure PVB	PVB/TTC	PVB/TTC/NBC	PVB/TTC/MBI
Unirradiated	394	395	382	409
50	398	400	412	424
200	380	420	430	410–424

irradiation to various doses. The tensile strength of PVB/TTC composites before irradiation is much lower than that of the unirradiated neat PVB. However, the elongation at break of PVB/TTC composite is almost twice that of neat PVB. The tensile strength of PVB/TTC composites was found to decrease by increasing irradiation dose from 50 to 100 kGy and then tends to increase with increasing irradiation dose up to 200 kGy. The elongation at break showed an opposite behavior. The TTC dye acts as a lubricant, which facilitates the slipping of PVB chains. The subsequent decrease in tensile strength of electron beam irradiated PVB/TTC composites is due to the occurrence of oxidative degradation of both PVB and TTC dye, while the increase at higher doses may be due to occurrence of crosslinking.

Figure 6 shows the different tensile mechanical properties of PVB composites with constant ratios of the TTC dye (3.5 wt %) and the antioxidant NBC (2 wt %) before and after electron beam irradiation to various doses. It can be seen that the combined effect of the TTC dye and the antioxidant NBC resulted in a gradual increase in the tensile strength and elongation with increasing irradiation dose up to 150 kGy and tends to decrease at higher dose. The introduction of the NBC molecules seems to enhance the compact structure of the composites against electron beam irradiation. However, the tensile strength of unirradiated PVB/TTC/NBC composites is lower than unirradiated PVB/TTC one and all is still lower than unirradiated neat PVB. Meanwhile, the electron beam irradiated composites are still higher than that of irradiated neat PVB at the same irradiation doses.

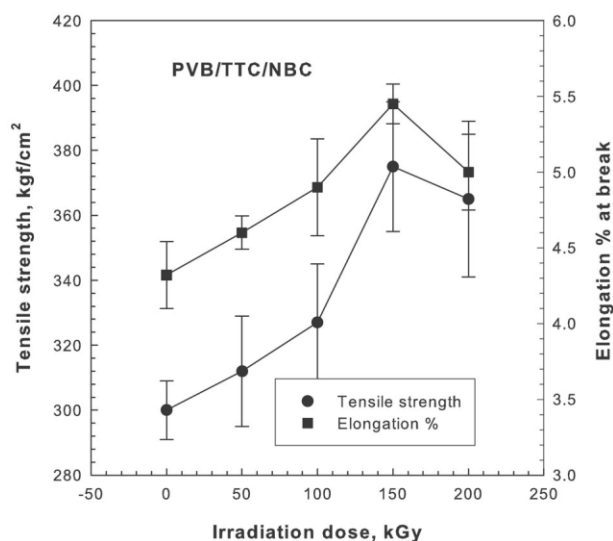


Figure 6 Tensile mechanical properties of PVB composites with constant ratios of the TTC dye (3.5 wt %) and the antioxidant NBC (2 wt %) before and after electron beam irradiation to various doses.

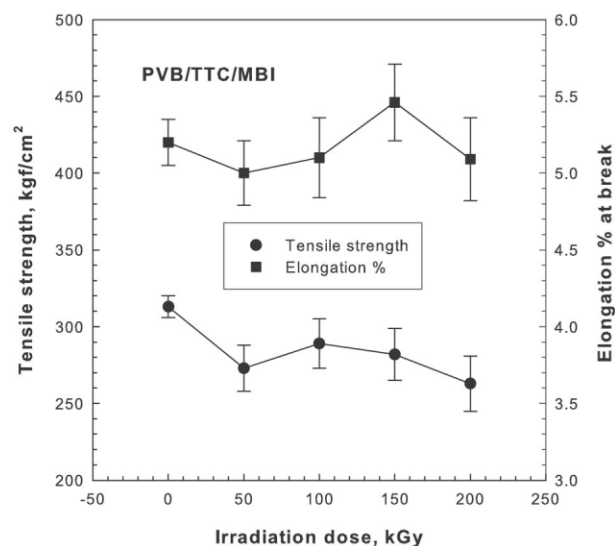


Figure 7 Tensile mechanical properties of PVB composites with constant ratios of the TTC dye (3.5 wt %) and the antioxidant MBI (2 wt %) before and after electron beam irradiation to various doses.

Figure 7 shows the effect of irradiation dose on the tensile mechanical properties of PVB composites with constant ratios of the MBI antioxidant of 2 wt % and the TTC dye of 3.5 wt %. The tensile strength is suddenly decreased at 50 kGy and then tends to seem unaffected with the dosage range 100–150 kGy. By increasing irradiation dose up to 200 kGy, the tensile strength tends to decrease. This behavior may indicate that the MBI antioxidant offered protection against electron beam irradiation due to the presence of the benzene ring, which dissipates the radiation energy through the delocalized electron cloud.

On the basis of the results of mechanical properties, few conclusions may be withdrawn. (1) The tensile strength of all the composites with the TTC dye or with the antioxidants, before or after electron beam irradiation, is lower than unirradiated neat PVB polymer. This is rather expected because the dissolution and casting of the additives results in a disruption of the PVB chains. (2) The tensile mechanical properties of the different PVB composites depend to a great extent on the type of additive. In this regard, the reduction in the tensile strength of unirradiated neat PVB caused by blending with TTC dye, TTC + NBC, and TTC + MBI was calculated to be 4, 20, and 17%, respectively. However, the electron beam irradiation of all these composites to a dose of 200 kGy results in an improvement in tensile strength of (6% with respect to the tensile of neat PVB irradiated to the same dose. (3) Exposure of all the PVB composites to a dose of 50 kGy, at which maximum color was developed and as recommended for practical applications, the reduction or increment in tensile strength can put as

follow : PVB/TTC (-7%), PVB/TTC/NBC (+4%), and PVB/TTC/MBI (-8%) with respect to the initial tensile strength of each composite.

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