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Modification of Structure and Properties of PBAT via Gamma and Electron Beam Irradiation

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Abstract

Poly(butylene adipate-co-terephthalate) (PBAT) samples were irradiated at the absorbed doses up to 50 kGy by gamma rays with a dose rate of 0.92 kGy h^{-1} and an electron beam (EB) accelerator with a dose rate of 3.5 kGy s^{-1} to modify properties for further applications. The results revealed that radiation modalities significantly affected PBAT. The peroxide value (PV) of the irradiated PBAT increased sharply with the absorbed dose of gamma irradiation, but this value only increased slightly by EB irradiation. The viscosity average molecular weight of PBAT significantly decreased by gamma ray irradiation, indicating that the chain scission predominantly occurred. In contrast, EB irradiation does not seem to have affected the Mv of the irradiated PBAT, suggesting the occurrence of competing radiation-induced reactions, including chain branching and crosslinking, which may limit chain scission. Furthermore, oxidative reactions also contributed to the structural changes in PBAT during irradiation, as indicated in FT-IR spectra. The comparative results suggested that gamma irradiation led to a gradual increase in the melt flow index (MFI) and a decrease in the tensile strength and elongation at break of irradiated PBAT with increasing absorbed doses. In contrast, the MFI of EB-irradiated PBAT leveled off with the absorbed dose. The tensile strength of the EB-irradiated PBAT was slightly reduced with the absorbed dose, but its elongation at break significantly decreased at doses below 20 kGy and then recovered with doses up to 50 kGy. These findings demonstrate that gamma and EB irradiation can be applied as effective tools to modify PBAT.

Keywords: PBAT; irradiation; peroxide value; viscosity; melt flow index

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1. Introduction

Poly(butylene adipate-co-terephthalate) (PBAT) is one of the most promising and popular co-polyesters, which can be produced by a polycondensation reaction of terephthalic acid (TPA), butanediol (BBO), and adipic acid (AA). The presence of aromatic butylene terephthalate units provides PBAT with good mechanical strength and thermal stability, whereas biodegradable aliphatic segments are responsible for its flexibility and biodegradability [1–3]. The crystallization of the aliphatic segments also contributes to

the semi-crystalline nature of PBAT. When metabolized in soil or compost under standard conditions, PBAT can completely biodegrade to carbon dioxide, water and biomass [1,4,5]. Therefore, PBAT exhibits excellent biodegradability while maintaining beneficial mechanical properties, making it a promising biodegradable material to replace traditional low-density polyethylene (LDPE) for a wide range of applications.

Because the properties of PBAT depend on its molecular weight, process and storage conditions, commercial PBAT usually requires structural modifications to improve its compatibility and tailor its melt viscosity, particularly when blended with other hydrophilic polymers [6–8]. Two common modification approaches are currently employed for PBAT. The first is chemical modification using initiators containing azo or diazo compounds, peroxides, or hydroperoxides [9–11]. The second is a physical process using ionizing radiation, with gamma irradiation being one of the most widely used techniques. During irradiation, PBAT undergoes chain scission, producing free radicals that can subsequently react with oxygen to form peroxide (hydroperoxide) species, like other polymers [12–15].

PBAT contains both aliphatic and aromatic units, which are differently affected by ionizing radiation, and various irradiation modalities have been explored to modify the properties of this biodegradable polymer [16,17]. Generally, gamma radiation has been reported to predominantly induce chain scission in PBAT [18], but gel fractions were also found in the polymer irradiated at doses over 150 kGy, indicating the formation of crosslinking structures under high-dose irradiation [19]. In contrast, electron beam (EB) irradiation has been reported to promote crosslinking in PBAT even at relatively low doses, with the crosslinking density increasing with the absorbed dose [20,21]. These findings suggest that the balance between chain scission and crosslinking in PBAT depends on the irradiation modality and conditions. However, the differences in the structural changes and their implications for the molecular properties of PBAT have not been fully elucidated. In addition, a comprehensive comparison of gamma and EB irradiation, considering oxidative reactions and the resulting changes in melt flow behavior and mechanical performance, is required.

In the present study, the effects of gamma and EB irradiation on the structure and properties of PBAT were systematically investigated and compared. Changes in PBAT's structure were investigated via the peroxide value (PV), FT-IR spectra, and the relationship between radiation-induced structural modifications, and the resulting material was elucidated using its molecular weight, melt flow index (MFI), and mechanical properties. The findings provide further insight into the different radiation-induced changes under gamma and EB irradiation and may contribute to the modification of PBAT properties for specific applications.

2. Materials and Methods

2.1. Materials

The PBAT used in the experiments was TH801 Tunhe grade, purchased from Xinjiang Blue Ridge Tunhe Polyester Co., Ltd., Changji, Xinjiang, China. Sodium thiosulfate ($Na_2S_2O_3$), potassium iodide (KI), and 99.8% acetic acid (glacial) were purchased from Merck, Vietnam. Chloroform and other solvents were purchased from Duc Giang Chemical Company, Hanoi, Vietnam.

2.2. Radiation Degradation of PBAT

Neat PBAT pellets were dried, vacuum sealed in radiation-resistant polyethylene bags, and then irradiated at various doses from 0 to 50 kGy by a gamma ray with a dose rate of 0.92 kGy h^{-1} at Hanoi Irradiation Center and by an EB accelerator with a dose rate of

3.5 kGy s^{-1} at Vinagama Center, Ho Chi Minh city, Vietnam, to determine the influences of different radiation modalities on PBAT properties and structures.

2.3. Determination of Viscosity Average Molecular Weight of PBAT

Stock solution of 1 g dL^{-1} PBAT was prepared and diluted with chloroform as the pure solvent to obtain solutions with concentrations of 0.8, 0.6, 0.4 and 0.2 g dL^{-1}. The Ubbelodhe viscometer (Canon 1B, Tokyo, Japan) was kept at room temperature (25 ± 0.1 °C) using a circulating water bath. The flow times of the chloroform (t_0) and polymer solution (t) through the capillary were measured to calculate relative viscosity (η_r) and specific viscosity (η_{sp}) of PBAT solutions. Intrinsic viscosity [η] was determined by extrapolating the reduced viscosity ($\eta_{red} = \eta_{sp}/C$) to zero concentration (C = 0) [16,22]. The viscosity average molecular weight (Mυ) of PBAT was determined using the Mark–Houwink equation as follows:

$$M\upsilon = ([\eta]/K)^{1/\alpha} \quad (1)$$

where K and α are the empirical parameters. For the PBAT, the chloroform system was set at 25 °C, and the K and α values were 8.5×10^{-3} mL g^{-1} and 0.76, respectively [3].

2.4. Determination of Peroxide Value of the Irradiated PBAT

The peroxide (hydroperoxide) value is a typical and key indicator used to evaluate the radiation-induced oxidation and degradation in polymers. In the presence of oxygen, ionizing radiation (gamma, EB/X-ray) breaks polymer chains and leads to the formation of peroxide and hydroperoxide groups. These oxidative products may decompose at elevated temperatures, producing free radicals that initiate subsequent chemical reactions, such as radiation grafting. In this study, peroxide values (PVs) of PBAT before and after irradiation were determined using the titration method [23].

Briefly, 5 g of PBAT sample was weighed to an accuracy of ±0.1 mg using analytical balance (Presica, Dietikon, Switzerland) and placed into a 250 mL conical flask. Then 29.5 mL of admixture solvent composed of acetic acid and chloroform (3:2 ratio) was added to dissolve PBAT then purged with nitrogen to remove oxygen. After that, 0.5 mL of saturated potassium iodide (KI) was added; the mixture was stirred thoroughly for 10 min and kept in the dark for a further 3 min, allowing the peroxides to oxidize the iodide (white) to iodine (yellow). It was gently shaken for 1 min, and then we added 30 mL of water to terminate the reaction. We titrated with sodium thiosulfate ($Na_2S_2O_3$ 0.01 mol L^{-1}) solution while continuously vigorously shaking. We added the titrant slowly until the there was almost no yellow color and added 5 mL of 0.1% starch solution and continued titration under vigorous shaking until the blue color was completely discharged. We record the volume of $Na_2S_2O_3$ solution consumed as V_s. A blank titration was performed using the same procedure without PBAT to determine V_b, the volume of $Na_2S_2O_3$ used for the blank titration. The peroxide value (PV), expressed in milliequivalents of active oxygen per kg of PBAT, was calculated using following formula:

$$PV\left(meq.kg^{-1}\right) = \frac{1000 \times C(V_s - V_b)}{2 \times m} \quad (2)$$

where PV is peroxide value (meq kg^{-1}); C (mol L^{-1}) is concentration of $Na_2S_2O_3$ solution (0.01 mol L^{-1}); V_s and V_b are the volumes of $Na_2S_2O_3$ solution consumed to titrate the PBAT and blank samples, respectively; and m is sample weighting.

2.5. FT-IR Spectroscopy

Effects of gamma and EB irradiation on the structure of PBAT were analyzed by using an FTIR spectrophotometer (Thermo Scientific, Nicolet Summit X, Cheshire, UK). FT-IR

spectra were recorded in the wavenumber range of 400–4000 cm^{-1} with a resolution of 4 cm^{-1}.

2.6. Melt Flow Index Analysis

MFI measures the flowability of PBAT at high temperatures, where MFI represents the weight of the polymer (in grams) that flows through a standard die in 10 min under specific temperature and load conditions. In this experiment, the MFI (g/10 min) of dried PBAT was determined according to ISO 1133-1:2011 [24] using an Extrusion Plastometer (Olsen, Cleveland, OH, USA) at 160 °C and with a 2160 g load.

2.7. Mechanical Properties

Thickness of PBAT film was measured using a micrometer with accuracy of 0.01 mm. Tensile strength and elongation at break of PBAT films were determined using a universal testing machine (AGX-50kNVD, Shimadzu, Japan). PBAT films with an approximate thickness of 1.2 mm were prepared in dumbbell-shaped molds with a 25 mm gauge length; then they were tested with a crosshead speed of 50 mm min^{-1} and 50 N load at room temperature according to ASTM D638 [25]. The obtained results were the average of three repeated measurements.

3. Results and Discussion

3.1. The Peroxide Value of the Irradiated PBAT

The effects of the gamma and EB irradiation on the peroxide value of the irradiated PBAT have been determined and are presented in Figure 1. A very small amount of peroxide (PV about 0.02–0.05 meq kg^{-1}) was detected in neat PBAT (non-irradiated), which may have formed during processing and storage. The PV of PBAT was significantly increased by both gamma and EB irradiation, providing that the ionizing radiation broke polymer chains and produced free radicals, which intermediately reacted with oxygen to form peroxyl radicals (ROO•) and subsequent peroxides (hydroxyperoxides) [12,17]. Because the density of free radicals increased with the radiation intensity, the number of peroxyl radicals and the PV also increased proportionally with the absorbed dose, as observed in Figure 1.

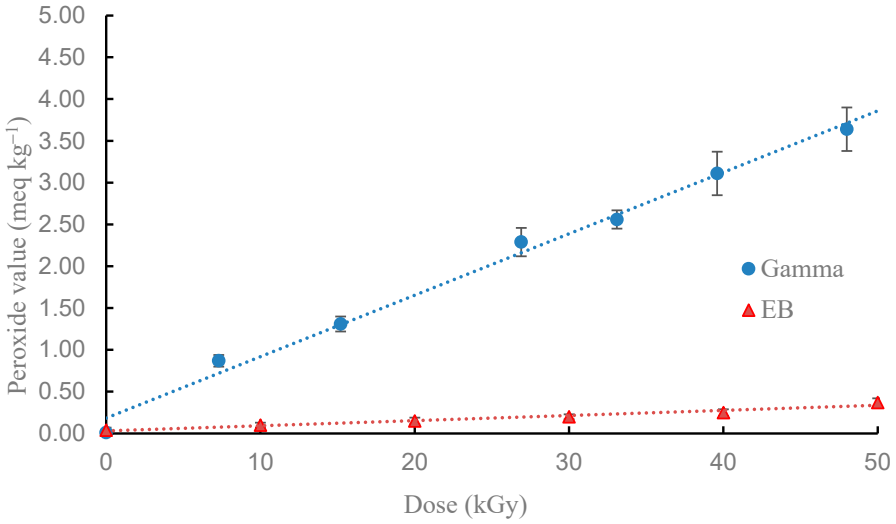


Figure 1. Peroxide values of the irradiated PBAT as the functions of absorbed dose.

At a similar radiation dose, the PV of gamma-irradiated PBAT was much higher than that of the EB-irradiated sample. For gamma irradiation, the PV of the irradiated PBAT increased from 1.1 ± 0.05 meq kg^{-1} at 10 kGy to 3.78 ± 0.25 meq kg^{-1} at 50 kGy, whereas

these values were 0.1 ± 0.02 and 0.37 ± 0.05 meq kg^{-1} for the PBAT irradiated by EB at 10 and 50 kGy, respectively. This may be explained by inherent differences between gamma and EB irradiation. Gamma irradiation requires a longer processing period, allowing oxygen to continuously diffuse into the polymer and react with free radicals, thereby increasing the peroxide value (PV). In contrast, EB irradiation produced large amounts of free radicals within a very short period. These radicals rapidly react with the oxygen present in the polymer matrix, leading to an early saturation of the peroxide formation. Moreover, EB irradiation generated a high concentration of free radicals in the polymer, which readily recombine, thereby limiting the peroxide formation.

3.2. Effects of Gamma and EB Irradiation on Mν of PBAT

Changes in the Mν of PBAT after irradiation were evaluated as a function of the absorbed dose, and the results are shown in Figure 2. The Mν of the neat PBAT was decreased by both gamma and EB irradiation, indicating the degradation of PBAT chains by high-energy ionizing radiation. Gamma irradiation caused a pronounced reduction in the Mν, whereas the decrease caused by EB irradiation was relatively small. This suggests that radiation-induced chain scission was dominant under gamma irradiation, while branching or crosslinking was also promoted in the PBAT matrix during EB irradiation. In general, chain scission and crosslinking can occur simultaneously at random sites in the amorphous regions of semi-crystalline polymers, and PBAT is readily degraded by irradiation [7,21,26,27]. Previous studies have also reported radiation-induced crosslinking in PBAT at gamma doses above 150 kGy [19] and EB doses of 20–200 kGy [20,28]. The high density of free radicals generated during EB irradiation likely accelerated radical recombination, leading to long-chain branching, such as Y- or T-type linkages, and possibly limited crosslinking within the PBAT matrix. These structural changes not only limited the peroxide formation in EB-irradiated PBAT but also increased its viscosity and molecular weight, explaining why the Mv of PBAT changed only slightly upon EB irradiation.

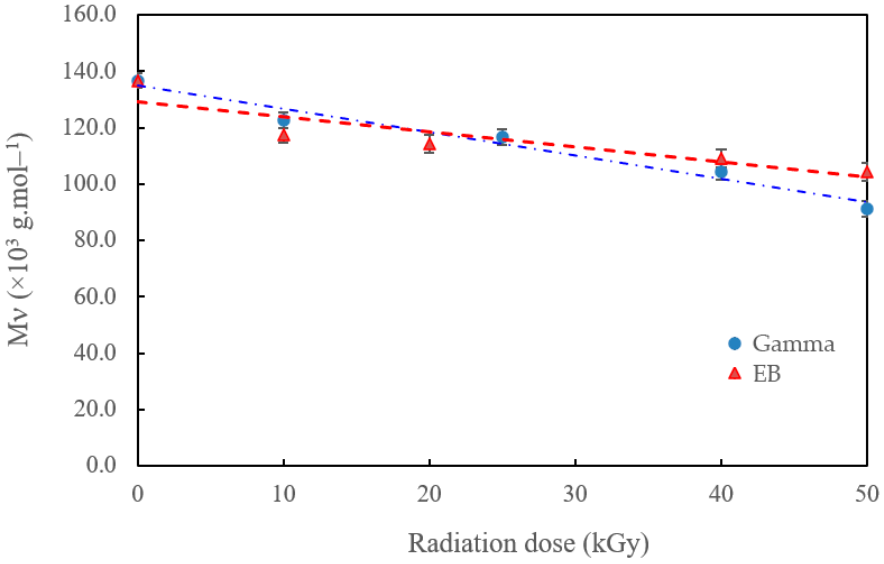


Figure 2. Changes in viscosity average molecular weight (Mν) of PBAT caused by gamma (●) and EB (▲) irradiation with absorbed doses.

3.3. Chemical Structure of PBAT and Irradiated PBAT Samples

The effects of gamma and EB irradiation on the functional groups of PBAT were analyzed using FT-IR. As observed in Figure 3, there are no significant differences in the FT-IR spectra of the neat PBAT and the irradiated PBAT, indicating that the gamma and EB

irradiation at a dose of 40 kGy did not affect the functional groups of PBAT. All spectra of PBAT and irradiated PBAT are typical, with main peaks in the same regions and a shape absorbance peak at 1715 cm^{-1}, caused by a carbonyl (C=O) group of esters [8,26,29]. A small broad peak at 3000–2800 cm^{-1} was attributed to the stretching vibration of the CH_2 and CH_3 [5,8], and the peak at 1150–1270 cm^{-1} was attributed to the asymmetric stretching of the C−O−C bond [5].

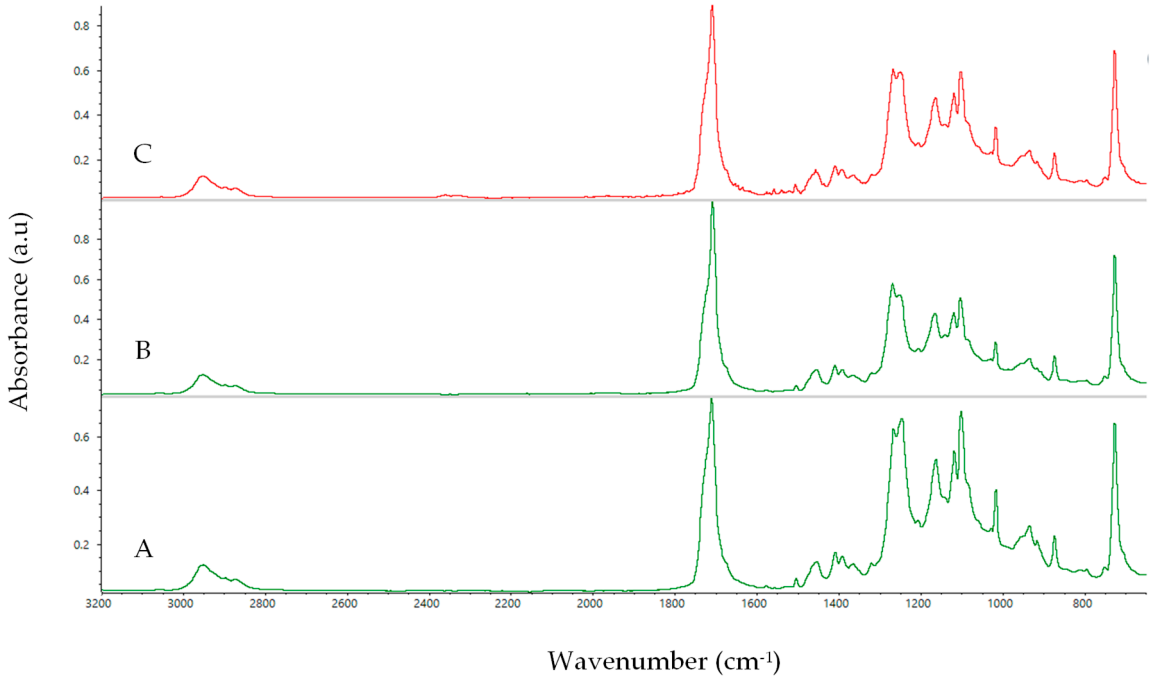


Figure 3. FT-IR spectra of neat PBAT (**A**) and the PBAT irradiated by EB (**B**) and gamma (**C**) at a dose of 40 kGy.

The FT-IR spectra also revealed that the intensity of the absorption peak at 1715 cm^{-1} increased progressively from the neat PBAT to EB-irradiated PBAT, reaching its maximum in the gamma-irradiated samples. Longer exposure periods of gamma irradiation promoted radiation-induced oxidation and produced carbonyl and peroxyl groups, which enhanced the intensity of the C=O peak as observed in the FT-IR spectra. In contrast, the high dose rate of the EB irradiation induced rapid oxygen depletion, thereby limiting the extent of oxidative chain scission. The reduction in the intensity of the C−O−C peak also proved the aliphatic segments were broken down by irradiation.

3.4. The Melt Flow Index of the Irradiated PBAT

Figure 4 shows the melt flow index (MFI) of the non-irradiated and irradiated PBAT samples. Under gamma irradiation, the MFI quickly increased from 4.45 ± 0.22 g 10 min^{-1} for neat PBAT to a maximum value of approximately 16.5 ± 0.62 g 10 min^{-1} for the PBAT irradiated at 50 kGy. The increase in the MFI can be attributed to the radiation degradation of PBAT molecules, leads to a reduction in the molecular weight and then lowers its melt viscosity, thereby enhancing the melt flowability of the irradiated PBAT. Furthermore, the progressive increase in the MFI with the absorbed dose indicates that the chain scission of PBAT is dose-dependent. These results are consistent with the decrease in PBAT's molecular weight, presented in Figure 2. Although chain scission improves melt processability, the resulting reduction in molecular weight may compromise the mechanical properties of PBAT.

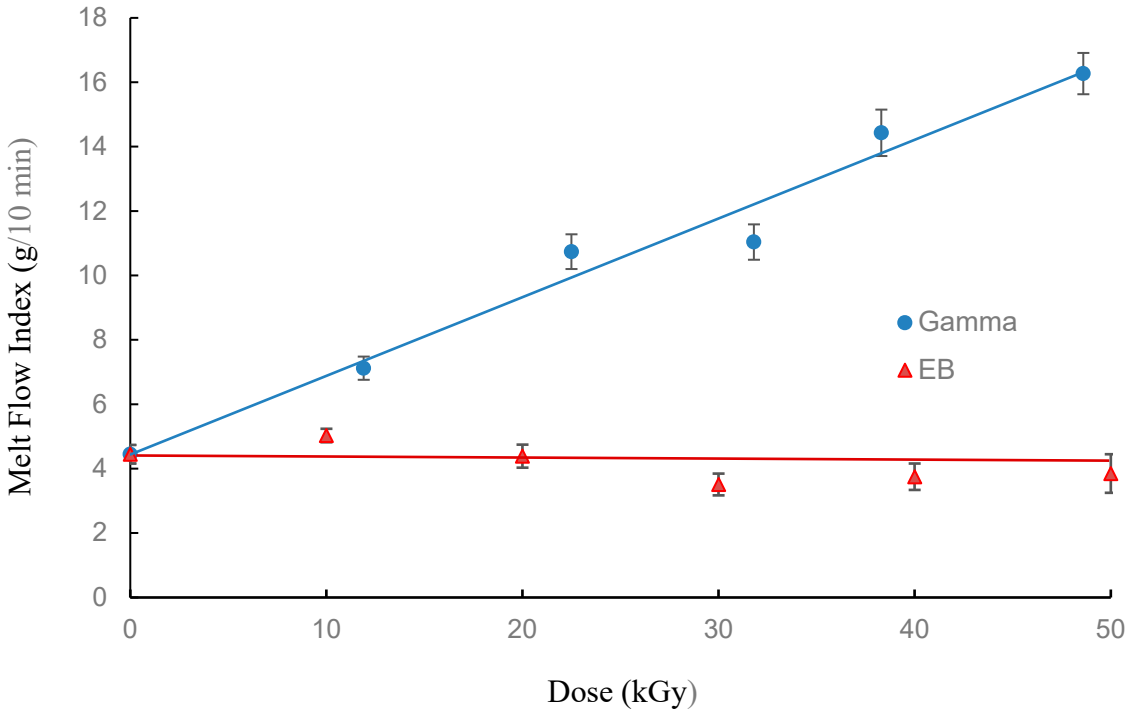


Figure 4. Effects of gamma (●) and EB (▲) irradiation on the MFI of PBAT.

In contrast, under EB irradiation, the MFI of the irradiated PBAT increased slightly to 5.63 ± 0.25 g/10 min at 10 kGy but subsequently decreased with increasing doses, reaching a minimum of 3.85 ± 0.56 g/10 min at 50 kGy, which was even lower than that of the non-irradiated PBAT. This behavior suggests a dose-dependent competition between chain scission and chain branching or crosslinking, resulting in complex and non-linear changes in the molecular structure of the EB-irradiated PBAT. Consequently, the decrease in the Mv, as estimated using the viscometric method, may not accurately reflect the actual changes in molecular weight. At relatively low doses, chain scission may contribute to the increase in the MFI, whereas at higher doses, the formation of branched and crosslinked structures appears to become increasingly dominant, resulting in an increased resistance to melt flow.

These findings are consistent with those reported by Rzepna et al. [21], who identified crosslinking as the predominant process in PBAT during EB irradiation over a dose range of 25–200 kGy. They also recorded an increase in the M*v* and a corresponding decrease in the MFI with increasing doses. However, our results further reveal an initial increase in the MFI at 10 kGy, suggesting that chain scission may contribute to the early-stage response to EB irradiation before branching or crosslinking becomes dominant at higher doses. The formation of branched and crosslinked structures, together with increased chain entanglement, can restrict molecular mobility and increase melt viscosity, thereby reducing the MFI.

Furthermore, Rzepna et al. [21] reported that long-term storage was accompanied by the deterioration of the M*v* and mechanical properties. This oxidation degradation was attributed to radical-mediated oxidation reactions that accelerated over time, particularly in irradiated PBAT.

3.5. The Effect of Gamma and EB Radiation on the Mechanical Properties of PBAT

The results for the mechanical properties of the PBAT and the irradiated PBAT are presented in Figure 5. The initial PBAT exhibited typical characteristics of a semi-crystalline and ductile polyester, with a tensile strength of 27.9 MPa and an elongation at break of 850%. Under gamma irradiation, both tensile strength and elongation at break decreased with

increasing doses, although the reduction in tensile strength at doses below 30 kGy was not statistically significant (Figure 5A). This behavior is consistent with radiation-induced chain scission, which progressively reduces the molecular weight and impairs the mechanical integrity of PBAT.

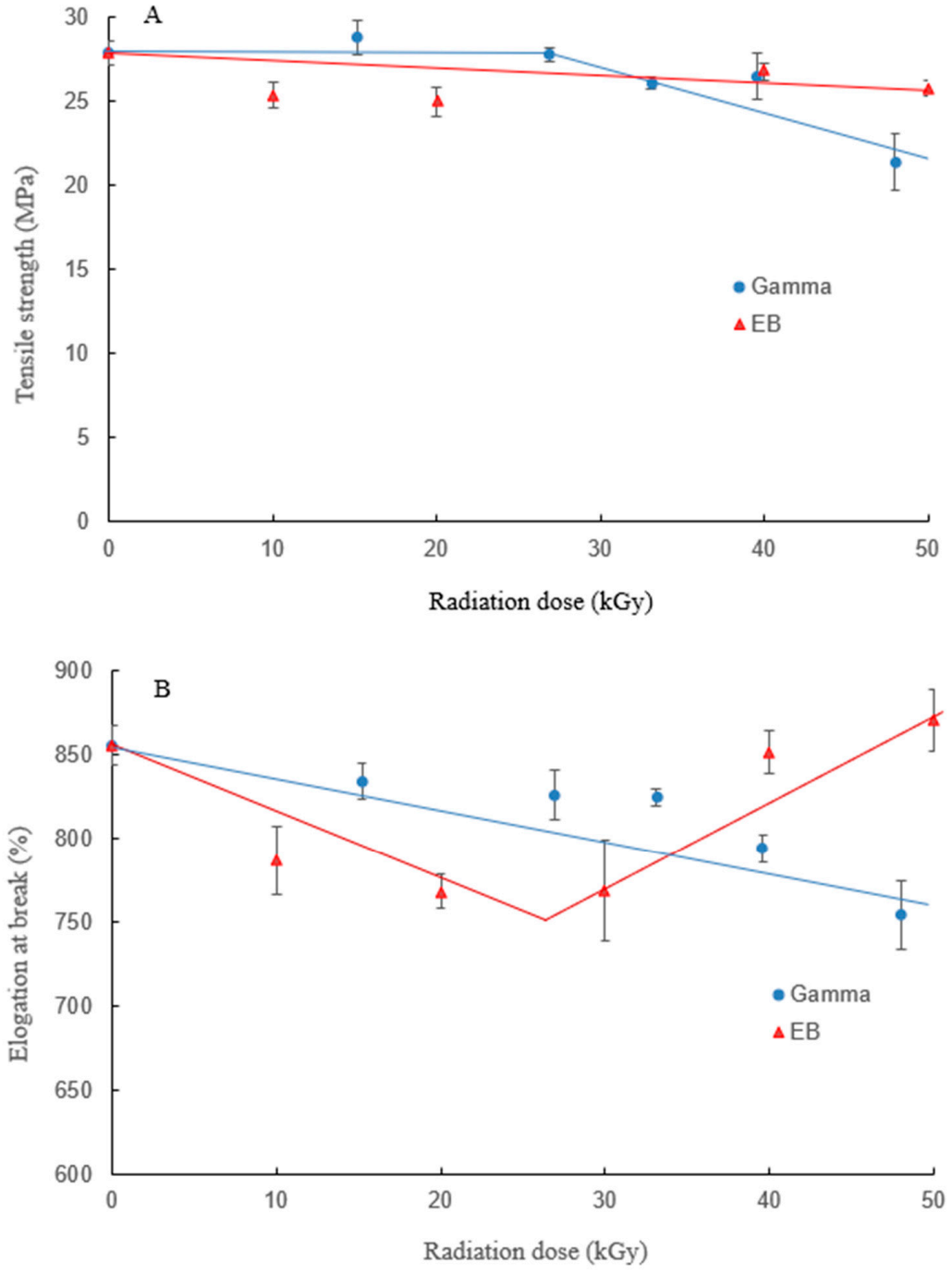


Figure 5. The effects of gamma (●) and EB (▲) irradiation on tensile strength (**A**) and elongation at break (**B**) of the irradiated PBAT specimens.

In contrast, while the tensile strength of the EB-irradiated PBAT declined with increasing doses, the elongation at break exhibited a non-linear trend, decreasing sharply up to 20 kGy before recovering at higher doses up to 50 kGy (Figure 5B). This behavior suggests that the structural changes induced by EB irradiation are more complex than gamma irradiation. The partial recovery of elongation at higher doses may be associated with changes

in molecular architecture, potentially involving chain branching and competing radical reactions, rather than the extensive formation of a crosslinked network.

These results differ from those reported by Hwang et al., who identified crosslinking as the predominant process in PBAT irradiated by EB under vacuum [20]. These differences may be attributed to the variation in specimen preparation and irradiation conditions. In particular, the presence of oxygen during EB irradiation in our study may promote oxidative reactions and limit the recombination of free radicals, thereby influencing the formation of branched or crosslinked structures.

Taken together with the MFI results, the distinct mechanical responses observed in our work suggest that EB irradiation in air induces a different pattern of structural modification in PBAT from that reported for EB irradiation under vacuum. While chain scission appears to dominate the response to gamma irradiation, EB irradiation in air may involve competing chain scission, branching, and crosslinking reactions, with their relative contributions varying with the absorbed dose.

4. Conclusions

PBAT pellets were irradiated with gamma rays and electron beams (EBs) at different absorbed doses, and the effects of two irradiation modalities on the structure and property of PBAT were investigated and compared for further applications. The results demonstrated that ionizing radiation induced significant structural changes in PBAT, with various responses to irradiation modality. Under gamma irradiation, chain scission was the predominant reaction, leading to a continuous reduction in the viscosity average molecular weight. The resulting polymer radicals could react with oxygen, promoting oxidative reactions and peroxide formation. As a result, the peroxide value (PV) of the irradiated PBAT increased significantly with the absorbed dose. The reduction in the molecular weight was accompanied by a continuous increase in the melt flow index (MFI), indicating enhanced melt flowability, and a deterioration of mechanical properties.

In contrast, the EB irradiation resulted in lower PVs, which may be attributed to the recombination of resulting free radicals at high radical concentrations, thereby competing with their reactions with oxygen. The structural changes induced by EB irradiation appeared to involve competing chain scission and radical recombination reactions, potentially leading to chain branching. This interpretation is supported by the variation in the MFI, which initially increased at low doses before decreasing at higher doses, as well as the distinct dose-dependent changes in elongation at break. Overall, the findings indicate that the irradiation modality may play an important role in the modification of PBAT properties.

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