

Recycling polypropylene waste packaging. Technical requirements for radiation processing

Julio Harada^a, Juliana Arquinto^a, Maria da Conceição Costa Pereira^a,
Maria Elizabeth Maués dos Santos^a, Dione Pereira de Castro^{a,*}, Traian Zaharescu^{a,b},
Leonardo Gondim de Andrade e Silva^a

^a IPEN-CNEN, Av. Professor Lineu Prestes 2242, 05508-000, São Paulo, SP, Brazil

^b INCIE ICPE-CA, Radiochemistry Center, 313 Splaiul Unirii, 030138, Bucharest, Romania

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ABSTRACT

This study addresses the use of radiation processing in the reuse of polypropylene (PP) waste derived from municipal products (MSW), evaluating its physical and chemical properties after e-beam (EB) irradiation in a wide range of doses between 12.5 and 500 kGy. The results show that EB irradiation up to 50 kGy has proven more efficient than conventional recycling methods, promoting molecular modifications and achieving convenient molecular rearrangements. Chemical and thermal analyses show the irradiation interference in PP after 25 kGy, and the melting temperature decreased when irradiated with a dose of up to 250 kGy, impacting the morphological and crystallinity phases, due to molecular cross-linking, especially in the PPre samples. It was observed that the Vicat tests did not interfere with PPre recycling, for secondary applications. The doses exceeding 100 kGy up to 500 kGy crosslinking allow PP to be a proper material as fillers, industrial applications, or utilized as an energy source, enhancing environmental sustainability. The use of EB in polymer processing presents a significant advantage as an alternative energy source, positioning it as a more sustainable and eco-friendly approach compared to traditional plastic recycling methods.

1. Introduction

One of the challenges in material technologies is reaching convenient durability during irradiation processing (Spadaro et al., 2017; Sirin et al., 2022; Fathy et al., 2024). Polypropylene (PP) is among the largest polymers either in its native form or in various modified forms as blends (Dahal and Kim, 2013; Suljovrujic et al., 2024) or in the production of composites (Xiao et al., 2022; Naeema et al., 2024; Tatár et al., 2025; Yu et al., 2025). The widely used commodity thermoplastic resins like polypropylene show outstanding properties, which include low density, excellent processability, superior cost-to-performance ratio, and convenient chemical and thermal resistance (Wang et al., 2019). Due to its significant industrial usefulness and academic interest, the structure and radiation modeling of polypropylene systems have been extensively studied (Zaaba et al., 2018; Wang et al., 2019; Liu et al., 2024; Dalinkovich et al., 2023; Allayarov et al., 2024; Naeema et al., 2024). Consequently, the radiation technologies applied for polymer processing

are gaining increasing attention in the research range as an appropriate alternative to chemical methods for modifying the structure (Chaudhari et al., 2007; Dahal and Kim, 2013; Kaizuka et al., 2024). In this context, EB irradiation can significantly alter the molecular structure of these polymers, thereby affecting their properties (Maeda et al., 2016; Svoboda et al., 2021; Wang et al., 2018).

According to the radiolysis-induced effects, several investigations on polypropylene recycled (PPrec) blends were carried out (Burillo et al., 2002; Burgstaller et al., 2021; Zaaba et al., 2018). Burillo et al. (2002) were among the first research groups to study and publish review articles on the potential application of radiation technology for polymer recycling, particularly using high-energy (e.g., EB and γ -radiation). The mechanistic assays reported that polypropylene is unstable when it is irradiated in air (George et al., 2007; Oliani et al., 2010). As the dose continuously rises, a higher number of shorter branches per molecule is achieved due to the advanced fragmentation (Burgstaller et al., 2021; Maeda et al., 2016; Svoboda et al., 2021). The oxidative degradation

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* Corresponding author.

E-mail address: dione_herrera@hotmail.com (D.P. de Castro).

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occurs in the irradiated PP due to the advanced scission of chemical bonds (Spadaro et al., 2017; Dalinkevich et al., 2023).

PP belongs to the very sensitive polymers in the radiation fields, even for small dose ranges (Chaudhari et al., 2007; Mikac et al., 2024; Suljovrujic et al., 2024). It was recently demonstrated (Yusoff et al., 2024) that the EB grafting of polyethylene/PP fibers using a glycidyl methacrylate (GMA) and the functionalization with two aliphatic amines progress satisfactorily at 30 kGy. The amplitude of the radiolysis effect depends on various factors, including the chemical structure, morphology, irradiation conditions, and the applied post-treatment. The fragmentation of PP chains follows the role of the strength of C–C bonds related to the degree of substitution. It means that the propylene moieties that contain tertiary carbons will be easily broken. (Liu et al., 2024; Wang et al., 2018). In the last period, several authors have conducted comparative studies on the different PP grades (Burgstaller et al., 2021; Pramanik et al., 2021; Suljovrujic et al., 2024).

The main goal of this study is to assess the compatibilization of isotactic PP neat polymer and PPreC by EB irradiation. The detailed results concerning the radiation processing of pristine/recycled PP obtained by various physical and chemical investigations are presented, completing the previous knowledge on the valorization of PP used products. The novelty of this study lies in the correlation between the different radiation doses to modify the characteristics of polypropylene and the resulting properties that recommend radiation processing as an appropriate recycling technology for these materials.

2. Materials and methods

2.1. Materials

The recycled polypropylene pellets (PPrec) used in this work came from JKS® industrial company (Guarulhos, Brazil) as the agricultural trays obtained by injection molding and provided by CLEAN ECO® selective waste collection (MSW) (Curitiba, Brazil) for planting seedlings (Fig. 1). PPreC are suitable for making agricultural trays production from industrial waste and MSW products.

2.2. Sample preparation

The selected granulated material was processed using a Battenfeld® injection molding machine (SmartPower Machine™ model; Vienna, Austria) to produce testing specimens. The specimens were molded with a thickness of 3.0 mm, following the international standards. Injection molding was employing a temperature profile of 250, 250, 260, and 270 °C across the four heating zones (feed to nozzle), an injection

pressure of approximately 160 bar, a mold temperature of 60 °C, an injection speed of 22 mm min⁻¹, an injection cooling time of 20 s, and a total cycle time of 35 s per sample. The aspect of the PPreC injected specimens used for characterization is illustrated in Fig. 2.

2.3. Irradiation

The PPreC samples (test specimens and trays) were subjected to EB exposure for further characterization. The sample irradiation treatment was performed in the Dynamitron® Job 188 electron accelerator (Radiation Dynamics, Inc.) available at the Instituto de Pesquisas Energéticas e Nucleares (IPEN-CNEN, Brazil). The samples were irradiated with 12.5, 25, 150, 200, 250, 300, 350, 400, 430, and 500 kGy (dose rate 19.99 kGy/s, energy: 1.146 MeV, beam current: 4.31 mA, tray speed 6 m/min). Alanine pellet dosimeters (93% L-alanine, diameter 4 mm and thickness 2.2 mm) were obtained from Aerial (Illkirch-Graffenstaden, France) and employed for radiation dose measurements. The actual absorbed doses, calculated after dosimetry adjustments for all samples, are presented in Table S1 of the Supporting Information. Additionally, the injected agricultural trays were exposed to various low radiation doses to assess potential toxicological effects.

2.4. Measurements

The melting temperature and fluidity index (MFI) were measured using ZwickRoell® equipment (Mflow™ model, Germany), under the standards: ISO 11357 (12.5 and 25 kGy) and ASTM D 1238, respectively. Vicat B softening temperature (50 N load, 50 °C h⁻¹, according to ASTM D1525), and thermal deflection temperature (HDT) (12.7 N load, 120 °C h⁻¹, as ASTM D648 standard indicates), were performed in Instron® equipment (CEAST™ model, USA). The cross-link content was determined based on the equilibrium swelling of samples in xylene. The samples were immersed in xylene under reflux in the Soxhlet extraction system, and the weight of the samples was measured after 24 h. When the weight of samples was established at constant values, the equilibrium swelling was reached.

2.5. The preliminary characterization of recycled polypropylene (PPrec)

The PPreC samples were characterized before and after the irradiation process, following several methods: Fourier-transform infrared spectroscopy (FTIR) was used to investigate the chemical modifications caused by EB irradiation. The resulting oxidation compounds in PPreC samples were highlighted. The spectral measurements were performed at the 450–4000 cm⁻¹ (PP neat specimen) and 450–2000 cm⁻¹ zoomed-in range (PPrec samples), using 32 scans and a 4 cm⁻¹ resolution, in a PerkinElmer Spectrum 3 FT-IR unit (PerkinElmer®, United Kingdom) spectrometer. The samples were analyzed in a UATR accessory. The



Fig. 1. Agricultural trays made of PPreC grafted trays.



Fig. 2. Injected PPreC specimens.

Raman study was carried out using a Raman Horiba® confocal microscope, model XploRA™ PLUS, provided with a multi-laser with 785 nm in the spectral range of 400 to 2000 Raman shift cm^{-1} with a resolution of 1 cm^{-1} . These measurements were performed directly on the surface of the samples, without any previous treatments.

Thermogravimetric analysis (TGA) was conducted in a TGA-DSC SDT Q600 thermobalance (TA Instruments®, USA) from 25 to 600 °C at a 10 °C min^{-1} heating rate under N_2 flow. After the measurement, all graphs were plotted from 200 to 600 °C range, to select the main thermal modifications achieved during EB irradiation. For analysis of the heating effects induced by the EB processing, the samples were studied using a differential scanning calorimeter DSC 6000 (PerkinElmer®, USA). The inert atmosphere (nitrogen) was used as a purging environment. The tests were performed in an isothermal regimen at temperatures of 50 °C at 200 °C.

The XRD patterns were obtained using a Rigaku Multiflex™ diffractometer (Rigaku®, Tokyo, Japan) set to operate at 40 kV, with $\text{Cu-K}\alpha$ radiation ($\lambda = 1.541 \text{ Å}$). The XRD measurements were conducted over a 2θ range up to 60° , with a step increment of 0.06° and a scanning rate of 4 s. Cross-linking content analysis involves assessing the degree of cross-linking within a PPrec network. The analysis was conducted using a Soxhlet system with xylene solvent under reflux for 24 h.

2.6. Statistical method

Inferential statistical analyses were conducted on the obtained thermal properties (melting point, MFI, Vicat, and HDT) and the apparent cross-linking content. The normality and homogeneity of variances for each group (NI and irradiated samples) were verified by one-way ANOVA, and mean comparison tests were performed according to Fisher's LSD procedure at a 95% confidence level ($n = 3$). Different uppercase letters in the figures indicate statistically significant differences among the means for each sample. All statistical analyses were carried out using Statgraphics® software, version 16.1.03 (Spanish version).

3. Results and discussion

3.1. Spectroscopic analysis

3.1.1. FTIR

Fig. 3 presents comparatively the recorded spectra of neat PP and

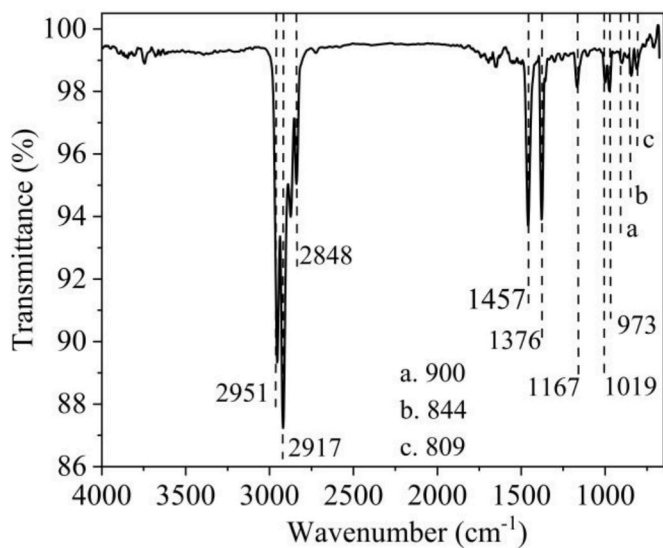


Fig. 3. FTIR spectra of a neat polypropylene specimen with its transmittance peaks.

non-irradiated (NI) PPrec, while Fig. 4 illustrates the modifications that occurred in the EB process PPrec, with 12.5, 25, 150, 200, 250, 300, 350, 400, and 500 kGy. As it was previously stated (Fifield et al., 2021; Wang et al., 2018), the exposure at 70 kGy or higher doses is a suitable conditioning for packaging materials.

FTIR peaks observed at 2951 cm^{-1} , 2917 cm^{-1} , and 2848 cm^{-1} correspond to the asymmetric stretching of the C-H bond in CH_2 . The absorbance peaks at 2920, 2850, 1460, 1370, and 1150 cm^{-1} , attributed to the stretching vibration of C-C, C-H, and CH_3 , respectively (Wang et al., 2019), are identified. The peaks associated with carboxylic groups from oxidized products were detected at 1719 cm^{-1} , 1717 cm^{-1} , and 1709 cm^{-1} , while the unsaturated groups appear in the 1650 cm^{-1} region (Boronat et al., 2024; Harada et al., 2025; Svoboda et al., 2021; Liu et al., 2024; Rodríguez-Alba et al., 2019; Suljovrujic et al., 2024). The peak at 1457 cm^{-1} is characteristic of C-H bending and asymmetric deformation in CH_3 , whereas the symmetric bending of the methyl group's CH bond occurs between 1376 cm^{-1} and 1365 cm^{-1} . The peaks between 1315 cm^{-1} and 1303 cm^{-1} correspond to CH bending modulus, while those at 1186 cm^{-1} , 1083 cm^{-1} , and 1045 cm^{-1} represent C-O-C stretching vibrations, while the peaks at 1167 cm^{-1} and 1019 cm^{-1} represent the C-O-C stretching vibration (Rodríguez-Alba et al., 2019; Wang et al., 2018). Additionally, peaks at 972 cm^{-1} , 997 cm^{-1} , and 1165 cm^{-1} are associated with CH_3 group vibrations, and the peak at 720 cm^{-1} is attributed to the crystalline phase of the polymer (Boronat et al., 2024; Harada et al., 2025). As it may be remarked from other previous records (Fathy et al., 2024), in a dose up to 25 kGy, the structure of PP is not meaningfully affected by the irradiation dose. In

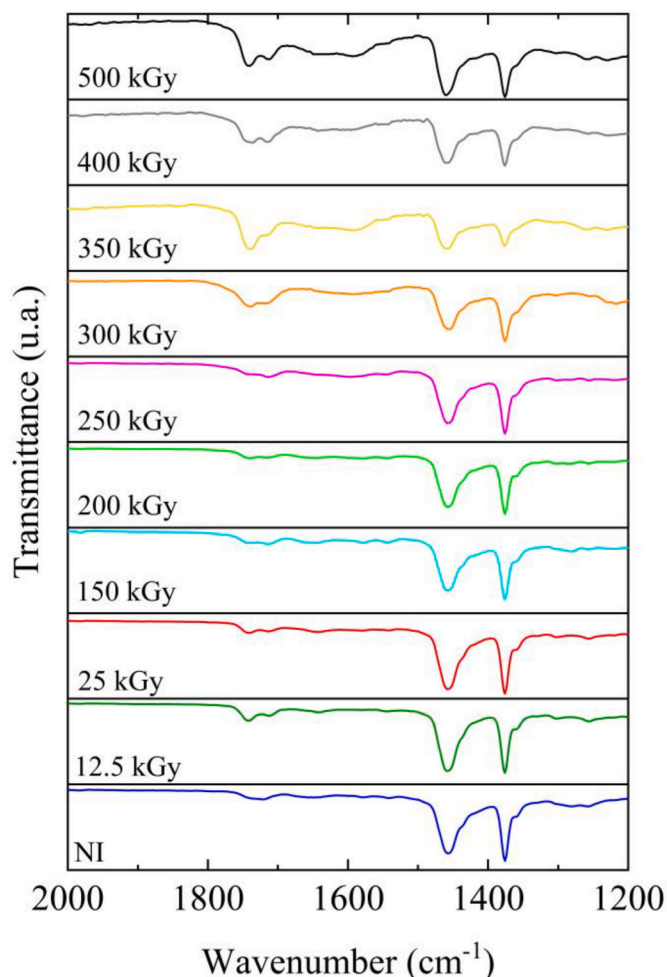


Fig. 4. FTIR spectra of non-irradiated (NI) and irradiated PPrec samples, in the range $450\text{--}2000 \text{ cm}^{-1}$.

the other study (Massoud et al., 2024), the FTIR spectra relate the presence of a new band (1744 cm^{-1}) at a low γ -radiation dose corresponding to carbonyl groups.

The peak at 1170 cm^{-1} is ascribed to the stretching $-\text{C}-\text{CH}_3$ branch on the PP chain (Wang et al., 2018). The peaks at 973 cm^{-1} , 997 cm^{-1} , and 1165 cm^{-1} are associated with CH_3 group vibrations (Boronat et al., 2024), and the peak at 720 cm^{-1} corresponds to the crystalline phase of the polymer (Boronat et al., 2024). The symmetric CH_3 bending vibration (1376 cm^{-1}) reported the presence of a carbon atom with one methyl group, which in turn supported the presence of PP (George et al., 2007; Harada et al., 2025). The EB irradiation in the air may allow for the formation of oxidized groups from chain scission reactions as a result of the presence of oxygen, an increase in the transmittance values (Fintzou et al., 2007; Suljovrujic et al., 2024; Wang et al., 2018). The FTIR results obtained for both PPre samples irradiated showed no significant change in the composition of the materials present in the PPre samples between 150 and 250 kGy.

The advanced oxidation was noticed at the 1700 cm^{-1} region for the $-\text{C}=\text{O}$ group in the FTIR spectrum in all samples studied (Dahal and Kim, 2013; Fintzou et al., 2006; Park et al., 2020; Liu et al., 2024). An increase in the absorbed dose, even up to 400 kGy, did not influence the cumulative absorbances of $\text{C}=\text{C}$ and $\text{C}=\text{O}$ groups. Raman studies with different phase assignments (Andreassen, 1999; Kaizuka et al., 2024) may be correlated with data from other techniques, such as wide-angle X-ray scattering (WAXS), to assess crystallite orientation of PP (Andreassen, 1999).

3.1.2. Raman spectroscopy

The Raman spectra recorded on the irradiated PPre samples are illustrated in Fig. 5 with 12.5, 25, 150, 200, 250, 300, 350, 400, and 500 kGy. The association of in-plane $\text{C}-\text{H}$ bonds to $\text{C}-\text{C}$ stretching vibrations exhibits a decrease in their intensity for 12.5 to 25 kGy. This phenomenon was observed in the 1315 cm^{-1} range. This reduction may result from perturbations in conjugated electron systems, leading to diminished band intensity upon irradiation (Mikac et al., 2024). The irradiation at 25 kGy is slightly higher than that of non-irradiation PPre samples shown in the 794 cm^{-1} peak (Huang et al., 2017). Kaizuka et al. (2024) describe that the 825 cm^{-1} band, which appears as a shoulder of the 841 cm^{-1} band, is due to the PP amorphous phase, and thus, the existence of the negative peak suggests the diminution of the amorphous phase of PP. The changes in the crystallinity appear more clearly at a 25 kGy dose. Our results show the EB effect on the surface PPre samples over 12.5 kGy, especially in the range 825 cm^{-1} , indicating the enlargement of the crystalline phase.

In combination with FTIR analysis, Raman spectroscopy was utilized to examine PPre samples of different irradiation doses as previously suggested (Nielsen et al., 2002). Some prominent band peaks were identified (Andreassen, 1999; Boronat et al., 2024; Harada et al., 2025; Kaizuka et al., 2024; Nielsen et al., 2002): 2955 , 2879 , and 2838 cm^{-1} , linked to $\nu\text{CH}_3\text{ asym.}$, $\nu\text{CH}_3\text{ sym.}$, and $\nu\text{CH}_2\text{ sym.}$ vibrations; 1458 cm^{-1} , which correspond to $\delta\text{CH}_3\text{ asym.}$, and δCH_2 vibrations; 1329 cm^{-1} , associated with δCH and τCH_2 vibrations; 1152 cm^{-1} , related to νCCb , $\nu\text{C}-\text{CH}_3$, δCH and ρCH_3 vibrations; 972 cm^{-1} , corresponding to ρCH_3 and νCCb vibrations; 841 and 808 cm^{-1} , due to ρCH_2 , νCCb , $\nu\text{C}-\text{CH}_3$ and ρCH_3 vibrations; and 398 cm^{-1} , linked to ωCH_2 and δCH vibrations. In this Raman range, the main characteristic bands of PP are placed at 1445 cm^{-1} (CH_2 scissoring) (Huang et al., 2017), which is more visible in the EB processing at 200 and 250 kGy. It may be observed that the irradiation dose applied at 150 to 250 kGy induces changes on the surface of PPre, especially from 1000 to 1750 cm^{-1} , including a reduction of the crystallinity of PP.

Our results didn't indicate the structural changes in the analyzed samples for high doses. However, it is important to notice that the position and intensity of these Raman bands can fluctuate due to various factors, including polymer molecular mass distribution, crystallinity degree, and manufacturing processes, among others (Boronat et al.,

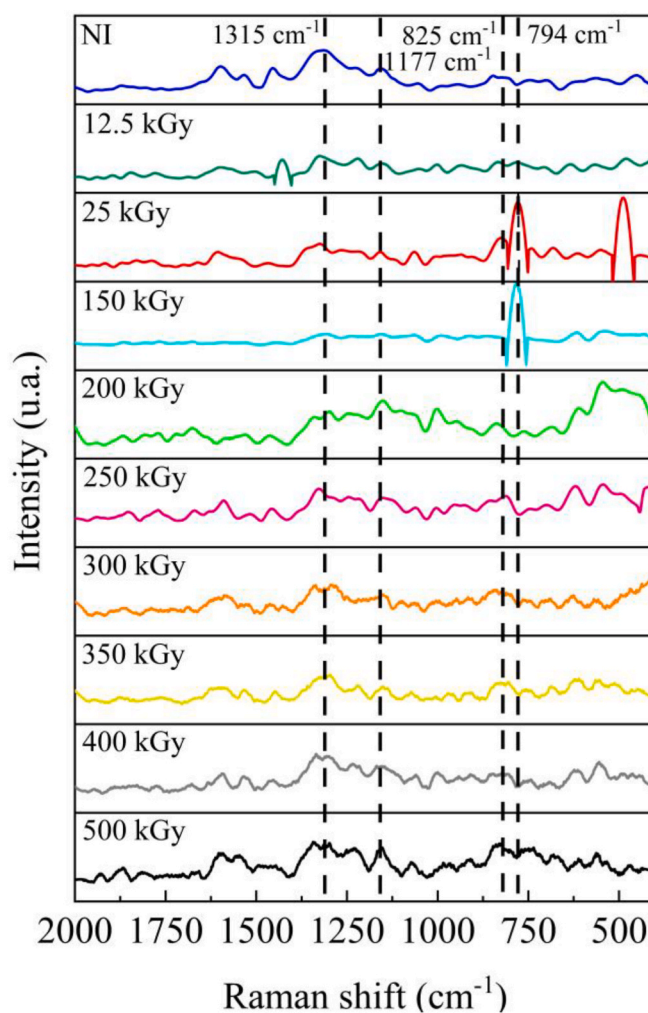


Fig. 5. Raman spectra of PPre samples irradiated up to 500 kGy.

2024). Moreover, Raman spectra indicate the resistance to further degradation in high irradiation doses (Harada et al., 2025), associated with a slight reduction in peak intensity for both PPre samples with 300 kGy. PPre exposed to 500 kGy exhibits minor structural changes when it is compared with other spectra recorded on samples irradiated, which increases the CH_3 asymmetric bending and CH_2 bending vibrations, and PPre exposed to 350 kGy shows negligible changes, at 300 kGy, suggesting no apparent polymer chain degradation during EB exposure. In the 800 to 1500 cm^{-1} region, the irradiation can cause structural changes in the PP in the surface region caused by irradiation (Mikac et al., 2024). The bands in the $\text{C}-\text{H}$ stretching region are more intense in the non-irradiated samples and are not shown in other samples over 300 kGy. Such changes between 350 and 500 kGy cannot be detected by Raman optical images, and this phenomenon can be seen in the morphological aspects of the PP surfaces, under Fig. 6, and were related in the previous Raman results.

3.1.3. XRD

Fig. 7 presents the XRD diffraction curves for the PPre with 12.5, 25, 150, 200, 250, 300, 350, 400, and 500 kGy. The diffraction peaks at $2\theta = 13.1^\circ, 15.9^\circ, 17.6^\circ, 20.9^\circ$, and 24.6° are assigned to the planes of the α -form isotactic structure of PP (Fathy et al., 2025). Harada et al. (2025), Manas et al. (2013), and Manas et al. (2014) show the α -phase in the non-irradiated specimen in (110), (040), and (130) crystallography planes for PP. Harada et al. (2025) investigated and described the structural modifications in γ -irradiated polypropylene (PP) samples, evidenced by the variations in the diffraction peaks observed at 2θ

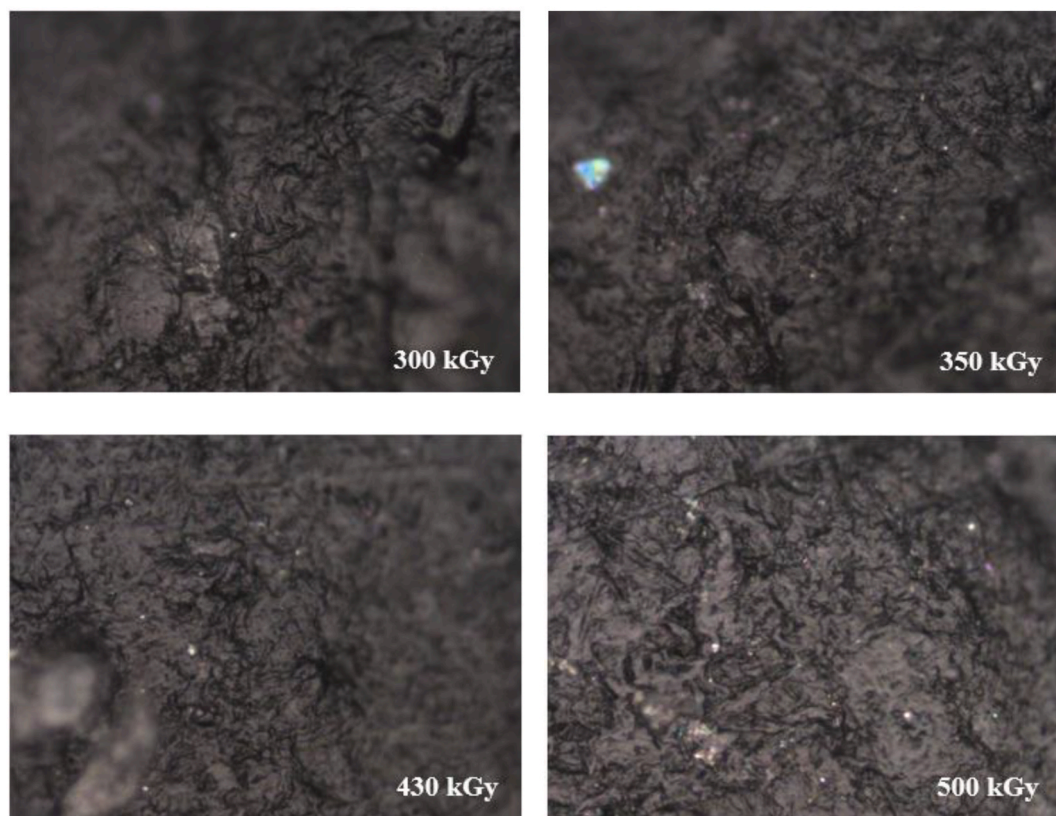


Fig. 6. Illustrative Raman images of PPREC samples irradiated at doses of 300, 350, 430, and 500 kGy, reported by Harada et al. (2025).

values of 14° , 16° , 18° , 21° , and 23° , which correspond to characteristic reflections of the crystalline phases of PP. The doses exceeding 15 kGy do not show crystallography peak changes. The crystalline fraction (%) was determined by integrating the region where the peak appears relative to the entire diffraction pattern. For PPREC, the crystalline fractions were 6.15% for the non-irradiated sample and 5.44% and 5.43% for samples irradiated at 12.5 and 25 kGy, respectively. These results indicate that EB exposure produces a reduction in the crystallinity degree (Maeda et al., 2016; Svoboda et al., 2021; Oliani et al., 2010), confirming that no structural modification occurs; Suljovrujic et al. (2024) investigated PP modified by EB radiation, exhibiting a peak increase in its crystallinity at doses around 50 kGy, followed by a decline as the radiation dose continues to increase. These results are compatible with an increase in the peaks for pristine and 25 kGy exposed samples as shown in Fig. 7. Fathy et al. (2025) state that the ionizing radiation does not affect all polymer regions equally; the amorphous regions, which contain tie molecules connecting crystallites, are more susceptible to radiation, whereas the crystalline regions are relatively less impacted with 20 kGy dose. Due to the material's high recrystallization rate, rapid heating is required to prevent reorganization (Krause et al., 2006).

The analysis of samples exposed to 150, 200, and 250 kGy, revealed a breakdown of the crystalline structure and the emergence of species closer to the carbon lattice with oxygen bonds at 11° , along with an increase in amorphous carbon in the region from 5° to 30° is possible due to the damage of smaller crystals by high energy radiation (larger crystals remain), generating β -phase up about 60 kGy, where the intensity at $2\theta = 16^\circ$ starts to decrease (Liu et al., 2024; Manas et al., 2013; Svoboda et al., 2021) in the irradiated samples at 150, 200 and 250 kGy is visibly modified (Massoud et al., 2024). As a result, certain crystalline planes may lose atoms, while other ones may become enriched with them (Fathy et al., 2025). However, for the 150 kGy specimens, crystallinity returns to levels similar to those observed in the pristine sample (Maeda et al., 2016). Manas et al. (2013) observed that the irradiation

up to 90 kGy overall crystallinity decreased by up to 9%, and a gradual loss of β -phase for PP samples. Massoud et al. (2024) observed a diminution of the crystallinity in the raw PP samples with $\sim 16\%$ less crystallinity of the unirradiated PP and irradiated up to 200 kGy.

In the presence of oxygen, EB radiation typically causes chain secondary scissions through a well-known chemical mechanism, leading to the formation of hydroperoxides ($-O-OH$), dialkyl peroxides ($-O-O^*$), tertiary alcohols, and methyl ketones (Scheme 1) (Boronat et al., 2024; Svoboda et al., 2021; Yang et al., 2022; Sirin et al., 2022; Liu et al., 2024).

The peak recorded at 300, 350, 430, and 500 kGy decreases but also shifts to lower angles between 15° and 20° . This phenomenon is attributed to the radiation-induced destruction of smaller crystals, while the larger crystals remain intact with doses over 120 kGy for PP (Maeda et al., 2016; Svoboda et al., 2021). PP has three polymorphs, one of them being metastable. The resulting structure in irradiated PP corresponds to the same monoclinic α phase present in the pristine sample (Maeda et al., 2016). The irradiation induces a change in the crystallinity by combining two opposing effects: an increase caused by chain scission and a decrease induced by branching and crosslinking on the lamellae surfaces/thickness, creating defects in the crystalline phase (Andreassen, 1999; Liu et al., 2024; Suljovrujic et al., 2024). Liu et al. (2024) related to iPP polymer, as the total dose increases, a characteristic diffraction peak of the β -crystal emerges at 16.0° , indicated by a black dashed line, with its intensity gradually increasing. A reversion to the crystalline state for PPREC, however, has not been previously reported.

3.2. Thermal analysis

3.2.1. TGA analysis

Fig. 8 presents the TGA curves for non-irradiated (NI) PPREC and PPREC irradiated at doses of 12.5, 25, 150, 200, 250, 300, 400, and 500

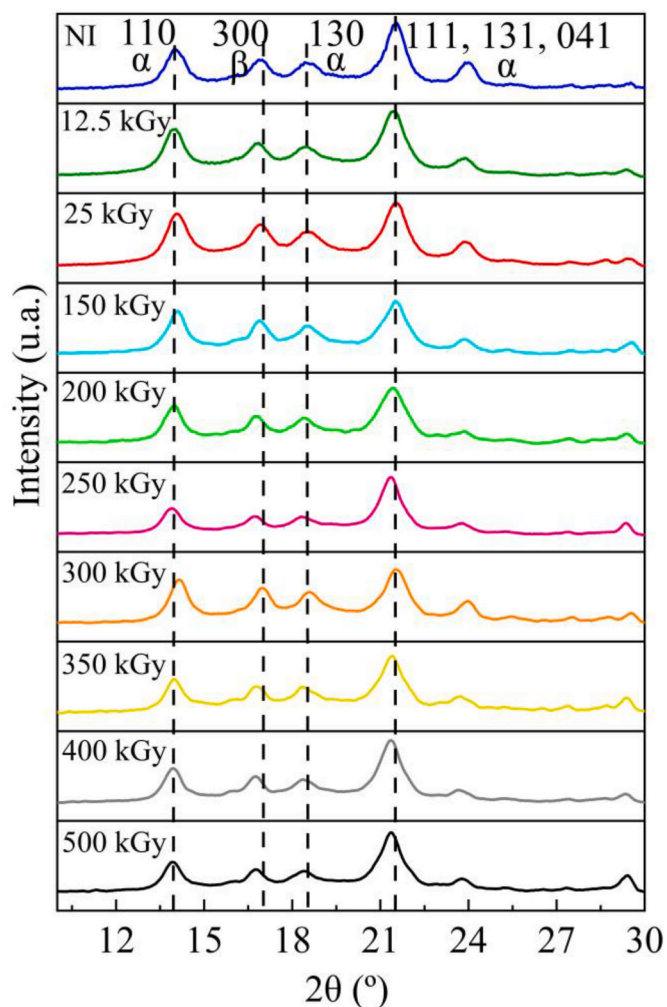


Fig. 7. XRD curves of PPreC samples, non-irradiated (NI) and irradiated up to 500 kGy.

kGy. The results indicate no significant mass loss up to 400 °C under the studied radiation conditions. The major mass losses caused by degradation occurred between 400 °C and 480 °C; further decomposition observed beyond 480 °C is revealed in the PPrec formulations. The EB

irradiation may crosslink polymers when a complex network structure is formed; it improves the thermal stability of the material (Dahal and Kim, 2013; Sirin et al., 2022; Fathy et al., 2025). While the investigations on the improvement in the thermal stability improvement for PP neat from 245 to 269 °C (Fathy et al., 2025) after the irradiation of PP at 20 kGy,

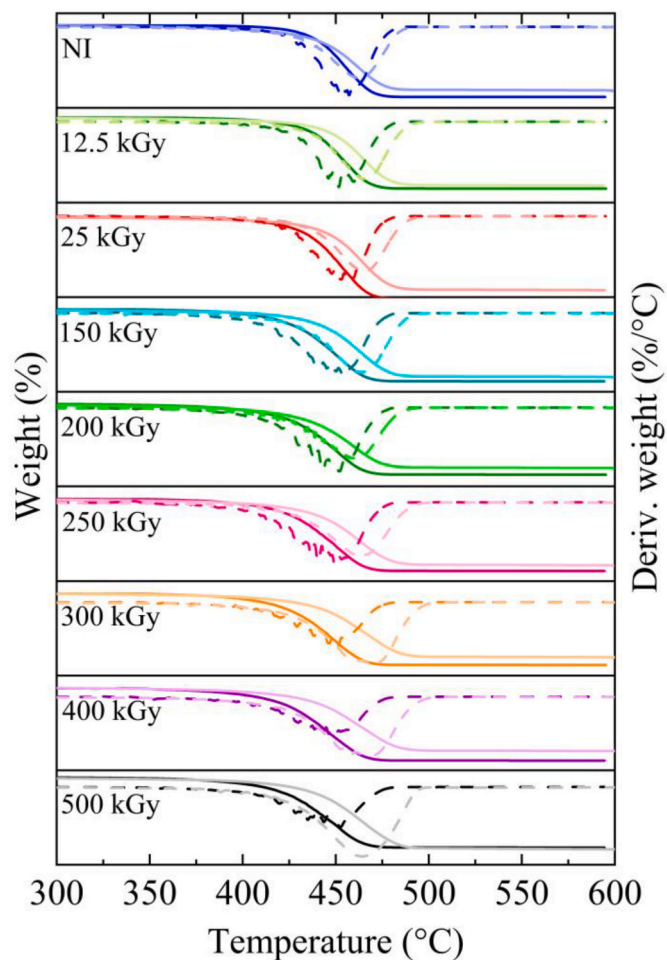
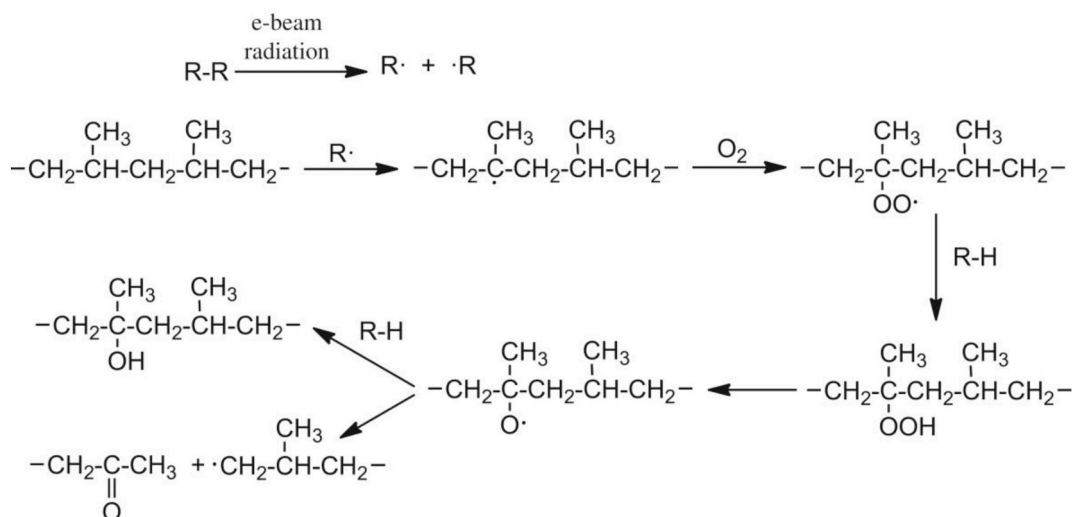


Fig. 8. TGA curves of non-irradiated (NI) and irradiated PPreC samples up to 500 kGy.



Scheme 1. The radiation oxidation mechanism of PP by EB, proposed by [Svoboda et al. \(2021\)](#) and [Massoud et al. \(2024\)](#).

another study found the start of thermal decomposition at 461.7 °C after the same processing dose (Yang et al., 2022). However, it was reported (Fathy et al., 2024; Oliani et al., 2018) that the PP starts its degradation (T_{onset}) at a temperature lower than 51.6% after receiving 20 kGy. After the thermal degradation of PP exposed to 30 kGy, the remaining solid residue (ash content) obtained consists of accumulated materials such as calcium carbonate, titanium dioxide, carbon black, zinc stearate, silica, and other components (Yusoff et al., 2024).

The enhancement in thermal stability was attributed to the formation of intermolecular bonds within the polymer chain or the development of a more compact three-dimensional crosslinking network in the crystalline region upon heating (Yang et al., 2022; Zaaba et al., 2018). Fathy et al. (2024) investigated the γ -irradiation for enhanced properties of PP composites, with thermal stability reaching up to 380 °C after receiving 20 kGy, and 17% residual ashes. When the effect of increasing γ -irradiation doses on pure PP was examined, the samples, particularly those exposed to 70 and 100 kGy, exhibited a more yellowish color (Sirin et al., 2022), reached by the material degradation.

The results obtained for the exposures of 150, 200, and 250 kGy doses show that there was no great loss of mass up to 400 °C according to the comparison of TGA curves of the non-irradiated and irradiated samples. Rodríguez-Alba et al. (2019) evaluated the thermal decomposition of the PP specimen at 10% weight loss, detected at 428 °C. The major mass losses (degradation) occurred from 400 up to 480 °C, and after 480 °C, the solids content of the PPREC formulations remained within a range of less than 1%. The solids present (ash content) in accumulated mass are calcium carbonate, titanium dioxide, carbon black, zinc stearate, silica, and others. Ali and Saleh (2014) investigated the thermal stability of LDPE/iPP blends when a decrease in the thermal stability is attained as the isotactic polypropylene (iPP) content or γ -irradiation dose (20 to 600 kGy) rises. No significant differences among the samples irradiated with 300 up to 500 kGy are obtained (Fig. 8). Based on these results, the dissociation temperature of irradiated PPREC gradually decreases with increasing γ -irradiation doses, particularly at high doses (Massoud et al., 2024).

Though the crystal form of irradiated PP remained unchanged, as confirmed by the Raman spectra and XRD information, this aspect suggests that PP became more amorphous after irradiation. These findings are consistent with previous studies, which observed and analyzed a decrease in T_m for PP with increasing doses of EB irradiation up to 120 kGy (Fintzou et al., 2007a,b; Fintzou et al., 2007). Other studies conducted on PP samples irradiated at 250 kGy (Massoud et al., 2024; Wang et al., 2018) provide a similar statement.

3.2.2. DSC analysis

The effect of EB irradiation on the melting temperature (T_m) and enthalpy of fusion (ΔH_m) of PPREC samples is summarized in Table 1. Fig. 9 presents the DSC curves during the heating and cooling cycles of non-irradiated (NI) PPREC samples and those irradiated at doses of 12.5, 25, 150, 200, 250, 300, 350, 400, 430, and 500 kGy. A decrease in the

crystalline melting temperature (T_c) and enthalpy of crystallization was observed (Krause et al., 2006; Krupa and Luyt, 2001; Svoboda et al., 2021; Zaaba et al., 2018), ranging from 117 to 128 °C, with a shift of approximately ± 1 °C in the comparison of non-irradiated samples with respect to those exposed to 12.5 and 25 kGy. Yusoff et al. (2024) investigated the PP polymer fibers, having the first T_m peak at 126.83 °C, while PE/PP grafted resulted in the second endothermic melting peak at 168.21 °C. Allayarov et al. (2024) show that the melting temperature in the samples can change when PP receives high doses, for example, 4000 kGy. After irradiation, the T_m values are also changed (~ 163 to ~ 97 °C), and the melting and crystallization peaks of the polymer move towards lower temperatures.

It is evident that, in the absence of chemical compounds and/or co-agents, numerous structural modifications due to the branching in PP occur. These defects create steric hindrances during chain folding, leading to a reduction in crystallinity. Consequently, it is linked to specific melting enthalpy (Fintzou et al., 2007a,b; Krupa and Luyt, 2001), associated with chain destruction and accompanied by a decrease in the molecular weight of the polymer depending on the input absorbed dose (Sirin et al., 2022; Allayarov et al., 2024).

Therefore, the endothermic peak, which appears at much higher temperatures due to the melting of the crystalline phase, arises not only from the melting of the smectic crystalline phase present at room temperature but also from the crystal portions formed through additional crystallization during heating (Oliani et al., 2018; Suljovrujic et al., 2024; Zaaba et al., 2018; Allayarov et al., 2024); the decreases in the melting temperature and the percent crystallinity after EB operation are attributed to the formation of slight crystal imperfections and a reduction in crystallite size (Fathy et al., 2024; Pramanik et al., 2021) based on the comparison with amorphous phase in PP (Kremser et al., 2020). In some studies, the irradiation does not affect the crystallization temperature (Kremser et al., 2020; Krupa and Luyt, 2001). This result is in good agreement with our XRD and TGA results.

Table 2 shows the results of the DSC analysis of the PPREC samples irradiated with doses of 150, 200, and 250 kGy. The results indicate that as the processing doses increase, both the melting temperature (T_m) and crystallization temperature (T_c) decrease across all samples of PP shifted to a lower temperature with a shoulder peak (Krause et al., 2006; Liu et al., 2024; Wang et al., 2018; Yang et al., 2022). It may be attributed to the ease of entangling, untangling, and folding of the short chains formed during the initial irradiation stage, while the increased crosslinking at higher irradiation doses limits chain mobility (Liu et al., 2024; Yang et al., 2022), decreasing the length of the macromolecular chains, crosslinking, and other modifications in the crystalline PP are accomplished (Fintzou et al., 2006; Fintzou et al., 2007a,b; Fintzou et al., 2007). Specifically, the PPREC samples irradiated at 150, 200, and 250 kGy showed a reduction in their crystallization temperature to 120.2 °C, 116.9 °C, and 115.5 °C, respectively. During the second melting, the melting point differs, and the degree of melting point depression is greater than that observed in the first DSC scan at the 150 kGy specimen, which was described (Svoboda et al., 2021; Wang et al., 2018). These results suggest that the occurrence of the degradation on the PP chains reduces the number and the size of the crystalline domains after the cooling recrystallization (Dahal and Kim, 2013; Pramanik et al., 2021; Wang et al., 2018). Tatár et al. (2025) reported that during the first heating cycle of samples, after receiving 150 and 200 kGy, the degree of crystallinity remained largely unchanged in response to irradiation. However, it decreased significantly in the second heating cycle.

Based on the DSC investigation on the PPREC samples irradiated in the dose range of 150 to 250 kGy, the reducing molecular weight generated as a function of the absorbed dose may be noticed. As a result, several smaller spherulites were formed, which accounts for the lower T_c values and the appearance of a shoulder melting peak at higher absorbed doses (Wang et al., 2018). These results suggest there has been occurred some structural change has occurred in the sample in comparison with the low doses used. The DSC assay for the determination of

Table 1
DSC analysis data of the non-irradiated (NI) and irradiated PPREC samples.

	NI		12.5 kGy		25 kGy	
Analysis steps, ΔH (J/g)	ΔH_{onset}	ΔH_{endset}	ΔH_{onset}	ΔH_{endset}	ΔH_{onset}	ΔH_{endset}
1st heating	7.00	35.21	10.04	25.44	18.27	30.31
2nd heating	10.52	32.05	9.54	25.13	18.03	30.31
Cooling	-10.94	-57.33	-7.92	-38.46	-6.38	-21.37
T_m - 1st heating, °C	128.02	165.54	127.67	165.85	128.31	165.33
T_m - 2nd heating, °C	128.52	163.19	128.72	159.88	129.65	159.58
	T_{endset}	T_{onset}	T_{endset}	T_{onset}	T_{endset}	T_{onset}
T_c (Cooling), °C	117.63	123.68	118.01	122.43	117.76	121.43

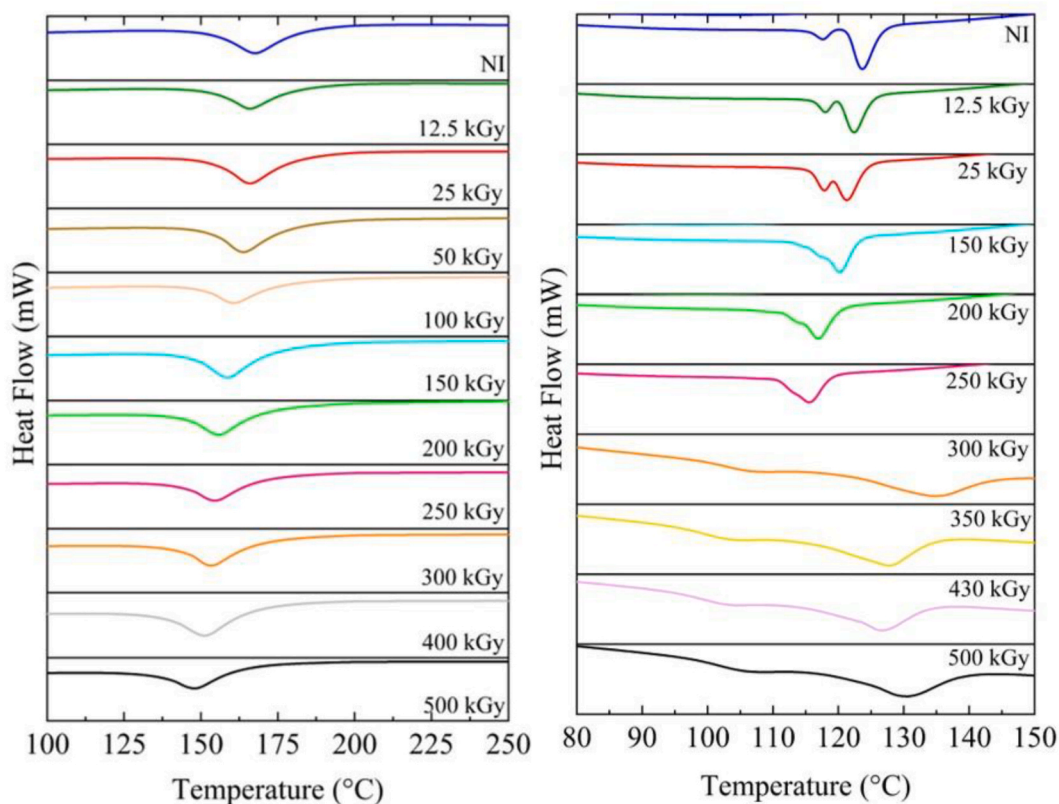


Fig. 9. DSC curves in the heating (left) and cooling (right) cycles of non-irradiated (NI) and irradiated PRec samples up to 500 kGy.

Table 2

DSC analysis of PRec samples irradiated with doses of 150, 200, and 250 kGy.

	150 kGy		200 kGy		250 kGy	
Analysis steps, ΔH (J/g)	ΔH_{onset}	ΔH_{endset}	ΔH_{onset}	ΔH_{endset}	ΔH_{onset}	ΔH_{endset}
1st heating	4.74	25.60	12.08	27.50	5.14	18.94
2nd heating	2.70	28.36	5.93	26.63	0.51	21.69
Cooling	—	72.40	—	74.44	—	60.01
T_m - 1st heating, °C	12.88	158.94	125.26	148.60	123.68	145.79
T_m - 2nd heating, °C	125.73	152.94	125.26	148.60	123.68	145.79
T_c (Cooling), °C	120.21		116.92		115.57	

the melting temperatures (T_m) of PRec in the dose range from 300 kGy to 500 kGy indicates a minimum value at a temperature of ~ 130 °C at 500 kGy. Ali and Saleh (2014) report the increase in the crosslinking degree as the irradiation dose or HCB content enhances the LDPE/HCB matrix or the LDPE/iPP/HCB blend. This curing growth prevents the structural reorganization during cold drawing shown in the DSC analysis, especially for high doses up to 600 kGy.

3.2.3. Melting temperature and MFI analysis

Fig. 10 presents the melting temperature values for NI and irradiated PRec samples. The formation of a crosslinked phase in the composite system can be induced by the EB irradiation process, as a higher number of radicals are generated in the polymer matrix and fillers through radical decay (Zaaba et al., 2018). DSC analysis reveals that heating and melting enhance the chain mobility of polymer fragments, increasing the likelihood of free radical recombination reactions. This occurs because polymer irradiation typically generates free radicals (Fintzou et al., 2007; Allayarov et al., 2024). Similarly, as the temperature

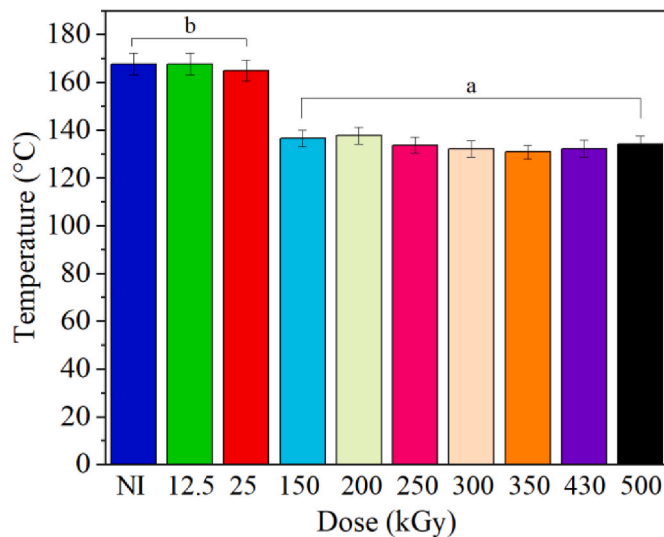


Fig. 10. Melting temperature results of PRec samples, non-irradiated (NI) and irradiated up to 500 kGy.

approaches the melting point, and moves quickly. These processes lead to some changes in the thermal properties of polymers (Fathy et al., 2024). There is a threshold radiation dose between 12.5 and 25 kGy that marks the limit at which polypropylene can maintain stable melting temperature resistance compared to the non-irradiated sample (NI). Burgstaller et al., (2021), when investigating the viscosity of PP, found a decrease in final viscosity, indicating some chain scission happening up to 100 kGy.

The radiation with a dose of 25 kGy induces a drastic drop of viscosity in PP due to main chain scissions, resulting in a significant

decrease in molecular weight in the MFI analysis (Turker et al., 2018). Fig. 11 presents the MFI values for PPreC samples, including both pristine samples and the irradiated specimens at doses of 12.5, 25, and 150 kGy. The significant increase in the fluidity index of the irradiated samples was caused by the shearing (splitting) of the molecular chains because PP belongs to the degradable polymer class under ionizing irradiation (Turker et al., 2018). At this stage, the former crosslinking networks restricted the mobility of the generated radicals, trapping them within the network structure (Turker et al., 2018; Zaaba et al., 2018). Due to the irradiation treatment, the diffusion rate of PP chains slows down according to the dose of radiation applied. It produces increasing over time MFI values after irradiation (Chaudhari et al., 2007; Fathy et al., 2024; Yang et al., 2022) and gel content (Krupa and Luyt, 2001; Wang et al., 2019; Zaaba et al., 2018). Another study reported an increase in MFI due to the induced crosslinking by EB exposure (Krämer et al., 2024) and γ -irradiation (Krupa and Luyt, 2001; Turker et al., 2018; Yang et al., 2022). The samples had an increase from 5.52 to 104.4 g/10 min for PP neat and 20.9 to 110 g/10 min for PPreC.

The MFI values for PP neat samples and PPreC (Fig. 11) that were NI and were irradiated with doses of 12.5, 25, and 150 kGy demonstrate that it was impossible to measure the fluidity index of the PP neat and PPreC samples with doses of 150 kGy and above due to the cross-linking of the PP. Chaudhari et al. (2007) established that MFI values shift following the irradiation dose used, and for PP is dependent on the hindrance chain PP (-CH₃), with chain segments of PP irradiated. Liu et al. (2024) state that when the total dose increases, the density polymer exhibits a decreasing trend. From 12.5 to 250 kGy, the density polymer declines rapidly. However, as the irradiation dose continues to rise to 500 kGy, the rate of decrease slows.

3.2.4. Vicat B, and HDT analysis

Fig. 12 presents the results of the Vicat B softening temperature tests for both non-irradiated PPreC samples and those irradiated at doses of 12.5 and 25 kGy. The irradiated PPreC samples showed a slight increase of 1.3 °C in the Vicat B softening temperature compared to the non-irradiated sample. Fig. 13 shows the results obtained for the HDT (°C) of the non-irradiated PPreC samples (NI) and those irradiated with doses of 12.5 and 25 kGy under conditions 12.7 N and 120 °C/h.

The thermal deflection temperature of the irradiated samples increased by 4 °C, likely influenced by the molecular alignment generated during the injection conditions (pressure, cooling, and injection speed). The irradiation may help relieve post-injection stresses in the PPreC specimens, resulting in this modification compared to the non-

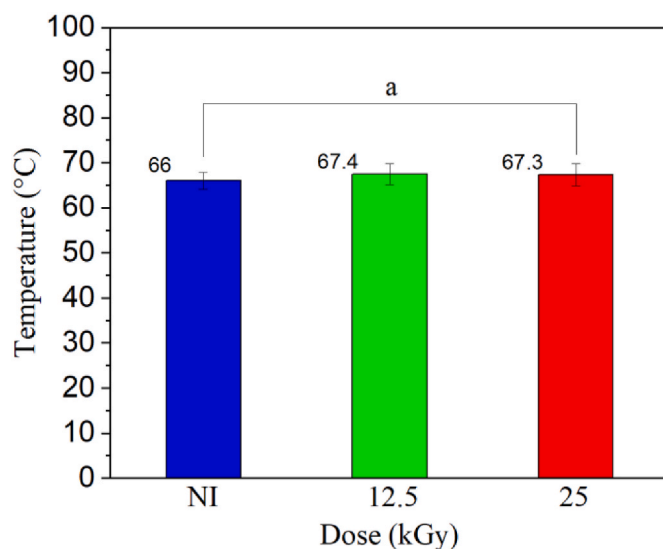


Fig. 12. Vicat B softening temperature tests on PPreC samples, non-irradiated (NI) and irradiated with doses of 12.5 and 25 kGy.

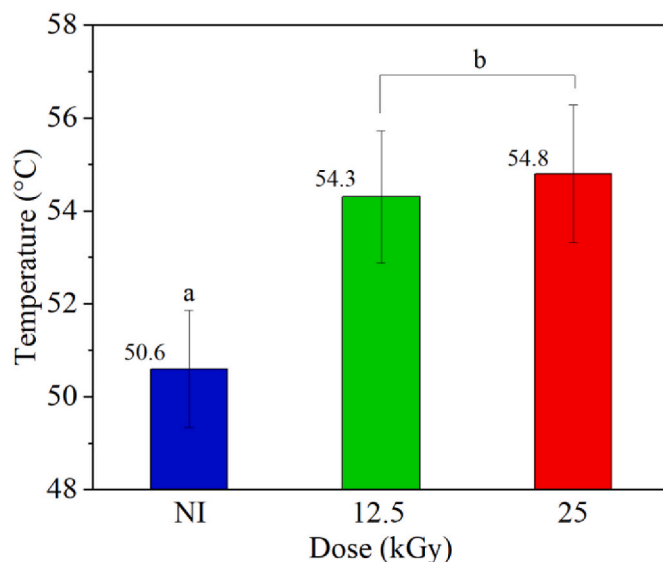


Fig. 13. HDT results of PPreC samples, non-irradiated (NI) and irradiated with doses of 12.5 and 25 kGy.

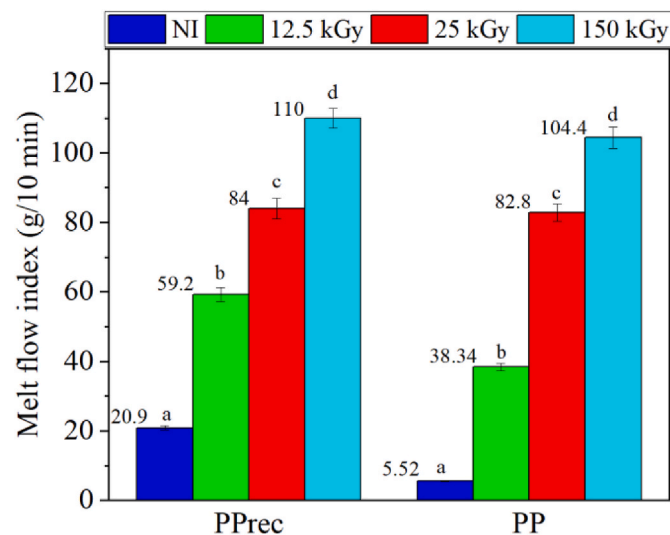


Fig. 11. MFI of PPreC and PP neat samples, non-irradiated (NI) and irradiated with doses of 12.5, 25, and 150 kGy.

irradiated sample.

The results of the thermal analysis and melting temperature for non-irradiated and irradiated PPreC samples at doses of 150, 200, and 250 kGy. The transformation of the chain topology in blends after EB irradiation processing not only affects the melt fluidity but also alters the chain regularity, influencing both the melting and crystallization behavior of PP samples (Liu et al., 2024; Yang et al., 2022). The decrease in melting temperature resistance observed in the samples irradiated at these doses was due to the shearing of the molecular chains, as PP is highly sensitive to EB radiation. However, the irradiation didn't change in the samples between 150 and 250 kGy.

For the MFI tests on irradiated PPreC samples at doses of 150, 200, and 250 kGy, the fluidity tests could not be performed on the irradiated samples because the irradiated materials lacked viscosity (melt strength) and had cross-linked bonds. Even at a lower temperature of 190 °C (typically used for PE), the cross-linked material irradiated at 150 kGy did not exhibit the necessary flow properties, such as those shown at 230 °C (for PP).

The significant increase in the fluidity index of the irradiated samples was attributed to the shearing (breaking) of the molecular chains, the disruption of the crystalline structure, and the cross-linking of PPreC, which is highly sensitive to EB radiation, compared to the non-irradiated sample. This phenomenon was previously studied (Krämer et al., 2024). Yang et al. (2022) investigated the PP γ -irradiated with 100 kGy, obtaining 102.66 g/10 min for PP neat, and 145.92 g/10 min for 80 kGy; whereas this study obtained 104 g/10 min (for PP) and 100 g/10 min (for PPreC) samples using EB radiation. Fintzou et al. (2006) described the melting temperature as well as specific melting enthalpy decreased with increasing irradiation doses of 30 to 120 kGy. In another study, Fintzou et al. (2007) reported that the melting temperature exhibits no statistically significant differences between γ -irradiation in air and under vacuum. However, the decrease in the melting enthalpy is more pronounced after γ -irradiation under vacuum than in air in all the samples evaluated.

The variation in melting temperature observed in samples exposed to 300, 350, 430, and 500 kGy was caused by the shearing of the molecular chains, followed by the formation of cross-links. However, the samples analyzed did not show differences between 300 and 500 kGy.

3.2.5. Cross-linking content analysis

Fig. 14 shows the degree of cross-linking of the non-irradiated PP samples and PP irradiated for comparison with similar PPreC samples. A lower irradiation dose results causes a slower gel formation rate, leading to lower gel content values. In contrast, higher irradiation doses generate a greater number of radicals, which are expected to produce higher gel content (Ali and Saleh, 2014). The samples irradiated at 100 and 150 kGy especially exhibit EB-induced cross-linking in PP neat, inducing the crosslink chain and reducing average polymer molar weight (Fifield et al., 2021; Krämer et al., 2024). PP neat irradiated at doses up to 100 kGy does not cross-link its molecular chains but only shears them, which accounts for the significant increase in fluidity index and the reduction in its mechanical, thermal, and chemical properties (Yang et al., 2022). PP neat irradiated at 100 kGy begins to cross-link, and at 250 kGy, it reaches its maximum cross-linking peak. However, beyond 250 kGy, cross-linking begins to decrease as the cross-linked chains undergo degradation due to intense exposure to very high radiation doses.

Between 12.5 and 25 kGy, PPreC did not undergo cross-linking, but instead chain scission, resulting in a significant increase in fluidity index and reduction in mechanical properties. Manas et al. (2014) show that the gel content for the PP specimen irradiated with 15 kGy was 75 %, in comparison with the value found in this study. Kremser et al. (2020) reported that the molar mass (kg/mol) was significantly lower depending on the irradiation method used. At 45 kGy, γ -radiation had a more pronounced effect than EB. Despite these changes, the material remained suitable for tray applications with radiation doses up to 25 kGy without compromising its safety for vegetable planting and other uses. Cross-linking tests were not performed on PPreC samples irradiated at 50 and 100 kGy. The progressive crosslinking of PPreC occurs at doses ranging from 150 to 500 kGy. However, it did not reach the same extent as natural polypropylene due to the presence of foreign elements in the material originating from the Municipal Solid Waste (MSW) collection.

4. Conclusions

It is widely recognized that high doses of ionizing radiation can cause significant chain scission and crosslinking in polymers. While cross-linking can enhance tensile strength, it may also reduce MFI test results. Additionally, radiation-induced oxidation can lead to polymer discoloration at lower doses than those needed to noticeably affect mechanical properties. The findings provide valuable insights by analyzing the effects of EB radiation on PPreC. The thermal (TGA, DSC, MFI, Vicat B, HDT) and morphological (XRD) properties observed under ionizing radiation enable a broader exploration of PPreC applications in various

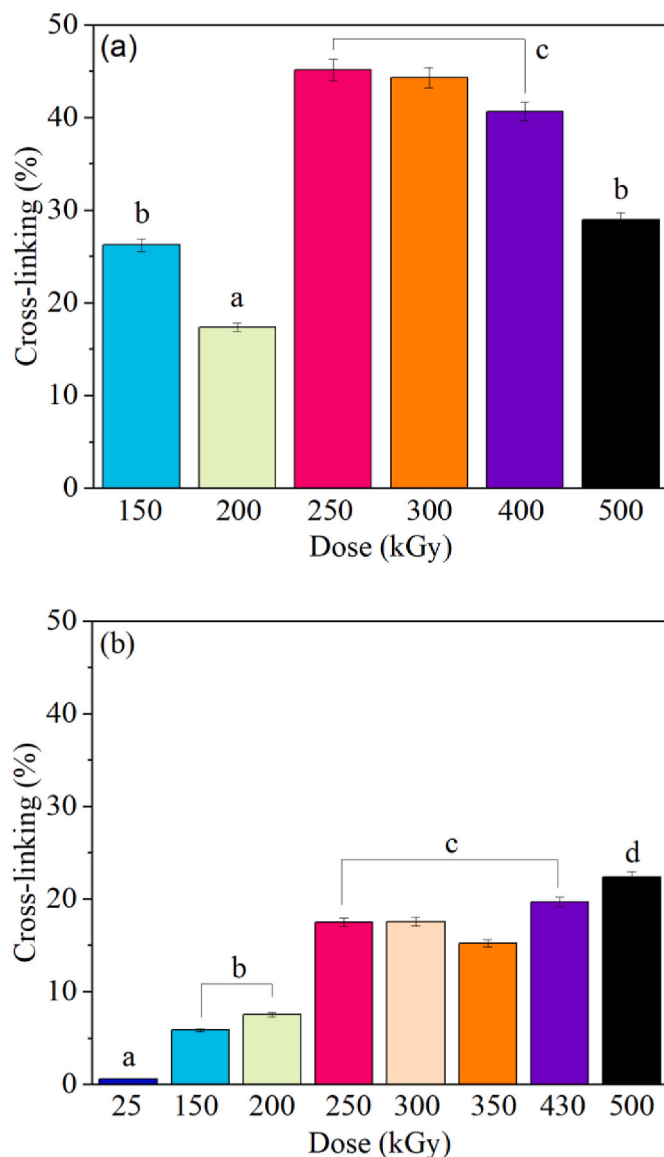


Fig. 14. Cross-linking obtained for PP neat (a) and evaluated for PPreC (b).

plastic components, facilitating a critical assessment of recyclability, cost, and environmental impact.

Ionizing radiation up to 25 kGy has been identified as beneficial for the recycling and reuse of PP, provided that the resulting materials are not exposed to extreme thermal, chemical, or mechanical conditions. However, comprehensive cost and performance evaluations are necessary to ensure its viability, being the application of EB radiation in polymer processing is highly significant. There are still significant research gaps in this field that must be addressed with advancements in technology and new applications of radiation processing. Peak intensities in the 1700–1750 cm^{-1} range, corresponding to carboxylic groups, rose steadily as the radiation dose reached 500 kGy, and similarly, a reduction in crystallinity was observed, attributed to the dose-dependent degradation of the crystalline structure of PPreC. The results of the thermal and chemical properties with a dose of 25 kGy were considered admissible for similar applications, not showing changes in the PP chain structure. Above this dose of 25 kGy, these properties were compromised and are not recommended where parts are needed for these requirements. The melting temperature decreased when irradiated with a dose of up to 250 kGy due to molecular shear and then increased when irradiated with a dose of 250 up to 500 kGy due to molecular

cross-linking of the PPrec.

MFI tests revealed molecular shear occurring at radiation doses up to 50 kGy. Within this range, ionizing radiation effectively controlled molecular shear, producing shorter molecular chain lengths in PPrec. These modified polymers could serve as carriers for additives in paints, adhesives, and plastics or act as flow modifiers for the injection molding of large or thin-walled parts. Beyond 50 kGy, cross-link formation began, resulting in cross-linked polypropylene, which could be utilized as fillers for asphalt, construction materials, and other applications.

The Vicat softening temperature and thermal deflection temperature properties did not interfere with PPrec recycling. However, DSC tests indicated that increasing radiation doses reduced the crystalline melting temperature, potentially affecting the recyclability of PPrec and subsequent processing methods such as injection molding, extrusion, and other transformation techniques. TGA analysis showed minimal changes in mass loss at elevated temperatures, with degradation commencing near 400 °C in irradiated samples. Each application must consider the physical, thermal, and chemical properties of PPrec, with the radiation dose used. A radiation dose of up to 12.5 kGy, or even lower, proved sufficient for reusing trays and other plastic products, including injection-molded, blow-molded, thermoformed, and container-based items made from PPrec, reducing waste and promoting sustainability. It is widely recognized that high doses of ionizing radiation can cause significant chain scission and crosslinking in the processed polymers. While crosslinking can enhance tensile strength, it may also reduce MFI values. Additionally, radiation-induced oxidation can lead to polymer discoloration at lower doses than those needed to noticeably affect mechanical properties. Evaluation via DSC, MFI, and crosslinking content indicated that an irradiation dose of 150 kGy is optimal for iPP intended for use in extreme conditions. The findings provide valuable insights related to the effects of EB radiation on PPrec. The thermal characteristics evaluated by TGA, DSC, MFI, Vicat B, and HDT, as well as the morphological (XRD) properties observed under the action of ionizing radiation, enable a broader exploration of PPrec applications in various plastic components, facilitating a critical assessment of recyclability, cost, and environmental impact.

CRediT authorship contribution statement

Julio Harada: Writing – review & editing, Writing – original draft, Visualization, Validation, Project administration, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Juliana Arquinto:** Writing – review & editing, Validation, Supervision, Data curation. **Maria da Conceição Costa Pereira:** Writing – review & editing, Visualization, Validation, Formal analysis. **Maria Elizabeth Maués dos Santos:** Visualization, Validation, Investigation. **Dione Pereira de Castro:** Writing – review & editing, Investigation, Formal analysis, Data curation. **Traian Zaharescu:** Writing – review & editing, Validation, Supervision, Investigation, Formal analysis. **Leonardo Gondim de Andrade e Silva:** Writing – review & editing, Visualization, Supervision, Project administration, Funding acquisition, Formal analysis.

Declaration of competing interest

The authors don't have a conflict of interest in this manuscript.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.radphyschem.2026.113711>.

Data availability

Data will be made available on request.

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