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Chemical, Structural and Thermal Analysis of PET Flakes induced by Electron Beam Irradiation

Nor Batrisya Ismail[1], Mohd Hamzah Harun[1*], Izzuddin Mohamad Zaharuddin[1,2], Norfazlinayati Othman[2], Mahathir Mohamed[1], Mohd Sofian Alias[1], Mohd Faizal Abd Rahman[1], Khairil Nor Kamal Umar[1], Nurul Huda Mudri[1], Khairul Azhar Abdul Halim[1], Rida Tajau[1], Rusdyana Natasa Binti Ghazali[2], Siti Nur Eyisha Wafa Mohd Aminuddin[1]

[1] Radiation Processing Technology Division, Malaysian Nuclear Agency, Bangi, 43000, Kajang, Selangor, Malaysia
[2] Department of Physics, Faculty of Science, Universiti Putra Malaysia, 43400, Serdang, Selangor, Malaysia

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Corresponding Author:
Mohd Hamzah Harun
hamzah@nm.gov.my

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Abstract: This study presents a comprehensive chemical, structural, and thermal characterization of polyethylene terephthalate (PET) flakes subjected to electron beam irradiation at doses of 0, 40, and 120 kGy. Post-consumer PET bottle flakes were analyzed using Fourier Transform Infrared Spectroscopy (FTIR), X-ray Diffraction (XRD), and Thermogravimetric Analysis (TGA) to determine irradiation-induced modifications. FTIR results reveal noticeable variations in key functional groups, particularly in the ester (C=O and C–O) and aromatic regions, indicating chain scission, partial cross-linking, and oxidation with increasing radiation doses. XRD analysis shows a progressive reduction in peak intensity and an increase in peak broadening, signifying decreased crystallinity and enhanced amorphous character due to structural disorder. TGA measurements demonstrate improved thermal stability of irradiated PET, with major degradation shifting from ~450 °C in the non-irradiated sample to ~480 °C after irradiation, and a clearer two-stage degradation pattern associated with structural rearrangements. These findings confirm that electron beam irradiation induces significant yet controlled modifications on PET's molecular structure, crystallinity, and thermal behavior. Such property alterations highlight the potential of electron beam treatment as an effective pre-processing approach to enhance the recyclability and performance of waste PET, contributing to more sustainable plastic waste management strategies.

Keywords: Electron beam; Irradiation; PET flake

Introduction

Over the past few decades, there has been an extraordinary rise in the consumption of poly (ethylene terephthalate) (PET) products, leading to the growth of massive quantities of PET trash (Jamalzadeh and Sobkowicz, 2022). PET is a soft, transparent thermoplastic with a high melting point of 265 °C and excellent mechanical strength, at least up to 175 °C due to the aromatic ring in the polymer backbone. Applying radiation in polymers is essential because its use helps to achieve desired enhancements in the properties of the polymer. Considering ecological and economic considerations, chemical recycling has been considered to be one of the best ways of dealing with the problem of PET waste accumulation (Jamdar et al., 2017). PET is considered to be the most recyclable commodity plastic; at present, since the recycled product is rather costly, only a small portion of this polymer actually gets recycled. The main issue that renders recycling impractical tends to be its inefficiency especially when it comes to energy consumption. Research on PET recycling is being confronted with new issues such as determining how to recycle PET at a reasonable cost, optimizing the process to enhance conversion to monomeric units, reducing reaction times, and developing eco-friendly alternatives for the process (El-Saftawy et al., 2014). Therefore, understanding the characteristics of PET waste is crucial in order to overcome the problem. In the present work, PET flakes

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were exposed to electron irradiation and several characterizations have been made in order to investigate its properties include structural, thermal and chemical characteristics.

Method

The waste plastic bottles used for mineral or drinking water as PET sources were used. After being collected, the bottles were sent a crusher to be crushed into a fine flakes from micron to submicron size. After being cleaned, the material was filtered to get rid of bigger particles, producing a fine PET powder that could be used for more research. Following that, we made seven PET samples, each weighing roughly five kilograms. The electron beam radiation doses that were intended to be applied to these samples were 0, 40 and 120 kGy. The arrangement of samples were done carefully in order all samples received the same amount of electron beam irradiation and the sample arrangement can be seen in Figure 1.

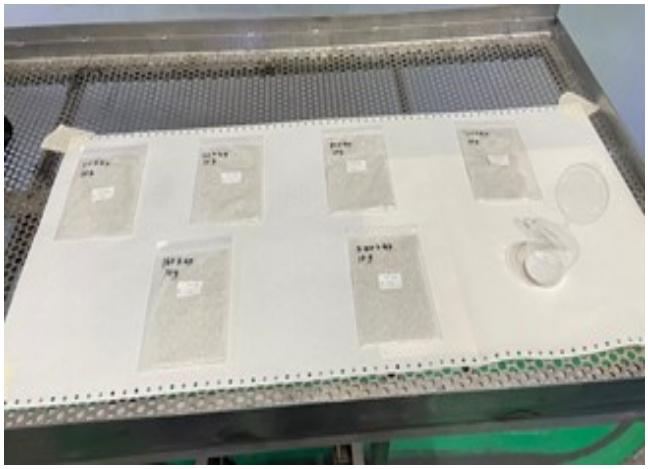

Figure 1. PET samples setup for electron beam irradiation Characterization

The IR spectra for the PET flakes were observed using FTIR spectrophotometry (Bruker Tensor II, Spectrum 2000). All spectra were obtained using the attenuated total reflection (ATR) method. A total of 32 scans were collected within a wavenumber range of 500–4000 cm−1, with a resolution of 4.0 cm−1 for all recorded spectra. The thermal analysis was measured using TGA model TG 209 F3 Tarsus (Netzch). The thermogravimetric analysis (TGA) records the weight fluctuation, which is the weight loss during heating as a function of temperature corresponding to the evolution of volatile compounds, thermal degradation of the PET material. The crystallinity degree of the PET flakes was characterized using X-ray diffraction (XRD) of a PAN Analytical X'pert PRO MPD with Cu Kα1 radiation source (λ = 1.5406 Å), operating at 45 Kv and 40 mA.

Result and Discussion

FTIR Result

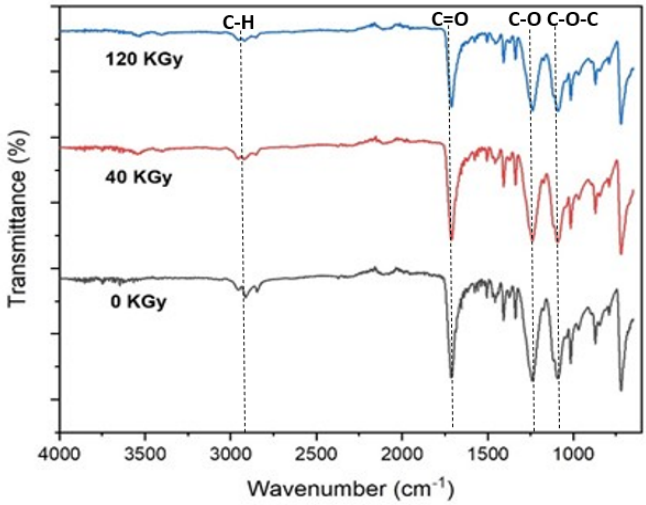

Figure 2. FTIR spectra for PET samples at different e-beam irradiation exposures

The Fourier Transform Infrared Spectroscopy (FTIR) graph shows the transmittance (%) of PET samples exposed to different doses of electron beam irradiation (from 0, 40 and 100 kGy). The control sample (cs), which has not been exposed to any electron beam radiation show peaks in the spectrum correspond to various functional groups and bonds within the PET polymer. As the radiation dose increases, changes in the peak intensities and positions can be observed, indicating alterations in the polymer structure. On 1715 cm^{-1} peak shows a prominent peak, indicating the presence of ester groups in PET. The intensity of this peak slightly decreases with increasing radiation dose, suggesting possible scission or alteration of ester groups due to electron beam exposure (Jamalzadeh and Sobkowicz, 2022). At the wavenumber 1245 cm^{-1} a clear peak is present, typical for ester bonds in PET. As radiation doses increase, the peak remains but shows slight changes in intensity, implying minor modifications in the ester linkage. For 1103 cm^{-1} representing the ether linkages in PET. Peak intensity variations indicate some structural changes in the ether linkages, with higher doses possibly causing more significant alterations. Meanwhile at peak 870 cm^{-1} there are presence of characteristic of aromatic rings in PET.

As the radiation doses increase, the peak intensity remains relatively stable, suggesting the aromatic structure is less affected by the radiation compared to other groups. On the other hand, 732 cm^{-1} is typically related to the benzene ring vibrations. Shifts or changes in these peaks can indicate changes in the aromatic structure of the polymer due to radiation exposure. The peak shows minimal changes, indicating that the aromatic rings in PET are fairly resistant to electron

beam radiation. The increase in the intensity of the carbonyl peak with higher radiation doses suggests the formation of new carbonyl containing degradation products likely from the scission of the polymer chains and subsequent oxidation. Changes in ester and ether Peaks (1245 cm^{-1} and 1103 cm^{-1}) indicates variations in these peaks can be attributed to alterations in the ester and ether linkages which may result from chain scission or cross-linking induced by gamma irradiation (Singh et al., 2018). For peak 870 cm^{-1} and 732 cm^{-1}, shifts or changes in these peaks suggest modifications in the aromatic ring structure, potentially due to the breakage of the aromatic rings or changes in the ring-substituted groups. Table 1 shows the the list of functional groups identified.

Table 1. IR spectroscopy of specific groups involved in the absorption of the PET flakes

Name	Absorption Curve (cm^{-1})	Corresponding Species	Vibration Types
C-H	2967	C H	Stretching
C=O	1714	Carboxylic acid	Stretching
C-O	1241	Ester	Stretching
C-O-C	1017	Ester	Bending

XRD result

PET often shows distinctive diffraction peaks at particular 2θ values that can be used as benchmarks to locate crystalline areas. This peak (2θ ≈ 17°) indicates a particular crystallographic orientation within the polymer and corresponds to the PET (010) plane. It is one of the primary PET's crystalline structure reflections. This peak (2θ ≈ 26°) corresponds to PET's (100) plane, which is another notable reflection that shows crystalline regions exist inside the polymer matrix. From the graph, the observations that can conclude are characteristic peaks, peak intensity peak broadening and shift in peak position. For characteristics peak, the XRD patterns display two major diffraction peaks, one around 2θ≈17∘and another around 2θ≈25∘. These peaks are characteristic of the crystalline regions in PET, corresponding to specific planes in the crystalline lattice. For peak intensity, the control sample shows distinct peaks with relatively high intensity indicating a certain degree of crystallinity in the PET polymer. As the radiation dose increases, there are slight variations in peak intensities. Generally, the peaks tend to become less sharp and slightly lower in intensity at higher doses. Meanwhile for peak broadening, the width of the peaks appears to increase slightly with higher radiation doses, particularly noticeable in the 25° peak. This broadening suggests an increase in structural disorder and a decrease in crystallite size due to radiation exposure. There is no significant shift in the peak positions with increasing radiation dose, indicating that the overall lattice parameters remain relatively unchanged (El-Saftawy et al., 2014).

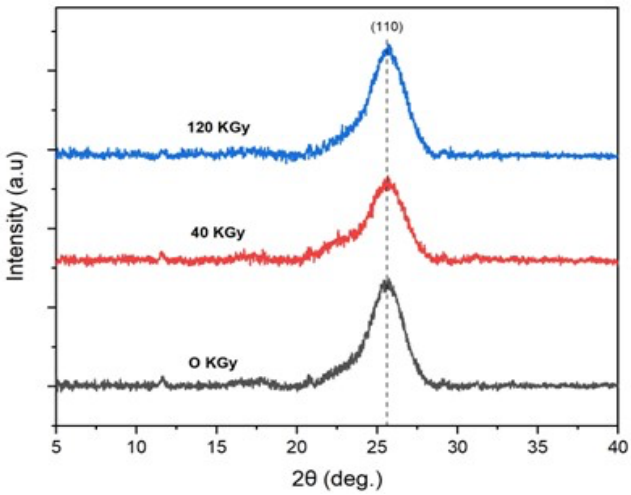

Figure 3. XRD spectra of PET samples at different e-beam irradiation exposures

The primary changes are in the intensity and sharpness of the peaks. The intensity and sharpness of the XRD peaks are indicative of the degree of crystallinity in the PET samples. The control sample has sharp peaks, indicating a well-defined crystalline structure. With increasing doses of electron beam radiation, the peaks become broader and less intense indicate a reduction in crystallinity. This implies that radiation induces some amorphization or disruption of the crystalline regions in PET. The broadening of the peaks can be associated with a decrease in crystallite size and an increase in structural disorder.

Thermogravimetric Analysis

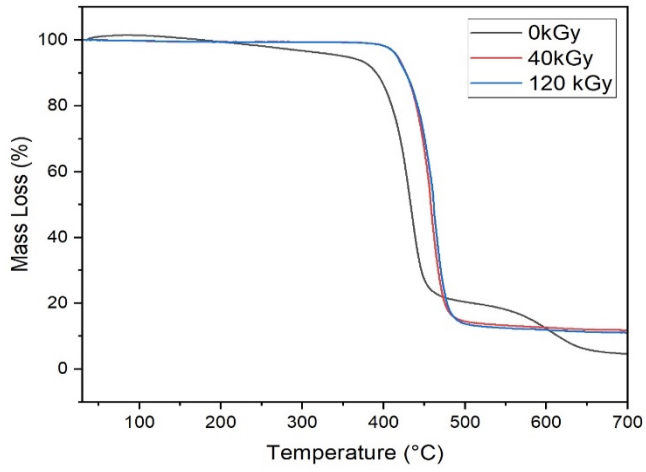

Figure 4. TGA curves of PET samples at different e-beam irradiation exposures

The thermal stability and thermal degradation electron beam induced PET flakes was studied by TGA analysis with a temperature range from 35 ℃ to 700 ℃. It is quite obvious that there are two stage degradation where the first region is approximately at 200 ∘C. It is attributed to the quaternized degradation due to graft

PET. Approximately 6% weight loss distinguished at this stage. At about 480 °C, the major weight loss occurred at about 78% of the total weight which is due to the evaporation of carbon and oxygen. In addition, the major weight loss for unirradiated PET flakes occurred earlier which is approximately at 450 °C. This indicates the thermal stability of PET flake is significantly improved after exposing to electron irradiation {Ragaert et al., 2017).

Conclusion

The FTIR analysis indicated changes in the functional groups of PET polymer with increasing radiation doses. Significant peaks corresponding to characteristic functional groups such as ester (C=O) and alkyl (C-H) groups showed variations in intensity and position. These changes suggest radiation-induced modifications such as chain scission, cross-linking, and oxidation, affecting the chemical structure of PET. The XRD analysis revealed changes in the crystalline structure of PET due to radiation exposure. There were noticeable shifts in peak positions, intensity, and broadening of diffraction peaks with higher radiation doses. These observations indicate a reduction in crystallinity and the introduction of defects and amorphous regions in the polymer matrix. The study demonstrates that electron beam radiation induces significant structural modifications in PET polymer, affecting both crystalline and amorphous regions. These modifications include chain scission, cross-linking, and oxidation. The induced structural and thermal changes can affect the material's mechanical properties, barrier performance, and suitability for specific uses. The thermal composition of the irradiated PET improved particularly on the residue formation at higher The PET irradiated properties effect is very minor due to the stable pristine structure of the PET that resulted to the small and waver enhancement of the PET, although after being irradiated by high ionization energy. Understanding the effects of radiation on PET is crucial for optimizing its performance in various applications.

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Author Contributions
Conceptualization, M. H. H, M. H and M. S. A; methodology, K. A. K.; software, N. O and K. A. K.; validation, M. H. H, M. H, R. T., M. F. A. R. and M. S. A; formal analysis, K. A. K, I. M. Z. and S. N. E. W. M. A.; investigation, K. A. K, I. M. Z. and S. N. E. W. M. A.; resources, M. H. H.; data curation, K. A. K, I. M. Z., K. N. K. U. and S. N. E. W. M. A.; writing—original draft preparation K. A. K. and M. H. H.; writing—review and editing, M.H. and M. S. A.; visualization, M. H. H. and M. S. A; supervision, M. H. H and M. S. A.; project administration, M. S. A. and M.H. H.; funding acquisition, M.H.H. All authors have read and agreed to the published version of the manuscript.

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Conflicts of Interest
The authors declare no conflict of interest.

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