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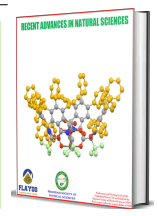

# Impacts of high-dose gamma irradiation on the mechanical, structural and thermal properties of doum fiber reinforced High-Density Polyethylene (HDPe)

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ABSTRACT

High-density polyethylene (HDPe) has widespread application in pharmaceutical packaging and medical devices since it is biocompatible and stable. Research on the impact of high-dose gamma irradiation on structural, thermal, and mechanical properties of Doum fiber-reinforced HDPe composites at 0 wt.%, 20 wt.% and 40 wt.% is required due to the composite nature of the material. The research involves synthesis and analysis of gamma irradiation impacts properties of biomass-reinforced HDPe. The samples were irradiated at 0-150 Gy, and then subjected to mechanical testing, thermogravimetric analysis (TGA), and Fourier transforms infrared spectroscopy (FTIR). The findings illustrated that gamma irradiation at 100 Gy greatly improved tensile strength, hardness, and impact strength because of enhanced fiber-matrix interaction and crosslinking effects. Tensile strength was improved from 16.93 MPa (0 Gy) to 26.80 MPa, hardness was improved from 53.83 Kgf/mm$^2$ to 70.43 Kgf/mm$^2$, and impact strength was optimum at 1.0187 J/mm. Mechanical properties at 150 Gy were compromised because of degradation of the polymer via chain scission. TGA analysis indicated improved thermal stability at 100 Gy, as manifested by the increased onset decomposition temperature, while at 150 Gy, degradation and oxidative effects led to decreased stability. FTIR analysis indicated structural change, in accordance with higher crosslinking at 100 Gy and degradation at 150 Gy. The results confirm that the modest irradiation (100 Gy) is accountable for optimum mechanical and thermal properties and higher irradiations (150 Gy) cause degradation. It also optimizes the performance of Doum fiber-reinforced HDPe composites to a maximum at 40 wt.% fiber content, and to meet high-performance structural applications in medical, pharmaceutical, and aerospace applications.



## 1. INTRODUCTION

The increasing demand for lightweight, high-performance, and environmentally friendly materials has led to significant research

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into polymer composites reinforced with natural fibers [1, 2]. High-Density Polyethylene (HDPe), a thermoplastic polymer widely used in packaging, automotive, and construction industries, offers excellent chemical resistance, process ability, and mechanical properties. However, its inherent limitations, such as low mechanical strength and thermal stability, restrict its use in high-performance applications [3]. To address these challenges, reinforcement of HDPe with natural fibers has been a viable alternative.

Among various natural fibers, Doum palm (Hyphaene thebaica) fiber is especially notable due to its mix of mechanical behavior, availability at the local level, biodegradability, and affordability. Available in high volume in African dry and semi-dry regions, Doum fiber is readily available at low cost and hence a very prospective substitute for other natural fibers such as flax, jute, or sisal. Moreover, Doum fiber possesses excellent mechanical properties such as high tensile strength and stiffness, which are ideal for structural reinforcement of polymer matrices [4, 5]. Doum palm fruits, primarily found in arid regions of Africa, has shown promising reinforcement capabilities by improving tensile strength, impact resistance, and flexural strength in polymer matrices [4]. However, fiber-matrix interfacial adhesion remains a critical factor affecting the overall performance of these composites, necessitating surface modifications and processing techniques to optimize their properties.

One effective methods for enhancing polymer composites is gamma irradiation, which induces polymer cross-linking, thereby improving mechanical strength, thermal stability, and fiber-matrix adhesion [6]. Gamma irradiation has been reported to enhance the structural integrity of HDPe-based composites by modifying crystallinity, reducing moisture absorption, and increasing resistance to environmental degradation [3, 5]. However, despite the extensive research on gamma-irradiated polymer composites, limited studies have specifically examined the effects of high-dose gamma irradiation on HDPe reinforced with Doum fiber.

The lack of sufficient data on optimal irradiation dosage and its effects on mechanical, structural, and thermal properties has hindered the full utilization of these composites in demanding applications [7]. This study is driven by the following research questions: What is the effect of high-dose gamma irradiation on the mechanical, thermal, and structural properties of HDPe/Doum fiber composites? What is the optimal dose of irradiation that yields the best composite performance without compromising structural integrity? How do the 20 wt.% and 40 wt.% fiber contents influence the irradiation response of the composites?

The general hypothesis is that moderate gamma doses (e.g., 100 Gy) would enhance the thermal and mechanical behavior of HDPe/Doum fiber composites due to stronger crosslinking and increased adhesion between fibers and matrix, while higher gamma doses (e.g., 150 Gy) can lead to degradation and decreased performance. To test this hypothesis, HDPe composites reinforced with 20 wt.% and 40 wt.% Doum fiber were synthesized and exposed to various doses of gamma radiation. Tensile strength, thermal stability, and chemical structure were assessed by mechanical testing, thermogravimetric analysis (TGA), and Fourier Transform Infrared Spectroscopy (FTIR). Findings of this research are aimed at informing the formulation of optimized, radiation-modified bio-composites for use in demanding industries like aerospace, automotive, and medical.

In this research, HDPe/Doum fiber composites were fabricated and subjected to high-dose gamma irradiation to evaluate the impacts of gamma irradiation on their mechanical, structural, and thermal properties. The study aimed to determine the optimal radiation dosage that enhances the composite's overall performance while maintaining structural integrity. The findings provide critical insights into the potential industrial applications of HDPe/Doum fiber composites in sectors requiring materials with superior mechanical and thermal stability, such as aerospace, automotive, and construction. By addressing existing gaps in knowledge, this research contributes to advancing the development of radiation-modified natural fiber-reinforced composites for high-performance applications.

## 2. MATERIALS AND METHOD

### 2.1. SYNTHESIS OF DOUM-HDPE COMPOSITE

Doum palm leaves were collected from Lau, Taraba State, Nigeria and the leaves were sorted, washed, and dried. The dried leaves were then shredded into 5 millimeters (mm) pieces using a laboratory shredding machine to ensure uniform size for composite fabrication. The High-Density Polyethylene (HDPe) polymer, with a purity of 99.9 percent (%), was used as the matrix material for reinforcement. Three Doum- HDPe composites were synthesized with varied weight percentages (wt.%s) of Doum namely 0, 20 and 40 % while the corresponding wt. %s of HDPe in the samples were 100% for sample A, 80% for sample B and 60% for sample D. In the mixing process, of HDPe pellets were softened on a two-roll mill at 210 degree Celsius (°C) for five minutes before gradually incorporating the shredded Doum fiber. The materials are mixed thoroughly for three minutes to achieve a uniform composite structure. The resulting composite is sheeted out and labeled accordingly for further processing. The composite was molded using a hydraulic hot press. It was placed in a metal mold of dimensions 120 millimeters (mm) x 100mm x 5mm and subjected to 160°C and 2.5 Mpa pressure for five minutes. After hot pressing, the sample is cooled using a hydraulic non-hot press and removed from the mold as shown in Figure 1. The composite is then cut into the required dimensions for mechanical testing and gamma irradiation studies as shown in Figure 2.

### 2.2. MECHANICAL AND THERMAL CHARACTERIZATIONS

Mechanical and thermal testing was done, including tensile strength, impact strength, hardness, and thermogravimetric analysis (TGA).

Tensile strength was performed as per ASTM D-638. Dumbbell-shaped sample A was applied with a tensile force and tensile strength, tensile modulus percentage elongation at break of every sample were computed and recorded automatically by the machine and the findings were on the certificate.

Impact test was performed according to the standard provided ASTM D-256. The specimen was cut to dimensions 64mm × 12.7 mm × 3.2 mm and 45° notched were inserted in the middle of the test specimens of all the composite samples prepared. Impact energy test was performed on Izod Impact Tester (Resil impactor testing machine). The test specimen was clamped ver-

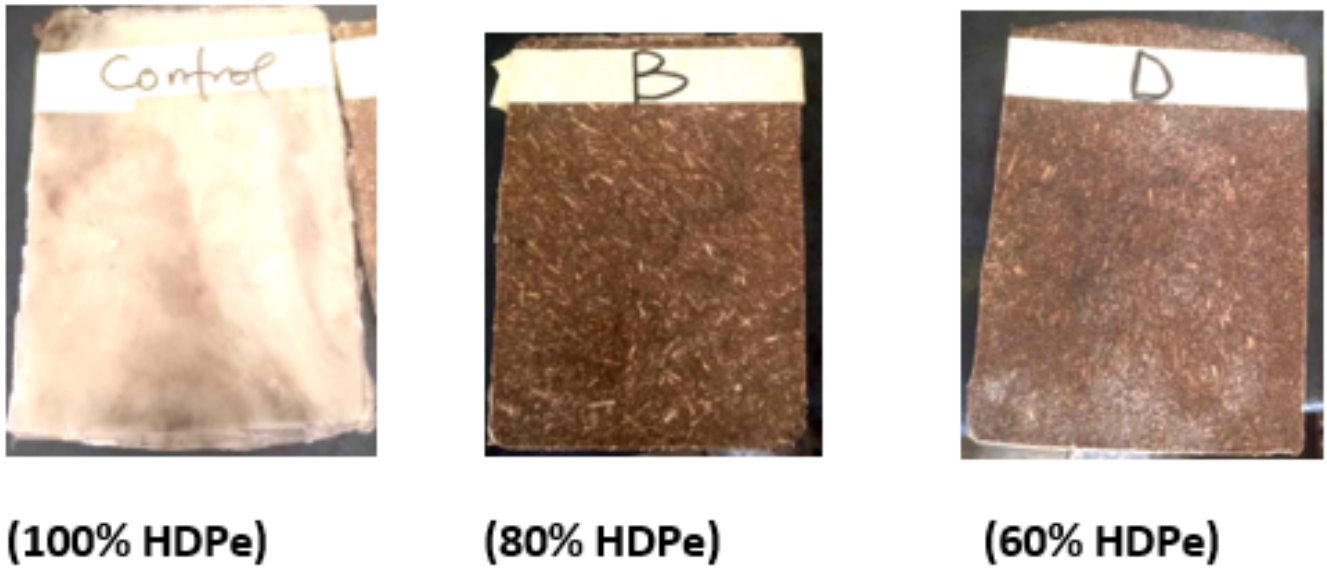


**Figure 1.** Synthesized Doum-HDPe Composites.

tically on the machine jaw and 1500 Newton (N) weight hammer was dropped from an inclined angle of 152°. Impact energy for corresponding test specimen was recorded and measured.

The hardness test was carried out in accordance with ASTM E18-79 a MicoVicker Harness Tester. The test was carried out at different positions on each sample before and after gamma irradiation and average hardness was calculated using the formula:

$$\mathrm{Hv} = \frac{1.854 \times \mathrm{F}}{\mathrm{d}^2}, \tag{1}$$

where F is the applied load in kilograms-force (kgf), d is the diagonal length of the indentation measured in millimeters (mm) [8].

### 2.3. THERMOGRAVIMETRIC ANALYSIS (TGA)

The Q5000 IR thermo-analyzer (TA Instruments) was used for thermogravimetric analysis (TGA) in order to determine the thermal stability of the samples and quantify their fraction of volatile compounds. TGA involved determination of the sample's weight change with heating under constant rate within atmospheres of nitrogen and air. The machinery functioned on a principle that constantly weighed the sample as it warmed, where an inert gas swept over the sample. Gaseous products evolved during processing were pumped off and the mass changes in the leftover samples were registered automatically. The samples were placed in an alumina crucible with masses ranging from 11 to 18 milligram (mg). Composite samples were scanned in an open platinum (Pt) pan from 25 to 800 °C at a heating rate of 10 °celsus per minute (C/min) for the irradiated composites.

The thermal stability of the composite is influenced by the degradation kinetics under gamma irradiation. The weight loss during thermal decomposition can be modeled using the Arrhenius equation:

$$\frac{dW}{dT} = A exp\left(-\frac{E_a}{RT}\right), \tag{2}$$

where A is pre-exponential factor, $E_a$ activation energy for thermal decomposition (J/mol), R is universal gas constant (8.314 J/mol·K), T is absolute temperature (K) [8].

The activation energy ($E_a$) can be calculated from TGA data using the Kissinger equation:

$$ln\left(\frac{\beta}{T_p^2}\right) = -\frac{E_a}{RT_p} + ln\,\frac{AR}{E_a}, \tag{3}$$

where $\beta$ is heating rate Kelvin per minute (K/min), $T_p$ is peak temperature of degradation (K) Changes in $E_a$due to gamma irradiation indicate alterations in the thermal stability of the composite [8, 9].

### 2.4. FTIR CHARACTERIZATION

The FTIR spectrums of the composite samples were examined using the FTIR thermo Nicolet with Model No. Nexus 870 at Ahmadu Bello University Zaria, a background spectrum without sample was run for calibration. Using tweezers the sample placed on the crystal surface. Carefully the sample was spread on the crystal until it is covered, then the pressure clamp assembly was swing so that the tip is right above the sample. Then the clamp was Screw down until the sample is firmly trapped between the tip and the opposing base. On the computer, Sample measurement was clicked to analyze the sample. The sample was removed carefully to make sure no particle remain on the crystal, then using a cotton bud the crystal surface was wipe off. The samples were analyzed at a range of 4000 – 650 per centimeter ($cm^{-1}$) wave number. This process was repeated for all the samples before and after gamma irradiation.

### 2.5. GAMMA IRRADIATION

Irradiations were performed using a gamma radiation source and a properly calibrated Cobalt-60 (Co-60) ionization chamber connected to an electrometer. The ionization chamber was set at an 80 cm Source to Surface Distance (SSD) and a radiation field size of 35 centimeters (cm) × 35 cm. Prior to irradiation, the ionization chamber was pre-irradiated in air with a buildup cap to stabilize the response and provide reproducible dose rate output. Irradiation was performed at a controlled dose rate of 0.21 Gy/min, which was maintained constant throughout the exposure time by real-time monitoring and feedback control. The dose of gamma photon irradiation needed was calculated from the relation:

$$\text{Dose} = \textit{Dose Rate} \times \textit{time}, \tag{4}$$

The dose rate was controlled and monitored during exposure to achieve the desired radiation dose levels of 100Gy and 150Gy. The HDPe-Doum fiber composite samples were systematically exposed to the gamma radiation field under controlled conditions. The respective individual samples were exposed to the respective doses under uniform exposure to prevent variations in radiation absorption.

The Doum fiber in the composite is primarily composed of cellulose, which undergoes degradation under gamma irradiation. The degradation degree of cellulose can be expressed as:

$$D_c = -\frac{DP_r}{DP_0}, \tag{5}$$

where $D_c$ is degree of cellulose degradation, $DP_0$ and $DP_r$ initial and residual degree of polymerization of cellulose higher $D_c$ values correspond to more significant degradation of the fiber, affecting the composite's overall properties.

## 3. RESULTS AND DISCUSSION

### 3.1. MECHANICAL TEST RESULTS OF THE COMPOSITES

The mechanical properties of HDPe Reinforced Doum Fiber Composites were evaluated under different gamma irradiation doses (0Gy, 100Gy, and 150Gy) to assess the impact of high-dose gamma irradiation on the mechanical properties. The results from tensile strength, impact strength, density, and hardness are analyzed in the Table 1.

For sample A, 100 Gy irradiation caused a ~22% increase in the tensile strength while for 150 Gy irradiation the tensile strength of the material was seen to have only increase by ~10%. For sample B with 20 wt. % of doum fiber, the tensile strength was seen to increase with 32% after irradiation with 100 Gy of gamma beam. This demonstrates that both the irradiation (with 100 Gy) and incorporation of doum fiber in the polymer metrix had impacted of the tensile strength of the composite. However, in all three samples, irradiation at 150 Gy had resulted in reduced tensile strength suggesting that the optimized dose for irradiation of doum reinforced HDPe is 100 Gy. The tensile strength of the composites increased significantly with gamma irradiation up to 100 Gy, as shown in Table 1 indicating enhanced crosslinking within the polymer matrix. At 0 Gy, the control sample exhibited the lowest tensile strength (16.93 Mpa), while samples B (19.59 Mpa) and D (20.98 Mpa) showed slightly improved strength, due to fiber reinforcement. After irradiation at 100 Gy, a notable increase in tensile strength was observed across all samples. The control increased to 20.62 Mpa, while samples B (25.95 Mpa) and D (26.80 Mpa) exhibited the highest tensile strength. This suggests that irradiation enhanced fiber-matrix adhesion and polymer crosslinking, improving mechanical performance. However, at 150 Gy, tensile strength decreased for all samples. The control dropped to 18.62 Mpa, while B (20.65 Mpa) and D (23.46 Mpa) still maintained higher strength than the unirradiated samples. The reduction in tensile strength at higher irradiation levels may be due to excessive chain scission, leading to polymer degradation.

The results align with [7], who observed that natural fiber reinforcement in HDPe composites can lead to a 20-25% improvement in tensile strength. The results here suggest good fiber dispersion and adhesion, even before gamma irradiation, as the tensile strength of fiber-reinforced samples increased by up to 24% compared to the control sample. Statistical calculation (one-way ANOVA, $p < 0.05$) confirmed that improvement of tensile strength at 100 Gy was considerably different from those of 0 Gy and 150 Gy levels, specifically in the fiber-reinforced ones.

The result for Hardness test showed a consistent increase with irradiation up to 100 Gy. The control sample increased from 53.83 Kgf/mm$^2$ to 58.43 Kgf/mm$^2$, while B and D reached 70.43 Kgf/mm$^2$ and 68.80 Kgf/mm$^2$, respectively as shown in Table 1. This suggests that gamma irradiation at moderate doses improved the material's resistance to deformation. At 150Gy, a slight decline in hardness was observed across all samples (57.57 Kgf/mm$^2$ for control, 68.67 Kgf/mm$^2$ for B, and 66.80 Kgf/mm$^2$ for D). This reduction may be due to overexposure leading to polymer degradation and microstructural defects. This result aligns with [7], who demonstrated that natural fiber-reinforced composites tend to have higher hardness compared to neat polymer matrices.

The impact strength follows a similar trend. At 0 Gy, the control sample showed an impact strength of 0.1373 J/mm, while B and D were 0.1399 J/mm and 0.1272 J/mm, respectively. Upon irradiation at 100 Gy, the impact strength increased significantly in samples B (1.0187 J/mm) and D (0.8917 J/mm), suggesting enhanced energy absorption capacity due to improved fiber-matrix interaction. However, at 150 Gy, impact strength decreased (0.8073 J/mm for the control, 0.887 J/mm for B, and 0.7897 J/mm for D), likely due to excessive radiation-induced brittleness, which reduced toughness. These results suggest that while doum fibers improve tensile strength and hardness, they reduce the composite's toughness, making the material more brittle as in Table 1. A significant increase in density was observed after 100 Gy irradiation, with the control sample increasing from 0.996 g/cm$_3$ to 1.6627 g/cm$_3$. Similarly, samples B and D showed increased density values (1.7760 g/cm$_3$ and 1.7840 g/cm$_3$, respectively). This increase can be attributed to polymer densification due to irradiation-induced crosslinking. However, at 150Gy, the density decreased slightly (1.6397 g/cm$_3$ for sample A, 1.6293 g/cm$_3$ for B, and 1.6373 g/cm$_3$ for D), which may indicate partial degradation and void formation in the material. The small variations in density as shown in Table 1 indicate that the inclusion of Doum fibers and the fabrication process did not drastically alter the composite's density. This result is consistent with the findings of [10], who reported minimal changes in den-

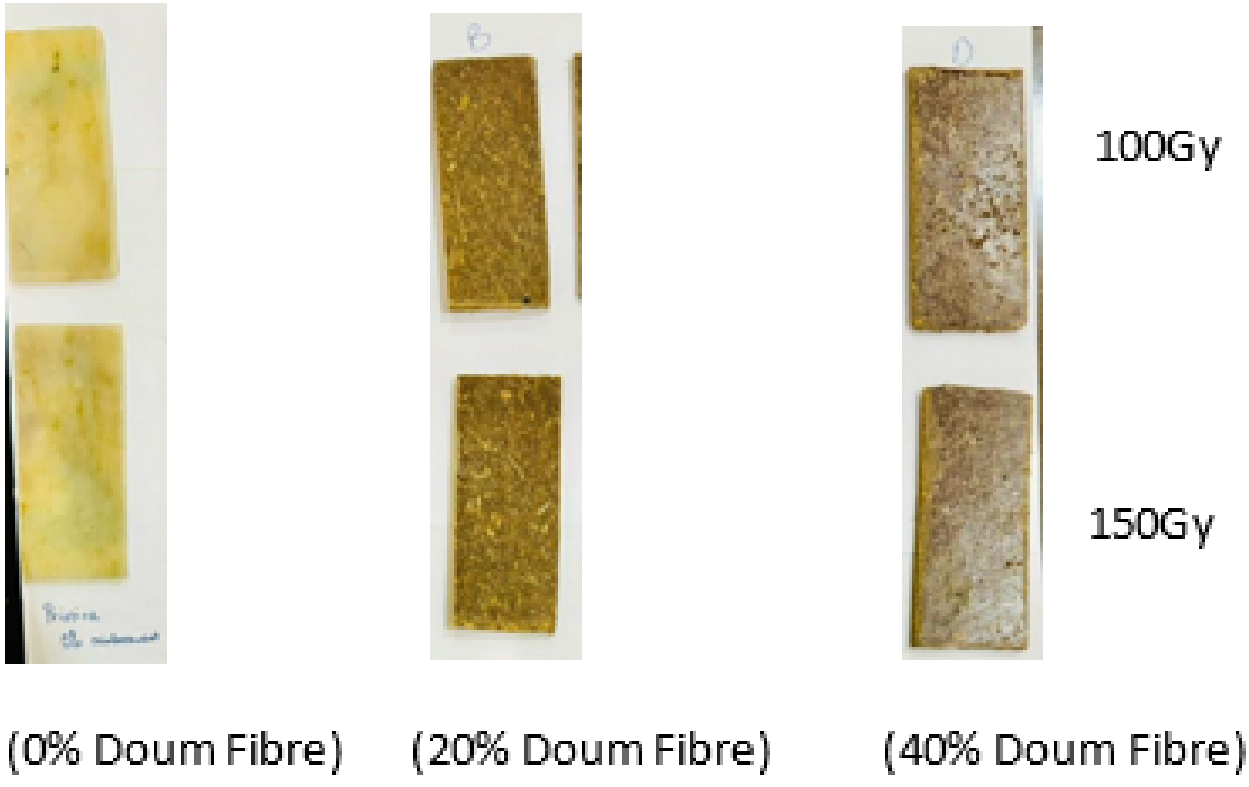


**Figure 2.** Composite samples for gamma irradiation.

**Table 1. Mechanical test results.**

| Dose | Sample ID | Tensile Strength (Mpa) | Impact strength (J/mm) | Density (g/cm$^3$ ) | Hardness (Hv) |
|---|---|---|---|---|---|
| 0 Gy | A | 16.93 ± 1.421 | 0.14 ± 0.001 | 0.996 ± 0.014 | 53.83 ± 0.252 |
| | B | 19.59 ± 0.806 | 0.14± 0.115 | 0.986± 0.020 | 60.67 ± 0.896 |
| | D | 20.98 ± 1.017 | 0.13 ± 0.008 | 0.994 ± 0.008 | 63.10± 0.173 |
| 100 Gy | A | 20.62 ± 0.445 | 0.993 ± 0.007 | 1.6627±0.0378 | 58.43 ± 0.153 |
| | B | 25.95 ± 0.641 | 1.019 ± 0.015 | 1.78 ± 0.046 | 70.43 ± 0.100 |
| | D | 26.80 ± 0.44 | 0.89 ± 0.006 | 1.78 ± 0.037 | 68.80± 0.100 |
| 150 Gy | A | 18.62 ± 0.240 | 0.81 ± 0.045 | 1.64 ± 0.049 | 57.57 ± 0.252 |
| | B | 20.65 ± 0.035 | 0.89 ± 0.009 | 1.63 ± 0.081 | 68.67 ± 0.153 |
| | D | 23.46 ± 0.440 | 0.79 ± 0.006 | 1.64 ± 0.055 | 66.80 ± 0.100 |

NB: sample A has 0 wt. % of doum fiber, sample has 20 wt.% of doum fiber and sample D has 40 wt.% of doum fiber

sity when natural fibers were added to polymer matrices. The slight decrease in density in some samples could be attributed to the relatively low density of natural fibers compared to HDPe, which has been observed in other studies, such as those by, where fiber-reinforced composites showed density changes of less than 2% [10].

The observed improvements in mechanical properties at 100 Gy align with findings in existing literature. Abdullahi *et al.* Ref. [11] reported that X-ray irradiation at doses of 5-20 Gy led to substantial improvements in tensile strength and thermal stability of waste coffee grounds reinforced HDPe composites. Similarly, [12] reported that gamma irradiation induced significant changes in the physical and mechanical properties of HDPe due to chain scission and crosslinking activities. However, the decline mechanical properties observed at 150 Gy suggests that higher doses of gamma irradiation may lead to polymer degradation [13]. Louahem [13] Noted that gamma irradiation can cause oxidative degradation or cross-linking in HDPe, affecting

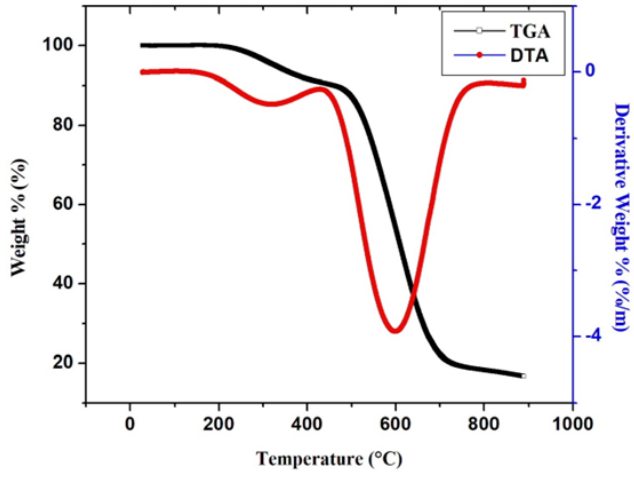


**Figure 3.** TGA/DTA Plot 0% Doum Fiber, Pre-Irradiated (0 Gy).

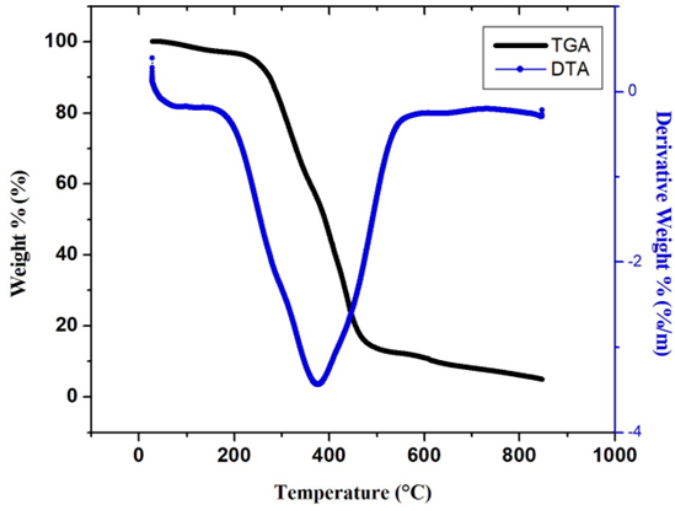


**Figure 4.** TGA/DTA Plot 0% Doum fiber, post irradiation (100 Gy).

its mechanical strength properties. Gamma irradiation at moderate doses (around 100 Gy) enhances the mechanical properties of HDPe Reinforced Doum Fiber Composites, while higher doses (150 Gy) may lead to degradation, corroborating findings in literature. The mechanical test results confirm that gamma irradiation at a dose of 100 Gy yields the optimum enhancement in tensile strength, impact strength, hardness, and density for Doum fiber-reinforced HDPe composites. Performance is decreased by scission and degradation of polymer chains at higher doses. The findings are statistically reliable and are consistent with previous results in the literature, indicating the potential of Doum fiber as an effective natural reinforcement for radiation-induced composites.

### *3.2. RESULTS OF THERMOGRAVIMETRIC ANALYSIS (TGA)*

#### 3.2.1. TGA results for sample A (0 Gy)

The Thermogravimetric analysis/Differential Thermal Analysis (TGA/DTA) plot depicts the weight loss of the control sample (0% Doum Fiber, Non-Irradiated) as a function of temperature. The curve shows two distinct phases of decomposition, indicating multiple degradation processes as shown in Figure 3. From Figure 3, the initial slight weight loss (below 200 °C) is attributed to the evaporation of moisture or other volatile compounds present in the polymer matrix. This is common in polymer, where low-temperature degradation primarily reflects the loss of absorbed moisture [13]. Main decomposition was observed between 300 and 450 °C showing that major weight loss occurs in this range, corresponding to the thermal degradation of HDPe matrix. HDPe typically undergoes significant decomposition between 350 and 450 °C, with the breakdown of polymer chains. The rapid weight loss in this region suggests that HDPe is thermally unstable above 300 °C.

The DTA plot (Figure 3), the heat flow associated with the polymer as it undergoes physical and chemical changes during heating. The first endothermic peaks (Below 150 °C) can be attributed to the evaporation of moisture and other low-energy processes, which aligns with the initial weight loss observed in the TGA curve. This suggests that the moisture content in the composite is relatively low. The sharp exothermic peak (around 350 to 450 °C) corresponds to the thermal degradation of the HDPe matrix. This peak represents the main combustion and degradation process of HDPe. The DTA peak correlates with the TGA's in this region thereby suggesting significant weight loss in the region. The TGA/DTA data of Sample A suggest typical thermal behavior of HDPe prior to gamma irradiation.

#### 3.2.2. TGA Results for Sample A Post Irradiation (100 Gy)

TGA/DTA result for Sample A post Irradiation (with 100 Gy) is presented in Figure 4 The results from the TGA and DTA plots highlight shifts in the decomposition patterns, thermal stability, and material integrity due to the irradiation.

Similar to the non-irradiated Sample A in Figure 4, the initial phase (below 200 °C) shows slight weight loss, likely due to the evaporation of moisture and low-molecular-weight volatiles. However, the 100 Gy irradiated sample shows a slightly reduced weight loss in this range compared to the non-irradiated sample, suggesting a potential cross-linking effect within the polymer matrix, which might have trapped some volatiles. This behavior is consistent with findings by Louahem [13], who reported that gamma irradiation induces cross-linking in polymer matrix, reducing the emission of volatiles. Main decomposition which indicates –polymer degradation, occurs at a slightly higher temperature for the 100 Gy irradiated sample A compared to the non-irradiated. This delay in thermal degradation suggests t gamma irradiation at 100 Gy has improved the thermal stability of the HDPe.

Manas *et al.* [10] observed a similar behavior in HDPe, where gamma irradiation at moderate doses increased the polymer's resistance to thermal degradation due to the formation of cross-linked networks within the polymer. For the irradiated sample, degradation starts later and progresses more slowly compared to the non-irradiated, which implies that gamma irradiation has strengthened the polymer bonding. Both the irradiated and non-irradiated samples show a small percentage of residual mass (at high temperatures (above 500 °C), but the irradiated sample retains slightly more mass. This indicates an increase in the ash

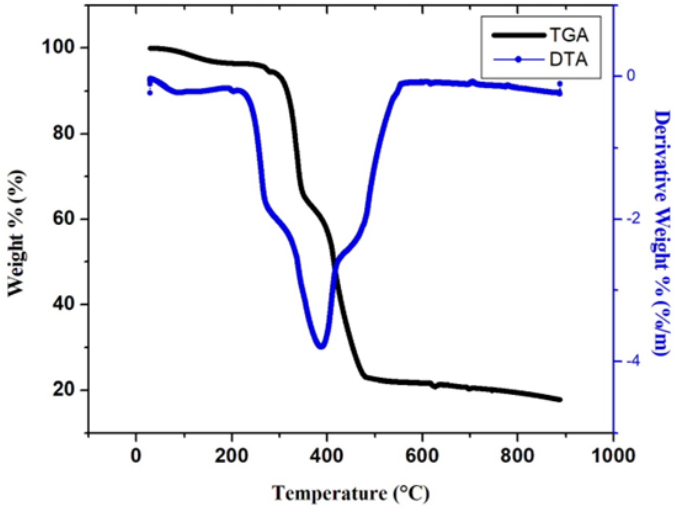


**Figure 5.** TGA/DTA Plot 0% Doum fiber, post irradiation (150Gy).

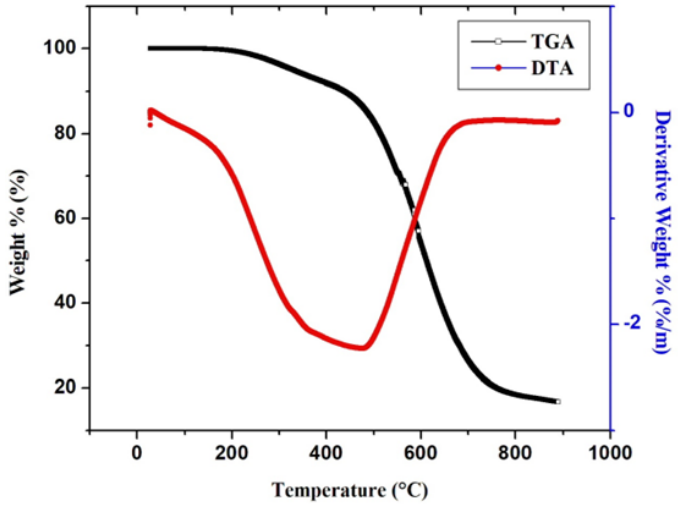


**Figure 6.** TGA/DTA plot 20% Doum fibers non- irradiation (0 Gy).

content or non-decomposable residues due to irradiation. As reported by Cota *et al*. [12], increased cross-linking often results in higher char yield, which is observed in this study.

DTA of Sample A after (100 Gy) Irradiation is similar to the non-irradiated control, the irradiated sample shows endothermic Peaks (below 150 °C), activity in this region, corresponding to moisture loss. However, the magnitude of the endothermic peak is smaller, indicating that the irradiated material has absorbed less moisture, likely due to the densification or cross-linking effects that reduce the polymer's porosity. The exothermic peak (350 °C – 450 °C) associated with the main thermal degradation of the polymer occurs at a slightly higher temperature for the irradiated sample compared to the control, reinforcing the observation that gamma irradiation improves the thermal resistance of the polymer. The delayed and broader exothermic peak suggests that the polymer undergoes degradation more gradually.

In this study, the 100 Gy dose had a beneficial effect on the thermal stability of the doum-HDPe composite. This behavior is consistent with the findings of Ahmed *et al*. [4], who observed that moderate doses of gamma irradiation (50–150 Gy) enhances the thermal stability of HDPe polymer by promoting cross-linking. However, excessive doses can lead to polymer degradation through chain scission, which would manifest as a reduction in thermal stability and earlier decomposition onset.

### 3.2.3. TGA results for sample A (150 Gy Irradiation)

TGA/DTA Results post Irradiation (150 Gy) for Sample A reveals distinct changes in its thermal stability and decomposition characteristics compared to both the non-irradiated and 100 Gy irradiated Sample A. TGA and DTA curves indicate how increasing gamma irradiation affects the HDPe's thermal performance as shown in Figure 5.

In Figure 5, initial decomposition (below 200 °C) is similar to the 100 Gy irradiated sample, the 150 Gy irradiated sample A also exhibits moisture loss and volatile evaporation in this temperature range, but the weight loss is slightly higher than in the 100 Gy sample, suggesting that increased irradiation have induced more chain scission at lower doses. This pattern is consistent with [17], who reported that while low to moderate doses of gamma irradiation promote cross-linking, higher doses result in chain fragmentation, which increases the mobility of low-molecular-weight volatiles. The main decomposition phase for the 150 Gy irradiated sample A starts at a slightly lower temperature compared to the 100 Gy sample, indicating a decrease in thermal stability at higher irradiation doses. This is in contrast to the improvements in thermal stability observed at 100 Gy.

This finding suggests that while the 100 Gy dose enhanced cross-linking, the 150 Gy dose may have caused some degree of polymer chain degradation, leading to earlier onset of decomposition. This observation is supported by Grzelak *et al*. [14], who noted that high doses of gamma irradiation tend to break down polymer chains, reducing the thermal stability of polymers. The delayed degradation at 100 Gy, compared to the 150 Gy dose, underscores the threshold behavior of gamma irradiation. Beyond a certain dose, the cross-linking effect is outweighed by chain scission, leading to compromised thermal properties. Residual Mass (above 500 °C) the residual mass at higher temperatures is slightly lower for the 150 Gy irradiated sample A compared to the 100 Gy of the same sample. This suggests a greater degree of material degradation. This decrease in residual mass is linked to the breakdown of more stable polymer structures at higher irradiation doses. Nia *et al*. [15], also observed that higher doses of irradiation lead to a reduction in char yield due to increased polymer breakdown.

DTA after Irradiation Endothermic Peaks (Below 150 °C) with the previous samples, the endothermic peak in this region corresponds to moisture loss. The 150 Gy irradiated sample shows an endothermic peak similar in magnitude to the 100 Gy sample, implying that higher irradiation doses have not significantly altered the polymer's moisture absorption or release behavior. –The exothermic peak corresponding to the main thermal degradation occurs at a slightly lower temperature in the 150 Gy irradiated sample A than in the 100 Gy sample, indicating that the polymer undergoes degradation earlier as a result of the higher irradiation dose. This earlier degradation suggests that the increased irradiation has weakened the polymer-fiber bonding, which is con-

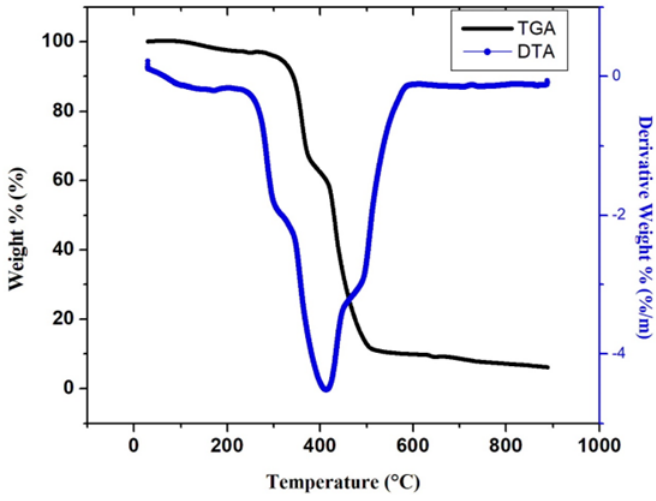


**Figure 7.** TGA/DTA plot 20% Doum fibers post irradiation (100 Gy).

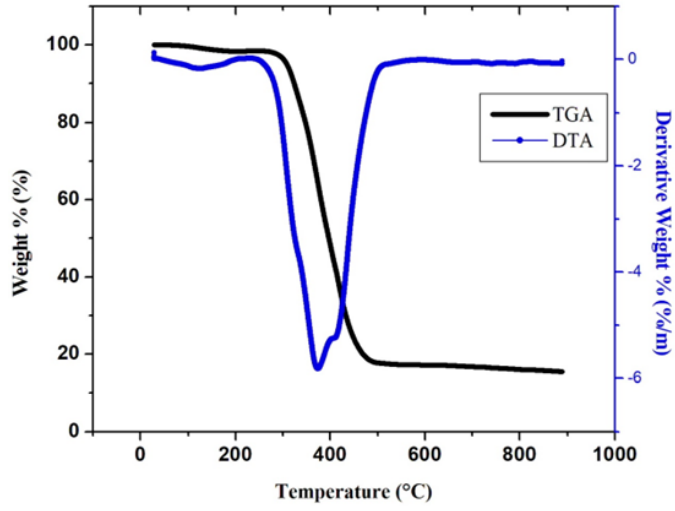


**Figure 8.** TGA/DTA plot 20% Doum fibers post irradiation (150 Gy).

sistent with findings from Grzelak *et al.* [14], who reported that high doses of gamma irradiation can lead to chain scission, reducing the thermal stability of polymer. The peak is also broader, indicating a more gradual degradation process, which could be attributed to the mixed effects of both cross-linking and chain scission within the polymer structure. The thermal analysis of the HDPE after 150 Gy of gamma irradiation reveals a trade-off between cross-linking and chain scission.

### 3.2.4. TGA results for Sample B (0 Gy)

The thermal decomposition behavior of Sample B before exposure to gamma irradiation, as shown in the TGA and DTA plots, highlights several important thermal transitions as shown in Figure 6.

The TGA curve (Figure 6) indicates a small weight loss below 150 °C, which is associated with the evaporation of moisture and low-molecular-weight volatile compounds. This initial weight loss aligns with the typical behavior of natural fiber composites, as moisture is commonly retained in natural fibers such as doum fibers. The magnitude of weight loss is consistent with the findings of Naikwadi *et al.* [16], who reported a similar early-stage weight loss in kenaf fiber-reinforced polymer composites due to the presence of bound water. The major weight loss occurs between 300 and 450 °C which is the main decomposition phase. The weight loss is associated with the thermal degradation of both the HDPe matrix and the doum fibers. The onset of degradation begins at approximately 320 °C, with maximum decomposition occurring around 400 °C. This behavior indicates the composite's thermal stability, which is primarily governed by the decomposition of the polymer matrix and the degradation of the cellulose and lignin in the doum fibers. The temperature range for thermal degradation corresponds well with the decomposition profiles observed in other natural fiber composites, as noted by Sahnoune *et al.* [17] for barley husk and coconut shell-reinforced polypropylene composites. The residual mass after 500 °C is around 5-10%, indicating the presence of non-volatile, thermally stable materials such as char or carbonized residues.

DTA endothermic peaks (below 150 °C); the DTA curve (Figure 6) reveals an endothermic peak corresponding to the moisture evaporation phase. The magnitude of this peak is typical of natural fiber composites and is linked to the moisture absorbed by the doum fibers during processing. This result is similar to the thermal profiles of other natural fiber composites, such as jute-reinforced polymers reported by Sahnoune *et al.* [17], where a similar moisture evaporation peak was observed. Exothermic Peaks (300 °C – 450 °C): The DTA plot exhibits a prominent exothermic peak in the range of 350 °C to 450 °C, which corresponds to the oxidative degradation of the polymer matrix and fiber. The position and shape of this peak suggest a gradual degradation process, indicating a relatively stable thermal profile for the composite. This thermal stability is comparable to the findings from Sahnoune *at al.* [17], who demonstrated that fiber-reinforced composites exhibit similar thermal behavior due to the delayed degradation of cellulose and lignin structures in natural fibers.

When compared to recent literature, the thermal decomposition of Sample B shows good consistency with the behavior of other fiber-reinforced polymer composites, Gulati *et al.* [18], noted that the thermal stability of natural fiber-reinforced composites is largely influenced by the fiber treatment and fiber-matrix interface. In the case of Sample B, the relatively stable decomposition profile indicates that the doum fibers have good compatibility with the HDPe matrix, leading to a stable thermal response. The residual mass observed after decomposition is also in line with findings from Gulati *et al.* [18], who observed similar char yields in natural fiber composites, suggesting that the thermal degradation of the fibers and polymer results in a stable char residue.

The TGA and DTA results for Sample B before irradiation indicate that the doum fiber-reinforced HDPe composite has a relatively stable thermal profile, with significant thermal degradation occurring in the range of 300 °C to 450 °C. The thermal behavior is consistent with other natural fiber-reinforced polymer composites, as reported in the literature. The data suggests that the composite is suitable for applications requiring moderate thermal stability, although further irradiation studies will be needed

to assess the effect of gamma radiation on its thermal properties.

### 3.2.5. TGA results for Sample B (100 Gy)

After subjecting Sample B (HDPe reinforced with doum fiber composite) to 100 Gy gamma irradiation, the TGA and DTA results show significant changes in its thermal decomposition behavior compared to the non-irradiated sample as shown in Figure 7.

Similar to the non-irradiated sample, the irradiated sample also shows a small moisture loss and initial weight loss below 150 °C, primarily attributed to moisture evaporation and volatile compounds. However, the percentage of weight loss in the irradiated sample is slightly reduced, indicating that gamma irradiation may have caused cross-linking in the polymer matrix, making it more resistant to thermal degradation in this early stage. This observation is consistent with the findings of Gulati *et al.* [18], where gamma irradiation was found to enhance the thermal stability of HDPe composites by inducing cross-linking. The main degradation phase, occurring between 300 °C and 450 °C, shows a shift in the onset of degradation toward higher temperatures in the irradiated sample.

The non-irradiated sample B began significant degradation around 320 °C, while the irradiated sample starts at approximately 350 °C, indicating enhanced thermal stability due to irradiation. The maximum decomposition temperature is also shifted slightly, suggesting that the irradiated sample's thermal resistance has improved. The residual mass after 500 °C is higher in the irradiated sample compared to the non-irradiated one. This could be due to increased char formation, which often results from cross-linking induced by gamma irradiation. The char stability suggests that gamma irradiation helps in forming a more thermally stable structure, similar to what observed in their study on gamma-irradiated polymer composites.

Endothermic Peaks (below 150 °C) as observed in the TGA, the DTA of the irradiated sample shows a smaller endothermic peak in the moisture evaporation region. This indicates that the irradiated sample retains less moisture, possibly due to changes in fiber-matrix interactions induced by irradiation, leading to a reduced ability to absorb moisture. Exothermic peaks (350 – 450 °C) the DTA curve shows a noticeable shift in the exothermic peak to higher temperatures compared to the non-irradiated sample, confirming the improvement in thermal stability post-irradiation. The intensity of the exothermic peak is also slightly lower, indicating a slower degradation process due to the irradiation-induced changes. This behavior correlates with the findings of Gulati *at al.* [18], who demonstrated that irradiation improved the thermal degradation profiles of natural fiber composites by slowing down the oxidation process.

The reduction in moisture absorption observed in the DTA, along with the increased char formation in the TGA, supports the notion that irradiation strengthens the polymer-fiber interface, reducing its susceptibility to thermal degradation and moisture retention. This aligns with findings from Ref. [19], who also reported improved thermal and moisture resistance in gamma-irradiated polymer composites. The TGA and DTA results for Sample B after 100 Gy gamma irradiation reveal enhanced thermal stability, evidenced by a higher onset of degradation temperature, reduced moisture absorption, and increased char formation. The gamma irradiation induced beneficial cross-linking effects in the composite, resulting in a more thermally stable material.

### 3.2.6. TGA/DTA of sample B post irradiation (150 Gy)

The thermal analysis results obtained from TGA/DTA for Sample B exposed to 150 Gy of gamma radiation shows significant changes compared to the results before irradiation and the results after 100 Gy exposure as shown in Figure 8, the pre-irradiated sample, thermal degradation occurs at higher temperatures, indicating greater thermal stability. However, after exposure to 150 Gy, the onset of degradation appears to shift to a lower temperature, suggesting a decrease in thermal stability. This behavior is due to chain scission in polymer matrices, where the irradiation weakens the molecular bonds.

The TGA curve for the 150 Gy-irradiated sample shows a more rapid decline in mass compared to the 100 Gy irradiated sample and the pre-irradiated state. The mass loss begins earlier, around 350 °C, and continues steadily until approximately 500 °C. This indicates that gamma irradiation at higher doses accelerates the degradation process, likely by inducing greater fragmentation in the polymer chains, leading to a lower thermal resistance. This behavior contrasts with the 100 Gy results, where the degradation was less pronounced, and the sample exhibited a slightly higher thermal degradation onset temperature. This suggests that the structural integrity of the HDPe matrix reinforced with Doum fiber begins to break down more severely with increasing gamma radiation doses, supporting previous findings from literature that radiation degrades polymeric materials by creating free radicals and disrupting molecular chains

The DTA for the 150 Gy irradiated sample further supports the TGA findings. The endothermic peaks representing the melting and degradation of the polymer are broader and shift towards lower temperatures when compared to the pre-irradiated sample and the 100 Gy irradiated sample. Specifically, the endothermic peak occurs around 380 °C, indicating thermal events such as melting or decomposition happening earlier at this higher irradiation dose. The increased peak broadness implies a more significant degradation process, where the material undergoes complex reactions leading to chain scission, which could be attributed to the higher exposure to gamma rays.

The TGA and DTA results for Sample B at 150 Gy show a marked decrease in thermal stability compared to both the pre-irradiated and 100 Gy irradiated samples. This suggests that high-dose gamma irradiation severely impacts the structural integrity of the Doum fiber-reinforced HDPe composite, in agreement with current literature.

### 3.2.7. TGA/DTA results for Sample D (0 Gy)

Figure 9 presents the thermal degradation behavior of the Doum fiber-reinforced HDPe composite (Sample D) before exposure to high-dose gamma irradiation. The The TGA curve in Figure 9 a single major degradation event, starting at approximately 300 °C, with the most significant weight loss occurring between 350 °C and 450 °C. This degradation is attributed to the breakdown of the HDPe polymer matrix, as well as the thermal decomposition of the Doum fiber reinforcement.

The onset temperature of degradation (300 °C) indicates good

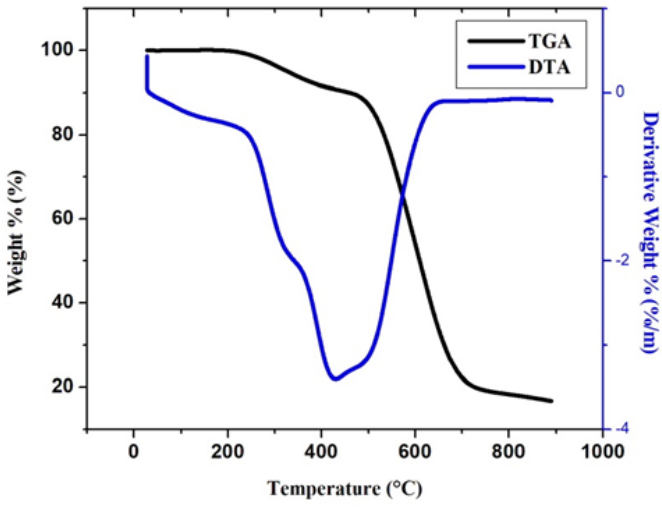


**Figure 9.** TGA/DTA plot 40% Doum fibers pre-irradiation (0 Gy).

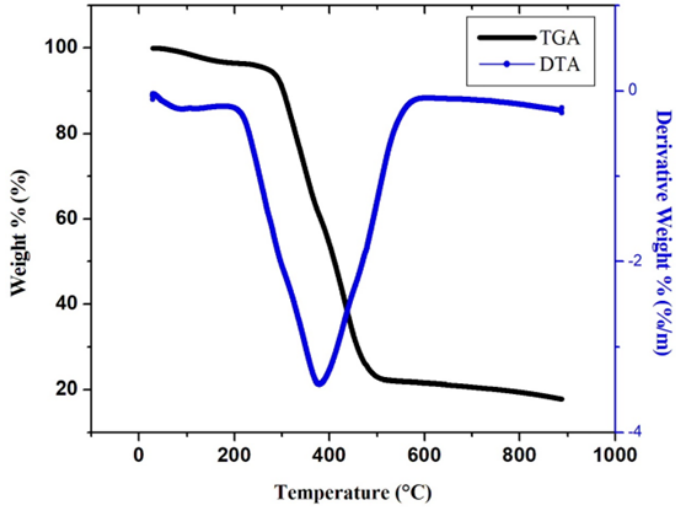


**Figure 10.** TGA/DTA plot 40% Doum fibers post irradiation (100 Gy).

thermal stability, which is typical for HDPe and fiber-reinforced composites. Literature reports similar degradation ranges for natural fiber-reinforced HDPe composites, often observing initial degradation onset between 270 °C and 320 °C [20]. The weight loss trend implies that both the fiber and polymer undergo simultaneous decomposition, with the final residue representing any inorganic content, such as residual catalysts from the polymerization process or mineral elements present in Doum fiber. The DTA curve (Figure 9) presents two prominent peaks: the first peak around at 100 °C represents moisture loss or evaporation, a common feature in natural fiber composites. The second exothermic peak around 350 °C corresponds to the major degradation process observed in the TGA curve, confirming the decomposition of the polymer matrix and fiber components. Exothermic peaks are typical in fiber-reinforced polymers due to the energy released during the breakdown of polymer chains and organic components in the fibers.

A study by Bansal and Arora [19] on coir fiber-reinforced HDPe composites reported similar degradation onset temperatures for natural fiber-reinforced HDPe composites, where the major decomposition occurred between 350 °C and 450 °C. The thermal behavior observed here aligns well with other bio-composite systems where the fiber reinforcement delays the thermal decomposition of the polymer matrix, offering increased thermal stability compared to pure HDPe [21]. The pre-irradiation thermal properties of the Doum fiber-reinforced HDPe composite show good thermal stability, with degradation occurring in typical ranges for fiber-reinforced HDPe. The DTA and TGA analyses confirm that the composite can withstand temperatures up to 300 °C without significant degradation.

### 3.2.8. TGA/DTA results of sample D (100 Gy)

TGA/DTA Results after Irradiation (100 Gy) for Sample D results are shown in Figure 10. The results highlight several changes in the thermal stability and degradation behavior compared to the non-irradiated sample.

From the TGA curve above, the major thermal degradation event for Sample D begins around 300 °C, which is consistent with the onset temperature observed in the pre-irradiated sample. However, there is a slightly lower degradation rate observed in the irradiated sample between 300 and 400 °C, which indicates that the exposure to gamma irradiation has induced changes in the polymer matrix. In the DTA curve above, the key peaks corresponding to moisture loss and degradation are still present, but there are notable shifts in peak intensity and position compared to the non-irradiated sample. The exothermic peak around 100 °C is still visible, though slightly less pronounced, indicating some moisture content in the composite, which may have increased as a result of microstructural changes after irradiation, allowing for greater absorption of moisture from the environment.

The major exothermic peak around 350 °C, which corresponds to the degradation of both HDPe and Doum fiber, appears broader and less sharp than in the pre-irradiated sample. This suggests that the irradiation has caused a broadening of the degradation range, potentially due to a mixed response from the polymer-fiber interface. The TGA/DTA results post-irradiation reveal that while the Doum fiber-reinforced HDPe composite maintains its primary thermal degradation temperature, gamma irradiation at 100 Gy induces polymer chain scission, leading to a more gradual degradation process. This decrease in thermal stability aligns with findings in existing literature on irradiated HDPe composites. The findings suggest that the composite could experience reduced performance in high-temperature applications after gamma irradiation, though it may still retain adequate stability for certain uses.

### 3.2.9. TGA/DTA results of sample D, post irradiation (at 150 Gy)

The TGA and DTA results for Sample D after exposure to a higher gamma irradiation dose of 150 Gy show further changes in the thermal stability and degradation behavior, compared to both the non-irradiated and 100 Gy irradiated samples in Figure 11.

In the TGA curve above, the primary degradation onset for the 150 Gy irradiated sample D remains around 300 °C, similar to the other samples. However, the slope of the weight loss curve is steeper than observed at 100 Gy, indicating a more rapid

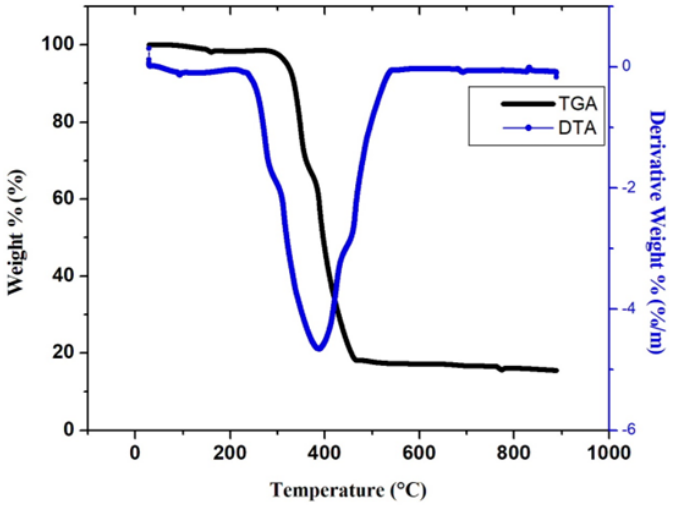


**Figure 11.** TGA/DTA plot 40% Doum fibers post irradiation (150 Gy).

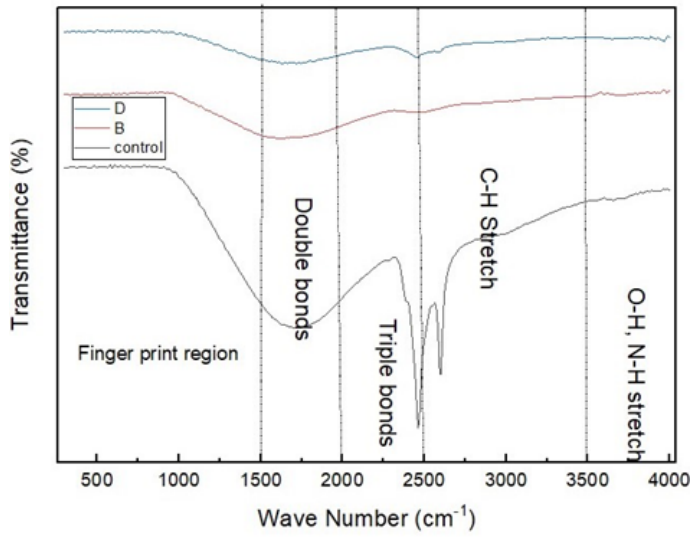


**Figure 12.** FTIR results of samples A, B and D pre-irradiation.

degradation process. This suggests that at higher doses, gamma irradiation significantly weakens the HDPe matrix through intensified chain scission and disrupts the fiber-matrix interface. The rapid degradation rate seen here aligns with studies indicating that polymer stability decreases as irradiation doses increase beyond certain thresholds, causing faster breakdown [21]. Compared to the 100 Gy irradiated sample, the residual mass at the end of the TGA is slightly lower, suggesting further loss of material integrity. This may be due to additional scission of polymer chains and degradation of the Doum fiber reinforcement at the elevated irradiation dose. The effect of radiation on natural fibers like Doum is known to include cellulose degradation, which can further compromise the structure of the composite [21].

The DTA curve (bottom plot) for the 150 Gy sample reveals a similar set of exothermic peaks as seen in the non-irradiated and 100 Gy samples, with a moisture release peak around 100 °C and a main degradation peak near 350 °C. However, the primary exothermic peak in the 150 Gy irradiated sample is broader and less defined than in both the non-irradiated and 100 Gy samples. This indicates more widespread breakdown, possibly due to cumulative damage to the polymer-fiber interface and weakening of the molecular structure within the HDPe matrix. The TGA/DTA results for Sample D after150 Gy irradiations indicate that the composite experiences more rapid degradation and reduced thermal stability compared to the non-irradiated and 100 Gy irradiated samples. These findings support existing literature on the impact of gamma

irradiation, showing that higher doses exacerbate polymer and fiber degradation, weakening the composite structure. For applications requiring thermal stability in irradiated environments, the results suggest a need for optimization of irradiation levels to balance material stability and degradation resistance.

### 3.3. COMPARISON OF TGA RESULTS BETWEEN PRE-IRRADIATION AND POST IRRADIATION OF SAMPLES

i. Onset of degradation: The onset degradations remains around 300 °C across all three samples (non-irradiated, 100 Gy, and 150 Gy), showing that gamma irradiation has not significantly altered the initiation temperature for degradation.

ii. Degradation rate: The 150 Gy sample shows a more rapid degradation rate in TGA compared to the 100 Gy sample and the non-irradiated sample, suggesting accelerated breakdown due to greater chain scission and fiber degradation at higher doses.

iii. Thermal stability: The sample irradiated at 150 Gy has reduced thermal stability compared to both the non-irradiated and 100 Gy samples, as indicated by the steeper degradation slope and broader degradation peaks in the DTA curve.
Sample D at 100 Gy demonstrates the best thermal stability, with the highest residual mass and delayed decomposition onset. This is attributed to optimal cross-linking between HDPe and 40 wt.% doum fiber. In contrast, the 150 Gy dose reduces stability across all samples, highlighting the detrimental effects of excessive gamma radiation.

### 3.4. FOURIER TRANSFORM INFRARED SPECTROSCOPY ANALYSIS (FTIR)

#### 3.4.1. FTIR results for samples A, B and D pre-irradiation

The results of the FTIR analyses of the 3 samples pre-irradiation are presented in Figure 12. The FTIR spectrum of sample (Sample A before gamma irradiation represented by the black spectrum. The black spectrum provides insight into the structural composition of the HDPe. The major peaks observed in the black spectrum correspond to the characteristic functional groups present in HDPe. Notable peaks include C–H (carbon-Hydrogen single bond) stretching vibrations peaks around 2916 $cm^{-1}$ and 2848 $cm^{-1}$ are attributed to the asymmetric and symmetric stretching vibrations of the methylene (-$CH_2$-) groups in polyethylene. C–H bending vibrations peaks near 1465 $cm^{-1}$ and 1377 $cm^{-1}$ are associated with bending vibrations of the methylene and methyl (-$CH_3$) groups.

C–C (carbon-carbon single bond) stretching vibrations Peaks in the range of 720 $cm^{-1}$ are indicative of crystalline regions in polyethylene. The spectrum aligns well with the characteristic

signatures of HDPe, confirming its molecular integrity before any external treatment. The absence of additional peaks suggests that the sample is free from impurities or significant crosslinking. The well-defined peak at 720 $cm^{-1}$ highlights the crystallinity of HDPe, which is a crucial parameter influencing its mechanical and thermal properties. The FTIR profile is consistent with other studies focusing on neat HDPe. For example, studies by Swilem *et al*. noted similar peak positions and intensities, indicating the stability of the polymer under standard conditions. These studies emphasize the utility of FTIR in verifying polymer purity and assessing the impact of treatments. FTIR studies of HDPe reinforced with natural fibers often show additional peaks corresponding to the functional groups of the fibers (e.g., hydroxyl or carboxyl groups). In contrast, the control sample lacks these modifications, serving as a baseline for subsequent analysis. For instance, in fiber-reinforced composites, peaks near 3400 $cm^{-1}$ often emerge, indicating hydroxyl stretching vibrations from the fiber content. This comparison highlights that the sample A has not undergone such modifications.

The red spectrum in Figure 12 is for Sample B and it reveals important functional groups that correlate with the composite's structure. Broad peak around 3300 $cm^{-1}$ is typically associated with -OH (Hydroxyl Group) stretching vibrations, indicating the presence of hydroxyl groups from the lignocellulosic fiber used in the composite. Peak around 2915-2850 $cm^{-1}$ correspond to C-H stretching vibrations, suggesting the aliphatic hydrocarbon chains from the HDPe matrix. Peak around 1720 $cm^{-1}$ corresponds to C=O (Carbon-Oxygen double bond) stretching vibrations, which may arise from the fiber or oxidation byproducts. Peak around 1000-1050 $cm^{-1}$ is characteristic of C-O (Carbon-Oxygen single bond) stretching, likely due to cellulose and hemicellulose in the fiber. The spectrum of sample B before irradiation reflects a strong integration of HDPe and doum fiber, evidenced by peaks representing both hydrophobic and hydrophilic components. The functional groups present suggest good dispersion of fiber within the HDPe matrix, which is crucial for the mechanical properties observed. The presence of hydroxyl groups (-OH) has been similarly noted in studies of fiber-reinforced composites, indicating that untreated fibers retain their natural functional groups. Studies like Ref. [22] suggest these groups can lead to hydrogen bonding, potentially improving tensile strength but reducing hydrophobicity. The aliphatic C-H stretches observed align with findings from composites with HDPe matrices. These peaks confirm the dominance of the polymer phase, critical for thermal and mechanical stability. Peaks in the C=O region might also indicate initial signs of oxidation, as noted in composites studied by Lee *et al*. [23], where oxidation was linked to fiber exposure.

The interaction between HDPe and fiber (evidenced by C-O and C=O peaks) suggests strong interfacial bonding, which supports the higher tensile strength of sample B (19.593 Mpa). This is consistent with other studies that attribute improved mechanical performance to strong matrix-fiber adhesion. Compared to the control sample, the inclusion of 20 wt.% fiber in sample B introduces polar groups (-OH and C=O) that enhance mechanical bonding but may reduce compatibility with the hydrophobic HDPe matrix. However, the mechanical test results demonstrate that this balance yields superior tensile and impact strength compared to pure HDPe.

The FTIR spectrum of Sample D represented by the blue spectrum in Figure 12 shows characteristic peaks associated with the functional groups of its composite materials. The prominent absorption bands observed can be summarized as follows C-H stretching vibrations the peaks near 2912 $cm^{-1}$ and 2848 $cm^{-1}$ correspond to the asymmetric and symmetric stretching of aliphatic C-H bonds in HDPe. These peaks are typical for polyethylene-based materials, indicating the presence of long hydrocarbon chains. O-H (Oxygen-Hydrogen bond) stretching a broad absorption band around 3400–3500 $cm^{-1}$ suggests the presence of hydroxyl groups, likely originating from the fiber component. This band indicates the hydrophilic nature of the fiber due to cellulose and hemicellulose. C=O stretching absorption peak near 1735 $cm^{-1}$ indicates the presence of carbonyl groups, which could be attributed to ester or ketone groups present in the fiber. This peak is often associated with lignin or oxidized cellulose in natural fibers. C-O and C-C Stretching Peaks in the region of 1016–1240 $cm^{-1}$ correspond to the C-O stretching vibrations, indicative of cellulose and hemicellulose present in the fiber. Crystallinity and HDPe backbone is the sharp peaks around 730 $cm^{-1}$ and 717 $cm^{-1}$, which correspond to the rocking vibrations of $CH_2$ (Methylene group). These peaks are characteristic of the crystalline structure of HDPe.

Sample A shows sharp peaks for C-H stretching and $CH_2$ rocking vibrations, emphasizing the high crystallinity and absence of fiber components. The lack of significant O-H and C=O peaks confirms the absence of natural fibers. Compared to Sample D, the spectrum of Sample B shows weaker O-H (Oxygen-Hydrogen bond) and C=O peaks due to the lower fiber content. The HDPe-related peaks dominate, as expected for a higher HDPe concentration. The FTIR results align with the mechanical property analysis. The addition of fiber in Sample D (40% fiber) enhances properties such as tensile strength and modulus due to the increased reinforcement effect but may reduce elongation at break due to the brittle nature of fibers. Existing literature corroborates that adding natural fibers improves stiffness but can affect ductility in polymer composites.

#### 3.4.2. FTIR results for samples A, B and D post irradiation at 100 Gy

The black spectrum in Figure 13 presents the characteristic peaks corresponding to the functional groups present in sample (control sample). The strong peak around 2915–2849 $cm^{-1}$ is attributed to C-H stretching vibrations of the aliphatic chains in polyethylene. Peaks near 1468 $cm^{-1}$ and 720 $cm^{-1}$ are associated with C-H bending and rocking vibrations. These peaks confirm the integrity of the HDPe polymer structure. Effect of Gamma Irradiation Compared to the control sample before irradiation, there is a reduction in peak intensity at certain regions, especially the C-H stretching vibrations, which suggests chain scission or molecular weight reduction due to gamma irradiation. Emergence or enhancement of a peak near 1710–1730 $cm^{-1}$ is indicative of the formation of carbonyl groups (C=O) which also suggests oxidative degradation of HDPe after 100 Gy irradiation. This agrees with findings in literature where gamma irradiation is known to induce oxidative reactions in HDPe due to the interaction of free radicals with oxygen. The lack of new, significant peaks indi-

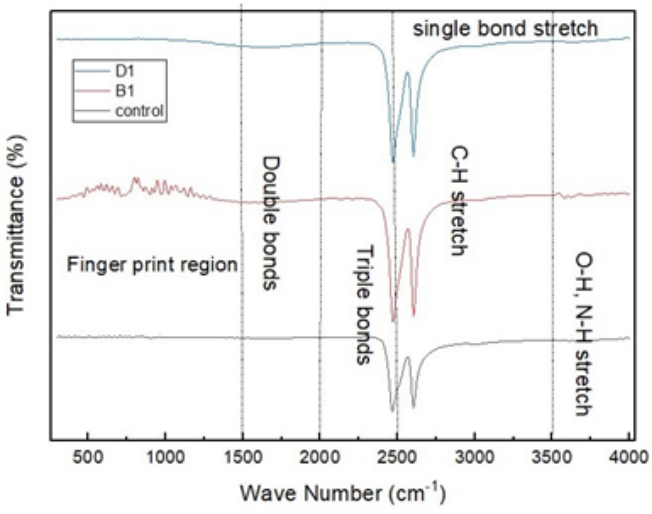


**Figure 13.** FTIR spectra post-irradiation (at 100 Gy).

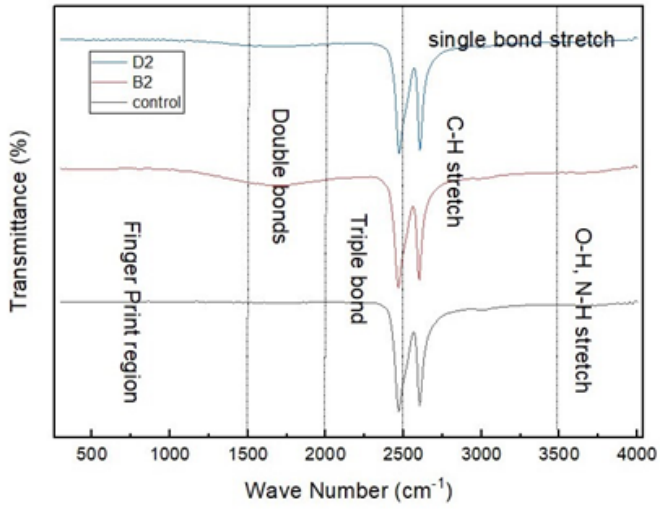


**Figure 14.** FTIR post-irradiation spectra (at 150 Gy).

cates that no major chemical cross-linking or substantial structural transformations occurred in sample A at 100 Gy exposure.

Existing studies confirm that HDPe exposed to gamma radiation undergoes oxidation, chain scission, and limited cross-linking, depending on the radiation dose and environment. The presence of carbonyl peaks (1710–1730 $cm^{-1}$ ) aligns with findings from similar studies, such as those by Jiang *et al.* [24] where irradiated HDPe displayed oxidative degradation when exposed to gamma radiation in the presence of oxygen. Literature also supports that low doses (<150 Gy) primarily result in chain scission with minor cross-linking, leading to reduced mechanical properties such as tensile strength and elongation at break. The unirradiated control sample showed stronger, more defined C-H stretching and bending peaks, reflecting an undisturbed polymer structure. The differences in peak intensities and the appearance of carbonyl peaks post-irradiation confirm the degradation pathways. The FTIR analysis demonstrates that gamma irradiation at 100 Gy induces oxidative degradation in HDPe, as evidenced by carbonyl group formation and reduced C-H vibrational intensity.

These findings are consistent with the well-established effects of gamma irradiation on polyolefins, as described in various studies. However, at 100 Gy, the changes remain moderate, indicating that HDPe can tolerate such doses with limited structural damage, making it potentially viable for applications where moderate radiation exposure is expected.

The FTIR spectrum for sample B after exposure gamma irradiation (100 Gy) is represented by the red spectrum in Figure 13. The spectrum exhibits significant changes in chemical structure compared to the pre-irradiation spectrum. Sample B, a composite containing 20% fiber and 80% HDPE, shows notable shifts in key absorption peaks, indicating radiation-induced modifications in polymer-fiber bonding and molecular structure. C-H Stretching (2800-3000 $cm^{-1}$) a reduction in the intensity of peaks associated with C-H stretching vibrations is observed, reflecting degradation or scission of alkyl chains within the HDPe matrix under gamma irradiation. This is consistent with polymer oxidation and chain scission reported in literature. Carbonyl (C=O) stretching (1700 $cm^{-1}$) appearance or intensification of a peak around 1700 $cm^{-1}$ indicates oxidative degradation, leading to the formation of carbonyl groups. Such behavior is typical for polymers exposed to ionizing radiation as free radicals react with oxygen. The hydroxyl (-OH) Stretching (3200-3600 $cm^{-1}$) which is an increased intensity in the hydroxyl band suggests the introduction of hydroxyl groups due to radiation-induced oxidation or interaction between degraded fiber and the HDPe matrix.

Fiber Contribution (1030 $cm^{-1}$), the peak near 1030 $cm^{-1}$, corresponding to cellulose functional groups, remains prominent but displays slight broadening, likely due to irradiation-induced modifications in the fiber's molecular structure. Before irradiation, the FTIR spectrum of sample B demonstrated stronger C-H vibrations and minimal carbonyl or hydroxyl activity, indicative of the intact HDPe structure and unaltered fiber. The changes post-irradiation point to chemical modifications, including cross-linking, chain scission, and oxidation, which align with observations in gamma-irradiated polymer composites from existing studies. Gamma irradiation has been widely documented to induce oxidative degradation in HDPE-based composites, leading to the formation of carbonyl and hydroxyl groups. For instance, studies highlight those composite materials with moderate fiber content (e.g., 20-30%) exhibit enhanced degradation resistance compared to pure HDPe due to the protective role of fibers. However, excessive fiber content can exacerbate degradation as fibers themselves are susceptible to radiation-induced damage.

The FTIR spectrum for Sample D after exposure to 100 Gy gamma irradiation is represented by the blue spectrum in Figure 14. It highlights key changes in the polymer-fiber composite structure, reflecting the combined effects of HDPe and the 40% fiber content under irradiation. Retention of major functional groups Peaks at 2915 $cm^{-1}$ and 2849 $cm^{-1}$ (C-H stretching vibrations) remain prominent, indicating that the hydrocarbon backbone of HDPe is stable despite irradiation. Peaks around 1460 $cm^{-1}$ and 720 $cm^{-1}$ ($CH_2$ bending and rocking modes) are also retained, consistent with the semi-crystalline HDPe structure. Formation of Oxidative Degradation Products which is a notable peak near 1720 $cm^{-1}$ corresponds to carbonyl groups (C=O), likely formed due to gamma-induced oxidative degra-

dation. This suggests that Sample D experiences some level of oxidative chain scission, similar to trends observed in irradiated HDPe composites.

Transmittance reduction in regions around the fiber-associated bands indicates potential cross-linking or interaction effects between the HDPe matrix and the fiber content under gamma irradiation. Fiber Contribution Peaks at 1030–1050 $cm^{-1}$, possibly related to Si-O (Silicon-Oxygen bond) or C-O stretching (from fiber content), suggest that the fibers in the composite influence the stability and interaction dynamics after irradiation. Before irradiation, Sample D displayed fewer oxidative degradation markers, with minimal or no carbonyl peak at 1720 $cm^{-1}$. The irradiation-induced carbonyl peak and changes in transmittance confirm the role of gamma exposure in driving oxidative degradation and interaction changes within the composite.

Research on HDPe composites has consistently shown that gamma irradiation introduces carbonyl groups due to oxidative chain scission, particularly at doses above 50 Gy. The incorporation of fibers (40% in Sample D) is reported in the literature to enhance mechanical reinforcement but may also act as oxidative degradation sites, accelerating structural changes under irradiation. The FTIR results for Sample D at 100 Gy irradiation demonstrate significant oxidative degradation, evidenced by carbonyl group formation, with stable hydrocarbon backbones in the HDPe matrix. The interaction of fiber and HDPe under irradiation appears to enhance cross-linking or localized degradation, as seen in reduced transmittance and fiber-specific peaks. This suggests a balanced compromise between stability and degradation in Sample D under gamma exposure, aligning well with prior studies on HDPe-fiber composites under similar conditions.

### 3.4.3. FTIR results for Samples A, B and D P Irradiation at 150 Gy

The FTIR spectrum for the control sample exposed to 150 Gy gamma radiation is represented by the black spectrum in Figure 14 and it reveals several notable changes in the functional group regions when compared to the control sample before irradiation and post irradiation at 100 Gy. These changes can be correlated to alterations in the molecular structure of HDPe due to gamma irradiation, which induces chain scission and crosslinking. The transmittance peaks in regions associated with C-H stretching vibrations (around 2800–3000 $cm^{-1}$) exhibit slight reductions in intensity, indicative of chain scission of aliphatic groups. A notable decrease is also seen in the regions attributed to C-H bending (1460 $cm^{-1}$) and rocking vibrations (730–720 $cm^{-1}$), signifying molecular fragmentation.

Emergence or shift of carbonyl groups, peaks around 1700 $cm^{-1}$ become more prominent, which may correspond to carbonyl (C=O) groups. This is consistent with oxidation reactions induced by gamma irradiation, forming ketones or aldehydes. Widened hydroxyl peaks in the 3200–3600 $cm^{-1}$ range, broadening and increased intensity of O-H stretching vibrations suggest hydroperoxide or alcohol formation due to oxidative degradation. At 150 Gy, the chemical changes (e.g., oxidation) are more pronounced than at 100 Gy, indicating cumulative effects of irradiation. The control sample at 150 Gy exhibits significant structural degradation compared to the non-irradiated sample, as evident by peak intensity reductions and functional group transformations. The formation of new functional groups (carbonyls) and degradation of existing C-H bonds aligns with established findings that irradiation promotes free radical formation and oxidation.

As for sample B, it is represented by red spectrum in Figure 14, which indicates the presence of characteristic functional groups for HDPe and fiber in Sample B (20% fiber and 80% HDPe). Major peaks at 2914.4 $cm^{-1}$ and 2847.8 $cm^{-1}$ represent the $CH_2$ asymmetric and symmetric stretching vibrations typical of HDPe. These peaks are present but show slight changes in intensity after 150 Gy irradiation compared to 100 Gy and the unirradiated sample. Oxygenated products which is a a notable peak appears around 1718.5 $cm^{-1}$ after 150 Gy irradiation, corresponding to carbonyl (C=O) stretching vibrations. This suggests gamma irradiation induced oxidative degradation, forming carbonyl-containing byproducts such as ketones or aldehydes. Fiber Influence peaks around 1050-1100 $cm^{-1}$, representing C-O stretching (related to fiber structure), remain consistent, indicating the fiber component's stability even after 150 Gy exposure.

As per radiation dose effect, comparing the spectra at 150 Gy and 100 Gy, there is an increase in the carbonyl index (intensity of the 1718.5 $cm^{-1}$ peak relative to a reference peak). This highlights dose-dependent oxidative degradation. Studies on HDPe exposed to gamma irradiation report similar degradation patterns, such as the formation of carbonyl groups and reduction in $CH_2$ peak intensities. For example, irradiation in air induces oxidation due to free radical generation, which reacts with oxygen to form peroxides, subsequently decomposing into carbonyl products.

Literature on natural fiber-reinforced polymer composites reveals that fibers enhance thermal and mechanical stability but are not immune to irradiation effects. However, their presence can restrict extensive chain scission of the HDPe matrix by providing structural reinforcement. Sample B shows a balance between mechanical performance and thermal stability before and after irradiation compared to the control and Sample D (40% fiber and 60% HDPe). However, increased fiber content might reduce radiation-induced oxidation in the sample. These findings align with studies suggesting that gamma irradiation modifies polymer composites based on dose and composition. Future work could involve stabilizers to mitigate radiation-induced degradation effects.

The FTIR spectrum for sample D after exposure to 150 Gy gamma irradiation is represented by the blue spectrum in Figure 14 which reveals significant alterations in chemical functional groups compared to both the unirradiated sample and the sample exposed to 100 Gy. Key regions in the spectrum, such as the O-H stretching (broad band 3200-3500 $cm^{-1}$), C-H stretching (2800-3000 $cm^{-1}$), and C=O stretching (1700 $cm^{-1}$), exhibit notable changes, which suggest oxidation and structural reorganization due to gamma irradiation. These findings indicate enhanced chemical scission and crosslinking as the radiation dose increases. An increase in intensity at the hydroxyl region (3200-3500 $cm^{-1}$) suggests the formation of hydroxyl groups, likely due to oxidative degradation of HDPe and fiber components. Compared to the unirradiated sample D and the 100 Gy spectrum, this peak appears more pronounced, indicating that the radiation dose amplifies the oxidation process. The carbonyl peak C=O Stretching (1700 $cm^{-1}$) becomes more intense and sharper after

150 Gy irradiation compared to the 100 Gy spectrum. This intensification confirms oxidative degradation, likely resulting in aldehydes, ketones, and carboxylic acids.

Literature supports this trend, with studies reporting increased carbonyl index values in polymeric materials post-radiation exposure. Reduced intensity in the C-H Stretching (2800-3000 $cm^{-1}$) in this region points to chain scission of alkyl groups within HDPe. This degradation leads to the release of smaller hydrocarbon fragments. In the fingerprint region (600-1500 $cm^{-1}$) , there are changes in peaks within the region, such as at 720 $cm^{-1}$ (rocking vibration of $CH_2$), suggesting rearrangement and scission of polymer chains. Comparison with the 100 Gy spectrum indicates that higher doses lead to more pronounced changes in polymer backbones and fiber-matrix interactions. Similar studies in polymer composites show that gamma irradiation at doses above 100 Gy promotes oxidation and degradation, resulting in increased brittleness and reduced mechanical properties.

Literature suggests that higher fiber content, as in sample D (40% fiber), exacerbates oxidative degradation under irradiation due to the fiber's susceptibility to radiation-induced chain scission and oxidation. However, it also provides enhanced initial mechanical properties, mitigating the effects of irradiation compared to pure HDPe. Other research also corroborates that gamma irradiation induces both crosslinking and chain scission, with the dominant mechanism depending on the dose. At 150 Gy, chain scission appears dominant, leading to a decline in structural integrity. The FTIR results for sample D at 150 Gy confirms significant oxidative degradation compared to both the unirradiated sample and the sample exposed to 100 Gy. These findings align with existing literature on the effects of high-dose gamma irradiation on polymer composites, highlighting the interplay between oxidative degradation and chain scission.

In summary, Sample B (20% Doum Fiber) irradiated with 100 Gy gamma dose shows a balance between oxidative degradation and structural integrity. While degradation markers (carbonyl and hydroxyl peaks) are present, the fiber reinforcement limits extensive chain scission and maintains mechanical properties. Sample D (40% fiber) experiences higher oxidative stress due to greater fiber content, and the control sample exhibits the least resistance to gamma-induced degradation.

## 4. CONCLUSION

The study demonstrates that gamma irradiation significantly influences the mechanical, thermal, and structural properties of Doum fiber-reinforced HDPe composites. Among the doses used, 100 Gy was identified as the optimum dose that provided the maximum improvements in tensile strength, hardness, impact resistance, and thermal stability. The improvement is primarily attributed to effective polymer crosslinking and improved fiber–matrix adhesion at intermediate irradiation doses. As regards fiber content, both composites of 20 wt.% and 40 wt.% Doum fiber evidenced superior mechanical and thermal properties when compared to pure HDPe matrix, the largest values of tensile strength and hardness being provided by 40 wt.%. The material presents a good chance for reinforcement. When, nonetheless, the dose of irradiation was increased up to 150 Gy, the mechanical and thermal properties decreased as a consequence, apparently, of too high a chain scission, polymer degrading, and microstructural flaws. FTIR analysis revealed structural modifications consistent with crosslinking at 100 Gy and structural degradation at 150 Gy.

These findings show that Doum fiber-reinforced HDPe composites irradiated to 100 Gy, particularly with 20–40 wt.% fiber loading, are suitable for applications requiring improved mechanical, thermal characteristics and structural modifications, including medical devices, radiation-resistant packages, automotive components, and light structural panels. Overall, moderate gamma irradiation enhances composite properties, making them suitable for applications requiring improved mechanical and thermal stability, while higher doses result in material deterioration.

## DATA AVAILABILITY

Data will be made available by the corresponding author on request.

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