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# Enhanced Mechanical Performance of Silica-Filled NR/HDPE Composites via Electron Beam Irradiation

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## Abstract

Thermoplastic elastomers (TPEs) are increasingly explored for structural and energy-related engineering applications due to their recyclability and processing versatility. However, their mechanical limitations restrict wider utilization in high-performance environments. This study examines the combined effects of silica reinforcement and electron beam (EB) irradiation on the mechanical, structural, and morphological properties of natural rubber/high-density polyethylene (NR/HDPE) composites. NR and HDPE were melt-blended with silica loadings of 2 and 10 phr, followed by compounding and subsequent EB irradiation at 50 and 100 kGy. Results show that silica significantly enhances tensile strength, from 5.25 MPa (0 phr) to 18.53 MPa (2 phr) and 25.65 MPa (10 phr), while EB irradiation further increases tensile performance by up to 87.2% at 2 phr and 77.5% at 10 phr. Fourier transform infrared spectroscopy (FTIR) analysis confirms the presence of Si-O-Si and Si-OH bonding structures, while scanning electron microscopic (SEM) observations reveal a transition from ductile to brittle fracture morphology with increasing irradiation, indicating higher crosslink density. The findings demonstrate that silica-polymer interactions, combined with EB-induced crosslinking, provide an effective pathway for producing stronger and more durable NR/HDPE-based TPEs suitable for engineering applications requiring improved mechanical integrity and long-term performance. These results highlight the synergistic role of silica loading and EB irradiation in tailoring the stiffness and deformation behavior of NR/HDPE composites for demanding engineering applications.

**Keywords**

Thermoplastic Elastomers, NR Composites, Silica Reinforcement, Electron Beam Irradiation, Mechanical Properties

## 1. Introduction

Thermoplastic elastomers (TPEs) are an important class of polymeric materials that combine the elasticity of rubbers with the processability of thermoplastics. They find applications in diverse fields, including automotive, medical, and construction industries, due to their flexibility, impact resistance, and mechanical durability [1] [2]. Among TPEs, high-density polyethylene (HDPE) and natural rubber (NR) blends have garnered interest because of their recyclability and adaptability. Nevertheless, the intrinsic limitations of TPEs, such as modest thermal stability and insufficient mechanical strength, continue to restrict their deployment in demanding operational environments. Consequently, numerous studies have examined reinforcement strategies, including the incorporation of inorganic fillers, to enhance the structural and mechanical performance of TPE systems [3] [4].

In the context of power and energy engineering, material performance and durability play a critical role in ensuring reliability, safety, and long-term operational efficiency. Polymer-based components such as seals, insulating layers, vibration-damping elements, and protective covers are increasingly used in energy systems due to their low cost, ease of processing, and design flexibility. However, exposure to mechanical loading, heat, radiation, and environmental stresses often leads to degradation, limiting their service life in demanding applications. Enhancing the structural integrity of thermoplastic elastomers is therefore essential for their broader adoption in power equipment, renewable energy technologies, and high-performance engineering systems. Strategies such as filler reinforcement and irradiation-induced crosslinking offer promising routes to improving mechanical stability, making them relevant to developing advanced materials for modern energy infrastructures.

Silica has been identified as a promising reinforcing filler due to its high surface area, low cost, and strong compatibility with TPEs. When added to TPEs, silica contributes to improved mechanical properties such as increased tensile strength, modulus, and resistance to fatigue and abrasion [5] [6]. However, a major challenge associated with silica-filled TPEs is the brittleness caused by poor adhesion between phases, leading to phase separation and compromised performance. Addressing this compatibility issue is crucial for the development of advanced TPE composites with superior mechanical characteristics.

One promising approach to overcoming these limitations is the use of electron beam (EB) irradiation. EB irradiation facilitates cross-linking in polymer matrices, thereby modifying their structural and mechanical properties. Studies have demonstrated that EB irradiation enhances polymer-filler interactions, leading to

improved mechanical and thermal stability [7]-[9]. For example, research on recycled polyethylene blends and ethylene-vinyl acetate ground tire rubber blends has shown significant property enhancements when exposed to EB irradiation [10]. Despite these findings, there is limited research on the specific effects of EB irradiation on NR/HDPE composites filled with silica, particularly in terms of optimizing silica content and irradiation dosage.

Previous studies on silica-filled TPEs have largely focused on conventional methods of reinforcement, such as chemical treatments or mechanical blending, without addressing the compatibility challenges posed by phase separation [11]-[13]. While EB irradiation has been investigated for other polymer systems, its role in modifying NR/HDPE/silica composites remains underexplored. Specifically, comprehensive studies are lacking on the effects of varying silica content on the mechanical properties of EB-irradiated NR/HDPE composites. Additionally, there is limited research on how different EB irradiation dosages influence crosslinking efficiency and overall composite performance. Furthermore, a deeper understanding is needed to correlate structural, morphological, and compositional changes with the enhancements in mechanical properties of EB-irradiated silica-filled TPEs.

To address these research gaps, this study aims to develop and characterize an irradiated silica-filled NR/HDPE composite with enhanced mechanical properties. It focuses on evaluating the influence of silica content on the mechanical performance of EB-irradiated NR/HDPE TPE composites while also examining the effect of varying EB irradiation dosages on their structural integrity. Additionally, the study seeks to establish correlations between the observed mechanical property changes and the morphological, compositional, and structural characteristics of EB-irradiated NR/HDPE/silica composites.

This research focuses on the development of NR/HDPE/silica TPE composites through an internal mixing process followed by EB irradiation treatment. Silica was incorporated in varying amounts (2 and 10 phr), and EB irradiation was applied at different dosages (50 kGy and 100 kGy) to study its effects on composite properties. The structural, morphological, and mechanical characteristics of the resulting materials were analyzed using scanning electron microscopy (SEM), Fourier transform infrared (FTIR) spectroscopy, X-ray diffraction (XRD), and mechanical testing.

## 2. Methodology

### 2.1. Materials

The materials used in this study include high-density polyethylene (HDPE), standard Malaysian natural rubber (NR), and silica as the primary components for composite preparation. HDPE was selected for its strength, recyclability, and compatibility with elastomers, while NR was chosen for its flexibility and mechanical resilience. The HDPE used was a commercial-grade polymer suitable for melt blending and composite fabrication, exhibiting a typical density of approximately 0.94 -

0.96 g/cm$^3$ and a melt flow index (MFI) in the range of 0.3 - 1.0 g/10 min (190˚C, 2.16 kg), which is characteristic of medium- to high-molecular-weight HDPE commonly employed in thermoplastic elastomer and composite systems.

The natural rubber used was Malaysian natural rubber, which typically possesses a density of approximately 0.92 - 0.94 g/cm$^3$ and a Mooney viscosity (ML 1+4, 100˚C) in the range of 60 - 80, making it suitable for melt blending with HDPE in thermoplastic elastomer formulations. Silica, known for its reinforcing capability, was incorporated to enhance the mechanical performance of the composites.

## 2.2. Sample Preparation

The preparation of the irradiated silica-filled TPE composites began with the formulation of mixtures consisting of HDPE, natural rubber, and varying amounts of silica. Specifically, silica was incorporated at 2 phr and 10 phr to investigate the influence of filler content on the mechanical properties of the composites. Control samples without silica were also prepared for comparison. Furthermore, different electron beam (EB) irradiation doses, namely 50 kGy and 100 kGy, were applied to enhance the overall performance of the materials. Table 1 shows the formulations of the samples used in this study.

Table 1. Formulations of the samples.

| Sample | Composite Composition | | | EB Irradiation |
|---|---|---|---|---|
| | HDPE (wt%) | NR (wt%) | Silica (phr) | (kGy) |
| HDPE/NR | 30 | 70 | 0 | 0 |
| HDPE/NR/EB50 | 30 | 70 | 0 | 50 |
| HDPE/NR/EB100 | 30 | 70 | 0 | 100 |
| HDPE/NR/Si-2 | 30 | 70 | 2 | 0 |
| HDPE/NR/Si-2/EB50 | 30 | 70 | 2 | 50 |
| HDPE/NR/Si-2/EB100 | 30 | 70 | 2 | 100 |
| HDPE/NR/Si-10 | 30 | 70 | 10 | 0 |
| HDPE/NR/Si-10/EB50 | 30 | 70 | 10 | 50 |
| HDPE/NR/Si-10/EB100 | 30 | 70 | 10 | 100 |

The HDPE and NR were initially blended using an internal mixer maintained at a temperature of 130˚C with a rotor speed of 55 rpm. This melt-mixing process was essential for achieving a preliminary homogeneous blend. Following this, the mixture was further compounded using a two-roll mill preheated to 130˚C to enhance the dispersion of silica within the polymer matrix [14]. During this stage, the temperature of the rolls was carefully monitored and adjusted as needed to maintain optimal mixing conditions. Inadequate heating could lead to poor filler dispersion, while excessive heat risked thermal degradation of the rubber. Additionally, the gap between the rolls was adjusted during mixing, as a smaller roll gap improves mixing and dispersion but requires higher rolling forces. After com-

pounding, the composites were shaped into sheets using a hot press, forming samples suitable for subsequent testing and analysis. Finally, the sheets were subjected to electron beam irradiation to induce vulcanization, facilitating crosslinking among the polymer chains [15] [16].

### 2.3. Characterization

Characterization in this study encompassed both the analysis of silica powder properties and the evaluation of NR/HDPE/silica composites. Silica powder was initially characterized using a particle size analyzer (PSA) to determine the median particle size distribution. In addition, scanning electron microscopy (SEM) was performed to observe the surface morphology and shape of the silica particles. These analyses provided essential data on particle size and morphology, both of which are key factors influencing filler performance in composites.

The characterization of irradiated silica-filled TPE composites focused on evaluating their mechanical, structural, and morphological properties. Mechanical testing was performed using a universal testing machine (UTM) to determine the tensile strength and elongation at break. Hardness testing was conducted using a durometer to evaluate the effect of silica content and irradiation on the material's surface resistance to deformation.

Structural analysis was carried out using Fourier-transform infrared (FTIR) spectroscopy. FTIR provided insights into the chemical structure and functional groups present in the composites, highlighting chemical changes from the blending of NR, HDPE, and silica. Morphological analysis was conducted using scanning electron microscopy (SEM) to examine the surface morphology and fracture characteristics of the composites. This analysis aimed to assess the dispersion of silica within the NR/HDPE matrix and evaluate the interfacial interactions influenced by electron beam irradiation.

## 3. Results and Discussion

### 3.1. Silica Powder Characterization

The particle size of silica plays a critical role in determining the processability and final properties of NR/HDPE composites. Smaller particles influence flow, mixing, and blend stability, while larger ones affect settling behavior and dispersion. The PSA results, as shown in **Figure 1**, indicate that the silica sample had a median particle size of approximately 20 μm, with a distribution range from 2.51 μm to 120.23 μm. This range falls within standard particle size specifications and reflects a relatively broad distribution, indicating the presence of both fine and coarse particles in the filler material [17].

The particle size distribution revealed that the most abundant particle size was 19.96 μm, representing 8.49% of the sample population. Although fine particles made up a very small fraction (0.02%), they may still contribute to surface area-dependent interactions in the composite. The overall distribution suggests that the silica used has characteristics suitable for reinforcing polymer matrices with-

out excessive agglomeration or instability.

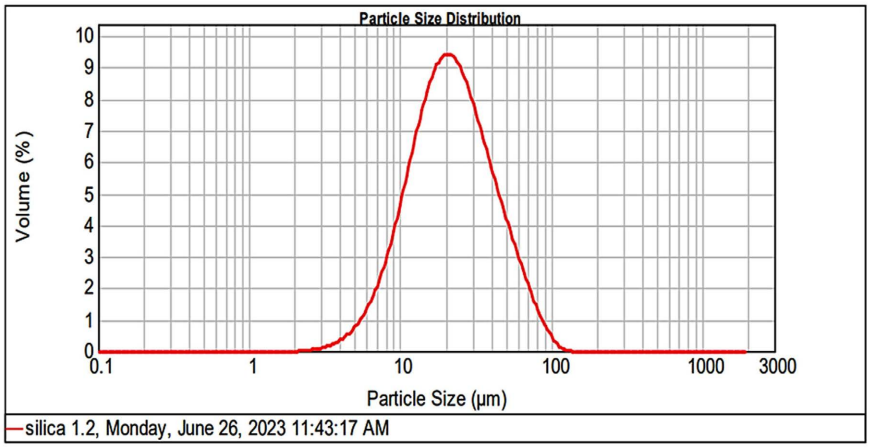



**Figure 1.** Particle size distribution of the silica powder.

SEM imaging further supported the PSA findings, showing that the silica particles were generally spherical and uniform in shape. As shown in **Figure 2**, the particles observed had an average diameter close to 20 µm and exhibited cenosphere-like morphology, in agreement with Nooney *et al.* [18], who also reported that silica particles tend to be nearly spherical. Additionally, SEM scans confirmed that the silica sample contained no visible impurities or foreign components, indicating high purity and compatibility with polymer blending processes.

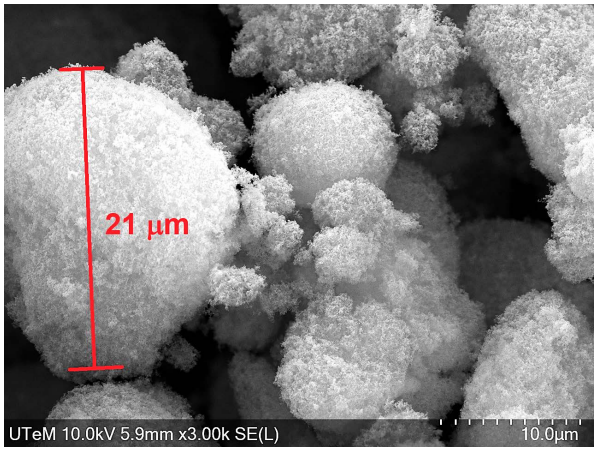



**Figure 2.** SEM image of silica particles at 3000x magnification.

## 3.2. Mechanical Properties of the Composites

The tensile strength of the NR/HDPE blend increases significantly with the addition of silica, as demonstrated in **Figure 3**. At 0 phr, the tensile strength is 5.25 MPa, while at 2 phr and 10 phr it rises to 18.53 MPa and 25.65 MPa, respectively. This indicates that silica acts as a reinforcing filler in the composite. The increase in tensile strength suggests good interfacial bonding between the silica particles and the NR/HDPE matrix. Silica likely enhances stress transfer and reduces poly-

mer chain mobility, resulting in improved mechanical performance.

Even a small addition of 2 phr silica leads to a more than threefold increase in tensile strength. This implies that minimal filler loading already brings about substantial reinforcement, which may be beneficial for cost efficiency and material processing. Although the silica particles used in this study are micron-sized (~20 μm), rather than nanoscale, they still contribute to reinforcement by enhancing load transfer and restricting polymer chain mobility. However, due to their larger particle size and lower specific surface area compared to nanofillers, the reinforcement efficiency may be lower, and localized stress concentration effects may occur, particularly at higher filler loadings.

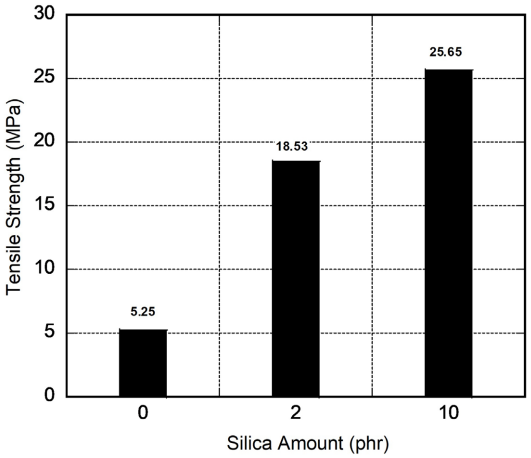



**Figure 3.** Tensile strength of NR/HDPE/Silica composites at various silica amounts.

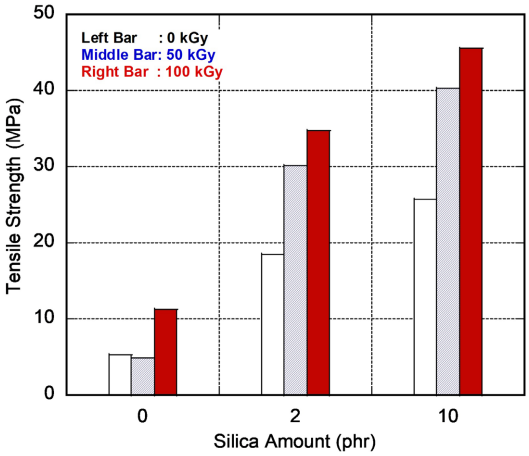



**Figure 4.** Tensile strength of NR/HDPE/Silica composites at various silica amounts and vulcanized at different EB dosages.

The tensile strength of the composites increases consistently with higher EB irradiation doses across all silica loadings. As evident in **Figure 4**, for each silica amount (0, 2, and 10 phr), samples irradiated at 100 kGy exhibit higher tensile strength than those irradiated at 50 kGy. At 0 phr silica, the tensile strength increased by approximately 114.5%, rising from 5.25 MPa (unirradiated) to 11.26

MPa at 100 kGy. Meanwhile, the tensile strength increased by 87.2% and 77.5% for 2 and 10 phr silica, respectively.

This trend clearly indicates that increasing the EB dose enhances the mechanical performance of the composites. The improvement is attributed to greater crosslinking density within the polymer matrix as the irradiation dose increases [19]. EB irradiation promotes the formation of covalent bonds between polymer chains, leading to a more robust, three-dimensional network structure that resists deformation under tensile stress [20].

Additionally, the results suggest that the magnitude of strength enhancement due to EB irradiation is more significant at lower silica contents but remains beneficial at all levels. This indicates that EB treatment is an effective method to reinforce polymer systems, regardless of filler amount.

As shown in **Figure 5**, the elongation at break of the NR/HDPE TPE increased with silica loading, from 113% at 0 phr to 116% at 2 phr and reaching 141% at 10 phr, indicating enhanced ductility. This improvement may be attributed to good interfacial bonding between silica and the polymer matrix, which improves stress transfer and prevents early failure, allowing the material to stretch more before breaking.

At 0 phr silica, the elongation at break also increased with EB irradiation, from 113% (0 kGy) to 119% (50 kGy) and 132% (100 kGy), likely due to radiation-induced crosslinking and improved phase interaction between NR and HDPE, which enhances stress distribution and enables the material to deform further before breaking.

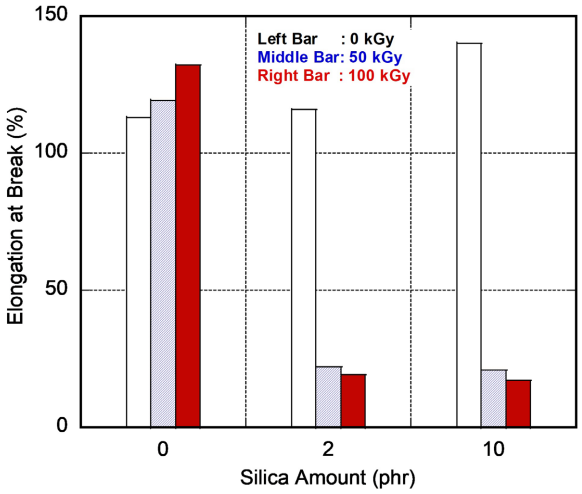



**Figure 5.** Elongation at break of NR/HDPE-Silica composites at various silica amounts and vulcanized at different EB dosages.

However, for the NR/HDPE blend containing 2 and 10 phr silica, the elongation at break decreased sharply upon EB irradiation. This drastic reduction may be caused by excessive crosslinking or chain scission initiated or accelerated by the silica particles under irradiation, resulting in increased stiffness and brittleness.

Additionally, silica may have disrupted the uniformity of the polymer network during irradiation or weakened interfacial bonding, creating stress concentrators that triggered early failure under tensile stress. These combined effects likely reduced the material's ability to elongate and absorb deformation energy.

This behavior can be attributed to the presence of silica particles during electron beam irradiation. The silica surface contains silanol (Si-OH) groups, which can promote localized radical formation at the filler-polymer interface under irradiation. This localized radical activity may accelerate chain scission or induce excessive crosslinking in the surrounding polymer matrix, leading to restricted chain mobility and increased brittleness. Consequently, the elongation at break of silica-filled NR/HDPE composites decreases more sharply than that of the unfilled system after irradiation.

The hardness of the NR/HDPE thermoplastic elastomer is strongly influenced by both silica incorporation and electron beam (EB) irradiation, as shown in Figure 6. The unfilled and unirradiated NR/HDPE sample exhibits the lowest hardness value (37.1 Shore A), reflecting the high elastomeric character and chain mobility of the blend. In contrast, the highest hardness, approximately 74 Shore A, is observed for the NR/HDPE composite containing 10 phr silica and irradiated at 100 kGy, indicating a substantial increase in material rigidity.

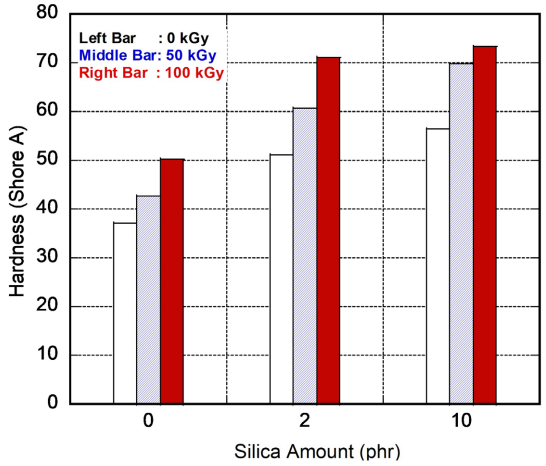



**Figure 6.** Hardness (Shore A) of NR/HDPE-Silica composites at various silica amounts and vulcanized at different EB dosages.

The addition of silica plays a dominant role in hardness enhancement, with increases of approximately 38% and 52% observed at 2 phr and 10 phr silica loading, respectively. This improvement is attributed to the reinforcing effect of silica particles, which restricts polymer chain mobility and enhances resistance to surface deformation. Comparatively, EB irradiation at 100 kGy results in a hardness increase of about 35%, suggesting that irradiation-induced crosslinking further contributes to stiffness but to a lesser extent than filler loading.

Nevertheless, EB irradiation is effective in further increasing the hardness of silica-filled NR/HDPE composites. The synergistic effect of silica reinforcement

and irradiation-induced crosslinking leads to a denser polymer network, resulting in improved resistance to indentation. These findings indicate that while silica content primarily governs hardness enhancement, EB irradiation serves as an effective secondary mechanism for further stiffness improvement in NR/HDPE/Si thermoplastic elastomers.

### 3.3. Structural Characterization of the Composites

All samples were subjected to FTIR spectroscopy to identify functional groups and assess chemical interactions within the NR/HDPE/Si composites. Figure 7 displays the FTIR spectra of the control NR/HDPE blend, as well as those containing varying silica loadings and subjected to EB irradiation. Meanwhile, the characteristics of peaks are shown in Table 2.

A prominent absorption band observed around 2915.8 $cm^{-1}$ in both NR and HDPE corresponds to the stretching vibrations of aliphatic C-H bonds, confirming the hydrocarbon nature of both polymers. The peak near 1639.2 $cm^{-1}$ is attributed to C=C stretching vibrations, which are typically present in the NR phase due to residual unsaturation [21]. Additionally, HDPE exhibits a notable peak at 1201.4 $cm^{-1}$, which can be assigned to C-O stretching, suggesting the presence of oxygenated functional groups, possibly from minor oxidative degradation or processing residues.

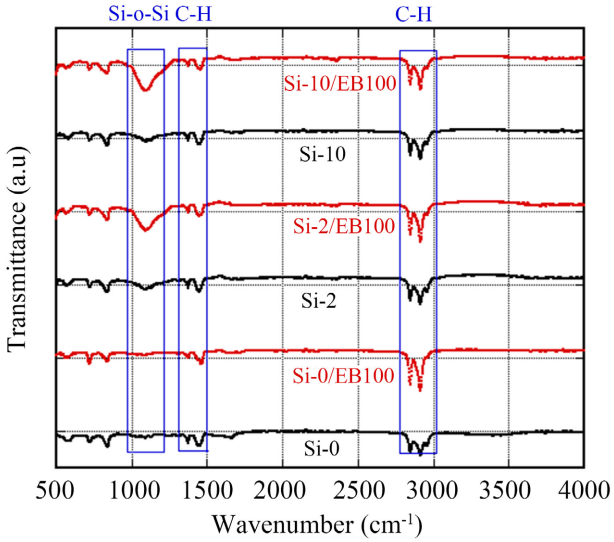



Figure 7. FTIR spectra for NR/HDPE/Si at various Si amounts and vulcanized using EB irradiation.

In the silica-containing samples, characteristic silica peaks are evident. The absorption band at 1095.4 $cm^{-1}$ corresponds to the asymmetric stretching of Si-O-Si bonds, indicating the siloxane network structure typical of silica. The broad peak observed at 3612.0 $cm^{-1}$ is assigned to the stretching vibrations of Si-OH groups (silanol), while the band at 1629.6 $cm^{-1}$ is related to the bending vibrations of these hydroxyl groups [22]. These features confirm the presence of surface hydroxyla-

tion in the silica powder, which may contribute to interfacial interactions with the polymer matrix.

**Table 2.** Characteristics of the FTIR peaks of HR/HDPE/Silica samples.

| Materials | Dominant Peak | Remark |
|---|---|---|
| NR | 1396.21 | Corresponding to C-H bending |
| | 1639.19 | Corresponding to C=C stretching |
| HDPE | 2915.84 | Corresponding to C-H stretching of -CH2- |
| | 1396.21 | Corresponding to C-H bending |
| Silica | 1095.37 | Stretching vibration of Si-O-Si bonding |
| | 3612.02 | Stretching vibration of Si-OH |

The comparison of spectra suggests that while NR and HDPE primarily consist of hydrocarbon chains, silica introduces silicon-based bonding structures (Si-O-Si and Si-OH), which are chemically distinct. These functionalities are relevant for potential filler-matrix interactions, particularly hydrogen bonding or physical entanglement with the polymer chains. The presence of these silica-related peaks in the composite spectra supports the successful incorporation of silica into the NR/HDPE matrix.

As evident in Figure 8, a pronounced increase in absorption at ~1095 $cm^{-1}$ was observed after EB irradiation of the NR/HDPE/Si (10 phr) sample, indicating stronger Si-O-Si stretching vibrations. Since the silica content remained constant, this increase is attributed to irradiation-induced condensation of surface silanol groups and interfacial restructuring of the silica network within the NR/HDPE matrix, rather than an increase in filler amount. This observation is consistent with the enhanced stiffness and reduced elongation observed in mechanical testing.

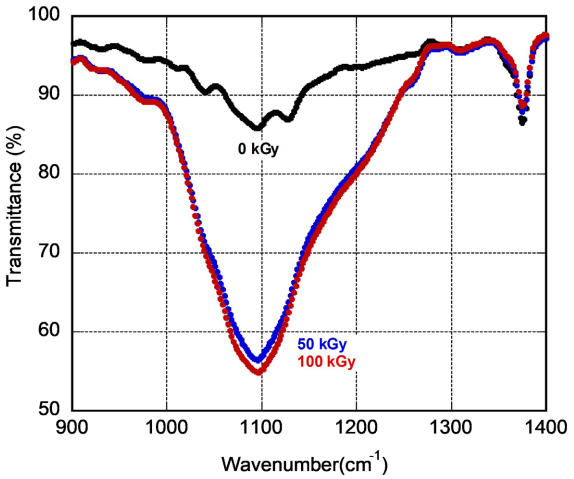


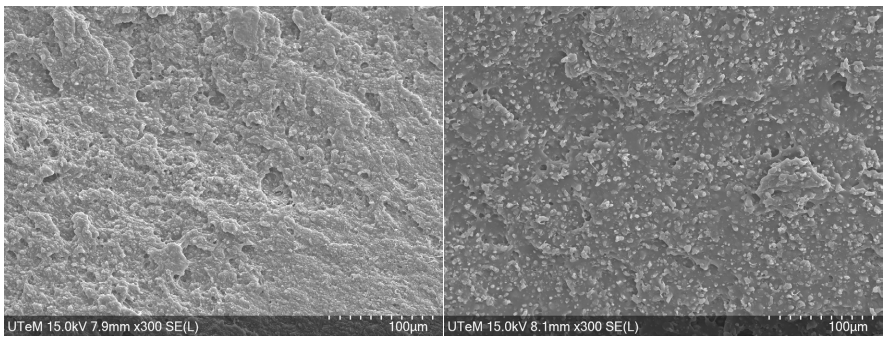

**Figure 8.** FTIR spectra for NR/HDPE/Si-10 vulcanized at different EB irradiation dosages.

### 3.4. Morphological Analysis

SEM was employed to examine the fractured surfaces of NR/HDPE/silica composites to gain further insight into the failure behaviour and compatibility of the phases. The morphological observations were correlated with tensile properties to evaluate the strengthening effect of EB irradiation and the compatibility between TPE and silica. The effects of silica loading and irradiation dose on tensile strength and elongation at break were qualitatively assessed based on the fractured surface morphology.

The tensile fractured surfaces of the unirradiated and EB irradiated NR/HDPE/silica composites are shown in **Figure 9(a)** and **Figure 9(b)**, respectively. As discussed earlier, EB irradiation significantly affected the mechanical properties, particularly reducing the elongation at break. The morphological observations support this behaviour and reveal distinct differences between irradiated and unirradiated samples.

The fractured surface of the unirradiated composite (**Figure 9(a)**) appeared rough and irregular, indicative of ductile failure with substantial plastic deformation before fracture. This roughness suggests greater energy absorption during failure and points to a more flexible matrix [23]. In contrast, the EB-irradiated samples (**Figure 9(b)** exhibited considerably smoother and more uniform fracture surfaces, consistent with brittle failure. The smoother morphology reflects restricted chain mobility and reduced deformability due to the formation of a densely crosslinked network induced by EB irradiation [24]. The SEM analysis aligns well with the tensile property trends, reinforcing the conclusion that EB irradiation enhances rigidity but reduces toughness in NR/HDPE/silica composites.




(a) (b)

**Figure 9.** SEM images of the fractured surface of (a) HDPE/NR/Sil-10, and (b) HDPE/NR/Sil-10/EB100 at 300x magnification.

## 4. Conclusion

This study demonstrates that the incorporation of silica filler and the application of EB irradiation significantly improve the mechanical performance of NR/HDPE/silica composites. The addition of silica, even at low loadings such as 2 phr, results in a notable increase in tensile strength, highlighting its effectiveness as a reinforcing agent. Furthermore, higher silica content coupled with increased EB irradia-

tion doses (up to 100 kGy) leads to a substantial improvement in tensile strength and modulus, attributed to increased crosslinking density within the polymer matrix. Morphological investigations, including SEM and FTIR analyses, reveal improved interfacial bonding and structural modifications induced by EB irradiation, which contribute to mechanical performance enhancements. However, beyond optimal silica loading and irradiation levels, excessive crosslinking may reduce ductility, as evidenced by decreased elongation at break. Overall, this synergistic approach of silica reinforcement and EB irradiation provides a promising pathway for developing advanced NR/HDPE/silica composites with superior strength and durability for various engineering applications. Future research should explore the long-term stability and potential application-specific performance of these composites to realize their commercial potential fully.

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## Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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