PAPER

Insight on the properties of thermoplastic elastomer-based natural rubber and recycled rubber post-treated with electron beam irradiation

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Keywords: Thermoplastic elastomer, recycle rubber, Radiation-induced crosslinking, recycling rubber, thermoplastics

Abstract
Thermoplastic elastomers are an indispensable group of materials used in various relevant industries. This study aims to enhance the properties of thermoplastic elastomers by blending the same ratio of natural rubber (NR) and recycled rubber (RR) with different ratios of high-density polyethylene (HDPE). Therefore, the study examined the effect of irradiation on the crosslinking of the main chain of thermoplastic elastomer (TPE) based on a 1:1 ratio of natural rubber and recycled rubber. TPE was prepared at different time periods and composition ranges by mixing recycled rubber with natural rubber together with HDPE. Using an internal Haake machine mixer, the mixing process was conducted, and the mix was standardized using a two-roll mixer. All samples were exposed under electron beam irradiation for structural modification. The effect of crosslinking in TPE was determined by analyzing the gel content. The high dose of irradiation resulted in a higher gel content, indicating an increase in crosslinking. The effect of irradiation and HDPE as a filler increased TPE’s tensile strength, while higher RR/NR compositions improved elongation properties. The irradiation dose of 50 to 150 kGy exhibited an increase in mechanical properties than the unirradiated composites. The FTIR analysis confirmed the presence of a carbonyl group formed after irradiation through a decomposition-oxidation reaction. The study found that high NR/RR ratios increase elongation and thermal characteristics. The homogeneous blending of NR/RR/HDPE was shown from the blending profile, crosslinking density, and morphology. The high amounts of NR/RR ultimately enhance TPE’s thermal stability.

Introduction
Numerous rubber products have resulted in an enormous amount of disposed waste rubber at stores and landfills. Waste rubber is non-degradable and therefore challenging to recycle. It has become a threat to the environment and human health. The best way to solve the problem is to reuse waste rubber, hence, numerous types of research on the recycling of waste rubber have been conducted worldwide [1]. Rubber footwear [2], the glove industry [3], and used tires are examples of the large quantities of waste that accumulate every year, requiring effective mitigation. However, such waste cannot be recycled as thermoplastics using reasonably cost-effective techniques or direct reprocessing (re-melting and re-molding). The reclamation technique is the best way to transform waste rubber into wealth since it possesses a re-vulcanizable output and a higher potential of mixing with the new natural rubber. Possible rubber recycling technologies have promoted considerable amounts of academic and industrial interests [4, 5]. A variety of waste rubber devulcanization techniques have
been published worldwide. These techniques include mechanochemical devulcanization [6, 7], chemical devulcanization [8, 9], microwave devulcanization [10], and irradiation devulcanization [11].

Thermoplastic elastomers (TPEs) are copolymers or a physical mixture of polymers (plastic and rubber) with thermoplastic and elastomeric properties. Several investigations have been made on the enhancement of TPE properties by introducing partially natural fiber-based materials such as kenaf [12] and rice straw [13]. These fiber replacements have shown to increase mechanical behavior. However, substituting new materials may increase the cost of production, therefore, they are not economically advantageous. TPEs based on recycled tire rubber have been thoroughly studied to enhance their recycling efficiency [14]. One feasible option is to ground waste rubber and use the resulting crumb rubber (CR) as thermoplastic filler or thermoset resins [15, 16]. Tan et al (2009) conducted research on TPE-filled waste rubber has shown significant improvements with excellent miscibility and compatibility of the blend’s mechanical strength, hydrophilic features, swelling resistance, and thermal properties. Studies have reported the development of polyethylene waste thermoplastic elastomers and the recovery of rubber compounds as well as their composite fly ash [3]. The properties of thermoplastic elastomers containing rubber tire powder derived from waste rubber were reported to have substantially improved [17].

The radiation processing of polymers has gained significant research attention since it can be used to manipulate polymers’ molecular structure as an alternative approach to more conventional chemical methods [7]. The effect of high ionizing radiation on rubber has been extensively discussed [7, 18]. Interaction leads to free radicals’ formation by dissociating molecules in the excited state or interacting with molecular ions. By directly connecting to polymer chains or by initiating grafting reactions, free radicals or molecular ions can react. The interaction process allows the macro-radical formation of elastomer chains recombined to cause structuring [19]. Compatibility is the key problem that may contribute to phase separation or poor adhesion between phases when preparing TPE-filled recycled rubber. By forming covalent bonds between the polymer phases, radiation treatment will allow polymers to be more compatible [20]. The enhanced effect of irradiation on TPE properties was also proven in previous research. It was further reported that the properties of glass fiber reinforced polyethylene waste/reclaimed rubber composites can be improved by irradiation treatment [21]. Studies have verified an enhancement in the crystallinity and dynamic mechanical properties of reclaimed waste tire rubber/EVA mixtures under electron beam influence [22]. The effect of gamma irradiation was investigated and reported on ethylene propylene diene rubber composite [23].

Embedding crumb rubber in the polymer matrix of a thermoplastic has demonstrated significant results, allowing them to mold into new products effectively. However, to avoid low mechanical properties, the substituted recycled rubber in the TPE formulation must be limited. Thus, we suggest using irradiation in our study for inducing crosslinking to enhance properties. This study prepared TPEs with a maximum 1:1 ratio of natural rubber and recycled rubber with a varying ratio of HDPE. Subsequently, the chemical, thermal, morphological, and mechanical properties before and after electron beam irradiation were evaluated. The effects of the absorbed dose variant on the different ratios of natural rubber (NR), recycled rubber (RR), and high-density polyethylene (HDPE) were analyzed. Infrared spectroscopy (FTIR), scanning electron microscopy (SEM), thermogravimetry analysis (TGA), and mechanical properties were then used to examine the efficiency of the compounding process.

**Methodology**

**Materials**

Recycled rubber produced from end-of-life tires was employed for the entire experiment. The mechanical grinding of automotive tires produced recycled rubber which was then screened and classified. The natural rubber
rubber in this study is non-vulcanized rubber supplied by the Malaysian Rubber Board (MRB). HDPE was provided by Lotte Chemical Sdn. Bhd., and the sodium dodecyl sulphate (SDS) used as a compatibilizer was supplied by Sigma Aldrich (Malaysia).

**Blending natural rubber and recycled rubber**
The natural rubber and recycled rubber mix was melted in the Rheo-Haake internal mixer equipped with two rotating blades and a 60 cm³ volumetric capacity mixing head. The rotor’s speed was set to 50 rpm, while the mixing temperature was set to 120 °C. The designated amount of natural rubber was initially fed into the inner mixing chamber and allowed to melt for two minutes, followed by the addition of RR. Both NR and RR were mixed for 4 min and HDPE has then added accordingly. The total time of mixing was 10 min. A built-in software recorded the melt-mixing torque-time. For further compression molding, the collected materials were kept in sealed plastic bags. Table 1 presents the weight percentage of the prepared mixtures.

To obtain the test specimens, materials acquired from the internal mixer were compression molded. The compounded materials were positioned on both sides of a steel frame mold covered with aluminum plates. The materials were pressed into sheets with a thickness of 1 mm at 130 °C. Using a hot pressing machine (LP-S-50 Scientific Hot and Cold Press), the molding cycles involved 3 min of preheating without pressure, 20 s of venting, and 3 min of compression under 14.7 MPa pressure. Cooling was immediately carried out between two cold press plates equipped with a chiller for 2 min at 20 °C.

**Irradiated TPE by electron beam accelerator**
The molded sheets were irradiated using a 3 MeV electron beam accelerator (NHV-EPS-3000 model) at doses ranging from 0 to 200 kGy. The acceleration energy, beam current, and the dose absorbed per pass were 2 MeV, 5 mA, and 50kGy, respectively. The electron beam irradiation procedure was performed in the air atmosphere.

**Characterization**

**Gel content**
Following ASTM D2765, the content of the gel samples was determined. The samples were placed within a 120 mesh-sized stainless-steel wire mesh and extracted using Soxhlet for 24 h in boiling Toluene. The samples were then collected and dried at 70 °C in the oven until a constant mass was obtained. The gel content was calculated according to the following equation:

\[
\text{Gel content} = \frac{W_1 - W_0}{W_0} \times 100
\]

where the dry weight of the sample is \(W_0\) and \(W_1\) before extraction and after extraction, respectively.

**FTIR analysis**
Infrared spectra were recorded in the Equinox 55 (Bruker, Karlsruhe, Germany) Fourier transform infrared spectrophotometer (FTIR) for all samples. The transmission mode samples were measured between 4,000 and 400 cm\(^{-1}\) at 4 cm\(^{-1}\) resolution. All results provided in this report are based on an average of three measurement data.

**Thermogravimetric analysis**
TGA was accomplished using TG 209 Tarsus F3 (Netzsch). The samples were heated within a temperature range of 25 °C–600 °C at a rate of 5 °C min\(^{-1}\) under a controlled dry nitrogen flow of 20 ml min\(^{-1}\).

**Scanning electron microscopy (SEM)**
A microscope, model JEOL JSM-5400, was used to take SEM images of the rubber blends’ fractured surfaces. The samples were sputter-coated with gold under a high vacuum with an image magnification of 7500 for 3 min.

**Tensile strength**
The mechanical properties of compounds were calculated following the ISO 527 standard on a Zwick Z250 universal testing machine. The load cell was 20 kN at ambient temperature, and the crosshead speed was 100 mm min\(^{-1}\). The absolute average deviations for tensile strength and elongation at break were calculated with 5 tests per compound.
Results and discussion

Effect of the blending process on recycled rubber

Figure 1 displays the blending profile of six different TPE formulations, as previously listed in Table 1. SDS was used as a surfactant to improve the dispersion of HDPE during the blending process [24]. The torque significantly increased at minute-1 due to the rotors’ resistance by the un-melted natural rubber. Suppose that NR is melted and mechanically sheared, the temperature within the chamber will then increase, resulting in a lower torque value. Recycled rubber (RR) was added into the mixing chamber at minute-2, but the torque only marginally increased due to the loose inter/intrachain bonding of RR [25]. The torque significantly increased at minute-4 after the addition of HDPE. The amount of HDPE used has an effect on the sufficient value of the torque. High HDPE results in higher torque compared to low HDPE [26]. Since NR and RR were adequately mixed with HDPE, the torque gradually decreased and stabilized at the end of 5 min. As shown in Figure 1, when comparing the stabilization torque at minute-5 for all formulations, it can be concluded that the stabilization torque increases with an increase in the mixture’s NR/RR content. The profile further demonstrates that all compounds were mixed homogeneously, shown as a straight line in minute-9.

Effect of radiation on crosslinking density of thermoplastic elastomer

The TPE blends were compressed into sheets and subsequently irradiated with an electron beam accelerator, from 50 to 200 kGy absorbed doses with a 25 kGy pass−1 dosage rate. Figure 2 presents the percentage of gel
content by weight in six separate TPEs and irradiated formulations. The gel content increased with the higher irradiation dosages for all formulations. A rise in gel content was also noticed with the increasing amounts of rubber-based materials in the blend. The incremental patterns suggest an improvement in the crosslinking chain caused by irradiation [27]. Crosslinking enhances TPE stiffness and, therefore, does not allow it to dissolve in the solvent. From these results, it can be concluded that a greater amount of gel content entails a higher degree of crosslinking between NR/RR/HDPE, which occurs during the mixing stage and is caused by irradiation.

### Chemical properties using FTIR analysis

The FTIR analysis was conducted on TPE at different ratios to observe the chemical bonding after blending. Figure 3 displays the TPE FTIR spectra, and table 2 presents the wavenumbers and the respective chemical bonds. The peaks at 2944 cm$^{-1}$ correspond to the $-$CH symmetrical stretching in the CH$_2$ group. The two peaks...
have confirmed the deformation of C–H at 1340 cm$^{-1}$ and 820 cm$^{-1}$. It was found that the intensity of these peaks increased as the ratio of NR/RR increased. The peaks at 1280 cm$^{-1}$ correspond to the S=O of recycled rubber, which underwent vulcanization. Other peaks at 1080 cm$^{-1}$ and 880 cm$^{-1}$ correspond to the presence of C–O–C and C–O, respectively [28]. These peaks are attributed to the chemical compounds that mainly originate from the structure of natural rubber and recycled rubber, which are key components of this mixture, as reported in other articles [29].

Polymer irradiation was noted for rapid crosslinking, chain-branching, unsaturation, chain-scission reactions, and degradation processes. The FTIR experiments were therefore conducted on TPE samples subjected to different absorption dosages. Figure 4 displays the FTIR spectra of 45NR/45RR/10HDPE irradiated at 50, 100, and 150 kGy. The new peaks at 1734 cm$^{-1}$ were observed to correspond to carbonyl groups' presence [30]. The presence of carbonyl groups is due to the degradation-oxidation reactions caused by ionizing radiation which occurs in air, as shown in figure 4 (right). However, carbonyl groups' appearance only increases at 150 kGy where the radical formation is high, thus allowing potential oxidative reactions [10, 31, 32].

Figure 5. SEM micrograph of various compositions of NR/RR/HDPE blends.

Figure 6. SEM micrograph of 45NR/45RR/10HDPE blends irradiated at different absorption doses.
Morphology and EDX
A comparison of the morphology of TPE blends was accomplished. Figure 5 presents five TPE micrographs at different loading ratios of NR: RR: HDPE. In the continuous HDPE phase, the high HDPE fracture surface (25NR/25RR/50HDPE blends) revealed that RR and NR were dispersed as domains. However, the two phrases seem to match and interconnect within a 3D network as the RR and NR loading ratio increases. High levels of HDPE led to coagulation between the rubbers, while high rubber levels assisted in shaping uniform surfaces.

The influence of different absorbed dosages on the morphology was also examined. Figure 6 displays the TPE blend morphology at 50, 100, and 150 kGy for the ratio of 45NR/45RR/10HDPE. As seen in the micrograph, TPE irradiated at 50 kGy had created irregular surfaces relative to TPE irradiated at 150 kGy. The TPE irradiated at 50 kGy can be explained by adjusting the adhesion force between the rubber particles, initiating mobility and exposing the HDPE network accordingly. The observed smooth surface for TPE irradiated at 150 kGy can be due to the higher absorption dose, forming an interparticle bond between the rubbers [33].

Thermal stability
The thermal behaviors of TPEs were studied by thermogravimetry analysis. A thermogram of NR, RR, HDPE, and the distinct TPE ratio blends are shown in figure 7. From the TG and DTG graphs, HDPE was the most thermally stable while NR was the least. Thermal degradation of HDPE primarily occurs by the random scission along the backbone chain with the formation of long radical fragments, which is the initial step with no weight loss. Subsequent reactions of intermolecular chain transfer then occur, leading to many low molecular weight hydrocarbon products such as methane and ethane. The NR displayed one degradation step whose onset temperature at 368°C corresponds to the isoprene unit’s degradation [34]. However, RR presented two degradation peaks. The first set of peaks at ∼220°C was assigned to the decomposition of processing oil and other low boiling point components, while the second set of peaks at ∼400°C was assigned to the decomposition of rubber components of reclaimed rubber, SBR, and NR [35]. The TPE blends exhibited two degradation peaks at 344°C and 468°C, which correspond to NR/RR and HDPE, respectively. It can be seen that the amount of NR and HDPE influences thermal stability, while no significant effect was shown from RR.

The effect of irradiation (50, 100, and 150 kGy) on thermal behavior is reflected in the overall percentage of degradation of TPE blends. It can be seen that the remaining weight is lower for TPE with high NR/RR than with low NR/RR. In addition, the degradation of TPE with high NR/RR presented high and broad peaks at the
temperature of 468 °C, whereas TPE with low NR/RR displayed small and narrow peaks. The irradiation effect significantly increased the extent of crosslinking, as shown by the gel content results [36]. The thermal results revealed that the principal crosslinking was between NR and RR instead of NR/RR and HDPE since HDPE was highly crystalline relative to NR/RR.

**Mechanical properties**

*Effect of irradiated thermoplastic elastomers on tensile strength*

The tensile properties of TPE sheets, with or without irradiation, were collectively examined. As shown in figure 8, the tensile strength drops with a decrease of more than 80% at 0 kGy as the NR/RR ratio increases. However, an increase of about 50% in the absorbed dose was observed due to irradiation, causing an improvement in tensile strength. This pattern can be explained by the high degree of crosslinking induced by irradiation, which was also seen in the gel content analysis and thermal analysis. Therefore, the presence of HDPE as a filler in this formulation is essential for enhancing the tensile properties of TPE.
Effect of irradiated thermoplastic elastomers on the elongation behavior

The influences of various ratios and absorbed doses on elongation properties were observed and calculated, as shown in figure 9. Most elongation properties typically improve with an increase in rubber content [37]. From the graph, the elongation at break increases as the NR/RR ratio increases. The existence of a high HDPE ratio had limited the mobility of the rubber matrix and the recycled rubber chain, thus decreasing the elongation at break [38]. The tensile strength was not compatible with the influence of the absorbed dose on elongation properties. The elongation at the break did not exhibit any improvement at a low NR/RR ratio. The increase was only observed in TPEs with a 35NR/35RR/30HDPE ratio, while TPEs with 45NR/45RR/10HDPE presented a decrease in elongation properties. The high ratio of HDPE restricted the elongation of rubber molecules; thus, the elongation at break remained constant. At 35/NR/35RR, and with a rubber ratio of 70, the TPE extended with HDPE molecules without any interruptions. With a rubber ratio of 90, where 45 is recycled rubber, the elastomeric properties will be less and elongation may decrease [39]. The effect of RR on elongation properties is therefore significant in the preparation of TPEs.

Conclusions

TPEs with a 1:1 NR:RR ratio and various HDPE ratios were successfully obtained. The presence of recycled rubber had a direct effect on the blending features of the thermoplastic elastomer. When the amount of NR/RR is greater than HDPE, the torque significantly increased. The gel content analyses, thermal behavior, and tensile strength confirm the increase in post-irradiation crosslinking. The FTIR spectra displayed a new peak due to the degradation-oxidation process during irradiation, corresponding to a carbonyl group. The RR presence is significant for enhanced elongation properties in the preparation of TPEs. Reduced elongation properties are due to the low elastomeric characteristics of RR. However, the existing RR in the TPE preparation contributes to favorable elongation characteristics. Lower elastomeric RR properties will reduce TPEs’ elongation properties.

Data availability statement

The data that support the findings of this study are available upon reasonable request from the authors.

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