Influence of Tire Reclaimed Rubber (TRR) Loadings on Cure Characteristics and Mechanical Properties of Natural Rubber/Styrene Butadiene Rubber (NR/SBR) Blends

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ABSTRACT

This investigation delves into the potential utilization of Tire Reclaimed Rubber (TRR) as a filler within Natural Rubber/Styrene Butadiene Rubber (NR/SBR) blends. TRR is obtained from discarded rubber tires and stands as a pertinent resource that necessitates its effective utilization in the production of eco-friendly, value-added rubber-based products. This is due to the rising of rubber waste tires which causes environmental concerns and all sorts of pollution. The TRR filled NR/SBR blends were fabricated using two-roll mills. The loadings of TRR were varied ranging from 20 phr to 100 phr. The aim of this study is to investigate the influence of TRR loading on the cure

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characteristics and mechanical properties of blends in terms of tensile strength, rebound resilience, hardness, and density. It is found that the M_H *and* Δ *torque exhibited a decreasing trend which would indicate a reduction in crosslink density due to high loading of TRR. Meanwhile, the* t_{s2} *and* t_{c95} *increased with the increase of TRR loading. Furthermore, the tensile strength, hardness, density, and rebound resilience of blends experience a steady reduction with the incorporation of TRR. The decrement of those properties might be due to the low molecular weight of TRR. Regarding the collective findings, TRR emerges as a potentially viable candidate for waste rubber repurposing, serving as a non-reinforcing filler with optimal utility achieved at a 30 phr loading level.*

Keywords: *Tire Reclaimed Rubber (TRR); Natural Rubber/Styrene Butadiene Rubber (NR/SBR) Blends; Potential Filler; Mechanical Properties; Cure Characteristics*

Introduction

One of the many issues in Malaysia has been concerns about the environment. Poor waste management directly impacts numerous habitats and species as well as air pollution and climate change. According to the Malaysian Rubber Export Promotion Council (MREPC), total world rubber production climbed to 26.9 million tons in 2016 compared to 2010 [1]. From 2017 through 2025, the International Rubber Study Group (IRSG) predicts that global total rubber consumption will grow at a rate of 2.8% per year on average [2]. Pneumatic tire production is the second largest rubber production in Malaysia. As the need for the production of tires increases, there will be more waste generated from the manufacturing of these tires. They are called waste tires. In Malaysia, waste tires are not classified as either solid trash or hazardous waste [3]. Because it is commonly seen as commercial or trade waste, there is currently no explicit rule or regulation governing waste tire handling. Tire dealers are put under a lot of pressure when discarded tires pile up on their premises, which sometimes leads to incorrect trash storage and penalties from the local government [3].

Malaysia's yearly waste tire production is expected to be 8.2 million, or about 57,391 tons [3]. Approximately 60% of trash tires are disposed of in unknown ways. Tire wastes take up not only space but are also hard to dispose of due to their crosslinked state. Reclaimed Rubber (RR) is waste rubber produced from the leftovers of rubbers during the production of rubber products in factories and would undergo various processes [4] in order to transform it into value-added reclaimed rubber. RR is a type of rubber that is obtained from vulcanized scrap rubber and is a mixture of rubber, carbon black, oil, zinc oxide, stearic acid, and other compounding ingredients that are used in the original rubber compound [5]. RR has existed in the rubber industry for quite some time and is slowly gaining the interest of rubber manufacturers and researchers around the world due to the alarming amount of rubber waste [6] which is still at large, especially involving the rubber-based tire industry. The incorporation of RR with virgin rubber is an alternative strategy to effectively utilize RR [7]. There have been numerous previous studies that were done on the incorporation of RR in NR composites [8] and SBR composites [9]-[10], however, there are lack of studies involving RR in polymer blends, especially NR/SBR blends. Hence, in this research study, TRR is used as a filler in NR/SBR blends.

This study could help combat the rubber waste issue in Malaysia as well as help in fulfilling one of the Sustainable Development Goals (SDG) namely, SDG12. This research is to see how TRR acts as a potential filler in NR/SBR blends. Previous studies have shown that TRR can be added to rubber as fillers and have shown significant results [11]. A study done by Ramarad et al. [11] presented pertinent facts concerning the use of waste rubber tires as a filler in polymeric materials. According to the study, one of the crucial elements of recycling waste tires is that the rubber must be grounded and shredded before being mixed into the polymer. The ground rubber was then surface-modified to improve the interaction between the rubber particles and the polymer matrix. Surface alteration was accomplished physically or chemically, as well as through the devulcanization or reclamation process [12]. In their review, they stated that rubber waste tires in rubber matrix are simple to process and that their composites have acceptable qualities but lack information on mechanical properties and cure characteristics of waste tire polymeric blends. Thus in this research, the effect of various loadings of TRR on the cure characteristics and mechanical properties of NR/SBR blends is studied and the optimum loading of TRR to be added as a filler in NR/SBR blends can be determined.

With the exploitation of the addition of TRR in NR/SBR blends, this study aims to develop rubber blends with the incorporation of RR, specifically TRR filled NR/SBR blends which can then be used to produce other types of rubber-based products at a cheaper cost, without deteriorating the mechanical properties. This way, an alternative solution to support the efforts of rubber recycling and reducing the ecological impact on the environment by incorporating RR as filler in polymer blends. This is indeed a simple, sustainable, eco-friendly, low-cost, and effective way in providing a promising method to obtain a high-performance RR filled polymer blend with improved mechanical properties. The influence of TRR loading on the cure characteristics and mechanical properties of NR/SBR blends was studied through the cure test, tensile test, hardness test, rebound resilience test, and density test. Therefore, this study can prove that the utilization of TRR as filler in NR/SBR blends can potentially be a suitable approach to achieve the circular economy of rubber waste's abundance. The incorporation of TRR in NR/SBR blends provides a sustainable alternative to producing green, eco-friendly rubber-based products which would be beneficial to the rubber industry.

Materials and Method

Materials

Natural Rubber (NR) and Styrene-Butadiene Rubber (SBR) of commercial grade along with other ingredients including Zinc Oxide (ZnO), Stearic acid, Calcium Carbonate ($CaCO_3$), Poly(1,2-dihydro-2,2,4-trimethylquinoline (Parmanax TMQ), Santoflex, 2,2'-Dibenzothiazyl Disulfide (MBTS), Diphenylguanidine (DPG) and Sulfur (S) were purchased from Malaysian Rubber Board (MRB). The $CaCO_3$ used was Hakuenka $CaCO_3$ with a particle size of 70 nm and an average surface area of 21 m^2/g. Tire Reclaimed Rubber (TRR) granules were obtained from Yong Fong Rubber Industries Sdn. Bhd.

Preparation of Tire Reclaimed Rubber (TRR) filled NR/SBR blends

The fabrication process involved the blending of NR and SBR using a two-roll mill (Qingdao Shun Cheong Machinery Co., Ltd., Qingdao, China) and in accordance with the American Standard of Testing and Material (ASTM) designation D3184-80. The process began by introducing the NR and SBR rubber bases onto the mill, where they were mixed to create the base blend. Subsequently, a stepwise addition of various ingredients was carried out to achieve the desired composite. First, Zinc Oxide (ZnO) was carefully added to the base blend, followed by Stearic Acid (HST) to facilitate dispersion and enhance processing, Calcium Carbonate ($CaCO_3$) as filler, Poly(1,2-dihydro-2,2,4-trimethylquinoline) (Parmanax TMQ) to serve as an antioxidant, Santoflex as antioxidants and anti-degradants to enhance the material's resistance to degradation, 2,2'-Dibenzothiazyl Disulfide (MBTS) as an accelerator, Diphenylguanidine (DPG) as co-accelerator, and finally Sulfur (S) to initiate the vulcanization process. The formulation of TRR filled NR/SBR blends is summarized in Table 1. Formulation 1 represented the control group without TRR, while formulations 2 to 9 encompassed varying loadings of TRR ranging from 20 to 100 phr.

Table 1: Formulation of TRR filled NR/SBR blends

Ingredients (phr)	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**
NR	50	50	50	50	50	50	50	50	50
SBR	50	50	50	50	50	50	50	50	50
TRR	0	20	30	40	50	60	70	80	100
ZnO	5	5	5	5	5	5	5	5	5
HST	1	1	1	1	1	1	1	1	1
$CaCO_3$	100	100	100	100	100	100	100	100	100
Parmanax TMQ	1	1	1	1	1	1	1	1	1
Santoflex	1	1	1	1	1	1	1	1	1
Accelerator	1.4	1.4	1.4	1.4	1.4	1.4	1.4	1.4	1.4
S	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5

Testing

Cure characteristics test

The evaluation involves subjecting the samples to analysis by utilizing an oscillating disk rheometer, adhering to the ASTM D 2048 standard. Rubber samples with a weight of approximately 5-10 g were tested and the cure characteristics were studied at 160 °C. This assessment of cure characteristics aims to derive essential parameters such as the scorch time (ts_2), optimum cure time (tc_{95}), maximum torque (M_H), minimum torque (M_L) values, and the cure rate index (CRI), all of which are derived from the resulting curve.

Tensile test

The rubber blends undergo a tensile test in accordance with ASTM D412-061, conducted at room temperature (25 ± 2 °C), utilizing a consistent crosshead speed of 500 mm/min. The samples are configured into dumbbell shapes. This tensile test enables the determination of critical mechanical properties of rubber, including tensile strength, elongation at break, and moduli (M100 and M300). Furthermore, the reinforcement index (RI), a key parameter reflecting material reinforcement, is calculated using the Equation (1):

$$Reinforcement\ Index, RI = \frac{M300}{M100} \tag{1}$$

where M300 signifies the modulus at 300% elongation and M100 denotes the modulus at 100% elongation.

Rebound resilience test

The determination of rebound resilience in the rubber specimens is carried out through a rebound resilience test performed using a dedicated rebound resilience tester. Following the guidelines outlined in ASTM D7121, the rubber samples are subjected to this assessment. Through this test, the rubber's percentage of rebound resilience is quantified and subjected to thorough analysis. The rebound resilience is quantified in percentage terms, calculated according to the equation:

$$Rebound\ Resilience, RR = \left(\frac{h_2}{h_1}\right) \times 100 \tag{2}$$

where RR (%) represents the rebound resilience, while h_2 (mm) signifies the height post-testing and h_1 (mm) stands for the initial height.

Hardness and density measurement

The hardness of a material is defined as the resistance of the material from having a permanent deformation. Hence, in order to assess the hardness of the rubber specimens, an IRHD Hardness tester (Shore A) was used, following the guidelines outlined in ASTM-D1415. The density is measured by the densitometer.

Results and Discussion

Cure characteristics

The cure characteristics of different TRR loading in NR/SBR were summarized in Table 2. Minimum torque (M_L) is the lowest value of torque recorded and represents the stiffness and viscosity of the unvulcanized compound. Maximum torque (M_H) is the highest torque recorded and increment of torque would indicate that crosslinking between polymer chains dominates. Once torque reaches the plateau, this shows that the curing process is complete and a stable network within the polymer blends has been formed. Delta torque, Δ t is the difference between M_H and M_L [13].

As can be seen, the Δt showed a significant decrease with increasing loading of TRR in NR/SBR blends. The Δt reduced by 56.1% when 100 phr of TRR was incorporated in the NR/SBR blends. The decrease in Δt is an indication that the NR/SBR blends would have a slight reduction in the hardness of NR/SBR blends. The decreasing Δt of the NR/SBR blends also meant a reduction of crosslink density, hence negatively affecting the modulus of the NR/SBR blends where a lower modulus would be obtained [14]. This is in accordance with the findings of M300 of the NR/SBR blends which is shown in Figure 3.

From the values of T_{c95}, it slightly increases with the increase in the amount of TRR in NR/SBR blends. The t_{c95} increased by 16.7% when 20 phr of TRR was added and had a significant increase of 30.1% when 100 phr of TRR was added. A higher t_{c95} would suggest a decrease in cure rate which is observed in Table 2. The cure rate index (CRI) showed that a lower curing reaction was achieved as the content of TRR increased. This could be due to the nature of calcium carbonate particles itself being isotropic. Therefore, causing a small nucleation effect [7]. Moreover, these results could be attributed to the interaction of the calcium carbonate in NR/SBR blends with curatives, such as activators and accelerators. They tend to absorb more of the active ingredients. Hence as a consequence, the curative contents needed for the sulphur vulcanization process were decreased, resulting in cure retardation. In addition, the existence of unreacted curative and precursors in the TRR may have contributed to the observed cure characteristics results.

Table 2: Cure characteristics of TRR filled NR/SBR blends

Properties	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**
Min. Torque (M_L), dNm	1.49	1.44	1.36	1.47	1.51	1.54	1.51	1.5	1.47
Max. Torque (M_H), dNm	9.67	8.42	7.76	6.87	6.86	6.19	6.05	5.6	5.06
Δ Torque (M_H–M_t), dNm	8.18	6.98	6.4	5.4	5.35	5.65	4.54	4.1	3.59
Scorch time (ts_2), min	1.27	1.51	1.53	1.63	1.57	1.73	1.7	3.54	2.08
Optimum Cure Time (tc_{95}), min	3.04	3.65	3.72	3.79	3.72	4.26	4.12	4.22	4.35
Cure Rate Index (CRI)	56.49	46.72	45.66	46.29	46.51	39.52	41.32	147.1	44.05

Tensile properties

Figure 1 illustrates the impact of varying TRR loadings, ranging from 0 phr to 100 phr, on the tensile strength of the NR/SBR blend. The reference sample, which contains 0 phr of TRR, serves as the control. Notably, as the TRR content increases, discernible changes occur in the tensile strength, demonstrating a consistent decline in tensile strength. This diminished tensile strength with higher TRR loading can be attributed to the partially cross-linked structure inherent to TRR. This structural attribute renders effective dispersion within the continuous elastomeric matrix of rubber blends challenging [15]. The presence of partially cross-linked structures introduces varying lengths of crosslink between the rubber molecules. This variance in crosslink lengths creates zones of stress concentration during deformation, primarily at the shorter crosslink. Consequently, this phenomenon leads to the observed decrease in tensile strength upon the addition of TRR to the blend. Besides, the presence of residual oil in TRR might also affect the stiffness and strength of the rubber blends. Higher oil content can lead to increased flexibility due to the lubricating effect of the oil on polymer chains.

The influence of varying TRR loadings on the elongation at break (EB) of NR/SBR blends is depicted in Figure 2. Notably, the EB exhibits a consistent upward trend as the TRR content increases, with a notable enhancement in EB observed at TRR loadings of 20 phr and 30 phr. This phenomenon can be attributed to the progressive rise in crosslinking within the rubber-filler network. The presence of pre-existing sulfur in TRR contributes to the establishment of additional crosslinks between the rubber and the filler. This augmented crosslink density fosters stronger connections between the polymer chains [4]. As a result, the improved intermolecular interactions provide greater resistance to fracture and deformation, thereby leading to the observed increase in elongation at break at TRR loadings of 20 phr and 30 phr.

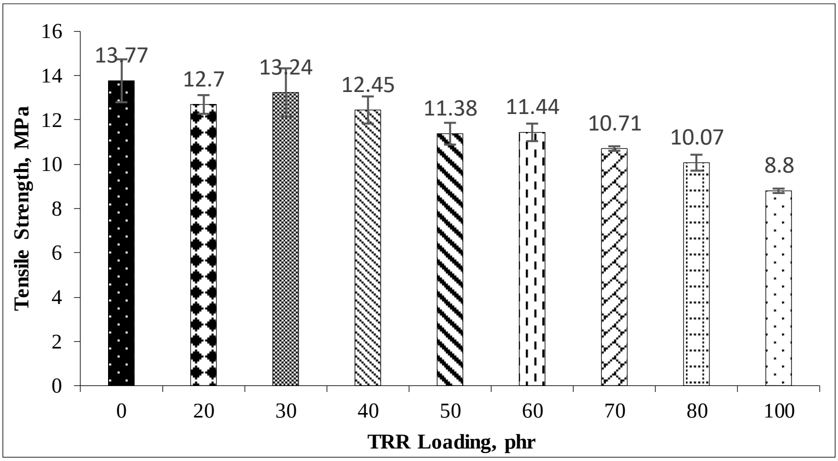

Figure 1: Effects of TRR loading on tensile strength of NR/SBR blends

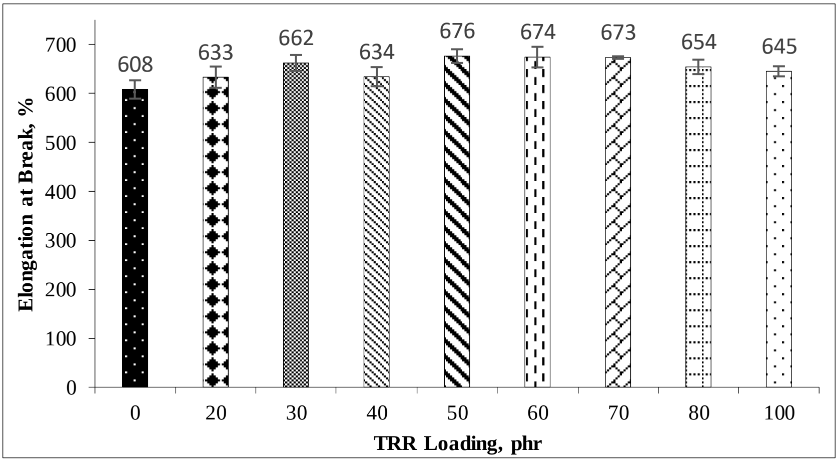

Figure 2: Effects of TRR loading on the elongation at break of NR/SBR blends

At the 40 phr loading of Tire Reclaimed Rubber (TRR), an observable dip in EB is followed by a declining pattern from 50 phr to 100 phr. This behaviour finds an explanation in the context of low interfacial bonding between TRR and NR/SBR, arising from the uneven distribution of TRR and NR/SBR [16]. The reclaiming process of TRR involves the potential breakdown of some crosslinks due to devulcanization [17]. This process could lead to the disruption of existing three-dimensional networks within the polymer chain structure of waste rubber. Consequently, the chain length between these networks becomes variable and non-homogeneous. This variability in chain length imparts an element of irregularity to the overall polymer structure. The fluctuating nature of EB stems from this non-uniform chain length distribution. The varying chain lengths create regions of differing mechanical properties within the rubber compound, which in turn influence its overall stretchability and deformation characteristics. This inconsistency in chain lengths contributes to the observed fluctuations in elongation at break.

The impact of TRR loading on the M300 of NR/SBR blends is demonstrated in Figure 3. Notably, the M300 values exhibit a consistent decreasing pattern. This behaviour can be attributed to the influence of the applied stress on M300. The decreasing trend in M300 indicates that the blends possess enhanced elasticity. This is due to the fact that M300 responds to the stress level applied. With diminishing M300, the blends display greater resilience upon deformation, highlighting their elastic nature. The observed fluctuations in the data could be ascribed to variations in crosslink lengths within the polymer chains - a consequence of the reclaiming process [17], as previously discussed and evident in Figure 2. When crosslink lengths are shorter, they experience higher stress levels, leading to initial breakage followed by propagation to adjacent regions. This unequal distribution of forces along the polymer chains leads to fluctuations in the modulus.

Basically, the decreasing trend in M300, denoting heightened elasticity, is a consequence of stress level variations impacting M300 behaviour. Fluctuations in modulus arise from the non-uniform crosslink lengths resulting from the reclaiming process, as shorter links endure greater stress and initiate a chain reaction of breakage.

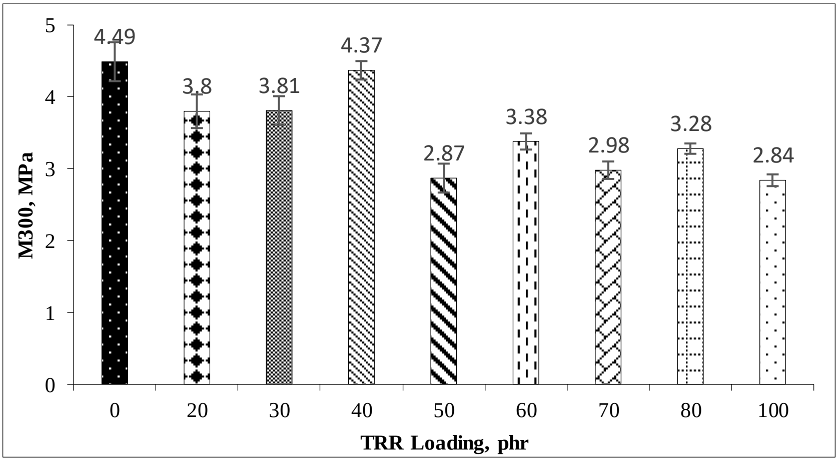

Figure 3: Effects of TRR loading on the M300 of TRR-filled NR/SBR blends

Reinforcement Index (RI)

Figure 4 illustrates the impact of TRR loading on the RI. This index serves as a gauge of whether a substantial reinforcing effect is present in the blend [18]. Notably, the addition of TRR loading appears to exert a limited influence on the RI, as the discrepancies between various TRR additions are relatively minimal. The data presented in Figure 4 underscores this observation, where the reinforcement index demonstrates a modest range, spanning from 2.02 to 2.2. This narrow span indicates that the incremental increase in TRR loading does not notably affect the reinforcement index of the blends. While this might render the blends less suitable for applications necessitating robust mechanical properties, it potentially positions them as candidates for non-reinforcing fillers in general rubber good products like children's toys, or as diluents within the rubber industry.

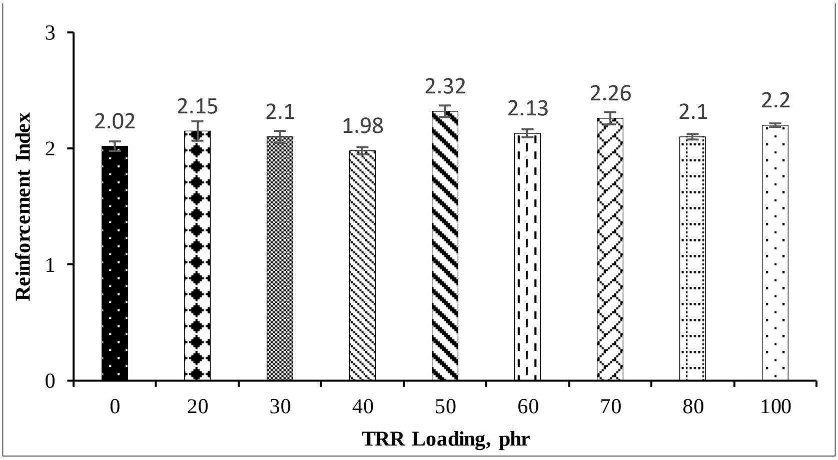

Figure 4: Reinforcement index of TRR Filled NR/SBR blends

Hardness and density

Figure 5 presents the density and hardness characteristics of TRR filled NR/SBR blends. Notably, both the hardness and density of the blends exhibit a decreasing trend as the TRR content increases. The interrelation between rubber hardness and density substantiates this correlation [19]. This reduction in hardness and density can be attributed to the comprehensive reclaiming process undergone by TRR, which leads to the disruption of 3D network structures [17]. This disruption contributes to diminished rigidity within the rubber, driven by the increased mobility of polymer chains [20]. Consequently, the incorporation of TRR into the blends fosters greater chain flexibility, ultimately leading to the observed decreases in hardness and density.

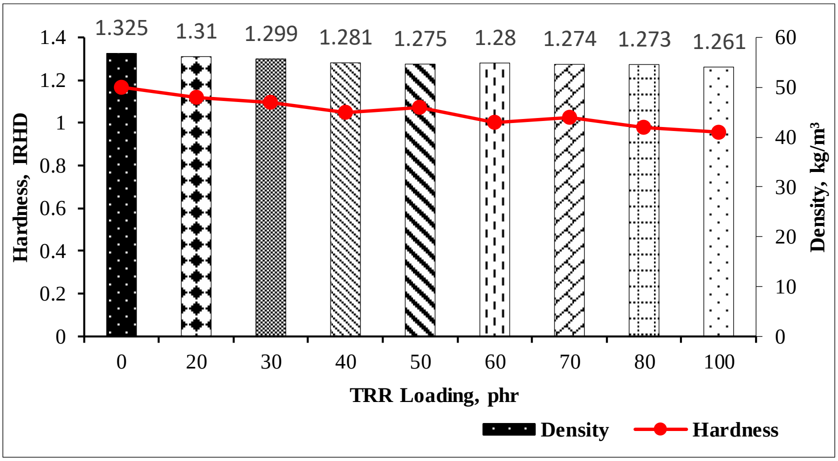

Figure 5: Hardness and density of TRR filled NR/SBR blends

Rebound resilience

Figure 6 presents the impact of TRR loading on the rebound resilience of NR/SBR blends. Rebound resilience, denoting the ratio of energy recovered to energy applied during deformation induced by a single impact, constitutes the essence of this assessment [21]. Notably, a higher resilience value corresponds to increased elasticity within the sample.

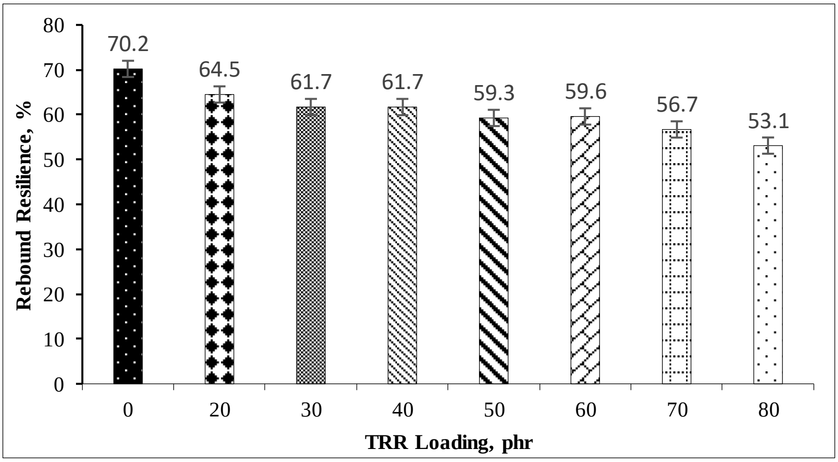

Figure 6: Rebound resilience of TRR-filled NR/SBR blends

The elasticity of the samples showcased a decline, a consequence of the non-uniform deformation attributed to the variable crosslink lengths present within TRR, as previously discussed. This variation in crosslink lengths [17], an outcome of the reclaiming process endured by TRR, contributes to the observed declining trend in rebound resilience as TRR loading increases. However, there was only a slight decrease in rebound resilience of NR/SBR blends when the loading of TRR increased. Even at the optimum loading of TRR where 30 phr of TRR was added, the rebound resilience still maintained at a high percentage of 65%. This way, utilization of TRR as a non-reinforcing filler in rubber blends provides a promising solution to reduce waste pollution generated by end-of-life rubber products [22]-[24], hence producing cost-efficient, sustainable general rubber goods applications such as toys and belts [25].

Conclusions

In conclusion, it can be said that TRR affects the curing characteristics and the mechanical properties of NR/SBR blends due to its low molecular weight. Moreover, the chain scission during the reclaiming process of TRR causes a decrease in molecular weight. Increasing the loading of TRR as filler in NR/SBR blends reduced the mechanical properties of NR/SBR blends. However, based on the results, 30 phr of TRR was the most optimum loading to be incorporated as filler in NR/SBR blends because it was able to achieve high tensile properties and maintained its high hardness and rebound resilience properties in comparison with other TRR loadings. Therefore, TRR can be a potential filler in NR/SBR blends when its optimum loading ratio is used. TRR can be judiciously employed as a non-reinforcing filler, ideally suited for rubber products that do not necessitate high mechanical attributes or as a means to curtail production costs. With this, the utilization of TRR as filler in NR/SBR blends can offer a promising solution to address the pervasive issue of waste pollution arising from the extensive use of rubber products.

Contributions to Authors

Conceptualization, formal analysis, S.N.L.M.; writing-original draft preparation, N.J.H.N.; methodology, S.N.L.M and D.K.; writing- review and editing, S.N.L.M and N.N.I.N.I. All authors have read and agreed to the published version of the manuscript.

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Conflict of Interests

The authors state no conflict of interest.

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