Preliminary Investigation on the Mechanical Properties of Carboxylated Nitrile Butadiene Rubber (XNBR) Blended with Different Loadings of Butyl Reclaimed Rubber (BRR)

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ABSTRACT

Reclaimed rubber (RR) from butyl tubes was evaluated as a partial and full replacement of a virgin rubber which is carboxylated nitrile butadiene (XNBR). This study involves the blending of carboxylated nitrile butadiene rubber with butyl reclaimed rubber (XNBR/BRR) in various loadings and

ISSN 1823-5514, eISSN 2550-164X © 2024 College of Engineering, Universiti Teknologi MARA (UiTM), Malaysia. https://doi.org/10.24191/jmeche.v21i1.25367 cured with sulfur by using a two-roll mill. The objective is to investigate the effect of RR loading on crosslink density and mechanical properties of XNBR/BRR blends. The mechanical properties such as hardness, rebound resilience, tear strength, tensile strength, elongation at break, and modulus were evaluated. In this study, the amount of reclaimed rubber varied from 0 phr to 161 phr. The results showed that the rebound resilience, tear strength, and tensile strength had a reduction of 43%, 27%, and 67%, respectively. Also, the hardness and modulus increased by 17% and 60% each. As for the elongation at break, it had a decrease of 69% and then an increment of about 77%. These results appeared due to the low molecular 1 weight of the BRR800 and the presence of processing oil and carbon black in the reclaimed rubber itself. This study could prove that reclaimed rubber can be fully utilized by putting it to good use in producing good quality rubber-based products.

Keywords: Reclaimed Rubber (RR); Butyl Reclaimed Rubber (BRR); Carboxylated Nitrile Butadiene Rubber (XNBR); Blending; Mechanical Properties

Introduction

Polymer blending is where two or more types of rubber are blended together to produce a new type of material that has excellent properties and are not found in individual rubber component [1]-[2]. By using this method, a newly developed material can be created, and polymer blending can be utilized to substitute present-day virgin polymer. Through the method of blending polymers together, different sorts of polymer blends can be produced. These polymer blends can either be a blend of thermoplastics or elastomers or even both thermoplastic and elastomer. With the combination of different compositions of polymers in a blend, different types of properties are achieved, and this is what makes each of these polymer blends rare and distinctive [2]. Each rubber product has its own required properties for the use of specific applications; hence the blends are varied to fulfill those requirements and needs. There are many benefits that can be obtained from polymer blending where polymer blending lowers both production cost and production time in comparison with producing new monomer or using a new polymerization method. Many previous studies were conducted to study polymer blending and the properties outcome of each blend. For example, Nitrile Butadiene Rubber (NBR/r-GTR) [3], Polylactide/ Polybutylene Adipate-co-terephthalate/ Carboxylated Nitrile Butadiene Rubber (ECOVIO/ XNBR) [4], and Ethyele Propylene Diene Monomer Rubber/ Nitrile Butadiene Rubber (EPDM/NBR) [5]. In the study involving NBR and r-GTR, the incorporation of Reclaimed Rubber (RR) with NBR led to an increment of density from 1.00 to 1.09 g/cm^3 , tensile strength (from 2.6 to 4.6-5.1 MPa), modulus at 100% (from 1.2 to 2.22.9 MPa), hardness (from 36 to 58-62 Shore A) and reduction in elongation at break (from 571% to 167-264%) [3]. As for the study regarding polylactide/poly (butylene adipate-co-terephthalate) blend (ECOVIO) in XNBR, the addition of ECOVIO in XNBR enhanced both the tensile modulus and tensile strength of the blend significantly. This could be a sustainable method of developing smart polymeric blends that could potentially substitute commodity plastic packaging products [4].

Nitrile Butadiene Rubber (NBR) is highly utilized in rubber-based industrial and automotive industries. NBR has a temperature range of -40 °C to +125 °C, hence it is able to resist a wide temperature range and is used in multiple automotive applications [6]-[8]. Nitrile compound 1 is better than most elastomers in terms of properties such as compression set, tear strength, and abrasion resistance, however, it has low resistance to ozone, sunlight, or weather [9]-[10]. As time goes by, more studies are done on NBR. The production of carboxylic elastomers was done first in the year 1930 and continued to increase studies involving synthetic elastomer materials which include Carboxvlated Nitrile Butadiene Rubber (XNBR) [11]. When the polymer backbone of NBR is introduced with carboxylic acid groups, this ultimately changes the properties of the rubber itself. This is due to the presence of additional crosslinking in the polymer, hence a polymer matrix that has enhanced tensile strength, tear strength, tensile modulus, and abrasion resistance is created. Moreover, the polymer matrix will have a decreased compression set, resistance to water, rebound resilience, and low-temperature properties [12].

It has been reported that approximately 10 million tons of rubber waste are discarded all around the globe. Rubber waste from used tires alone is a worrying matter and the numbers are still escalating up to this day [13]. Malaysia is one of the countries that consumes a huge amount of rubber which explains the abundance of rubber waste from all sorts of rubber products, and this could lead to environmental problems, pollution, and health safety issues [14]-[15]. The increment of rubber waste in the landfills needs to be overcome immediately and this rubber waste can potentially bring actual benefits to mankind [16]. These rubber wastes are usually discarded at landfills straight away, however since there has been a development in the latest technology, instead of immediate disposal at landfills, the rubber waste is reclaimed to be used in producing rubber-based goods. RR is an example of rubber where the source of these RR is from vulcanized rubber scraps, RR contains a mix of various compounding ingredients such as rubber, zinc oxide, stearic acid, reinforcing filler, accelerators, processing oil, and sulphur that are originally obtained from the reclaimed rubber compound [17]. There has been a high demand for RR recently due to their lost cost and ability to provide rubber with property improvement. An example of RR is BRR which is derived from reclaimed butyl inner tubes. One of the advantages of BRR is that BRR has low build-up when being processed and accelerated cure rate properties [18].

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BRR are proven to have a shorter chain therefore easing the processing steps in comparison with virgin butyl rubber [19]. A previous study was done to investigate the influence of a sulphur vulcanization system (conventional and efficient vulcanization) on the mechanical properties and heat aging resistance of Natural Rubber/Tire Tread Reclaimed Rubber blends (NR/TRR) [2]. The study resulted in an increment of hardness and modulus of blends as the content of RR increased. However, as for other mechanical properties, a decreasing trend was observed. With improved hardness and modulus, the blends in both curing systems are suited for certain uses and applications.

In this research, varying loading of BRR from 0 to 161 phr is blended into XNBR in order to study the influence of BRR loading on the crosslink density and mechanical properties of XNBR composites.

Experimental Section

Analytical test of BRR

This analytical test was conducted in a lab of a local research center in Sungai Buloh in order to find out the contents of the BRR used in this study. The analytical analysis concluded that the BRR used in this study contained a polymer content of 58.7%, a total of 34.3% of carbon black, an ash content of 3.2%, solvent extraction of 3.8%, zinc oxide of 0.2%, and total sulphur of 1.2%. As for the accelerators and antioxidants, they were not able to be detected as the detection limit of the analytical test was 0.01%. Hence, it shows that there is a very low content of accelerators and antioxidants in the BRR, making them insignificant and negligible. The polymer type in the BRR that was identified through this analytical test was a combination of Butyl and Ethylene Propylene Diene Monomer (EPDM). The formulation of XNBR/BRR is created based on the polymer content of BRR. The blend ratio of XNBR/BRR blends are 70/48, 60/65, 50/81, 40/97, 30/113, and 0/161 phr.

Preparation of XNBR/BRR Blends

XNBR and BRR were purchased from Aras Bakti Ventures and Yong Fong Sdn. Bhd., respectively. Table 1 shows the compounding ingredients used in this study. Blends with 70/48, 60/65, 50/81, 40/97, 30/113, and 0/161 phr ratio of XNBR/BRR which is equivalent to 70/30, 60/40, 50/50, 40/60, 30/70, and 0/100 % of BRR in each XNBR/BRR blends were prepared which can be seen in Figure 1. Each blend contained the same amount of chemical composition. The compounding process was done with a two-roll mill which is shown in Figure 2. XNBR/BRR blends were prepared by masticating the rubber and adding activators, fillers, and curing agents into the mix. After that, the final compounds were cured with a Monsanto Moving Die Rheometer at 160 °C to obtain their curing properties.

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Figure 1: Compounded XNBR/BRR blends



Figure 2: Two-roll mill machine used to prepare XNBR/BRR blends

Ingredients	Amount (phr)
XNBR	Variable
BRR	Variable
ZnO	3
Carbon black	35
Stearic acid	0.2
Accelerator	3.55
Sulphur	1.3

Table 1: Blend ingredients

Experimental Section

Crosslink density test

This test was conducted according to ASTM D6814 (2018). In order to determine the crosslink density, each rubber blend was immersed in toluene and acetone. Blend samples were weighed to about 0.2 - 0.25 g and this weight was noted as initial weight. After that, samples were put in bottles that had 10 ml of pure toluene and set aside to swell for a period of 24 hours at room temperature. These bottles containing samples were labelled accordingly which can be seen in Figure 3. Then, the toluene was taken out, replaced with a new set of toluene, and left for the next 24 hours. Once 24 hours were up, the swollen samples were weighed. Next, the bottles were filled with acetone for 30 minutes. Lastly, the acetone was drained and samples were left to dry until the following day. The calculation for crosslink density can be done with the Flory-Rehner Equation (1).

$$-In (1 - Vr) - Vr - \Psi V_{r^2} = 2\rho Vo [X] Vr_3^1$$
(1)

where Vr is the volume fraction of rubber; ψ is the rubber-solvent interaction parameter; ρ is the density of the polymer; and Vo is the molar volume of solvent.



Figure 3: Labelled bottles containing XNBR/BRR blend samples for crosslink density test

Tensile test

A tensile test is carried out to obtain tensile properties which are important for material specifications for quality guarantee. Through the tensile test, a few tensile properties can be obtained including tensile strength, elongation at break, and modulus. Specimens with a gauge length of 20 mm were used for this test. The tensile test was carried out according to ISO 37 specifications

and at a constant strain rate of 500 mm/min until fracture of the specimen occurred. The dumbbell-shaped specimens used in accordance with ISO 37 is shown in Figure 4.



Figure 4: Dumbbell-shaped test piece according to ISO 37 for tensile test

Crescent tear test

With a tear test, a material's tear strength can be studied. This test measures the force needed to initiate a tear in a material, in continuation of a prepared cut or nick or completely across the width of specific test pieces. In this study, the crescent tear test technique is used. The tear strength is measured according to specification ISO 34. ISO 34 defines tear strength as the measurement of the force required to tear a test sample divided by specimen thickness. The measurement of the specimen's average thickness was taken first, and specimens were then placed in the grips of the testing machine and pulled at a rate of 20 inches per minute until the specimen was ruptured. In this study, eXpert 7600 Single Column Testing Machine was used to measure tear strength. Tear strength is recorded as force per unit width (kN/m).

Rebound resilience test

The definition of rebound resilience is the ratio of energy being returned to the energy being applied during deformation which is due to the indentation at a single impact. Rebound resilience measures the elasticity of a rubber during exposure to varying stresses. Rebound resilience has sensitivity to temperature where a minor temperature difference could significantly affect rebound resilience results. This test was carried out with ISO 4662 specifications. Wallace Dunlop Tripsometer was used in this test.

Hardness test

The ability to resist permanent indentation is the definition of hardness. In this test, the hardness samples were pressed with an indenter and tested under a specific load for a period of time. The indention's size or depth was then measured. Figure 5 shows the test specimen for the hardness test. A hardness test was carried out to determine the suitability of a material for a specific use.

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This test was conducted according to ASTM D 2240 and a Durometer (Shore A) (Qingdao Shun Cheong Machinery Co., Ltd., Qingdao, China) was used.



Figure 5: Test specimen for hardness test

Results and Discussion

Tensile

The tensile strength results with the increment of BRR loading is indicated in Figure 6. It was observed that as the loading ratio of BRR increases, the XNBR/BRR blends undergo a reduction in tensile strength. The blend with 30% of BRR showed the highest tensile strength which was 12.92 MPa among the other blends. When 40% of BRR was added into the XNBR/BRR blend, the tensile strength was lowered to 7.19 MPa, a reduction of about 44.4% in comparison with the XNBR/1 BRR blend of 30% BRR. The incorporation of 50% BRR into the XNBR/BRR blend caused a slight increase in tensile strength where the tensile strength increased to 9.76 MPa. This slight increment could be contributed by the carbon black content of BRR itself (refer to analytical test). As the loading ratio of BRR in the XNBR/BRR blend increased to 60% and 70%, the tensile strength significantly reduced to 2.44 MPa and 1.66 MPa, respectively. The lower molecular weight of BRR can be one of the reasons contributing to the lowering of tensile strength [20]. BRR went through the reclaiming process. Hence, the high shear and temperature during the reclaiming process progressively break down the molecular chains in BRR into shorter segments. This resulted in a significant reduction in the tensile strength of the blends when this low-molecular-weight BRR is incorporated. Moreover, the decrease in tensile strength of XNBR/BRR blends might be associated with weak bonding between the rubber chains and its irregular networking too. 100% of BRR showed a considerably high tensile strength of 4.23 MPa. In fact, it is higher compared to XNBR/BRR blends with 60% and 70% of BRR. Based on the analytical test indicated that BRR contains about 34.3% of carbon black. The BRR composite became compact and reinforced due to reinforcement provided by the reinforcing filler, carbon black which was added into the composite and further aided by the 34.3% of carbon black content that was originally present in BRR.



Figure 6: Tensile strength of XNBR/BRR blends

Figure 7 shows the results of elongation at break. Elongation at break percentage decreased with the increment in BRR loading, ranging from 30% to 100%. As the blend has an increase of BRR 1 loading from 30% to 70%, a reduction in the elongation of break of the blends was observed where the value of elongation decreases from 323% to 99%. Among all the blends, the blend with a ratio of 70% BRR shows the lowest value of elongation at break as it contains a high ratio of BRR compared to its ratio of XNBR. Since BRR has a low molecular weight and has the presence of reinforcing filler, which is carbon black, this can cause the molecular orientations to be inhibited, resulting in the failure of the sample at lower elongation. The decrease in elongation at break is due to the reduction in flexibility of material where the higher the crosslink density, the bigger the restriction of the rubber chain's mobility [17], [21]-[22] which can be seen in Figure 8. This is also consistent with the lower tensile strength values obtained for the blends. However, in the presence of only BRR in the vulcanizate, it shows a high value of elongation of break which is 431% when the XNBR/BRR blend has 100% of BRR. Based on the analytical test, BRR has the presence of sulphur in its content, which results in the increased crosslink density between the rubber chains. Elongation at break decreases when crosslink increases because more crosslinks are able to prevent the rubber from being strained [23]. With high crosslinks between polymer chains, the chains cannot move relative to each other as easily [24]. It can also be said that as the loading of BRR increases, the crosslink density shows a decreasing trend. Blends with a higher ratio of BRR have low

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crosslink density could be due to the chain scission that occurred during the reclamation process of BRR which resulted in the reduction of the molecular weight of BRR itself [25]. The breakdown of crosslink and polymer chains caused this to occur [26]. Hence crosslink density is dependent on the loading of BRR added to the blends. The lower level of crosslinking at higher BRR loading could also be attributed to the fact that BRR is already partially crosslinked due to the sulphur being present in the BR originally. Therefore, there is relatively less reaction site available for any crosslinking to take place [27].



Figure 7: Elongation at break of XNBR/BRR blends



Figure 8: Elongation at break and crosslink density of XNBR/BRR blends versus percentage of BRR (%)

Figure 9 shows the modulus at 100% strain (M100) of XNBR/BRR blends containing varying loading of BRR. Modulus is a measure of stiffness of the material which is related to the material's network structure and crosslink density. Based on Figure 9, it can be seen that M100 decreased with the increasing loading of BRR. Elastic modulus is inversely proportional to the elongation in the rubber chains. This leads to a reduced 1 elastic modulus of the material and gives the material a spongy, compressible, elastic property.



Figure 9: M100 of XNBR/BRR blends

Crescent tear

The results for tear strength with increasing loading of BRR in the XNBR/BRR blends are shown in Figure 10. Tear strength generally indicates the materials' toughness and tear strength is usually proportional to the crosslink density which can be seen in Figure 11. It is shown that, the increasing ratio of BRR results in increasing in tear strength where tear strength was improved from 25.61 to 30.29 kN/m for XNBR/BRR blends. The increment and improvement of tear strength could be due to the presence of reinforcing carbon black in the BRR. As the amount of BRR increases and at the presence of only BRR in the vulcanizate, the tear strength obtained decreases and apparently shows a low value. This is due to the lower molecular weight of the RR itself and due to the absence of XNBR in the blend composition. During the tear test, propagation of the tear along the material is a crucial step. The presence of reinforcing filler can either arrest or deflect the crack front, leading to better and higher tear strength results [17], [21]-[22]. The effect of carbon black present in the rubber could have suppressed the BRR effect in the blends and the reinforcing filler is uniformly dispersed in the rubber matrix which can be attributed to the aggregation of BRR. This resulted in weak point formation in the rubber matrix, which then decreased the tear strength value and elastomeric strength [28].



Figure 10: Tear strength of XNBR/BRR blends



Figure 11: Tear strength and crosslink density of XNBR/BRR blends versus percentage of BRR (%)

Rebound resilience

As the proportion of reclaimed rubber in the blends increases, the rebound resilience of the blends decreases [22]. The results are shown in Figure 12. The lowering of rebound resilience can be due to the filler content of the reclaimed rubber [27]. The incorporation of filler 1 whereby the addition of BRR into the blends increased the chances of energy loss at the interfaces which then resulted in lower resilience. There is about a 40% reduction in rebound

resilience when the percentage of BRR in the blend increases from 30% to 70%. Reduced resilience can indicate a stiffer blend.



Figure 12: Rebound resilience of XNBR/BRR blends

Hardness

Figure 13 shows the hardness results for the XNBR/BRR blends at different loading of BRR ranging from 30% to 100% in the blend. It shows that the increment of the loading ratio of BRR in XNBR/BRR helped to maintain the hardness of the XNBR/BRR blend composite. When 30%, 40%, and 50% of BRR are incorporated into the XNBR/BRR blend, the hardness of the composite was maintained at 58 IRHD, 57 IRHD, and 57 IRHD, respectively. A higher loading of 60% BRR resulted in a slight increment of hardness where the hardness increased to 60 IRHD. As for the addition of 70% BRR in the XNBR/BRR blend, the blend had a slight decrease in hardness where the hardness of the blended composite reduced to 55 IRHD. However, it can be said that the incorporation of BRR in the XNBR/BRR blend led to a consistent hardness value, ranging from 55 to 60 IRHD. The slight reduction of hardness value when 70% of BRR is incorporated in the XNBR/BRR blend could be due to BRR containing a proportion of processing oil, which causes sliding and separation of rubber chains that results in the reduction ability to resist penetration surface. Hence, the vulcanizates become less stiff. Another explanation would be that the Durometer's hemispherical indenter penetrated and broke the vulcanizate slightly due to the microcellular 1 network was very brittle and dense [17], [21]-[22]. However, the blend which consists of 100% of BRR had a high value of hardness, 88 IRHD. The carbon black originally present in BRR which is mentioned in the analytical test may have contributed to the high value of hardness obtained by the blend which has 100% of BRR. The higher proportion of carbon black caused the blend to be stiff.



Figure 13: Hardness of XNBR/BRR blends

Conclusion

In this research, an effort was made to create sustainable rubber-based goods with XNBR/BRR blends. From the results, it can be deduced that the tensile strength, elongation at break, M100, tear strength, rebound resilience, and hardness of the blends are dependent on the loading of BRR. Higher loading of BRR caused a reduction in all mechanical properties except for hardness where the incorporation of BRR in the XNBR/BRR blend was able to maintain the high hardness of the XNBR/BRR blend composite. The low molecular weight of the RR, weak bonding between the rubber chains, the irregular networking, and the proportion of processing oil with the presence of reinforcing carbon black in BRR itself contribute to the results obtained. It can be said that the addition of BRR into XNBR creating XNBR/BRR blends at an optimum loading ratio does improve the mechanical properties of the XNBR/BRR blend composites. It was found that the optimum loading ratio of BRR is at 30% (blend ratio of 70/48 phr XNBR/BRR) was chosen as the best loading ratio compared to the others as it has moderate hardness, 1 high tear strength, high rebound resilience, high tensile strength, high elongation at break and high modulus too. With this, it shows that RR such as BRR has the potential to improve the properties of a rubber-based product and reduce the usage of virgin rubber like XNBR. This approach could be an alternative to decrease the abundance of rubber waste and gain benefits from rubber waste by turning it into RR for other sorts of rubber applications.

Recommendation

One of the recommendations that could be implemented for future studies would be the incorporation of any type of processing oil or lubricant into the formulation to ease the processing much more, reduce viscosity, and improve compatibility [29]. For instance, paraffin oil. This is because paraffin oil acts as a lubricant and it has been reported that mixtures with higher paraffin oil content would have better performance.

Contributions of Authors

The authors confirm the equal contribution in each part of this work. All authors reviewed and approved the final version of this work.

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Conflict of Interests

All authors declare that they have no conflicts of interest.

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