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Exploring The Effect of Devulcanized Nitrile Butadiene Rubber Glove (dNBRg) on the Cure Characteristics and Mechanical Properties of Virgin Nitrile Butadiene Rubber (vNBR) Blends

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ABSTRACT

The COVID-19 pandemic led to a significant surge in the use of disposable nitrile rubber gloves, raising environmental concerns due to the large volume of glove waste generated. Devulcanization presents a sustainable approach to addressing this issue by converting waste nitrile gloves into a value-added material known as devulcanized nitrile rubber glove (dNBRg), which can be utilized as a blend component. However, the awareness and application of dNBRg in rubber-related fields remain limited. This study investigates the effects of incorporating dNBRg at varying loadings (0–100 parts per hundred rubbers, phr) into virgin nitrile butadiene rubber (vNBR), with a focus on curing behavior and mechanical properties such as tensile strength and abrasion resistance. The blending of vNBR and dNBRg, along with compounding additives, was carried out using a two-roll mill. The results revealed that minimum torque (ML) increased with the addition of dNBRg, attributed to its higher viscosity, which is influenced by residual additives from both the original glove formulation and the new compounding ingredients. Maximum torque (MH) decreased at lower dNBRg loadings (20–40 phr) but began to increase at higher loadings (above 40 phr). Scorch

time (ts_2) remained relatively constant, while cure time (t_{90}) increased at higher dNBRg contents (70 and 80 phr), suggesting a slower vulcanization process. Fourier Transform Infrared (FTIR) analysis showed no new absorption peaks, indicating no significant chemical changes in the blend. Mechanically, tensile strength decreased with increasing dNBRg content due to a heterogeneous crosslink structure, where weakly crosslinked regions were more susceptible to chain slippage and microcrack formation under stress, leading to early failure. In contrast, elongation at break, modulus at 100% strain (M100), and volume loss all increased with higher dNBRg loadings, likely due to the increased crosslink concentration and non-uniform crosslink density within the rubber blends. These findings highlight the potential of dNBRg to partially replace vNBR in general-purpose rubber products that do not require high tensile strength, thereby promoting more sustainable rubber material development and offering a practical solution to the environmental and health hazards posed by the accumulation of rubber glove waste.

Keywords: Devulcanization; Devulcanized Nitrile Butadiene Rubber Glove; Virgin Nitrile Butadiene Rubber; Mechanical Properties

INTRODUCTION

Rapid technological advancements across various industries have driven a substantial rise in rubber demand, outpacing many other materials. Rubber's durability, flexibility, and cost-effectiveness make it indispensable in sectors such as automotive, construction, healthcare, and consumer goods. One significant application is in the production of rubber gloves, particularly for healthcare, industrial, and frontline use. According to the Malaysian Rubber Glove Manufacturers Association (MARGMA), global demand for rubber gloves is expected to grow by 12%, reaching approximately 368 billion pieces by 2025 [1]. Additionally, the Ministry of Investment, Trade and Industry (MITI) projects that the U.S. rubber glove market will grow to USD 4.17 billion by 2030, highlighting strong opportunities for Malaysian manufacturers [2]. During the COVID-19 pandemic, the global demand for rubber gloves surged dramatically, especially in Malaysia, where glove production doubled to meet personal protective equipment (PPE) needs [3]. Although production levels declined as the pandemic

subsided, the impact of this surge remains significant, particularly in terms of waste generation. Among the various glove types, nitrile butadiene rubber (NBR) gloves gained popularity due to their excellent chemical resistance and hypoallergenic nature, making them ideal for sensitive environments. Despite NBR having a relatively lower tensile strength compared to natural rubber, its resistance to oils, fuels, and solvents which are determined by its acrylonitrile content that makes it suitable for medical and industrial applications [4].

However, the sharp increase in glove production led to substantial amounts of waste, particularly from rejected gloves. Quality control measures, while essential, contribute to rejection rates of up to 15%, with defects such as pinholes and surface imperfections [5]. Improper disposal of these single-use or defective gloves poses serious environmental concerns, as most end up in landfills or are incinerated, leading to pollution and greenhouse gas emissions. The World Health Organization estimated that over 87,000 tonnes of PPE procured during the pandemic would eventually become waste, further stressing waste management [6]. To address this issue, recycling methods such as reclaiming and devulcanization have been explored. Reclaiming typically breaks both the carbon backbone and sulfur crosslinks, reducing material properties [7]. In contrast, devulcanization selectively targets sulfur crosslinks (C–S and S–S bonds), preserving the polymer backbone and partially restoring the elastomeric properties [8]. This allows devulcanized rubber to be reused in new formulations and the techniques include thermal, mechanical, chemical, physical, and biological methods [7,9]. Devulcanization of nitrile rubber glove waste (dNBRg) offers a promising route for recycling. It generates reactive sulfur-centered radicals and chain-end radicals that facilitate re-vulcanization during blending, enhancing material recovery and reducing waste. To further optimize performance, dNBRg can be blended with virgin rubber, allowing the development of sustainable materials with tunable properties for various applications.

This research proposes a novel approach by investigating the use of devulcanized nitrile rubber gloves (dNBRg) as a partial or full substitute for virgin nitrile butadiene rubber (vNBR). The study examines the effects of incorporating dNBRg at varying loadings from 0 to100 parts per hundred rubbers into vNBR based on the curing characteristics, crosslink density,

Fourier Transform Infrared Spectra (FTIR), and mechanical properties such as tensile strength, elongation at break, modulus, and abrasion resistance.

EXPERIMENTAL SECTION

Materials

This study utilized devulcanized nitrile butadiene rubber glove (dNBRg) with a Mooney viscosity of 35–50 ML (1+4), sourced from Bridge Fields Resources Sdn. Bhd. The dNBRg was produced from post-consumer nitrile glove waste through a combined mechanical and chemical devulcanization process using a novel devulcanizing agent. The primary rubber matrix, virgin nitrile butadiene rubber (vNBR) containing 35% acrylonitrile, along with all compounding ingredients, was obtained from Airelastic Industries Sdn. Bhd. The rubber compounding formulation included carbon black (CB), silica, zinc oxide (ZnO), stearic acid (HST), mercaptobenzothiazole disulfide (MBTS), tetramethylthiuram disulfide (TMTD), dioctyl terephthalate (DOTP), sulphur, bis[3-(triethoxysilyl) propyl] tetrasulfide (Silane 89), polyethylene glycol (PEG 4000), and Lowinox CPL.

Preparation of vNBR/dNBRg Blends

The vNBR was blended with dNBRg in various blend ratios at 80/d20, 70/d30, 60/d40, 50/d50, 40/d60, 30/d70, and 20/d80 by employing a conventional sulphur vulcanization (CV) system as shown in Table 1. Control samples consisting of 100 phr vNBR and dNBRg were also prepared. The rubber blends were mixed in accordance with ASTM D3182 by using a YF-150 two-roll mill at room temperature, with the mixing sequence detailed in Table 2. Cure characteristics of the blends were evaluated using a GoTech Moving Die Rheometer (MDR) in accordance with ASTM D5289, using approximately 4 g of each blend at 150 °C. The blends were then compression-moulded at 150 °C using a HI-TOP hydraulic hot press (HP-507) at 500 psi, based on the respective cure times (t_{90}). Moulds were preheated for 4 minutes prior to curing, and all samples were cooled at room temperature 24 hours before testing.

Table 1: Formulation of vNBR/dNBRg blends.

Ingredient	Loadings (phr)								
	vNBR	80/ d20	70/ d30	60/ d40	50/ d50	40/ d60	30/ d70	20/ d80	dNBRg
vNBR	100	80	70	60	50	40	30	20	0
dNBRg	0	20	30	40	50	60	70	80	100
ZnO	7	7	7	7	7	7	7	7	7
HST	3	3	3	3	3	3	3	3	3
MBTS/TMTD	1.3	1.3	1.3	1.3	1.3	1.3	1.3	1.3	1.3
Sulphur	2	2	2	2	2	2	2	2	2
Plasticizer	3	3	3	3	3	3	3	3	3
Lowinox CPL	1	1	1	1	1	1	1	1	1
Coupling agent	2	2	2	2	2	2	2	2	2
Silica	50	50	50	50	50	50	50	50	50
PEG 4000	2	2	2	2	2	2	2	2	2
CB	10	10	10	10	10	10	10	10	10

Table 2. Mill mix procedure of vNBR/dNBRg blends.

Step	Operation
1	Mastication of vNBR and dNBRg using two-roll mill for 2 to 5 minutes separately
2	Blending of vNBR and dNBRg until a uniform dispersion was visually confirmed
3	Addition of stearic acid and zinc oxide
4	Addition of lowinox CPL
5	Addition of silica alternately with silane 89 and DOPT
6	Addition of carbon black alternately with PEG4000
7	Addition of MBTS and TMTD
8	Addition of Sulphur
9	Sheet off

Testing

Cure Characteristics Test

The cure characteristics were determined following ASTM D5289 using the GoTech Moving Die Rheometer (MD-3000A) with oscillation frequency of 30-100 cycle/min. A sample weighing approximately 4 g was placed on the rheometer's die. The optimum cure time (t_{90}), scorch time (ts_2), minimum torque (M_L), maximum torque (M_H), and difference between M_H and M_L (M_H- M_L) also known as ΔT were then recorded.

Crosslink Density Measurement

A crosslink density was measured according to ASTM D471 using toluene, with the test conducted at room temperature (27 ± 2 °C). Three test samples measuring 1 cm × 1 cm were weighed and immersed in toluene until equilibrium swelling was achieved. Sample weights were recorded daily until no further change was observed, indicating saturation. The saturated weight represented the equilibrium swelling. After swelling, samples were dried in an oven at 70 °C for 20 minutes, cooled at room temperature for 15 minutes, and reweighed. Crosslink density was then calculated using the Flory-Rehner equation, as shown in Eq. (1).

$$-\ln(1-V_r)-V_r-\Psi V_r^{\,2} = 2\rho V_o[X]_{phy}V_r^{\,1/3} \quad (1)$$

where V_r denoted the volume fraction of rubber in the swollen gel, calculated using Eq. (2), was the rubber–solvent interaction parameter with a value of 0.6, Vo represented the molar volume of the solvent, which was 103.11, $[X]_{phy}$ indicated the crosslink density of the rubber blend, while ρ referred to the rubber density.

$$Vr = \frac{\frac{Dried\ weight}{\rho\ \text{of rubber}}}{\frac{Dried\ weight}{\rho\ \text{of rubber}} + \frac{Swollen\ weight}{\rho\ \text{of solvent}}} \quad (2)$$

Fourier Transform Infrared (FTIR) Analysis

This test was performed to identify the functional groups in the vNBR, dNBRg, and vNBR/dNBRg phases, as well as to evaluate any physical or chemical interactions that may have occurred during the blending of the two rubbers. The spectra were recorded using Perkin Elmer Spectrum One Series equipment with the attenuated total reflection (ATR) technique. Material of the ATR crystal used in this study is zinc selenide (ZnSe). A spectral resolution of 8 cm^{-1} with 16 scans was applied, and the scanning range was set from 4000 to 600 cm^{-1}.

Tensile Test

The tensile properties of the blends, including tensile strength, elongation at break, and modulus, were determined using an Instron 5569 series testing machine, following ASTM D412 standards. The tests were performed at room temperature (27 ± 2 °C) with a crosshead speed of 500 mm/min. The dumbbell die type use is ISO 37 type 2 with the specification of width 4 mm and gauge length 4.06 mm and range of thickness 4.09-4.64 mm.

Abrasion Resistance Test

The abrasion resistance of the blended samples was evaluated using a GoTech Din Abrasion tester (GT-7012-D), which applied a controlled force to a defined surface area in contact with Grit #60 abrasive paper. The test was performed in accordance with the ISO 4649:2017 Method A standard. Results were reported as volume loss (VL), calculated using Eq. (3). A standard rubber compound conforming to DIN specifications served as the reference material.

$$VL = \frac{M_t \times M_{const}}{P_t \times M_r} \tag{3}$$

where M_t is a mass loss (in mg), M_{const} is a mass loss (based on a standard reference compound), P_t is density of the rubber material (in mg/cubic millimeter), and M_r is mass loss of the reference compound.

RESULTS AND DISCUSSION

Cure Characteristics

Table 3 summarizes the cure characteristics of vNBR/dNBRg blends where unblended vNBR and dNBRg were acts as a control for blended. Key rheometric parameters analyzed include minimum torque (M_L), maximum torque (M_H), torque difference (ΔT), scorch time (ts_2), and optimum cure time (t_{90}). M_L, which indicates the viscosity and processability of the uncured compound, was lower for vNBR compared to dNBRg. This suggests that vNBR exhibits lower compound viscosity and provides ease of processing [10]. The higher M_L of dNBRg is attributed to the combined effect of residual additives from the glove formulation and the compounding additives used in this study, both of which indirectly contribute to increased viscosity. As the dNBRg content increased from 20 to 70 phr, the M_L also rose, indicating reduced processability due to restricted polymer chain mobility [7]. However, at 80 phr, M_L decreased slightly, which may be attributed to a reduction in crosslinking efficiency.

Table 3. Cure Characteristics of vNBR /dNBRg Blends.

Properties	Loadings (phr)								
	vNBR	80/ d20	70/ d30	60/ d40	50/ d50	40/ d60	30/ d70	20/ d80	dNBRg
M_L (dN-m)	6.57	7.56	6.00	6.65	11.81	14.26	17.99	15.25	21.57
M_H (dN-m)	15.77	13.39	9.81	8.90	15.95	17.21	35.07	31.58	30.70
ΔT (dN-m)	9.20	5.83	3.81	2.25	4.41	2.95	17.08	16.33	9.13
ts_2 (min)	1.39	1.24	1.29	1.44	0.40	1.50	1.23	1.05	1.00
t_{90} (min)	2.09	1.86	1.80	1.76	1.20	2.20	5.23	5.33	2.16

This reduction is likely caused by the formation of uneven rubber networks, potentially due to interference from residual additives present in the dNBRg. The higher M_L of dNBRg is attributed to the combined effect of residual additives from the original glove formulation and the compounding additives used in this study, both of which indirectly contribute to increased viscosity. Both M_H and ΔT exhibited non-linear behavior with increasing dNBRg content. M_H, which indicates the stiffness and crosslink density of the compound, was nearly twice as high for dNBRg compared to vNBR,

primarily due to the rigidity imparted by residual additives and pre-existing crosslinks. In the blends, M_H gradually decreased up to 40 phr dNBRg, likely due to dilution of the crosslink able vNBR phase. However, from 50 to 70 phr, M_H increased again as the dNBRg phase became dominant, with its inherent stiffness which contributed by residual fillers and partial crosslink structures playing a more significant role. The ΔT values, which represent the extent of new crosslink formation during vulcanization, initially remained similar between vNBR and dNBRg but increased significantly at 70 and 80 phr dNBRg, indicating the development of a denser crosslinked network at higher loadings. However, the reduction of ΔT on 40/d60 phr is due to low M_H value obtained and uncontrollable crosslink occurred in the rubber blend. Interestingly, despite the higher M_H observed in dNBRg, its ΔT was relatively lower than vNBR at earlier stages, which can be attributed to the presence of residual crosslinks and limited availability of free reactive sites. These residual crosslinks from previous vulcanization cycles reduce the number of new crosslinks that can form, while some curing agents may be consumed through side reactions with the devulcanized chains. Additionally, the presence of fillers such as carbon black restricts polymer chain mobility, further increasing torque values but hindering new crosslink formation. This explains the deviation from typical vulcanization trends observed in standard rubber compounds. The values of both scorch time (ts_2) and cure time (t_{90}) remained relatively unchanged with the addition of dNBRg to vNBR up to moderate loadings. Yet, the low ts_2 value of 50/d50 blend might be due to reactive site or residual chemical that reduce the induction time and heat distribution during mixing, leading to localized hotspots that accelerate early curing reactions. However, at 70 and 80 phr of dNBRg, the t_{90} values increased to 5.23 and 5.33 minutes, respectively. This increase may be attributed to a slower vulcanization process, likely due to delayed sulfur crosslinking between the double bonds in vNBR and the short-chain radicals present in the dNBRg

Crosslink Density

Swelling measurements are a commonly employed method to assess crosslink density. Figure 1 presents the crosslink density values for vNBR, dNBRg, and the respective blends. As shown, dNBRg exhibited a higher crosslink density than vNBR, primarily due to the presence of free radicals from short polymer chains generated during the chain scission of the original

crosslinked network in discarded gloves [11]. These radicals facilitated re-crosslinking either with other radicals or with sulfur-based crosslinkers used in the formulation [12]. The availability of these reactive sites in dNBRg promoted the formation of a denser three-dimensional network. As the dNBRg content in the blend increased, the number of short-chain radicals also rose, thereby enhancing the degree of re-crosslinking. This resulted in a progressive increase in the overall crosslink density of the blends with higher dNBRg loadings.

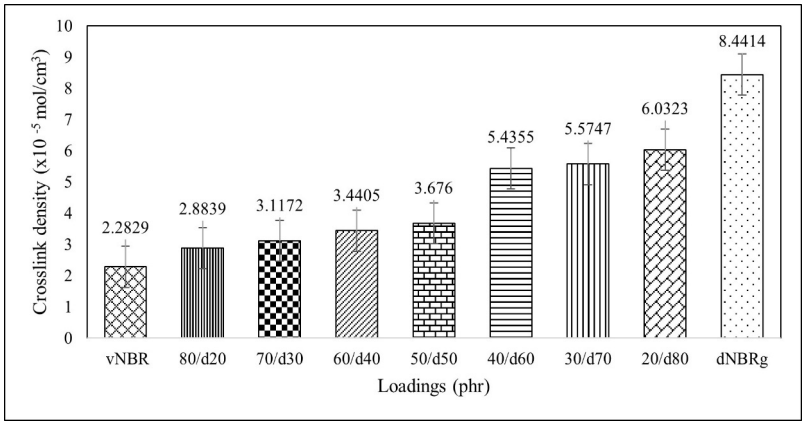

Figure 1: Crosslink density of vNBR, dNBRg and vNBR/dNBRg blends.

Fourier Transform Infrared Spectroscopy (FTIR)

FTIR analysis was performed to identify the functional groups and evaluate potential interactions between the vNBR and dNBRg phases. The corresponding wavenumber ranges and functional group assignments for vNBR, dNBRg, and their blends at 80/d20 and 50/d50 ratios are summarized in Table 4, while the FTIR spectra are illustrated in Figure 2. A prominent absorption band in the range of 2849.8–2919.2 cm^{-1} corresponds to the C–H stretching vibrations of CH_2 and CH_3 groups, indicating the presence of alkyl chains from butadiene and acrylonitrile units in the NBR matrix [13]. The peak observed at 2237.7–2239.0 cm^{-1} is attributed to C≡N stretching of the nitrile groups within the acrylonitrile segments. Peaks around 966.4–967.2 cm^{-1} are associated with C–H stretching of butadiene double bonds, while the presence of additional C=C signals further support the incorporation of butadiene structures [3]. A distinct band at 795.7–799.3 cm^{-1} is linked to

C–H bending vibrations in the butadiene units. Overall, the FTIR spectra of both the individual and blended samples display similar peak positions, with only slight shifts observed, indicating that blending did not significantly alter the molecular structure.

Table 4. Functional groups of vNBR, dNBRg and vNBR/dNBRg blends.

Range of wave number (cm^{-1})	Wavenumber of sample (cm^{-1})				Functional group	Classification of compound
	vNBR	80/d20	50/d50	dNBRg		
2400 - 3200	2918.9 - 2850.7	2918.9 - 2849.8	2918.9 - 2850.8	2919.2 - 2850.8	C-H stretching	Alkyl
2000 - 2400	2239.0	2237.5	2237.7	2238.3	C≡N stretching	Nitrile
1000 -1200	1081.3	1080.3	1081.4	1080.8	Si-O-Si	Silica, silane
800 – 1000	966.9	966.4	966.8	967.2	C-H, C=C	Alkenes
650 - 800	799.3	799.3	799.0	795.7	C-H	Alkenes

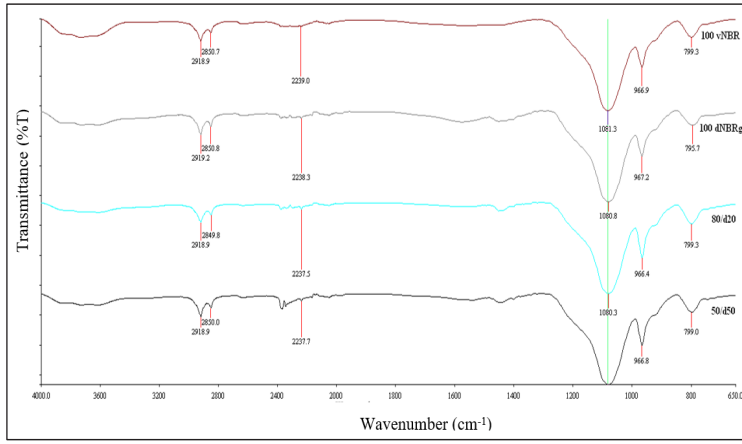

Figure 2: FTIR Spectra of vNBR, dNBRg and vNBR/dNBRg blends.

Tensile Properties

Tensile strength typically increased with higher crosslink density due to the formation of rigid and integrated networks that resisted deformation. However, as shown in Figure 3, the vNBR/dNBRg blends deviated from this trend, displaying an inverse relationship between tensile strength and crosslink density. This was attributed to the formation of non-uniform crosslink networks upon incorporating dNBRg into vNBR. In vNBR (Figure 4a), a uniform crosslink structure contributed to higher tensile strength approximately 19.2% greater than that of dNBRg that leads to even stress distribution and reducing stress concentrations that can initiate cracks. In contrast, blends containing dNBRg formed heterogeneous networks (Figure 4b) due to interactions between free radicals from short polymer chains and the crosslinkers or active sites in vNBR. These irregular networks introduced weak points and disrupted stress transfer within the rubber blends [14]. As the dNBRg content increased from 20 to 80 phr, tensile strength steadily declined by 3.7%, 4.7%, 11.6%, 12.7%, 13.2%, 14.1%, and 15.5%, respectively. Although crosslink density increased, the lack of network uniformity reduced the effectiveness of mechanical reinforcement, ultimately leading to lower tensile strength.

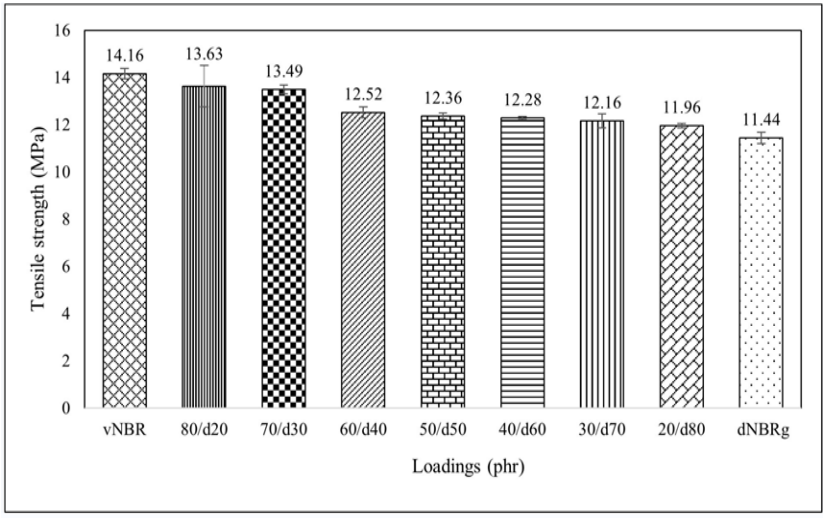

Figure 3: Tensile strength of vNBR/dNBRg blends.

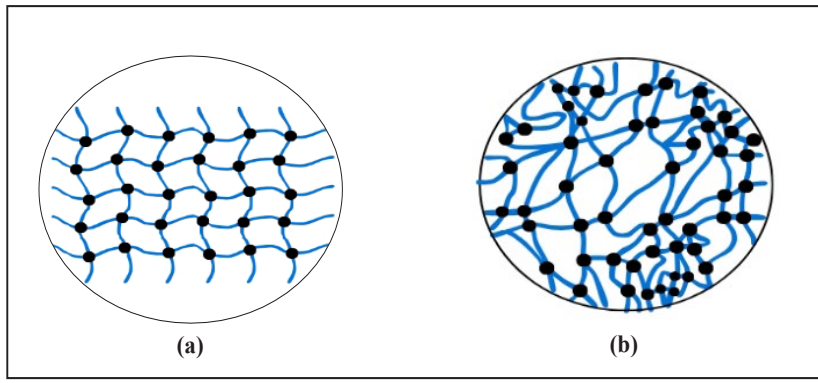

Figure 4: Proposed crosslinking network structures (a) uniform-vNBR (b) non-uniform-blends

Figure 5 shows the elongation at break (EB) values for vNBR, dNBRg, and their respective blends. vNBR exhibited the highest EB, attributed to its uniform crosslink network that allowed greater polymer chain mobility and stretchability under tension [15]. In contrast, dNBRg displayed lower EB due to its irregular network structure. When dNBRg was incorporated into vNBR at 20 to 50 phr, a slight increase in EB was observed. This improvement was likely due to a balanced interaction between the rigid crosslinked network and the flexibility provided by residual short-chain free radicals from dNBRg. However, as dNBRg content increased beyond 50 phr, EB progressively declined. At 80 phr, EB dropped sharply by approximately 54% compared to the 30/d70 blend. This significant reduction was attributed to the dominance of non-uniform crosslink regions, which introduced localized weaknesses and disrupted the network continuity. Although overall crosslink density increased, the resulting irregular structure hindered effective stress distribution, leading to premature failure under tensile strain. Consequently, blends containing 80 phr of dNBRg exhibited the lowest EB values, where the adverse effects of non-homogeneous crosslinking outweighed the mechanical benefits of increased crosslink density.

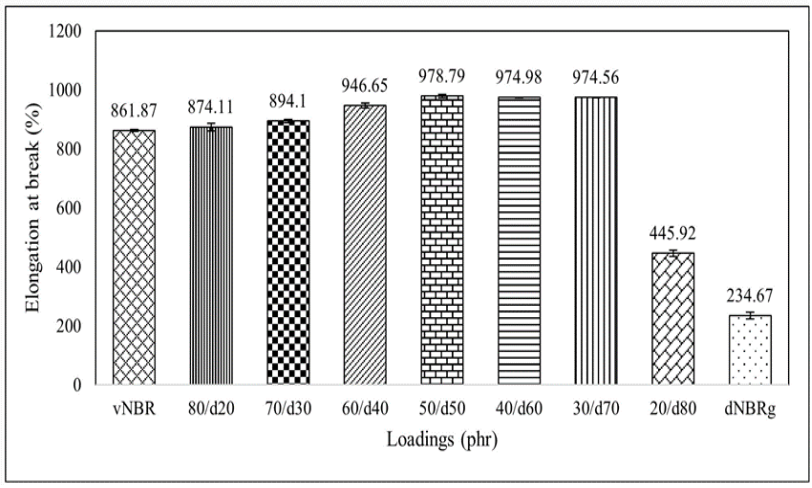

Figure 5: Elongation at break of vNBR/dNBRg blends.

Figure 6 presents the M100 values for both single rubbers and vNBR/dNBRg blends. The dNBRg sample exhibited a notably higher M100 value approximately 61% greater than that of vNBR indicating enhanced stiffness due to its higher crosslink density. The increased crosslinking restricts polymer chain mobility, contributing to greater rigidity within the vulcanized network. As the dNBRg content in the blends increased, a corresponding rise in M100 was observed, suggesting that both stiffness and rigidity were strongly influenced by crosslink density [15]. This trend aligns with established understanding, as modulus generally increases with crosslink density [16]. Swelling measurements supported this behavior by showing reduced chain mobility in more densely crosslinked systems. However, at a high dNBRg loading of 80 phr, a significant drop in M100 was recorded, approximately 44% lower than that of the 30/d70 blend. This unexpected decline is likely due to the dominance of dNBRg in the blend, which promoted the formation of a highly irregular and non-homogeneous crosslink network [17].

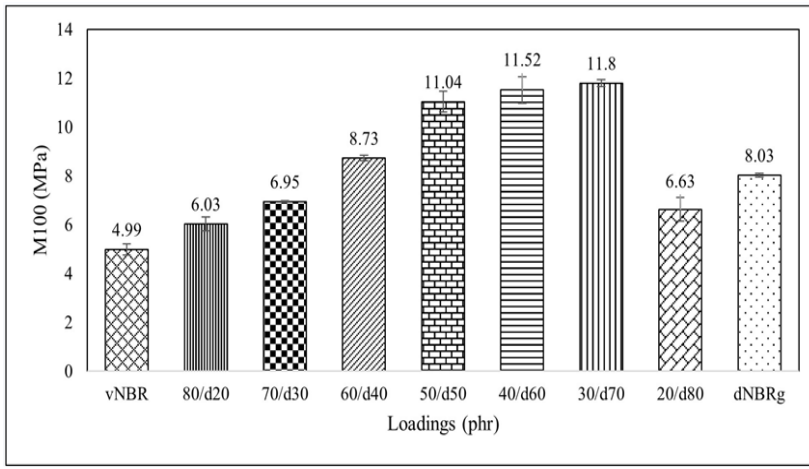

Figure 6: Modulus at 100% elongation (M100) of vNBR/dNBRg blends.

Abrasion Resistance

Figure 7 shows the volume loss (VL) data for vNBR/dNBRg blends. Among all samples, vNBR exhibits the lowest VL, indicating superior wear resistance and mechanical durability. This performance is primarily attributed to its uniform crosslink network, which allows for even stress distribution, preserving the material's integrity under mechanical and abrasive forces [15]. In contrast, dNBRg records the highest VL, reflecting its poor wear resistance. When blended with vNBR, VL values increase progressively with higher dNBRg content (from 20 to 80 phr). While the blends exhibit increased crosslink density, this is accompanied by the formation of non-uniform crosslink structures due to the incorporation of dNBRg. These irregularities introduce localized weak zones within the rubber blends, where bonding is less effective and stress distribution is uneven. As a result, the elasticity and ability of blends to relax under mechanical load are compromised, reducing its dimensional stability. The presence of these structural weak points makes the blends more vulnerable to micro-failures, detachment, and surface deformation during abrasive contact.

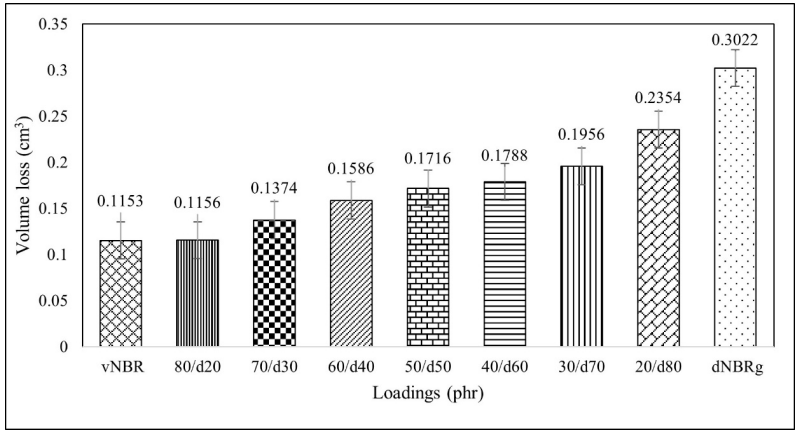

Figure 7: Volume loss of vNBR/dNBRg blends.

CONCLUSION

In conclusion, dNBRg shows promising potential as a partial replacement for vNBR, with the optimal blend ratio depending on the intended application in general rubber goods. The incorporation of dNBRg into vNBR results in an increase in M_L, primarily due to the higher viscosity of dNBRg caused by residual additives from reclaimed gloves. M_H initially decreases at lower dNBRg loadings (20–40 phr) but increases significantly beyond 40 phr. Similarly, the torque difference (ΔT) remains relatively stable between 20 and 60 phr, followed by a sharp rise at higher contents.Scorch time (ts_2) remains largely unaffected, while cure time (t_{90}) increases noticeably by up to 50% with increasing dNBRg content up to 60 phr. FTIR analysis confirms the preservation of key chemical structures, with no new peak formations or loss of characteristic functional groups, indicating no major chemical alterations. Mechanical properties show that tensile strength declines as dNBRg content increases. However, improvements are observed in elongation at break, modulus at 100% strain (M100), and VL, which are attributed to increased crosslink density and changes in the rubber network structure. Blends containing 20–40 phr dNBRg provide an optimal balance between processability and property retention. Overall, dNBRg is suitable for use in low-to-moderate performance rubber products where tensile strength and high abrasion resistance are not primary requirements.

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