

Friction and wear behavior of styrene butadiene rubber-based composites reinforced with microwave-devulcanized ground tire rubber

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ABSTRACT: In this work, the tribological properties of a new material obtained by revulcanization with styrene butadiene rubber (SBR) and devulcanized ground tire rubber (GTR) were investigated. GTR was devulcanized using the microwave method at a constant power while varying the microwave exposure time. Devulcanized rubber (DV-R) and untreated GTR were revulcanized by mixing with SBR at different rates (10, 30, 50 phr). To determine friction and wear characteristics of the samples, pin (ball) on disc and abrasion tests were conducted. Scanning electron microscopy (SEM) was employed to observe the worn surfaces of the composites to correlate the experimental test results to the wear mechanisms. All of these tests and experiments were performed on original vulcanized rubber samples for comparison. The composites exhibited different friction and wear behavior due to morphology, dispersion behavior and devulcanization functionalization of ground tire rubber. In general, DV-R/SBR composites exhibited improvement in both mechanical and tribological properties. However, the enhanced compatibility of DV-R resulting from the specific chemical coupling of DV-R with SBR was crucial for the mechanical, friction and wear properties. © 2015 Wiley Periodicals, Inc. *J. Appl. Polym. Sci.* **2015**, *132*, 42419.

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INTRODUCTION

Elastomers are a special class of polymeric materials due to their unique properties. In comparison with other polymers, elastomers are used in a wide range of specific applications because of their high elastic memory, high ultimate elongation and low elastic modulus.¹ Understanding the friction and wear properties of rubber materials is not only required for tire performance but also for other rubber products. The tribological properties of rubber differ in many ways from the tribological properties of most other solids because rubber has a very low elastic modulus and high internal friction.² The wear properties of rubber are governed by its fundamental visco-elastic bulk properties.^{3,4}

Shallamach published the first works about rubber friction. He suggested the deformation model, which describes the phenomenon as the propagation of deformations in a material called "Schallamach's waves".⁵ Although there are many studies related to this topic, a persuasive result of the origin of these waves has not been shown to date. Rubber friction has been well described by Moore, who suggested dividing the friction force between rubber and a rough (hard) surface into two components:

adhesion and deformation (hysteresis).^{5,6} It is widely accepted that adhesion, which is the result of samples exhibiting stick-slip behavior, plays a major role in the friction of materials exhibiting high modulus of elasticity. Additionally, hysteresis friction is the resulting behavior of samples when their internal friction is the more dominant component in the friction of materials with low moduli of elasticity.^{2,5-7} The wear behavior of rubber is a complex phenomenon and involves more than one mechanism. A phenomenon generally referred to as local mechanical rupture (tearing) and decomposition of the molecular network to a low molecular weight (smearing) was suggested by Gent.⁸ Grosch expresses that the wear of rubber is largely caused by fatigue.⁹ Fatigue wear, as a result of the detachment of particles as a result of cyclic stress variations, takes place when rubber slides against hard surface at low frictional force.¹⁰ Schallamach, Grosch, Kragelskii, Nepomnyashchil, Bhowmick, Viswanath, Rymuza, Stachowiak, and others have developed various forms of equations and relationships for the wear of rubbers.^{4,5,10-12} All of these models have stated variables, such as load, speed, sliding distance, and sliding duration, and they include properties, such as hardness of the counter face and asperity height.¹³

Table I. Formulations of the DV-R/SBR Composites²⁴

Ingredients (phr ^a)	1	2	3	4
SBR 1502	100	90.9	72.7	54.5
DV-R	0	10	30	50
Carbon black (N330)	67.75	66.85	65.05	63.25
Process oil	35.5	35.5	35.5	35.5
ZnO	3	3	3	3
Stearic acid	1	1	1	1
CBS	1.8	1.8	1.8	1.8
Sulfur	1.75	1.75	1.75	1.75

^aParts per hundred rubber.

Many studies have been performed and published on the mechanisms of devulcanization and mechanical properties of devulcanized rubber-virgin rubber composites.^{14–20} Moreover, some researchers, such as Karger-Kocsis *et al.*, Khan *et al.*, and Tangudoma *et al.*, have carried out detailed tribological investigations on rubbers containing different and novel reinforcing fillers; however, little has been studied on the friction and wear properties of devulcanized rubber-virgin rubber composites, which is the focus of this study.^{21–23} Devulcanized rubber can be revulcanized as a virgin elastomer or can be used in composites with other rubbers. Revulcanized rubber and rubber composites are widely used in different fields. Therefore, consideration of the tribological properties of these new materials is required for many rubber products.

In our previous paper,²⁴ devulcanized rubber was obtained from ground tires using the microwave method and combined with SBR matrix to produce new composite materials. Then, the curing characteristics, mechanical properties and morphology of these materials were investigated. The aim of this study is to investigate the friction and wear behavior of new materials obtained from revulcanization with SBR and devulcanized ground tire rubber. Additionally, the influences of devulcanized rubber content on the friction and wear characteristics and the relationship between tribological and mechanical properties during the run-in period were studied.

EXPERIMENTAL

Materials

The ground tire rubber was purchased from Ünsal Rubber (Turkey). Particle size analysis showed that the majority of GTR particles were in the 35–120 mesh (0.5–0.125 mm) range. The chemical composition of the ground tire rubber was determined from *thermogravimetric analysis* (TGA): 13% oil and highly volatile materials, 52% polymers and moderately volatile materials, and 35% carbon black and other additives.

The GTR was treated in a domestic microwave oven (Samsung MW71E). The power of the magnetron was set to 800 W. The processing variable was the exposure time: 4 and 5 min. After microwave treatment, the surface areas of the two kinds of DV-R (4 and 5 min) and untreated GTR were measured using a BET (Brunauer, Emmett and Teller) tester (*Micromeritics Gemini VII 2390*). Also, the surface morphologies of DV-R and

GTR particles were evaluated with SEM. The compounding formulation for the virgin SBR, other additives and various proportions of the DV-R (10, 30, 50 phr) were mixed for 15 min on an open two-roll mixing mill at room temperature. The formulations of the composites are presented in Table I. Formulation 1 does not contain DV-R and is a control sample. Formulations 2, 3, and 4 were prepared at 10, 30, and 50 phr DV-R. For comparison, untreated GTR was mixed with SBR in the same series. The amounts of the other additives and curatives in the formulations were based on 100 g of rubber without taking the DV-R into account because it was assumed that the additives in the DV-R originated from the parent compound and were inactive.²⁰ All other information about the devulcanized material was given in a previous study.²⁴

Friction and Wear Testing

Pin-on Disc Wear Test. Friction and wear characteristics were determined using a pin (ball) on disc wear tester (ASTM: G99-05). The rubber sheets were worn by one steel ball (100Cr6, diameter of 10 mm, hardness of 59 ± 1 HRC, and arithmetical roughness Ra of 1 mm). The test specimens were manufactured as square plates with dimensions of 40 × 40 mm² and thicknesses of *h* = 6 mm. Friction and wear tests were carried out under a constant normal-applied load of 15 N, a sliding speed of 0.3 m/s and a sliding distance of 500 m. The diameter of the rotation was 30 mm in each case. Tests were carried out at room temperature (23 ± 2°C) in ambient atmosphere without an abrasive. The friction coefficient was determined directly by measuring the tangential force with a strain gauge load cell and recorded continuously. The experimental data strongly fluctuated due to the stick-slip effect; therefore, the average values of the friction coefficients (*μ*) are reported for better comparisons between different samples.

DIN Abrasion Test. The abrasion resistance of the revulcanized rubber composites was measured using an abrasion tester according to ASTM: D5963-04. The cylindrically shaped specimen (16 mm in diameter and 6 mm in thickness) was punched from the compression-molded sheets. The specimen was placed at the starting position of the testing machine. The specimen was then abraded across a test abrasive paper of grade 60 at a constant force (10 N) and constant speed (0.32 m/s). After an abraded distance of 40 m, reaching approximately 84 revolutions, the specimen was automatically lifted from the test abrasive paper. The abrasion loss or DIN volume loss of the rubber vulcanizates was calculated from the following equation. The degree of abrasion was determined before and after each test through at least 3 measurements with the aid of a standard rubber sample.

$$\text{DIN volume loss (mm}^3\text{)} = \frac{\Delta m \times S_0}{\rho \times S}$$

where Δm is the mass loss (mg), ρ is the density (mg/mm³), S_0 is the value of nominal abrasive power (200 mg), and S is the average abrasive power (mg).

Morphology

The worn surfaces of the DV-R/SBR and GTR/SBR composites, which were obtained from the pin-on disc tests and the DIN

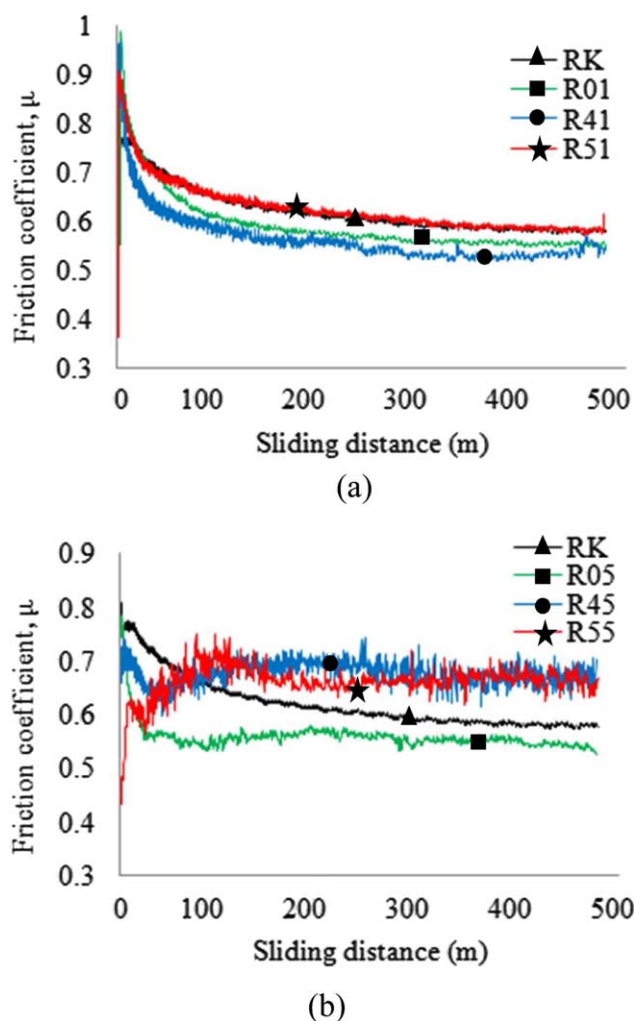


Figure 1. Comparison of the friction behaviors of different DV-R/SBR and GTR/SBR composites as a function of sliding distance (a) 10 phr and (b) 50 phr devulcanized rubber content. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

abrasion tests, were evaluated with a ZEISS EVO LS 10 scanning electron microscope. The sample surfaces were sputter-coated with gold powder using a Sputter Coater Auto 108.

RESULTS AND DISCUSSION

Friction and Wear Properties of Composites

Pin-on Disc Wear Test Results. Figure 1 shows the friction behavior of DV-R/SBR and GTR/SBR composites sliding against a steel ball. With the addition of DV-R and GTR (at 10 phr) to the composite, the friction curves did not show significant differences from the control sample. During sliding contact of these samples, the DV-R and GTR particles interacted directly with the counter surface, reducing the friction coefficient of all of the samples. This interaction could have also led to the formation of a DV-R and GTR film transferred to the counter surface, which led to significant reduction in the friction coefficient.

The friction behavior changed for the DV-R/SBR and GTR/SBR composites with increasing the content of DV-R and GTR. Upon increasing the content of DV-R and GTR in the composites, the friction behaviors of the composite samples were mainly affected by adhesion between the GTR or DV-R particles and the SBR matrix as in other mechanical properties.²⁵ The mechanical properties of the DV-R/SBR and GTR/SBR composites are given in Table II. The DV-R content and the microwave exposure time affected the mechanical properties of the composites. The results showed that the mechanical properties of DV-R/SBR were far superior to the properties of SBR filled with GTR at the same loading. The deterioration of the mechanical properties of the GTR/SBR composites was related to the weak adhesion between the GTR particles and the SBR matrix.^{15,20,24,26} Because the GTR particles were not well-dispersed in the SBR matrix, they represented weak sites for stress transmission, resulting in lower mechanical properties for the composite. The values of the properties for the composites that contained DV-R were much better because DV-R participated in the crosslinking reaction; the strong interfacial bond

Table II. Mechanical Properties of DV-R/SBR and GTR/SBR Composites²⁴

	RK	R41	R43	R45	R51	R53	R55	R01	R03	R05
Sample code ^a	Control		4 min			5 min		Untreated GTR		
DV-R (phr)	0	10	30	50	10	30	50	10	30	50
Elongation at break (%)	524.63	594.88	341.06	366.31	613.75	449.00	445.06	617.50	279.44	217.25
Tensile strength (N/mm ²)	7.49	7.17	5.35	4.74	7.82	6.50	4.91	9.20	4.29	6.39
100% Modulus (N/mm ²)	1.96	1.48	2.56	2.13	1.80	2.18	1.98	1.78	2.14	3.67
Hardness (Shore A)	57	58	64	66	56	67	67	57	64	64
Crosslink density (mol/m ³)	54.91	69.19	79.18	94.79	74.04	93.35	106.03	77.63	87.99	119.87

^aThe samples were identified as R followed by two numbers corresponding to the exposure time in minutes and the proportion of the DV-R or GTR in phr.

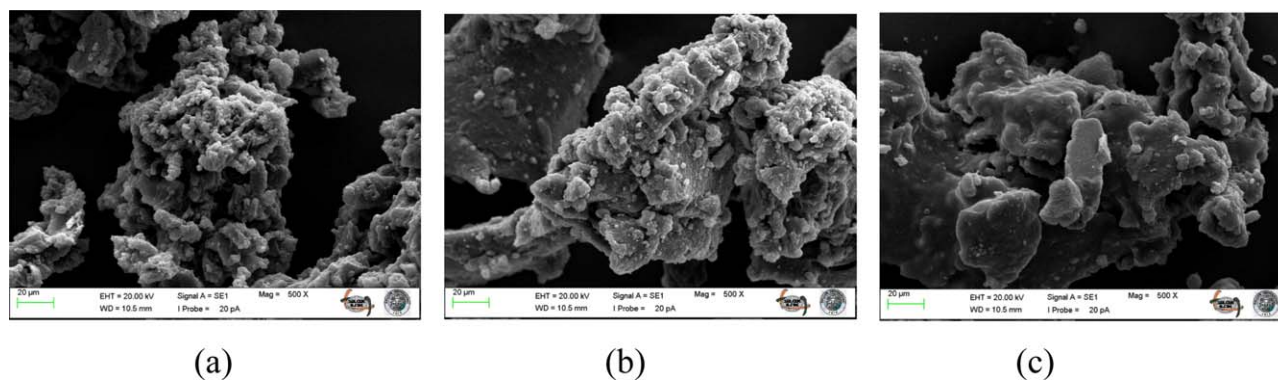


Figure 2. Morphology of particles (a) DV-R (4 min), (b) DV-R (5 min), and (c) untreated GTR. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

between the SBR matrix and DV-R led to good tensile properties.^{17,27}

The surface morphologies of DV-R and GTR particles are an important factor for the adhesion between the particles and the SBR matrix.^{28,29} Figure 2 shows the morphology of DV-R and GTR particles. The surface of devulcanized rubber, obtained by microwave treatment, is rougher than the surface of GTR particle. The morphology of the DV-R surface is one of the responsibilities of the strong adhesion between the particles and the SBR matrix, because GTR surface was activated by microwave devulcanization.^{28,29} In addition, the surface area of the treated particles that were exposed for 5 and 4 min was obtained from BET analysis 0.295 and 0.204 m²/g, respectively, while the surface area of the GTR particle is 0.0169 m²/g. This increase in surface area of the treated particles is significantly improved the adhesion between the particles and the matrix.^{25,30} Therefore, surface morphology is an important factor not only affecting tensile properties, but also friction behavior of composites.

Figure 1(b) shows that, with the addition of 50 phr of DV-R to the composite, the friction coefficient values are higher than the composites with GTR. This difference was the result of a good interface between the SBR and devulcanized rubber to the

counter surface caused by direct contact with the matrix materials. On the other hand, since GTR is already vulcanized, it is present as a filler in rubber blends and this material generally exhibit poor mechanical and viscoelastic properties owing to poor GTR-matrix adhesion. The addition of 50 phr of GTR to the composite showed significantly lower friction coefficient values because of the interaction of the GTR particulates in the wear track of the samples with the counter surface. Figure 3(a) shows that the DV-R/SBR composite present waves called the Schallamach's waves, and also the worn track suggests the occurrence of stick-slip mechanism during sliding. In contrast, the GTR/SBR composite did not show the characteristic stick-slip behavior due to the interaction facilitates easy sliding of the counter surface against a wear track covered with GTR particles [Figure 3(b)].

DIN Abrasion Test Results. The average values of the volume losses of rubber composites against the DIN abrader are shown in Figure 4. The lowest DIN volume loss from all of the composites, was obtained by GTR loading of 10 phr. The volume losses of composites with increasing GTR loading were lower compared to the other two types of composites under the same conditions. The wear mechanism primarily

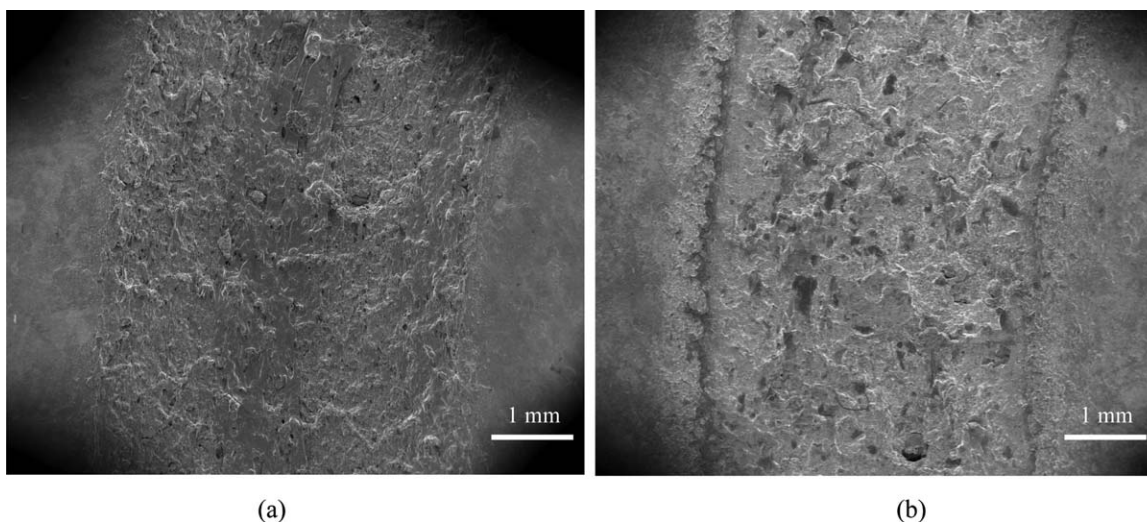


Figure 3. SEM pictures of wear track which were obtained from pin-on disc wear test: (a) 50 phr of DV-R (5 min) $\times 40$ and (b) 50 phr of GTR $\times 40$.

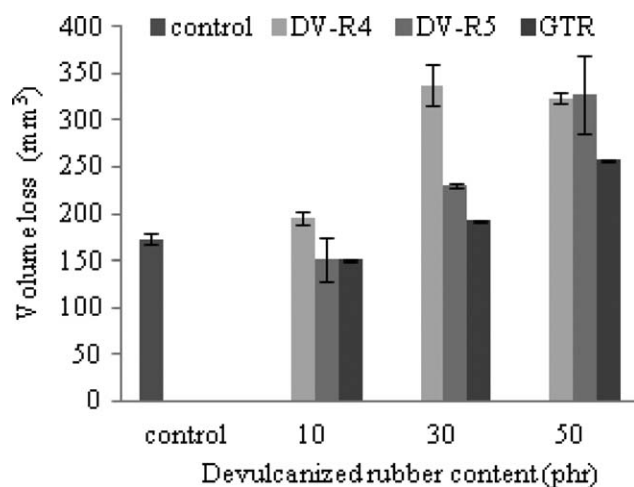


Figure 4. Effect of devulcanized rubber content on volume loss.

occurred because of the expelling of GTR powder from the surface of the rubber, which is in contact with the counter surface; there were not adequate interfacial bonds between the original rubber and the GTR powder. This mechanism also facilitated the counter surface to slip on the sample, obtaining a higher wear resistance.

With the addition of DV-R to the SBR, wear occurred in the form of rupture from the original rubber. This result could be explained in terms of stronger interfacial bonding between the SBR matrix and DV-R compared to those of the GTR/SBR composites, as shown by tensile test results and SEM images of their wear surfaces.

In addition, the wear resistance was higher in the samples (10 and 30 phr) that were exposed for 5 min than those for 4 min. The reason for this difference was because the higher crosslink density of these samples from revulcanization increased their wear resistance [Figure 5(a)]. Also wear resistance decreased as DV-R and GTR content increased for all of the samples.

The correlation of modulus and wear resistance of composites is illustrated clearly in Figure 5(b). With the addition of 50 phr of GTR to the composite, the 100% modulus and the wear resistance are higher than the composites with DV-R, because the GTR acted like reinforcement filler. This result is in agreement with the well-known fact that the reinforcement of elastomers is characterized by the increase in modulus and the improvement of abrasion resistance.³¹

Morphology

Depending on the texture of the counter surface which is abrasive or smooth surface, there are many different wear mechanisms, such as abrasive wear, fatigue wear and roll formation.³² Abrasive wear occurs on harsh surfaces, whereas the last two mechanisms types occur blunt and smooth surfaces, respectively. In this study abrasive papers were used and it is expected that the volume loss, is mainly due to the material removal from the surface as a result of the abrasive action of the abrasive paper. The removal of material is estimated due to the tearing and tensile rupture.^{33,34}

In order to understand the mechanisms of abrasion, the abraded surfaces of GTR/SBR and DV-R/SBR composites were

examined by SEM and pictures of the abraded surfaces of the composites are shown in Figures (6 and 11). As shown in Figure 6, wear scars have occurred in the SEM pictures of the control sample. Higher magnification shows the formation of wear debris [Figure 6(b)].

Ridges existed with fronts transversely oriented to the abrasion direction in the SEM images of the wear surfaces of GTR/SBR and DV-R/SBR composites. The formation of such an abrasion pattern was reported to be a characteristic of a low modulus of elastomer sliding with high friction against a counter surface.³² The magnified views reveal that upon rubber and abrasive surface contact, ridges occurred by deformation first and then detachment of solid material from the surface by a wear mechanism. The formation of the ridge patterns during abrasion of the DV-R/SBR composites is seen as more significant than that of the GTR/SBR composites.

The worn surfaces of GTR/SBR composites showed fine and narrow ridges and better abrasion resistance because of the expelling of GTR powder from the surface of the rubber, which is in contact with the counter surface (Figures 7, 8). Moreover, Figure 8 show that with the increasing GTR content, the

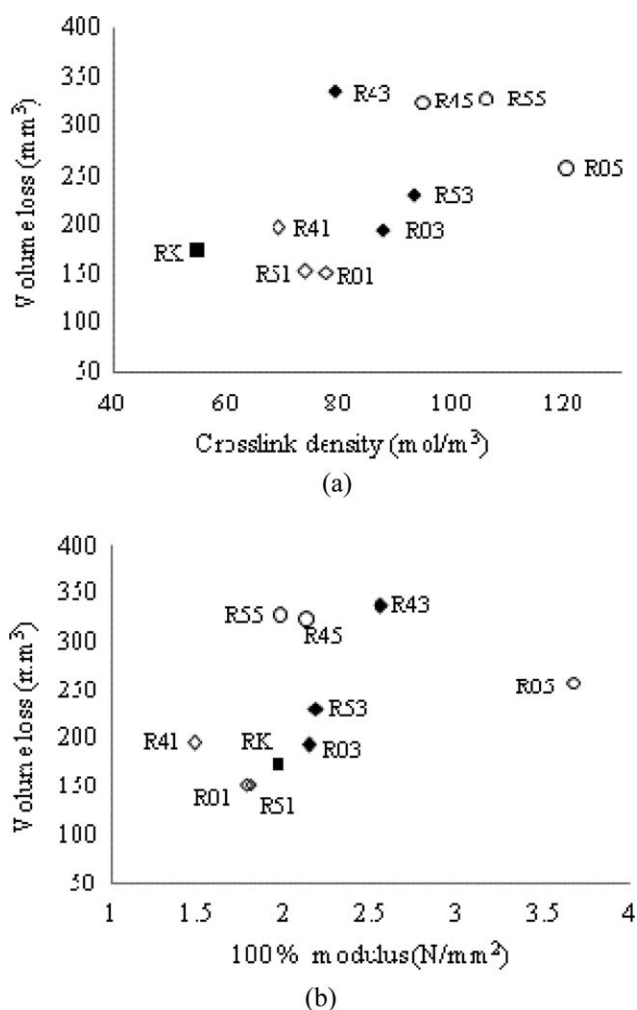


Figure 5. Volume loss versus (a) crosslink density and (b) 100% modulus for all samples.

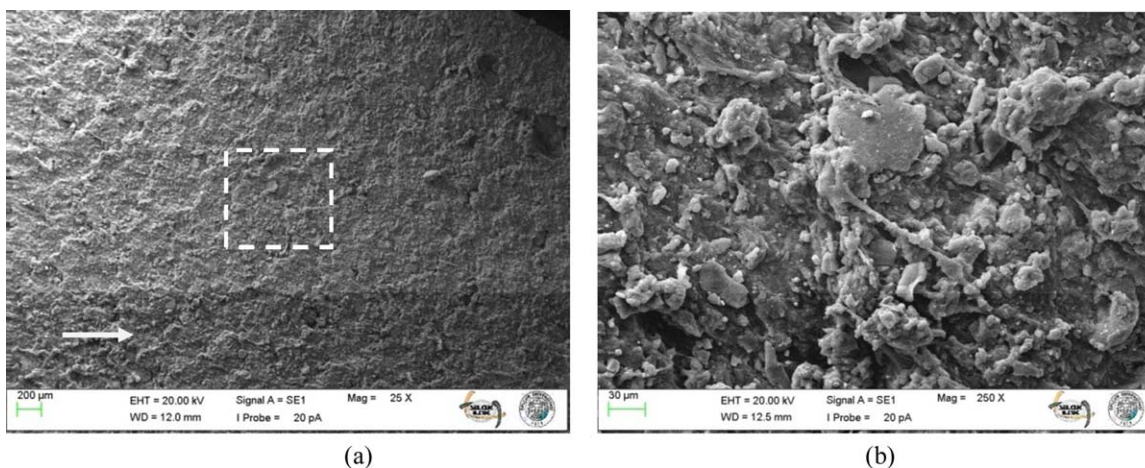


Figure 6. SEM pictures taken from worn surfaces of the control sample (a) $\times 25$ and (b) $\times 250$. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

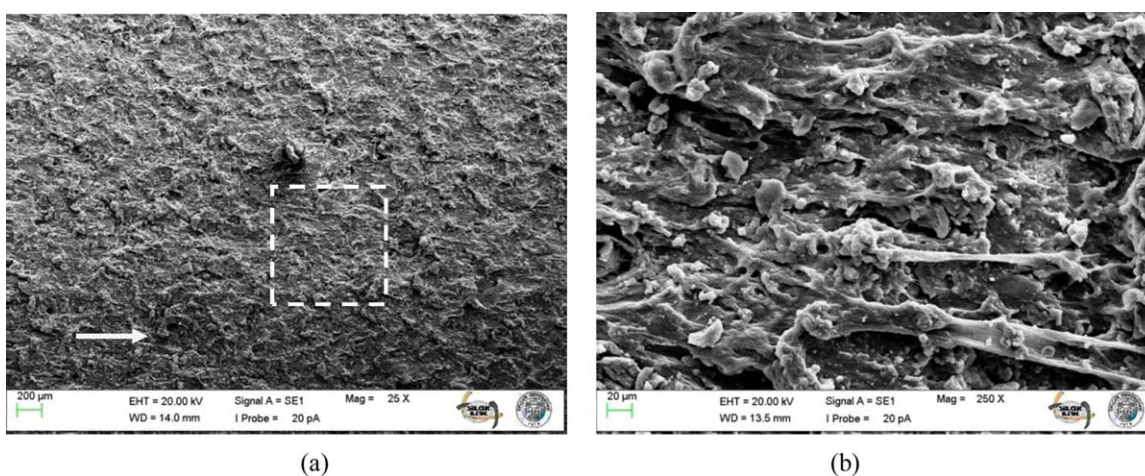


Figure 7. SEM pictures taken from worn surfaces with the addition of 10 phr of GTR to the composites (a) $\times 25$ and (b) $\times 250$. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

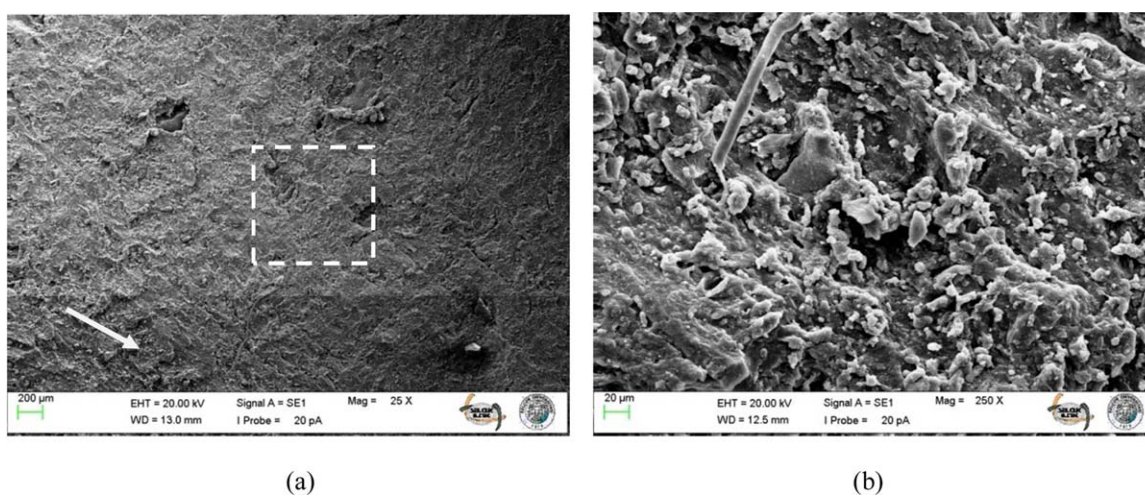


Figure 8. SEM pictures taken from worn surfaces with the addition of 50 phr of GTR to the composites (a) $\times 25$ and (b) $\times 250$. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

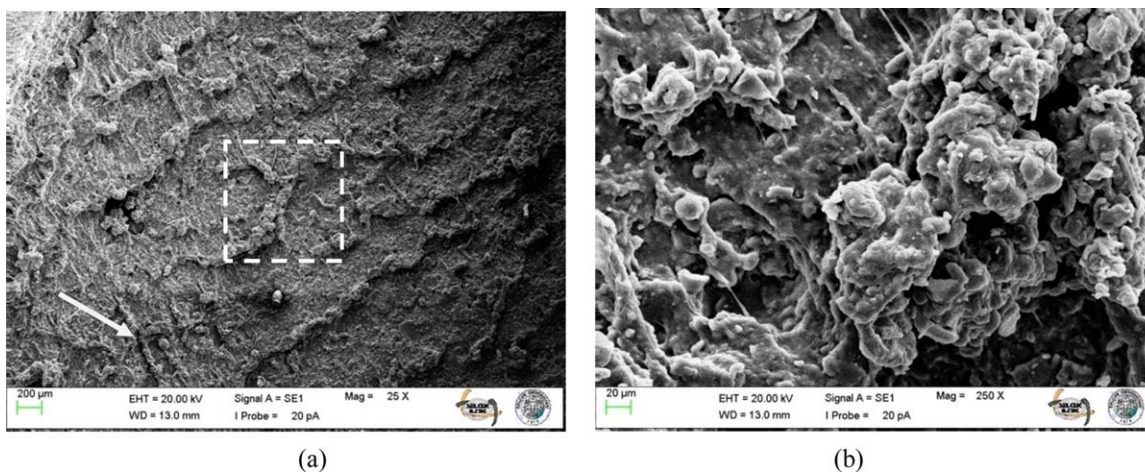


Figure 9. SEM pictures taken from worn surfaces with the addition of 10 phr of DV-R5 min to the composites (a) $\times 25$ and (b) $\times 250$. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

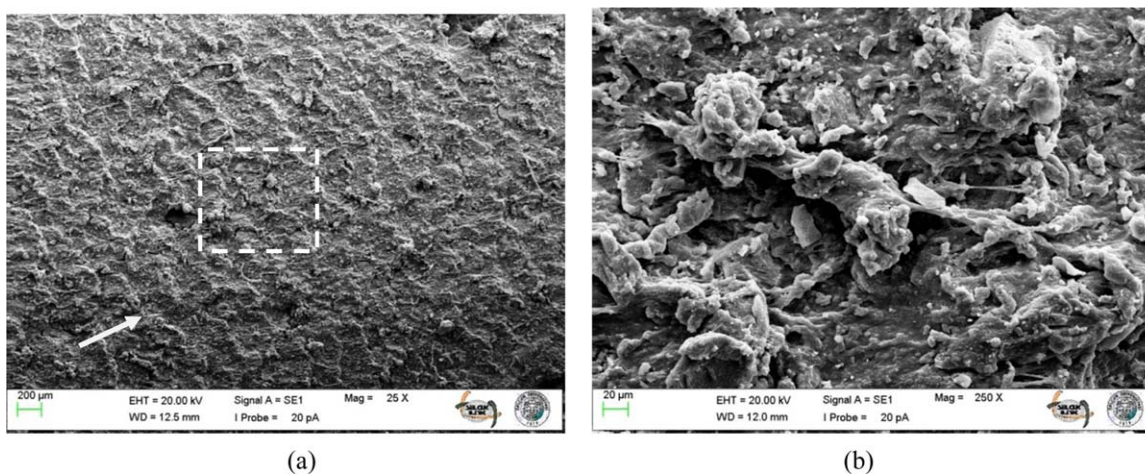


Figure 10. SEM pictures taken from worn surfaces with the addition of 50 phr of DV-R4 min to the composites a) $\times 25$ and (b) $\times 250$. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

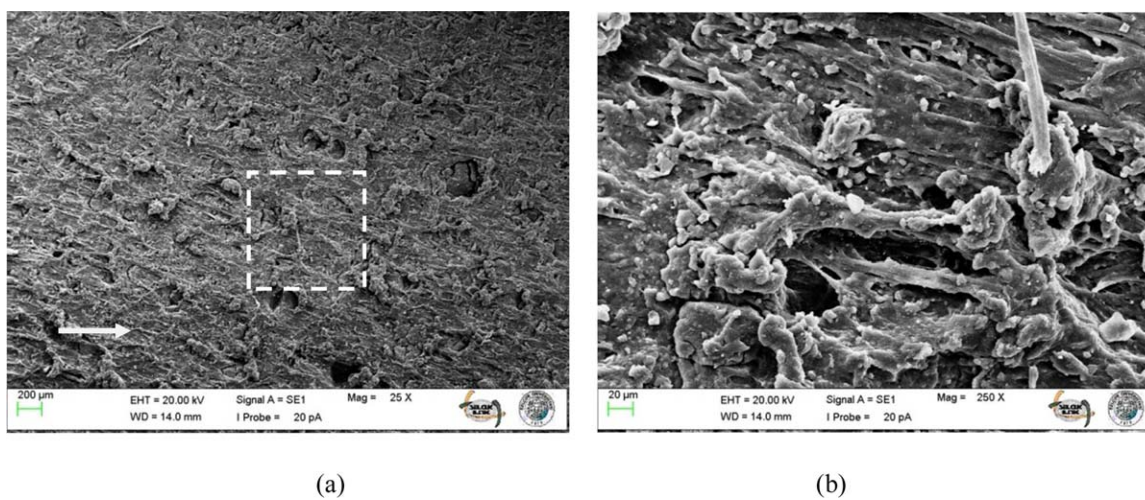


Figure 11. SEM pictures taken from worn surfaces with the addition of 50 phr of DV-R5 min to the composites a) $\times 25$ and (b) $\times 250$. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

formation of the ridge patterns of the GTR/SBR composites is not seen clearly and abrasive wear occurs by massive material detachment. This result was also supported by the low value of elongation at break and high value of modulus obtained with tensile testing.

The SEM images of worn surfaces of DVR/SBR composites show ridges with stretching wear particles (Figures 9–11). In addition, a type of roll formation wear was observed on the DV-R5 min/SBR composites with a high content of devulcanized rubber (Figure 11). Wear particles were produced by rolling and fracture of ridges.

CONCLUSIONS

The friction and wear behavior of new composites obtained from devulcanization with SBR and DV-R were tested. The following conclusions can be drawn:

- Improved composite materials, DV-R blended with virgin SBR in different proportions, exhibited a significant increase in mechanical properties compared to blending with untreated GTR. However, the friction and wear behaviors of the composites were different. While increasing the content of DV-R and GTR in the composites, the friction and wear behaviors varied, which depended on the modulus, crosslink density, interaction between DV-R and GTR with the SBR matrix, and friction and wear mechanisms of the rubber materials.
- Frictional behavior of the samples was varied, depending on their moduli and also interaction between the composites and the counter surface, by increasing the content of DV-R and GTR in the composites.
- Wear occurred in the form of rupture from the original rubber and was faster in the DV-R/SBR composites. This result was due to the stronger interfacial bonding between SBR and devulcanized rubber, and the counter surface made direct contact with the matrix materials. In contrast, when GTR was added to the composite, embedded GTR agglomerates appeared in the wear tracks of samples. This addition facilitated the counter surface to slip on the sample. These composites had a higher crosslink density due to the untreated GTR, obtaining a higher wear resistance.
- The SEM observations on the worn surfaces of the composites revealed that the ridges were existed with fronts transversely oriented to the abrasion direction. The ridges of the DV-R/SBR composites are seen as more significant than that of the GTR/SBR composites, and this is also evaluated an evidence that, wear of the GTR/SBR composites occurred by fracture, but the wear of DVR/SBR composites occurred not only by fracture but also by roll formation. In addition the SEM observation also revealed that the mechanical properties of the composites were important factors in the formation of wear scars.
- This research has shown that, the DV-R/SBR composites showed improved frictional properties on nonabrasive surfaces compared to the GTR/SBR composites and these composites also showed improved mechanical properties, especially SBR filled with DV-R (5 min), however, higher wear

resistance of GTR/SBR composites against abrasive surfaces make preferable for some rubber applications compared to the DV-R/SBR composites.

- Further investigations related to the influence of different devulcanization methods, devulcanized rubber content, morphology and state of crosslinking on devulcanized rubber filled with different virgin rubber are required. These investigations were necessary because they contribute to a deeper understanding of the friction and wear behaviors of devulcanized rubbers.

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