



MOONEY VISCOSITY, CURE BEHAVIOR, MECHANICAL PROPERTIES AND HEAT BUILD-UP OF SOLID TIRE TREAD COMPOUND CONTAINING GROUND TIRE RUBBER OF VARIED PARTICLES CHARACTERISTICS MIXED WITH TWO DIFFERENT ROTOR SPEED



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**Abstract:** The study investigates the effect of incorporating three mesh sizes of ground tire rubber (GTR) at 10 parts per hundred of rubber (phr) and 20 phr loading in tire tread compound on its properties. The compounds were mixed in internal mixer using 40 rpm and 60 rpm rotor speed respectively. The viscosity, cure behavior mechanical properties and heat build-up of the rubber vulcanizates were studied. The base elastomer used for the production of the vulcanizate samples was NR/BR blend. Mixing was conducted using a two-step mixing technique. Results show that at 10 phr GTR loading, increased rotor speed resulted to reduced viscosity of the rubber compounds filled with 40, 60 and 80 mesh sizes of the GTR; while at 20 phr GTR loading, increased rotor speed resulted to increased viscosity of the compounds filled with 40 and 60 mesh size GTR, while the viscosity of the compound filled with 80 mesh size GTR reduced. The mixing torque was influenced by the amount of GTR incorporated in the rubber matrix as well as the mixing speed, but the varied particles sizes did not have much effect on the torque. The rotor speed and the temperature at which the curatives were added influenced the scorch and cure time among the samples filled with GTR thereby affecting the cure rate. Combination of results show that the properties of the samples mixed with 40 rpm rotor speed were better than those of the compounds mixed with 60 rpm rotor speed.

**Keywords:** Ground tire rubber, tire tread, cure behavior, Mooney viscosity, and properties.

## Introduction

Demand for rubber products such as tires is very high. Both natural rubber (NR) and synthetic rubbers such as butadiene rubber (BR) and styrene butadiene rubber (SBR) are used in the production of tires. Blending of both NR and BR in tire production is very desirable as each contributes to improving the performance properties of tires. The desirable properties of NR are low compression set, resistance to inorganic chemicals, and good mechanical properties such high strength and rebound resilience. It however exhibits poor resistance to oil and organic liquids, high temperature and ozone resistance<sup>1</sup>. On the other hand, BR offers better abrasion resistance resulting to lower tread wear and low rolling resistance in vehicles which translates to fuel economy (Arti *et al.*, 2020). Optimization of NR/BR blending in the production of tires has been reported in previous works (Vishnu *et al.*, 2023). Tires are very important in the automobile industry. It was estimated that globally, about 1.5 billion tires were sold annually (Mohan *et al.*, 2021). The rubber in tires is in vulcanized state and therefore do not degrade easily when end-of-life tires are discarded. Annually, end-of-life tires are discarded in bulk and the trend keeps rising. End-of-life tires have been reported to be the major source of rubber waste to the environment (Leong *et al.*, 2023). It was estimated that the amount of end-of-life tires discarded to the environment will reach 1.2 billion in 2030 (Rashad, 2016). The problems caused by waste rubbers have become global issues. Waste rubbers pollute the environment; they create fire hazards, sites for pests and rodents which are harmful to man (Isayev, 2013; Yehia *et al.*, 2012). Waste rubbers are very difficult to recycle due

to their cross-linked nature. However, researchers are making conscious efforts on the re-use and recycling of waste rubbers through retreading of tires, reclaiming and devulcanization technologies, pyrolysis, and the use of recycled rubber in composites. Technology has improved and the grinding of end-of-tires to produce ground tire rubber (GTR) of different mesh sizes is now very effective. It has been reported that GTR can be used as filler in various types of matrices such as thermoplastics, thermosets and virgin rubbers (Rocha *et al.*, 2014; Kocovski *et al.*, 2012). The re-use of waste rubbers in composites is seen as a way to remove some wastes from the environment and to reduce the costs of the composite materials. Practically, the addition of GTR in rubber compounds has been reported to lower the elongation and strength of vulcanizates while increasing the hardness and stiffness (Klajn *et al.*, 2021; Majewska-Laks *et al.*, 2021; Hrdlička *et al.*, 2022). Researchers are therefore investigating various methods to improve the mechanical properties of rubber vulcanizates containing GTR. Some of the methods that have been investigated and reported are; surface modification of the GTR powders (Klajn *et al.*, 2021; He *et al.*, 2016; Elenien *et al.*, 2018; Narongthong *et al.*, 2018; de Carvalho *et al.*, 2018) and investigation on the effects of using various methods to grind the waste tire (Hrdlička *et al.*, 2022). Variation in the mixing parameters such as mixing time, temperature, fill factor and rotor speed influence the viscosity, dispersion and distribution of materials in the rubber compound, thereby changing the dispersion index, Payne effect and mechanical properties of rubber vulcanizates (Narongthong *et al.*, 2018). de Carvalho *et al.* (2018) reported that reducing the rotor speed during

dynamic vulcanization and increasing it on torque mixing improved dispersion of peroxide/bismaleimide cured polypropylene/nitrile rubber. Further, Thongpin and Tanprasert (2019) studied the effect two rotor speeds; 60 rpm and 80 rpm on the morphology and tensile properties of dynamically vulcanized natural rubber/polycaprolactone blend elastomer. Their report suggests that increased rotor speed changed the morphology of the blend and mixing at higher rotor speed resulted to reduced tensile strength, modulus and tension set (Thongpin *et al.*, 2019). In this work, the Mooney viscosity, cure behavior and mechanical properties of solid tire tread compound containing ground tire rubber of different mesh sizes mixed with two different rotor speed is presented.

## Materials and Method

### Materials

40 mesh size, 60 mesh size and 80 mesh size mechanically ambient ground GTR processed industrially were provided by Rubber Technology Research Centre, Mahidol University, Thailand. Thermogravimetric analysis of the GTR carried out showed the composition to be roughly 7.6% volatile matter, 40% natural rubber, 15% synthetic rubber (BR

and SBR), 30% carbon black and 7.4 residual mass. The particles size distribution of the GTR as obtained from particle sizes analysis were as follows; 90 vol% of the 40 mesh size GTR < 481.766  $\mu\text{m}$ , 50 vol% < 272.225  $\mu\text{m}$ , while 10 vol% < 134.047  $\mu\text{m}$  in particles sizes; 90 vol% of the 60 mesh size GTR < 298.841  $\mu\text{m}$ , 50 vol% < 186.746  $\mu\text{m}$ , and 10 vol% < 115.356  $\mu\text{m}$ ; while 90 vol % of the particles of 80 mesh size GTR < 256.178  $\mu\text{m}$ , 50 vol% < 151.962  $\mu\text{m}$ , and 10 vol% < 84.416  $\mu\text{m}$ . The other materials used for the research were natural rubber (NR) grade STR 20 (L. C. E. H Bangkok (Thailand) Co. Ltd.), butadiene rubber (BR) (BR 150) (Thai Synthetic Rubber Company Limited), N-(1,3-dimethylbutyl)-N'-phenyl-p-phenylenediamine (6PPD) (Eastman Chemical Switzerland LLC), zinc oxide (ZnO) (Thai-Lysaght Co., Ltd.), stearic acid (Asia Chem Co., Ltd.), poly(1,2-dihydro-2,2,4-trimethyl-quinoline) (TMQ) (Monflex PTE Ltd.), aromatic oil (P. S. P. Specialties Public Company Limited), Carbon black (N330) (Birla Carbon (Thailand) Public Company Limited), N-tert-butyl-2-benzothiazyl sulphenamide (TBBS) (Ningbo Actmix Rubber Chemicals Co., Ltd.) and sulfur (The Siam Chemical Public Company Limited). The formulations used for the production of the different samples of the vulcanizate are shown Table 1.

**Table 1.** Formulation used for the production of different tire tread vulcanizate samples

Sample code	Control	40/10	40/20	60/10	60/20	80/10	80/20
Compounding ingredient							
			Quantity (phr)				
NR (STR20)	80	72	64	72	64	72	64
Br (BR150)	20	18	16	18	16	18	16
GTR (40 mesh size)	-	10	20	-	-	-	-
GTR (60 mesh size)	-	-	-	10	20	-	-
GTR (80 mesh size)	-	-	-	-	-	10	20
ZnO	4	4	4	4	4	4	4
Stearic acid	2	2	2	2	2	2	2
6PPD	1.5	1.5	1.5	1.5	1.5	1.5	1.5
TMQ	1	1	1	1	1	1	1
Aromatic oil	10	10	10	10	10	10	10
Carbon black (N330)	60	60	60	60	60	60	60
Sulfur	1.8	1.8	1.8	1.8	1.8	1.8	1.8
TBBS	1.2	1.2	1.2	1.2	1.2	1.2	1.2

### Mixing methods used for the production of the vulcanizate samples

Each compound was mixed using two different methods. Two-step mixing was utilized for the first mixing method (tagged mix A). The step 1 of the mixing was carried using a 500 mL laboratory internal mixer (Brabender plasticoder lab station, Germany) at initial temperature of 60°C, fill factor of 0.78 and rotor speed of 40 rpm following the order of mixing presented in Table 2. After dumping the compound from the internal mixer and allowing it cool to room temperature, it was transferred to two-roll mill for mixing "step 2" which was carried out at 30°C for 5 minutes to ensure for homogenization.

**Table 2.** Order of mixing in the step 1 for Mix A and Mix C

Time (minute)	Mix A	Mix C
0	Loading STR 20, BR150 and GTR (where applicable)	
1 <sup>st</sup>	ZnO, stearic acid, 6PPD, TMQ, ¼ of carbon black	
3 <sup>rd</sup>	¾ of the carbon black, aromatic oil	
6 <sup>th</sup>	Sulfur + TBBS	Dumping
8 <sup>th</sup>	Dumping	-

The second mixing method (tagged mix C) also followed two-step mixing method. The first step of the mixing was done using the same internal mixer, fill factor, initial mixing temperature, at 60 rpm rotor speed in the order of mixing presented in Table 2. Two-roll mill was also employed for mixing "step 2" for Mix C and it was also carried out at 30°C for 6 minutes. In the step 2 mixing of Mix C, the curatives (TBBS and S) were added in the two-roll mill at the 3rd minute and the compound was thoroughly homogenized. After mixing, each rubber compound was stored for 24 hours before testing and further processing.

### Measurements of Mooney viscosity and cure behavior

The Mooney viscosity, MU (ML<sub>1+4</sub>, 100°C) of each compound was measured with Mooney viscometer (GOTECH, Taiwan) following ISO-289-1-2014 standard. About 25 g of uncured rubber compound was used for the measurement. The cure behavior of each rubber compound was determined with moving die rheometer (MDR-01, CGM Technology, Thailand) at temperature 150°C following ASTM D 2084-93 standard. The cure parameters of each compound reported include scorch time (t<sub>s1</sub>), optimal vulcanization time (t<sub>c90</sub>), maximum torque (MH), and minimum torque (ML). The cure rate index (CRI) for each compound was calculated from equation (1) (Zhao *et al.*, 2019). At least, 5 specimens of each sample were tested and the average values were taken.

$$CRI = \frac{100}{t_{c90} - t_{s1}} \quad (1)$$

### Production of the rubber vulcanizates via compression molding

Each compound was produced using hydraulic compression molding press (Wabash MPI, USA) under a molding pressure of 20 tons-force at 150°C. Different molds were used to produce the specimens for different tests. The optimal cure time (t<sub>c90</sub>) obtained for each sample from the cure curve was the time used for molding 2 mm thick rubber sheets for tensile and tear tests. The time used for molding the samples for Akron Abrasion testing (also used for hardness measurement) having internal diameter of 12.77 mm, external diameter of 63.95 mm and height of 12.73 mm was t<sub>c90</sub> + 10 minutes. The sample for heat build-up experiment having diameter of 16.6 mm and height 24 mm were molded using t<sub>c90</sub> + 15 minutes as the vulcanization time. The samples for testing rebound resilience measurement having diameter 28.5 mm and height 12.5 mm and those for compression set experiment having diameter 3 mm and height 6 mm were molded using t<sub>c90</sub> + 6 minutes respectively as the cure time. The vulcanized rubber samples were conditioned at room temperature for at least 24 hours before testing.

### Testing of the vulcanized rubber samples

The specimens for tensile testing were cut from the 2 mm molded vulcanizate sheet into dumbbell shape using cutting die. They were grouped into specimens for ageing and for non ageing. Tensile testing on the cut specimens were carried out at room temperature (23°C) on the universal testing machine (Instron model 5566, USA) at cross-head speed of 500 mm/min according to ASTM D412 standard. Extensometer attached to the testing machine was used to measure the elongation during the test. The specimens prepared for ageing were hung inside an air circulating oven (Gotech GT-7017-EL, Taiwan) and were maintained at 100°C for 22 hours before they were brought out and kept at room temperature for 24 hours (Imprastitichai *et al.*, 2009) and thereafter subjected to tensile testing. The ageing resistance of each vulcanized rubber sample was computed in terms of percentage retention of the tensile properties after ageing using equation (2) (Arayaprane *et al.*, 2013).

$$\text{Property retention (\%)} = \frac{\text{Value obtained after ageing}}{\text{Value obtained before ageing}} \times 100\% \quad (2)$$

The tear strength of each vulcanized rubber sample was measured on the universal testing machine at cross-head speed of 500 mm/min according to ASTM D624-00 (2020) standard. Hardness testing was carried out on each sample with shore A digital micro hardness tester (Wallace Cogenix H17a/2, England) following ISO 48-4:2018 standard. 5 indentations were made on different locations on the surface of the specimen and the average readings were recorded. Density measurement on each sample was carried out with electronic densimeter (Alfa Mirage model MD 200S, Japan) with resolution up to 0.001g/cm<sup>3</sup> in line with ASTM D297 standard. Abrasion volume loss of each vulcanizate sample was determined following BS 903-A9-2020 standard. Akron abrasion testing machine (Gotech GT-7012-A, Taiwan) was utilized and the test was carried out at room temperature (23°C) under a force of 45 N and counter weight of 26.69 N at an angle of 15°. The average of the weight losses of the 5 runs of 1000 revolutions was computed and used to calculate the volume loss (mm<sup>3</sup>) using equation (3).

$$\text{Volume loss, } \Delta v \text{ (mm}^3\text{)} = \frac{\text{weight loss, } \Delta m \text{ (g)}}{\text{density of specimen, } \rho \text{ (gcm}^{-3}\text{)}} \quad (3)$$

The compression set of each rubber vulcanizate sample was measured according to ASTM D395-18 standard using the compression set press (Gotech, GT-7049-1H, Taiwan). The height of each sample was measured before compression (H<sub>1</sub>) with micrometer gauge (Mitutoyo, Japan) and installed in the compression set press. The set containing the samples was put in air circulating oven, the temperature increased at the rate of 5 °C/min to 70°C and held for 24 hours before they were brought out, kept at room temperature (23°C) for 30 minutes and the height after compression (H<sub>2</sub>) measured. Compression set was calculated from equation (4).

$$\text{Compression set (\%)} = \frac{H_1 - H_2}{H_1} \times 100 \quad (4)$$

Heat build-up of each rubber vulcanizate sample were measured with flexometer (BFGoodrich Model II, USA) according to ASTM D623-07(2019)e1 standard under dynamic compressive force between the cross-section for 25 minutes, at constant frequency of 30 Hz, static force of 245 N, deformed distance of 3.19 mm and base temperature of 100°C.

At least, 5 specimens of each sample were tested and the average values were taken.

## Results and Discussion

### Mooney viscosity

The results of the Mooney viscosity of all the samples are presented in Table 3. The Mooney viscosities of all the compounds containing GTR were higher than that of the control sample because the introduced GTR was still in vulcanized state and the cross-links in the GTR inhibit chain mobility and that resulted to increased viscosity (Hrdlička *et al.*, 2022). Also, Mooney viscosity increased because carbon black was present in the GTR that was added. The presence of carbon black in the GTR that was added to the rubber compounds introduced additional filler and that resulted to higher viscosity. At 10 phr GTR incorporation, the compounds that were mixed using higher rotor speed exhibited lower Mooney viscosity.

The compound containing 40 mesh size GTR showed -1.55% reduction, the compound containing 60 mesh size GTR showed -10% reduction, while the compound containing 80 mesh size GTR exhibited -18.58% reduction in Mooney viscosity when the rotor speed of the mixer was increased from 40 rpm to 60 rpm. At 20 phr GTR addition to the rubber compound, the trend changed. The compounds containing 40 mesh GTR exhibited 16.67% increase in Mooney viscosity, the one containing 60 mesh size GTR showed 10.11% increase in Mooney viscosity, while the viscosity of the

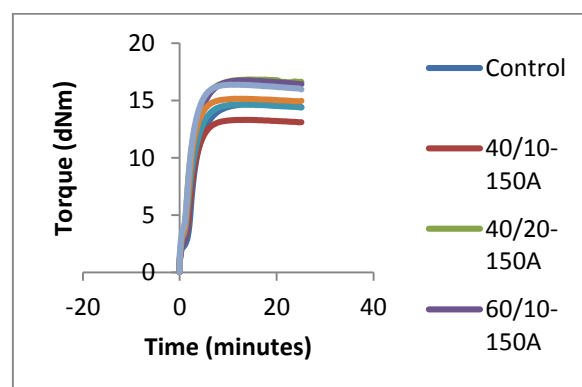
compound containing 80 mesh size GTR reduced by -9.22% when the rotor speed was increased from 40 rpm to 60 rpm. The reduced viscosity for samples containing 10 phr GTR with increased rotor speed could be attributed to breaking down of agglomerates to smaller sizes due to higher shear stress at higher rotor speed. The increased Mooney viscosity resulting from higher rotor speed for samples containing 20 phr GTR could be as a result of phase separations when the amount of GTR was increased at higher speed (Zhao *et al.*, 2019).

**Table 3.** Mooney viscosity of the rubber compounds

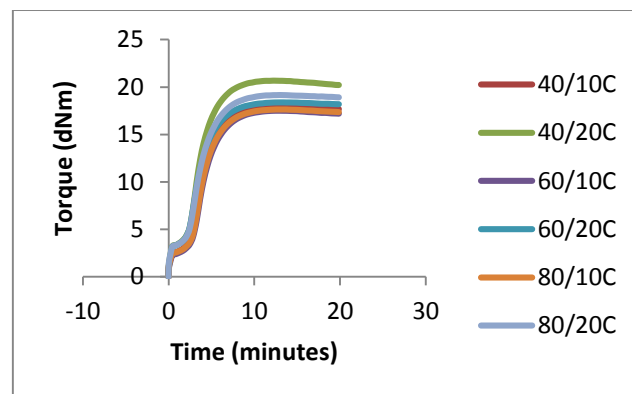
Sample	Control	40/10		60/10		80/10		40/20		60/20		80/20	
		Mix A	Mix C	Mix A	Mix C	Mix A	Mix C	Mix A	Mix C	Mix A	Mix C	Mix A	Mix C
MU	58.87	63.99	63.00	65.71	59.14	77.14	62.81	69.47	81.05	69.83	76.89	86.51	78.53
Std. dev.	0.26	0.43	0.56	0.53	0.40	0.42	0.15	0.64	0.38	0.22	0.47	0.89	0.44

**Cure behavior**

The cure behaviors of the rubber compounds are presented in Figure 1a (Mix A) and Figure 1b (Mix C), and the cure parameters are presented in Table 4. The minimum torque ( $M_L$ ) which is an indication of resistance to mixing follows the same trend as the Mooney viscosity. The  $M_L$  decreased when rotor speed was increased for all the samples containing 10 phr of GTR, and it increased when the amount of GTR was increased. The reason for that trend in  $M_L$  has been explained under Mooney viscosity. As expected, all the samples containing GTR exhibited higher  $M_L$  than the control sample. The scorch time ( $t_{s1}$ ) and cure time ( $t_{c90}$ ) of the rubber samples mixed with rotor speed of 40 rpm (Mix A) were shorter than those of the control sample, because both the GTR and curatives were added during the mixing of Mix A in the internal mixer. The increased bulk viscosity of the compounds resulted to higher shear heating during mixing, and that affected the thermal history. Formela and Haponiuk (2014) had suggested that the migration of accelerators from the GTR to the bulk rubber matrix during mixing is responsible for the higher cure rate of the compounds containing GTR compared to the control. Interestingly, the scorch time and cure time of the samples mixed at higher rotor speed (Mix C) were higher than those of Mix A suggesting lower cure rate. As a result of higher shear heating resulting from higher rotor speed, the curatives were not added in the internal mixer when the temperature was high for all the Mix C samples. The curatives were added in two-roll mill at lower temperature; therefore, their cure mechanism was different from those of Mix A samples due to different thermal history.



**Figure 1a.** Cure curves of Mix A samples



**Figure 1b.** Cure curves of Mix C sample

**Table 4.** Cure parameters from MDR tests for Mix A and Mix C

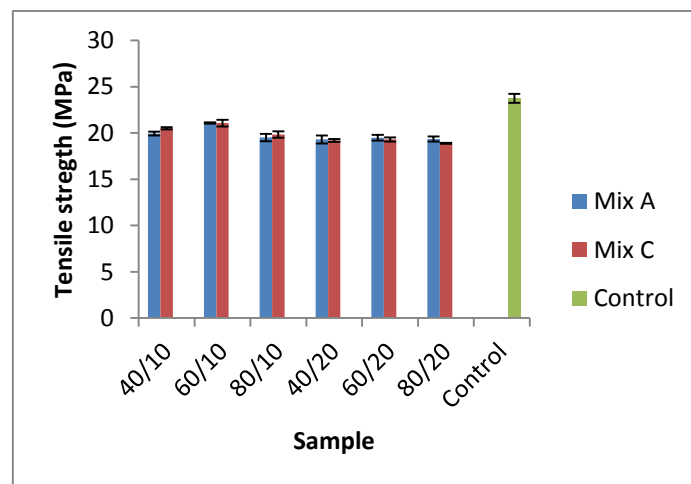
Cure parameters at 150°C for Mix A (initial mixing temp. 60°C and 40 rpm rotor speed)							
Parameter	Control	40/10A	40/20A	60/10A	60/20A	80/10A	80/20A
Maximum torque, M <sub>H</sub> , (dNm)	14.72 (0.42)	13.32 (0.21)	16.87 (0.26)	16.77 (0.35)	14.65 (0.78)	15.19 (0.3)	16.38 (0.23)
Minimum torque, M <sub>L</sub> (dNm)	2.23 (0.04)	2.66 (0.02)	3.15 (0.43)	2.75 (0.14)	3.23 (0.03)	3.43 (0.05)	3.87 (0.02)
M <sub>H</sub> – M <sub>L</sub> (dNm)	12.49 (0.44)	10.66 (0.19)	13.72 (0.38)	14.01 (0.34)	11.43 (0.76)	11.76 (0.34)	12.51 (0.23)
Scorch time, ts <sub>1</sub> (min)	1.72 (0.01)	1.52 (0.01)	1.05 (0.09)	1.30 (0.01)	1.33 (0.05)	1.26 (0.01)	1.19 (0.02)
Optimal cure time, tc <sub>90</sub> (min)	6.56 (0.23)	5.45 (0.12)	5.53 (0.16)	5.86 (0.11)	5.57 (0.08)	4.84 (0.15)	4.87 (0.02)
CRI (min <sup>-1</sup> )	20.68 (0.10)	25.50 (0.77)	21.62 (1.15)	21.94 (0.54)	23.60 (0.65)	27.84 (1.15)	27.15 (0.11)
Cure parameters at 150°C for Mix C (initial mixing temp. 60°C and 60 rpm rotor speed)							
		40/10C	40/20C	60/10C	60/20C	80/10C	80/20C
Maximum torque, M <sub>H</sub> , (dNm)		17.9 (0.22)	20.68 (0.19)	17.49 (0.09)	18.37 (0.46)	17.63 (0.38)	19.16 (0.48)
Minimum torque, M <sub>L</sub> (dNm)		2.58 (0.1)	3.35 (0.02)	2.39 (0.04)	3.30 (0.03)	2.57 (0.05)	3.23 (0.07)
M <sub>H</sub> – M <sub>L</sub> (dNm)		15.32 (0.31)	17.33 (0.21)	15.1 (0.06)	15.07 (0.48)	15.06 (0.04)	15.84 (0.51)
Scorch time, ts <sub>1</sub> (min)		2.29 (0.01)	2.08 (0.03)	2.44 (0.02)	2.11 (0.01)	2.40 (0.03)	2.12 (0.01)
Optimal cure time, tc <sub>90</sub> (min)		6.73 (0.07)	6.54 (0.21)	7.03 (0.05)	6.65 (0.14)	6.90 (0.09)	6.71 (0.08)
CRI (min <sup>-1</sup> )		22.52 (0.18)	22.42 (0.20)	21.79 (0.12)	22.03 (0.39)	22.22 (0.41)	21.79 (0.35)

(Standard deviations of the results of tests on 5 specimens are in the parenthesis)

**Properties of the vulcanizates**

The tensile strength of the rubber vulcanizates before and after ageing are shown in Figure 2 (a and b). In Figure 2a, improvement in tensile strength was observed in the samples containing 10 phr GTR when the rotor speed was increased from 40 rpm to 60 rpm for the three mesh sizes. This could be attributed to better distribution of materials in the rubber matrix due to higher shear stress as a result of higher rotor speed resulting to lower viscosity. Conversely, the tensile strength of the samples containing 20 phr of 40 mesh size and 60 mesh size GTR reduced by about 0.5% and 1% respectively while that of the sample containing 80 mesh size GTR reduced by about 2.5% when they were mixed with higher rotor speed as shown in Figure 2a. The slight decrease in tensile strength of samples containing more GTR when mixed at higher rotor speed could be attributed to phase separation which was dominant at high amount of GTR in the rubber matrix and high rotor speed. It can be seen from Figure 2a that change in rotor speed from 40 rpm to 60 rpm did not impart much on the tensile strength of the vulcanizate containing GTR, the major factor that imparted the tensile strength was the amount of GTR in the rubber matrix and the particles size of the GTR. This is because amount of GTR and particles sizes of the GTR influence the dispersion of the fillers in the matrix as they affect the bulk viscosity and shear stress within the rubber matrix. Those mixed with higher rotor speed however exhibited lower tensile strength after ageing (Figure 2b) as well as lower tensile retention (Figure 3) suggesting that they will age faster. Already, the incorporated GTR had a thermal history from vulcanization process, mixing with higher speed resulting to higher bulk temperature due to shear heating further imparted the thermal history of the GTR and therefore the samples containing GTR and mixed at higher rotor speed showed lower ageing resistance. The other tensile parameters including elongation at break, modulus at 100% and modulus at 200% elongation shown in Figures 4 and 5 (a-b) suggest that those mixed

with rotor speed of 40 rpm generally exhibited better properties than those mixed with rotor speed of 60 rpm. Higher modulus can also be indication of poor dispersion. The effect of rotor speed was more visible in those properties.



**Figure 2a.** Tensile strength of the vulcanizates before ageing

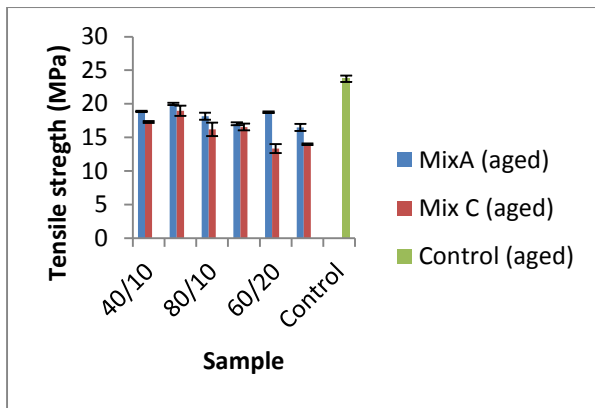


Figure 2b. Tensile strength of the vulcanizates after ageing

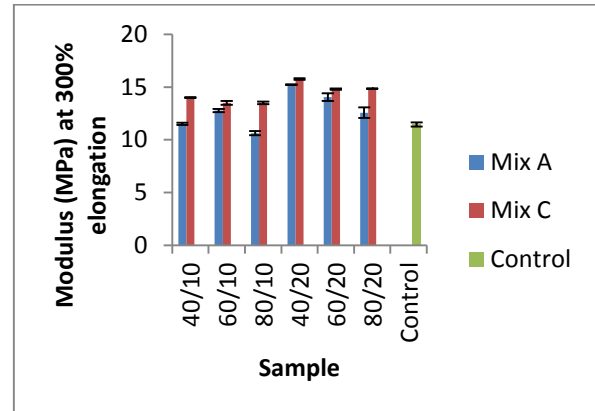


Figure 5b: Modulus at 300% elongation of the vulcanizate samples

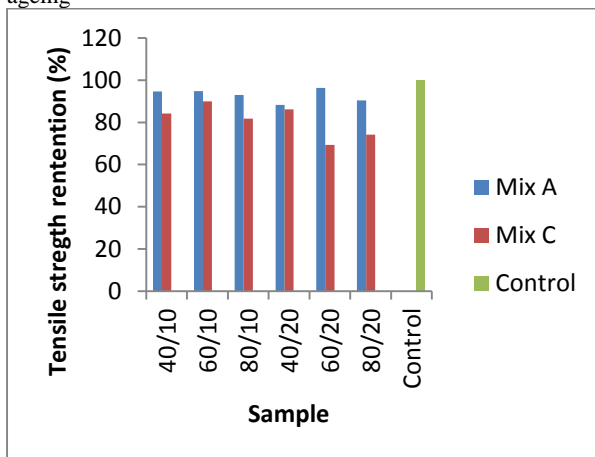


Figure 3. Tensile retention of the vulcanizate samples

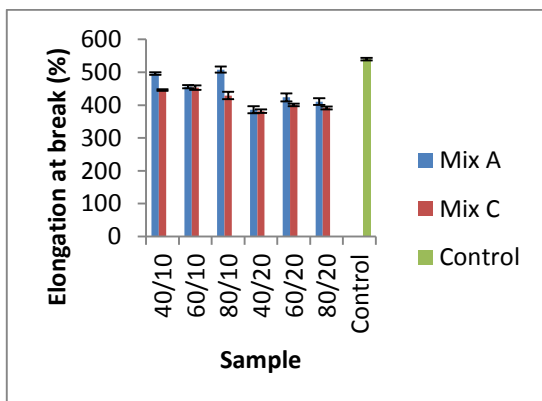


Figure 4. Elongation at break of the vulcanizate samples

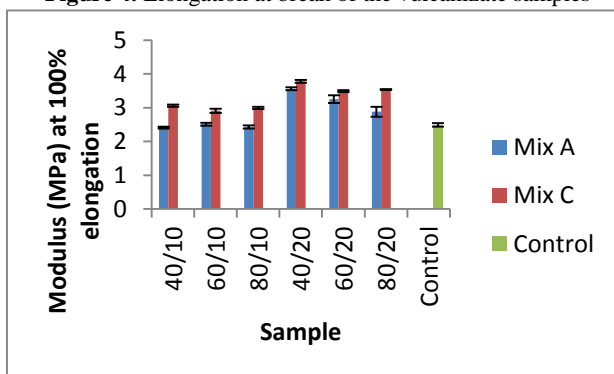


Figure 5a. Modulus at 100% elongation of the vulcanizate samples

The tear strength of the vulcanizates are shown in Figure 6 and the samples mixed with higher rotor speed exhibited lower tear strength. Earlier, de Carvalho *et al.* (2018) reported that mixing with lower rotor speed of internal mixer promotes better dispersion and that translates into improved properties. The results obtained in this research are in agreement with that. The hardness of the vulcanizates mixed with higher rotor speed were also higher than those of the ones mixed with lower rotor speed as shown in Figure 7. This is because of the rigidity (higher modulus) imparted in the vulcanizates as a result of shear heating during the mixing with higher rotor speed. All the vulcanizates containing GTR showed lower abrasion volume loss than the control as shown in Figure 8 and this is very desirable. Those samples mixed with higher rotor speed however showed lower abrasion loss because of their higher hardness. The compression set of the rubber vulcanizates are shown in Figure 9. Compression set is a permanent loss of shape of the vulcanizates as a result of applied static force over a period of time. All the samples mixed with higher rotor speed exhibited higher compression set, and that is not desirable. The higher compression set could be an indication of lower cross-linked density. The increase in compression set could also be as a result of phase separation and filler agglomeration resulting to impaired elasticity.

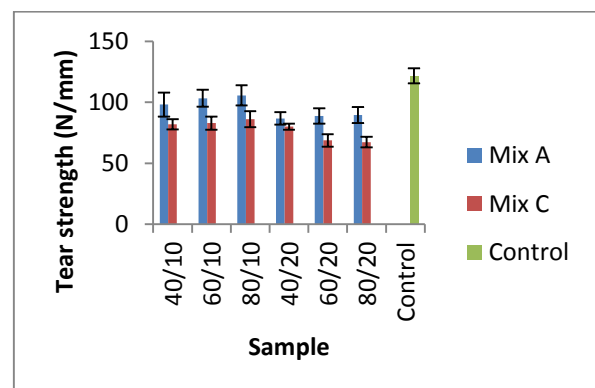


Figure 6. Tear strength of the vulcanizate samples

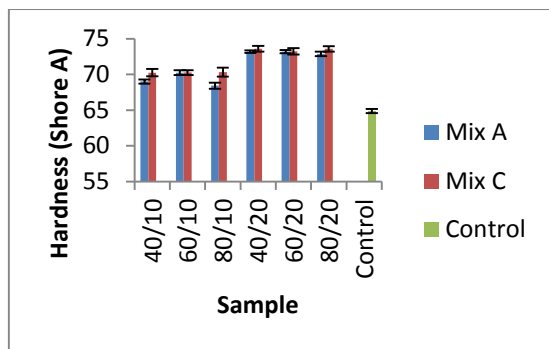


Figure 7. Hardness of the vulcanizate samples

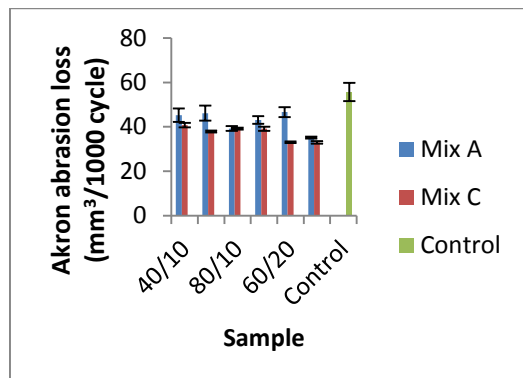


Figure 8. Akron abrasion loss of the vulcanizate samples

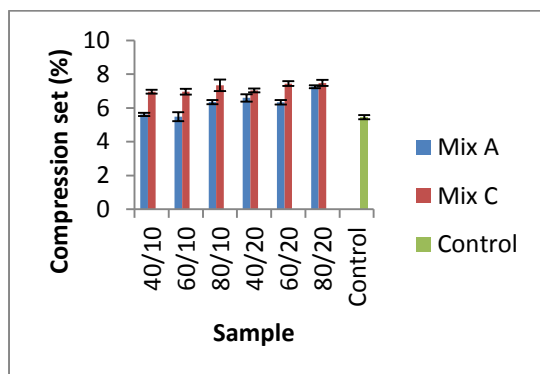


Figure 9. Compression set of the vulcanizate samples

The heat build-up of the rubber vulcanizates are shown in Figure 10. Generally, all the samples that contained GTR exhibited higher heat build-up than the control sample and the heat build-up increased further with increase in the amount of GTR in the matrix. This is because, introduction GTR in the matrix also increased the carbon black in the matrix and more filler loading will give rise to higher temperature development during cyclic testing due to the breakage of carbon black structure, viscoelastic losses and breaking and forming of anteraggregate bonds in carbon black (Park *et al.*, 2000). Also, the presence of GTR in the matrix increased the stress factor which contributed to heat development (Park *et al.*, 2000). Changing the rotor speed from 40 rpm to 60 rpm did not impart significantly on the heat build-up. The slightly lower heat build-up recorded by the samples containing 40 mesh size GTR at 10 and 20 phr and mixed with 60 rpm rotor speed when compared with those mixed with 40 rpm rotor speed was due to the high modulus of the vulcanizates as shown Figures 5 (a-b), which did not allow more filler break down during the cyclic loading, and thereby preventing the formation

of secondary network by carbon black that could be responsible for heat generation ((Park *et al.*, 2000; Zhang *et al.*, 2015). The same reason is responsible for the slight drop of heat build-up of the sample containing 80 mesh size GTR at 10 phr loading and mixed with 60 rpm rotor speed when compared to that of similar sample mixed with 40 rpm rotor speed. Although, the sample containing 80 mesh size GTR at 20 phr and mixed with 60 rpm rotor speed exhibited higher modulus when compared to modulus of the sample containing 80 mesh size GTR and mixed with 40 rpm rotor speed, the heat build-up could not reduce to a lower value because the viscosity dropped. The drop in viscosity may have resulted to poor distribution which increased the stress level (due to presence of GTR) in the rubber vulcanizate. The results generally point out that variation of heat build-up with change in rotor speed for the rubber vulcanizates filled with different particles sizes of GTR is a bit complicated.

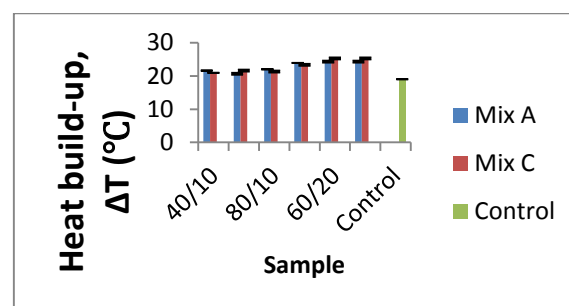


Figure 10. Heat build-up of the vulcanizate samples

### Conclusions

This work has presented the properties of solid tire tread compound containing GTR of three different mesh sizes and mixed with two different rotor speed. The addition of GTR to the rubber compounds increased the viscosity. Further increase was observed when the amount of GTR increased. Both particle sizes of the GTR and rotor speed used during mixing influenced the viscosity. The addition of GTR in the rubber matrix also affected the cure parameters compared to the cure parameters of the rubber compound that was not filled with GTR. Among the rubber compounds containing GTR, the particles sizes and the amount of GTR did not influence the scorch time and cure time much. The mixing torque was affected by those parameters due to change in viscosity. However, changing the rotor speed of the internal mixer and adding the curative at lower temperature changed the cure rate by influencing the scorch time and cure time. Generally, the addition GTR to the rubber matrix lowered the tensile strength and increased the heat build-up. The compounds containing GTR mixed with 40 rpm rotor speed showed a combination of properties better than those of the compounds mixed with 60 rpm rotor speed. However, the abrasion resistances of the compounds mixed with 60 rpm rotor speed were better than those of the compounds mixed with 40 rpm rotor speed.

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The authors declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

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