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Effect of Varied Cure Temperature on the Cure Behavior, Mechanical Properties and Heat Build-Up of Solid Tire Tread Compound Containing Different Particles Sizes of Ground Tire Rubber

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Abstract

This work investigates the effect of variation in vulcanization temperature on the cure behavior, tensile properties, tear strength, hardness, Akron abrasion loss and heat build-up of vulcanized tire tread compound in which the total rubber was replaced with 10% and 20% ground tire rubber (GTR) of 40, 60 and 80 mesh sizes. The first step was the characterization of the GTR using optical microscopy and a thermogravimetric analyzer. The first step of mixing each rubber compound was carried out using an internal mixer (Brabender) at 60°C and 40 rev/min rotor speed and the second step was done on a two-roll mill. The mooney viscosity of each rubber compound was investigated. the cure behavior of the rubber compounds was studied at 140°C, 150°C, and 160°C respectively. Results show that the introduction of GTR reduced vulcanization time, while increased cure temperature resulted in a lower vulcanization time. No significant difference was observed in the vulcanization time at a particular temperature of the rubber sample with different particle sizes and varied amounts of GTR. The introduction of ground tire rubber in the rubber vulcanizate however resulted in decreased tensile strength, tear strength and elongation, and increased hardness, abrasion resistance, modulus and heat build-up. An increase in the vulcanization temperature from 140°C to 150°C and 160°C did not impart a significant reduction in the tensile strength of the vulcanizates containing GTR. Vulcanization at higher temperatures resulted in reduced hardness, and modulus, slightly increased abrasion loss and increased elongation of the vulcanizates containing GTR. The heat build-up increased as the cure temperature increased. Generally, the control sample with no content of ground tire rubber exhibited the best properties. If GTR is to be introduced in rubber matrix at an amount up to 20%, the results suggest that 60 mesh size particles will impart better properties. The optimal vulcanization temperature recommended based on the findings from the work is 150°C.

Keywords: *Solid tire tread compound, Ground tire rubber, Vulcanization temperature, Properties.*

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1. Introduction

The recycling problems of vulcanized rubber in the form of end-of-life tire and other rubber goods after their service lives have posed a lot of challenges to the environment, industry and research. Vulcanized rubbers are cross-linked and do not degrade when discarded after service life and as such contribute to environmental solid waste problems. Researchers are actively exploring the possible methods for the re-use and recycling of end-of-life vulcanized rubber products such as pyrolysis, reclaiming and devulcanization, and the introduction of ground tire rubbers (GTR) as fillers in composites. Some works have been reported on the use of GTR in composites development. The introduction of GTR in concrete resulted in reduced thermal conductivity and density; however, a decrease in mechanical strength was observed and the incorporation of coarser particles of GTR resulted in more decrease in the strength of the concrete [1]. The incorporation of a GTR/recycled polyethylene blend in asphalt improved the high-temperature performance of the asphalt for road construction [2]. In thermoplastics and thermosets, GTR is used as a toughening agent [3-4]. The use of GTR in composites is geared towards removing waste from the environment, improving the properties of the composite material and reducing the cost of the composites. The use of GTR as filler and partial replacement of virgin rubber in rubber vulcanizates has been investigated [5-6]. The major challenge in the introduction of GTR in rubber compounds results in the decline of the strength and elongation of the vulcanizate compared to filled vulcanizates without GTR due to lack of adequate chain mobility in GTR and interaction with the matrices [7]. Research works have been carried out on various methods of surface modification of GTR to improve their compatibility with matrices and improve the properties of composites containing GTR. Surface functionalization of GTR through various methods such as the use of high energy gamma radiation [8], ultraviolet irradiation [9], plasma [10] and ozone [11] have been used to improve the performance of GTR in matrices. The use of chemical methods such as coupling agents, surface grafting, acids, and oxidizing agents have also been used to improve the interaction of GTR in matrices [12-15]. It has been reported that the variation in the cure temperature of rubber vulcanizates affects their mechanical properties [16]. This work investigates the effects of variation in cure temperature on the cure behavior, some of the mechanical properties and heat build-up of solid tire tread compound containing ground tire rubber.

2. Materials and method

2.1. Materials

Three different mesh sizes (40, 60 and 80 mesh sizes) of ground tire rubber (GTR) were provided by Rubber Technology Research Centre, Mahidol University Thailand. Natural rubber (NR) STR 20 crumb supplied in blocks from L. C. E. H Bangkok (Thailand) Co. Ltd., butadiene rubber (BR) (BR 150) was supplied by Thai Synthetic Rubber Company Limited, zinc oxide (ZnO) supplied by Thai-Lysaght Co., Ltd., stearic acid was supplied by Asia Chem Co., Ltd., N-(1,3-dimethylbutyl)-N'-phenyl-p-phenylenediamine (6PPD) was supplied by Eastman Chemical Switzerland LLC, poly(1,2-dihydro-2,2,4-trimethyl-quinoline) (TMQ) was supplied by Monflex PTE Ltd., aromatic oil was supplied by P. S. P. Specialties Public Company Limited, Carbon black (N330) was supplied by Birla Carbon (Thailand) Public Company Limited, sulfur was supplied by The Siam Chemical Public Company Limited and (N-tert-butyl-2-benzothiazyl sulphenamide (TBBS) was supplied by Ningbo Actmix Rubber Chemicals Co., Ltd. The formulation used for the production of the samples and the codes given to various samples for identification are presented in Table 1.

2.2. Characterizations of the GTR

The morphologies of the GTR of different mesh sizes were studied under high-resolution optical microscope (Olympus BX53M, Japan) at a magnification of 1000. Thermogravimetric analysis

(TGA) of the GTR was carried out on thermogravimetric analyzer (NetzschTG 209 F1 Libra, Germany) by heating about 10 mg of the powder sample in alumina pan under nitrogen atmosphere at a flow rate of 10 ml/min and heating rate of 20 ℃/min from room temperature (22℃) to 600℃ followed by heating in oxygen atmosphere at the same flow rate and heating rate from 600℃ to 850℃.

Table 1: Formulation of Different Samples of the Tire Tread Rubber Compound

Sample code	**Control**	**40/10**	**40/20**	**60/10**	**60/20**	**80/10**	**80/20**
Material	**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

2.3. Mixing of the tire tread rubber compound

The mixing was carried out by a two-step mixing method. The first step of the mixing was done using a 500 ml laboratory internal mixer (Brabender plasticoder lab station, Germany) at a starting temperature of 60℃, fill factor of 0.78 and rotor speed of 40 rpm. The order of mixing in the first step is presented in Table 2. The compound from the internal mixer was allowed to cool to room temperature after dumping before it was transferred to a two-roll mill for mixing step 2 at 30℃ for 5 minutes for homogenization. After mixing, each rubber compound was stored for 24 hours before testing and further processing.

2.4. Determination of the Mooney viscosity and cure behavior

The Mooney viscosity of each rubber compound was determined at 100℃ with the testing machine (GOTECH, Taiwan) in accordance with ISO-289-1-2015 [17]. About 25 g of each rubber compound was used for the experiment. The Mooney viscosity (ML_{1+4}, 100℃) at 4 minutes was recorded. The cure behavior of each rubber compound was determined with a moving die rheometer (MDR-01, CGM Technology, Thailand) at temperatures 140℃, 150℃ and 160℃ respectively in line with ASTM D 2084-2019 [18]. The parameters recorded from the MDR experiment were scorch time (t_{s1}), optimal vulcanization time (t_{c90}), maximum torque (MH), and minimum torque (ML) for each compound at the varied cure temperature. The cure rate index (CRI) was calculated from equation (1) [19].

$$\mathrm{CRI} = \frac{100}{t_{c90} - t_{s1}} \tag{1}$$

Table 2: Order of mixing in Step 1

Time (minute)	Operation
0	Loading STR 20, BR150 and GTR (where applicable)
1	ZnO, stearic acid, 6PPD, TMQ, ¼ of carbon black
3	¾ of the carbon black, aromatic oil
6	Sulfur + TBBS
8	Dumping

2.5. Curing of the rubber compounds

Each compound was cured on a hydraulic press compression mounding machine (Wabash MPI, USA) under a molding pressure of 20 tons-force at temperatures 140℃, 150℃, and 160℃ respectively. The optimal vulcanization time (t_{c90}) obtained for each compound from the rheometric measurements was used as the vulcanization time to produce 2 mm thick vulcanized rubber sheets for tensile testing and tear testing. The specimens for Akron abrasion testing (also used for the determination of the hardness) having an internal diameter of 12.77 mm, external diameter of 63.95 mm and height of 12.73 mm were molded with their specific mold using t_{c90} + 10 minutes as the vulcanization time. Samples for heat build-up having a diameter of 16.6 mm and height of 24 mm were molded with their designated mold using t_{c90}+15 minutes as the cure time. The vulcanized rubber samples were stored at room temperature for at least 24 hours before testing.

2.6. Characterizations of the vulcanized rubber samples

Dumbbell-shaped specimens for tensile testing were cut from the vulcanized rubber sheets using a cutting die and grouped into specimens for aging and non-aging. The specimens that were not aged were subjected to room temperature (23℃) tensile testing on the universal testing machine (Instron model 5566, USA) at a cross-head speed of 500 mm/min according to ASTM D412-16 (2021) standard test methods for vulcanized rubber and thermoplastic elastomers [20]. An extensometer was attached to the testing machine to measure elongation. 5 repeated tests were done on each sample and the average results were reported to ensure reliability. The specimens for aging were hung inside an air-circulating oven (Yasuda Geer aging oven N0. 102, Japan) and were maintained at 100℃ for 22 hours before they were brought out and kept at room temperature for 24 hours [21] and then subjected to tensile testing. The aging resistance of the vulcanized rubber samples was expressed in terms of percentage retention of the tensile properties after aging using equation (2) [22].

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

The determination of the tear strength of the vulcanized rubber samples on the universal testing machine at a cross-head speed of 500 mm/min followed the specification of ASTM D624-00 (2020) standard test method for tear strength of conventional vulcanized rubber and thermoplastic elastomers [23]. Shore digital A microhardness tester (Wallace cogenix H17a/2, England) was used to determine the hardness of each specimen in line with ISO 48-4:2018 standard [24]. 5 indentations were made on different locations on the surface of the specimen and the average of the readings was recorded as the hardness of each specimen. The density of molded rubber specimen was determined using an electronic densimeter (Alfa Mirage model MD 200S, Japan) with a resolution up to 0.001g/cm^3 in line with ASTM D297-21 standard [25]. The abrasion volume loss of each vulcanizate sample was determined in line with BS 903-A9-2020 standard [26] using an Akron abrasion testing machine (Gotech GT-7012-A, Taiwan) at room temperature (23℃) under a force of 45 N and counterweight of 6 lb (26.69 N) at an angle of 15^{c}. The average of the weight loss of the 5 runs of 1000 revolutions was computed and used to calculate the volume loss (mm^3) using equation (3).

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

Heat build-up and dynamic compression set were determined using molded cylindrical vulcanizate specimens of height 24.03 mm and diameter 16.6 mm with Flexometer (BFGoodrich Model II, USA) as specified by ASTM D623-07(2019)e1 standard test methods for rubber property-heat generation and flexing fatigue in compression [27] under dynamic compressive force between the cross-section for 25 minutes, at a constant frequency of 30 Hz, static force of 245 N, deformed distance of 3.19 mm and base temperature of 100℃. The dynamic set was calculated using equation (4) and H_1 and H_2 are the height of the specimen in mm before and after testing. Three specimens were tested for each sample and the average results were calculated and reported.

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

3. Results and discussion

3.1. Propertied of the GTR

The micrographs obtained from the optical microscopy of the GTR of the different mesh sizes are presented in Figures 1 (a- c). The particles of the 80 mesh size GTR appear finer, followed by the particles of the 60 mesh size GTR while the 40 mesh size GTR has the biggest sizes. Hakrama *et al.* [28] earlier reported that the white stains in the matrix of GTR observed under the microscope are foreign materials as a result of the incorporation of additives such as zinc oxide, stearic acid, sulfur,

silica, and others for different purposes during compounding. The irregular shapes of the crumb rubber particles shown in the optical micrographs imply that they were ground at ambient temperature [29].
The TGA and DTG curves of the GTR are shown in Figure 2. The TGA curves are similar to the earlier reported TGA curve of rubber crumbs obtained from scrap tires [30]. The major components of the GTR as deduced from the TGA curves are volatile substances, natural rubber, synthetic rubber, carbon black and residual ash. Generally, the degradation of each of the components overlapped but marked differences were seen at the different stages of the degradation. In the TGA curves, no significant degradation was observed up to about 276℃ because rubber undergoes both scission and cross-linking when heated within that temperature range [31]. The weight loss which was approximately -7.9% recorded within 276 - 350℃ from the TGA curve was due to the volatilization of volatiles such as moisture and plasticizers. Thereafter, the degradation of natural rubber started and stretched out to about 420℃ roughly recording a further -34.7% weight loss. The degradation of synthetic rubber (SBR and BR) followed and reached up to about 490℃ and recorded a further -22.7% weight loss. The decomposition of carbon black started at about 605℃ and proceeded to 695℃ recording about -27.15% weight loss. The residue left after the degradation process was about -7.42%. The major peaks from the DTG curve were observed at 391.6℃ , 445℃ and 667.9℃ signifying the temperature at which the maximum rate of weight loss occurred. The first and second peaks were the degradation of rubber and synthetic rubber respectively, while the third peak was that of carbon black.

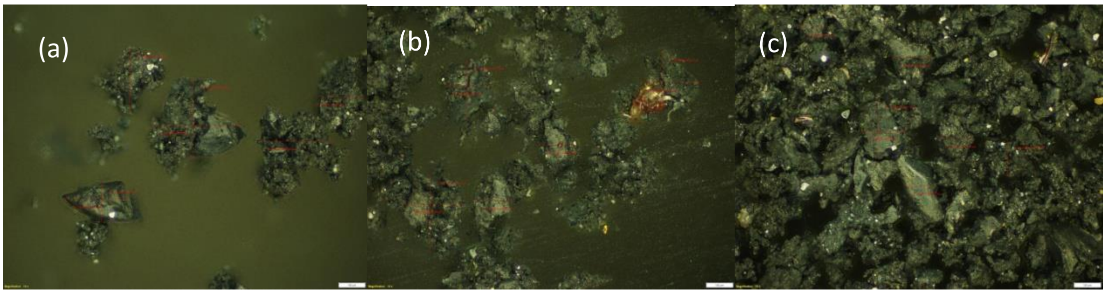

Figure 1. Images of the GTR from optical microscope (a) 40 mesh size (b) 60 mesh size (c) 80 mesh size

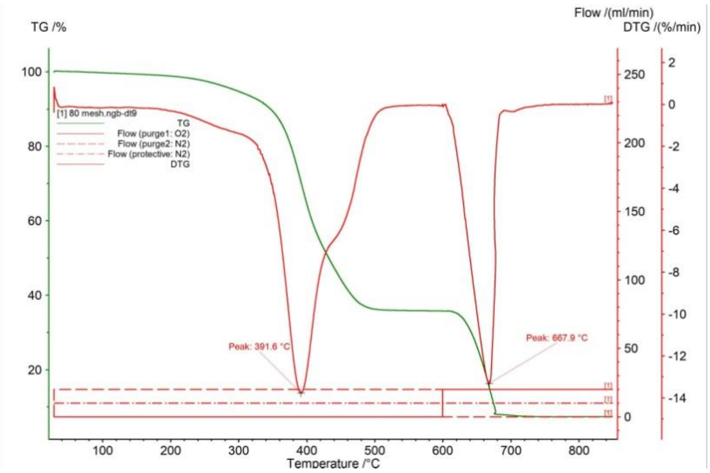

Figure 2. TGA and DTG curves of the GTR

3.2. Mooney viscosity

The results of the Mooney viscosity check on the tire tread compounds are presented in Table 3. Generally, the Mooney viscosity increased with the addition of GTR in the rubber compound and further increased with an increased amount of the GTR in the rubber compound. At the same loading of the GTR, while there is not much difference in Mooney viscosity of the rubber compounds containing 40 mesh GTR and 60 mesh size GTR, the compound containing 80 mesh size GTR showed higher Mooney viscosity. The increase in Mooney viscosity as a result of the addition of GTR has been reported to be a result of the presence of cross-linked gel in GTR which restricts rubber mobility and reduces ease of processing [5].

Table 3: Mooney viscosity (MU) of the rubber compounds

Parameter	**Control**	**40/10**	**60/10**	**80/10**	**40/20**	**60/20**	**80/20**
MU (ML 1+4, 100℃)	58.87	63.99	65.71	77.14	69.47	69.83	86.51
Standard deviation	0.26	0.43	0.53	0.43	0.64	0.22	0.89

3.3. Cure behavior

The cure curves at 140℃, 150℃ and 160℃ are shown in Figures 3(a-c) respectively while the cure parameters obtained at the various conditions are presented in Table 4. The minimum torque (M_L) followed the same trend as the Mooney viscosity as the addition of GTR in the rubber compound increased the minimum torque and further increased it with an increase in the amount of GTR in the rubber compound with the compound filled with 80 mesh size GTR having higher M_L at all conditions. The higher M_L indicates that it is more difficult to process rubber compounds containing GTR due to the cross-linked nature of GTR which imparts difficulty in mixing as a result of the formation of free space in the rubber resulting in higher viscosity [32]. Increased cure temperature from 140℃ to 150℃ resulted in increased minimum torque, while it decreased when the cure temperature was increased to 160℃. This is because, as the temperature increases, the mobility of the molecules will increase thereby lowering the viscosity [16]. The scorch time (t_{s1}) as shown in Table 4 reduced with the addition of GTR in the rubber matrix also due to the migration of sulfur from the rubber matrix to the GTR [32]. The reduced scorch time is also an indication of a higher cure rate in the rubber compound as a result of the addition of GTR. Since the control sample was cured at 150℃, it can be seen from Figures 3b and Table 4 that the addition of GTR to the rubber compound reduced both the cure time (tc_{90}) and scorch time (t_{s1}). A shorter cure time was observed with an increase in cure temperature. The lower cure time observed in the compounds containing GTR was reported to be due to the migration of accelerators from the GTR to the rubber matrix [16, 32-33]. Generally, an increase in cure temperature increases the vulcanization rate resulting in a lower cure time at a higher cure temperature. The variation in particle size of the GTR and its amount in the rubber did not impart significant change in the cure time at 10 phr and 20 phr replacement of rubber with GTR. The parameter that affected the cure time was the change in cure temperature. This suggests that at 10 phr and 20 phr replacement of rubber with GTR, the mechanism of curing of all the compounds containing the GTR is similar despite the variation in particle size of the GTR. The cure rate index (CRI) of the rubber compounds as shown in Table 4 generally increased with an increase in cure temperature. The slightly higher cure rate index of samples 40/10, 80/10 and 80/20 may be attributed to a higher amount of accelerator in the GTR that was added to the batch during mixing or the higher presence of unsaturated bonds resulting in competition between the cross-linking agents especially at higher curing temperature [32,34].

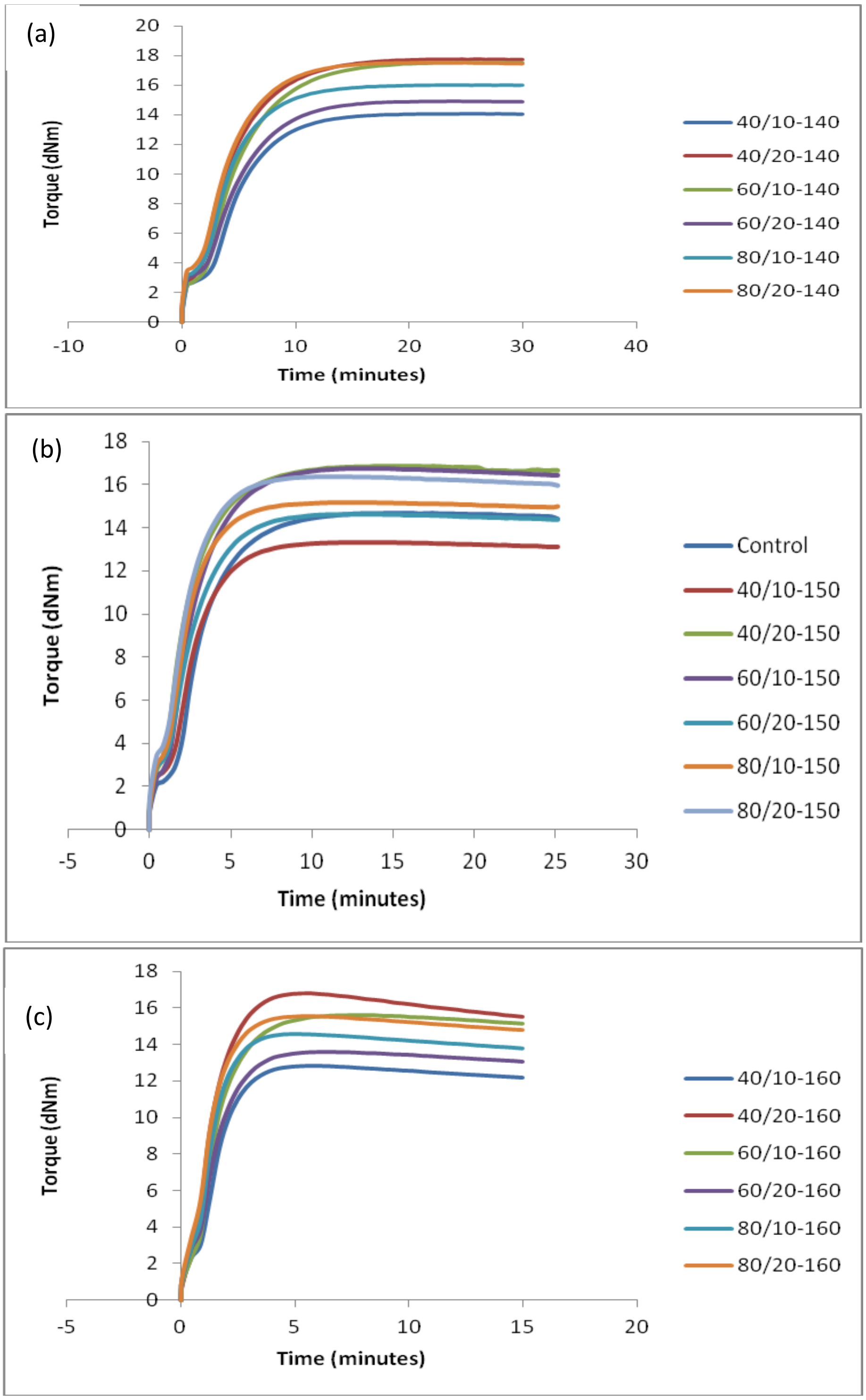

Figure 3. MDR rheograph of compounds at cure temperatures of (a) 140℃, (b) 150℃ and (c) 160℃.

Table 4: Cure parameters from the MDR measurement

Cure Parameters at 140℃							
Parameter		**40/10-140**	**40/20-140**	**60/10-140**	**60/20-140**	**80/10-140**	**80/20-140**
Maximum torque, M_H, (dNm)		14.06 ±0.01	17.75 ±0.12	17.56 ±0.3	14.94 ±0.17	16.04 ±0.26	17.52 ±0.18
Minimum torque, M_L (dNm)		2.64 ±0.06	2.95 ±0.12	2.67 ±0.14	3.10 (0.07)	3.29 ±0.04	3.65 ±0.04
$M_H – M_L$ (dNm)		11.42 ±0.05	14.4 ±0.19)	14.88 ±0.22	11.84 ±0.2	12.75 ±0.29	13.87 ±0.15
Scorch time, ts_1 (min)		2.60 ±0.03	1.74 ±0.1	2.10 ±0.12	2.28 ±0.07	1.96 ±0.03	1.80 ±0.02
Optimal cure time, tc_{90} (min)		9.88 (0.17)	9.91 (0.33)	10.75 (0.51)	10.08 (0.22)	9.06 (0.42)	9.04 (0.21)
CRI (min^{-1})		13.74 ±0.34	12.25 ±0.49	11.58 ±0.66	12.83 ±0.31	14.10 ±0.83	13.81 ±0.38
Cure Parameters at 150℃							
	Control	**40/10-150**	**40/20-150**	**60/10-150**	**60/20-150**	**80/10-150**	**80/20-150**
Maximum torque, M_H, (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_L (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
$M_H – M_L$ (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_1 (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_{90} (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^{-1})	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 160℃							
		40/10-160	**40/20-160**	**60/10-160**	**60/20-160**	**80/10-160**	**80/20-160**
Maximum torque, M_H, (dNm)		12.86 ±0.3	16.81 ±0.23	15.61 ±0.14	13.65 ±0.19	14.56 ±0.03	15.53 ±0.35
Minimum torque, M_L (dNm)		2.32 ±0.06	2.7 ±0.01	2.32 ±0.15	2.83 ±0.04	2.89 ±0.03	3.37 ±0.05
$M_H – M_L$ (dNm)		10.54 ±0.31	14.12 ±0.23	13.28 ±0.05	10.82 ±0.15	11.67 ±0.03	12.16 ±0.31
Scorch time, ts_1 (min)		0.89 ±0.01	0.63 ±0.01	0.76 ±0.01	0.82 ±0.05	0.70 ±0.01	0.63 ±0.01
Optimal cure time, tc_{90} (min)		2.98 ±0.03	2.88 ±0.01	3.32 ±0.07	3.14 ±0.10	2.61 ±0.02	2.68 ±0.03
CRI (min^{-1})		47.85 ±0.60	44.45 ±0.20	39.03 ±0.98	43.14 ±1.10	52.36 ±0.55	48.94 ±0.55

±Standard deviations

3.4. Properties of the vulcanizates

The results of the tensile properties before and after aging are shown in Figures 4-7. Comparing the result of the control sample (without GTR) and the others in Figures 4-7, it was observed that the introduction of GTR in the rubber vulcanizate reduced the tensile strength (Figure 4) and elongation (Figure 6), while the modulus increased (Figures 7a and 7b) in line with earlier reports [5-6, 35] due to the cross-linked nature of the GTR. The depreciation of the properties was more significant as the amount of GTR in the vulcanizate increased. At 10 and 20% replacement of rubber with GTR, the tensile strengths of the vulcanizates containing 60 mesh size of the GTR were better than those containing 40 mesh size and 80 mesh size; while the elongation was highest in the vulcanizate containing 80 mesh size, followed by those containing 40 mesh size GTR with the ones containing 60 mesh size showing the least elongation at 10% replacement of rubber with GTR. At 20% replacement, the trend in elongation showed dependence on cure temperature with samples containing 60 mesh size GTR exhibiting higher elongation at 150℃ and 160℃, while the ones containing 80 mesh size GTR showed the highest elongation at 140℃ cure temperature. The increase in vulcanization temperature from 140℃ to 160℃ imparted only a slight reduction in tensile strength as shown in Figure 4. The slight reduction in tensile strength as a result of an increase in vulcanization temperature is similar to the result earlier reported by Lee *et al.* [16].

On the contrary, the increase in vulcanization temperature of the tire tread compound containing GTR resulted in improved elongation as shown in Figure 6 for all the samples; although the control sample without GTR showed superior elongation than all the other samples at 150℃. The slight improvement in elongation as a result of higher vulcanization temperature was due to the reduction in modulus and hardness due to the increase in vulcanization temperature as shown in Figures 7 (a-b) and 8 respectively. The lower strength, hardness and modulus observed due to curing at higher temperatures have been attributed to be due to formation of a network with lower cross-link density [36]. Careful observation of the tensile strength (Figure 4) and tear (Figure 9) of the samples indicate that those cured at 150℃ exhibited a better combination of properties than those cured at 140℃ and 160℃. In the case of the rubber compound containing GTR, the reduction in hardness, elastic modulus and increase in elongation are highly desirable as the introduction of GTR in rubber caused excessive hardness, modulus and reduction of elongation. It is only the reduction of strength that is not desirable. The samples cured at 160℃ appear to exhibit slightly better aging resistance as shown in Figure 5. It has been reported that vulcanizing at temperatures higher than 160℃ will impart very poor mechanical properties in natural rubber-based vulcanizates [36-37]. The results of the Akron volume loss are shown in Figure 10. The addition of GTR in tire tread vulcanizate does not pose any abrasion problem as all the samples containing GTR showed lower volume loss than the control sample due to the higher hardness of the vulcanizate containing GTR resulting in less particle separation from the rubber and lower friction coefficient [35]. However, those cured at higher temperatures showed higher abrasion volume loss. The heat build-up is shown in Figure 11 while the dynamic set is shown in Figure 12. Earlier, Zhang *et al*. [36] suggested that curing temperature affects the structure and properties of natural rubber-based compounds. High curing temperature was reported to affect the stability of the bond and produce lower cross-links which results in higher heat build-up [36]. In this report, Figure 11 shows that the heat build-up increased as the curing temperature increased with the samples cured at 160℃ showing higher heat build-up at both 10 phr and 20 phr GTR replacement of rubber. Also, the higher amount of GTR in the rubber compound resulted in increased heat build-up. Samples with higher heat-up could be said to have weaker cross-links. The results of the dynamic set in Figure 12 also suggest that 150℃ is the optimal vulcanization temperature as the samples vulcanized at 150℃ exhibited a lower dynamic set compared to those cured at 140℃ and 160℃ respectively.

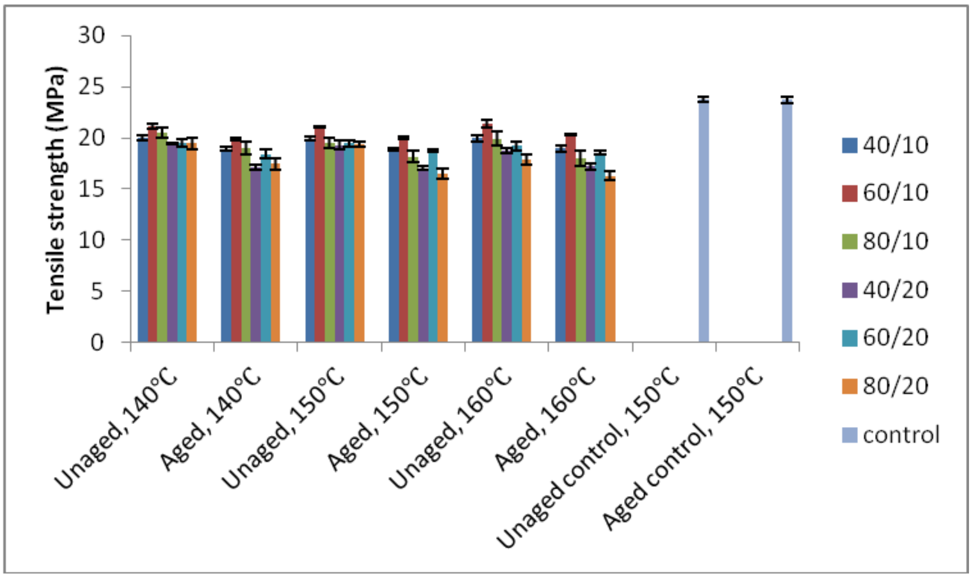

Figure 4: Tensile strength of the rubber vulcanizates before and after aging

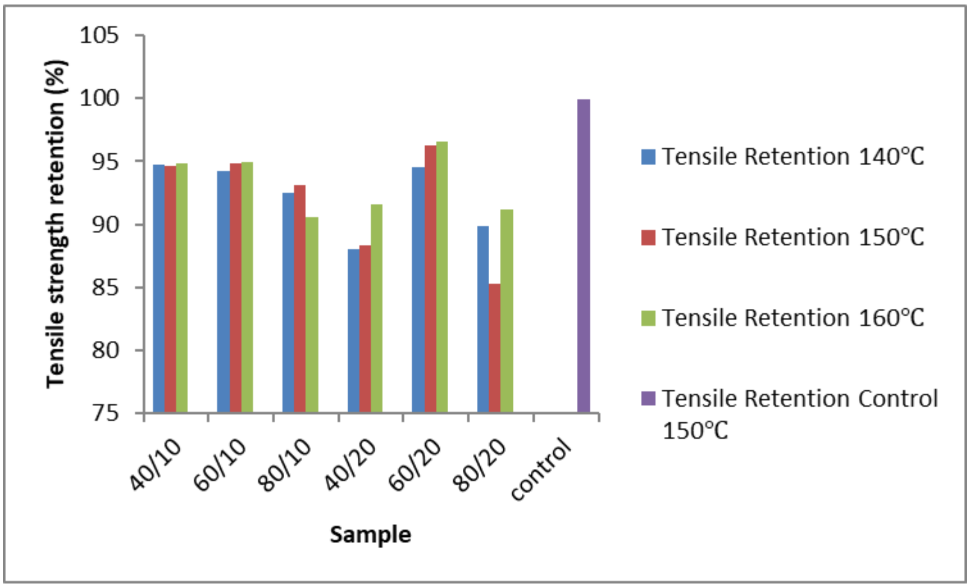

Figure 5: Tensile strength retention of samples at different mesh sizes of GTR in rubber vulcanizate cured at 140°C, 150°C and 160°C curing temperatures

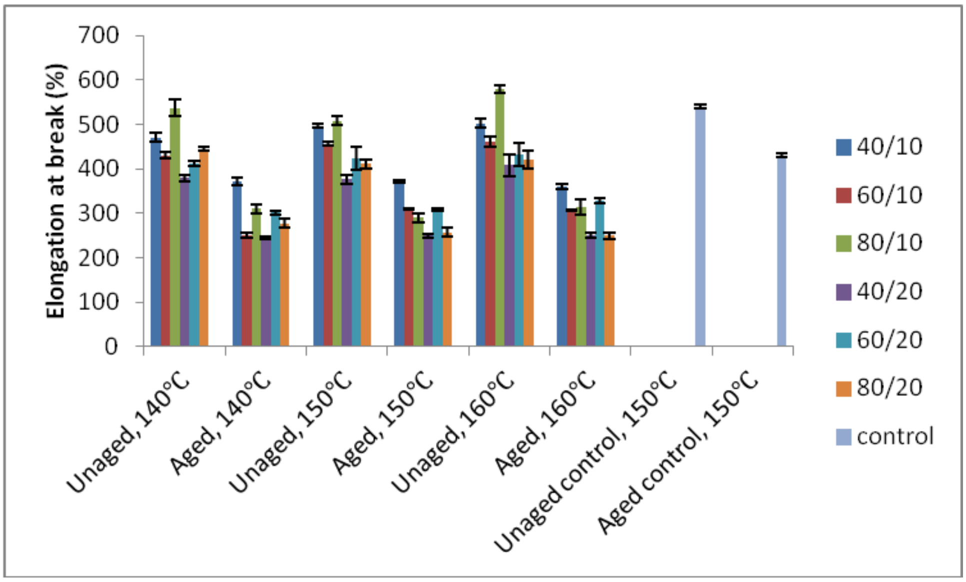

Figure 6: Elongation at break of the rubber vulcanizates before and after aging

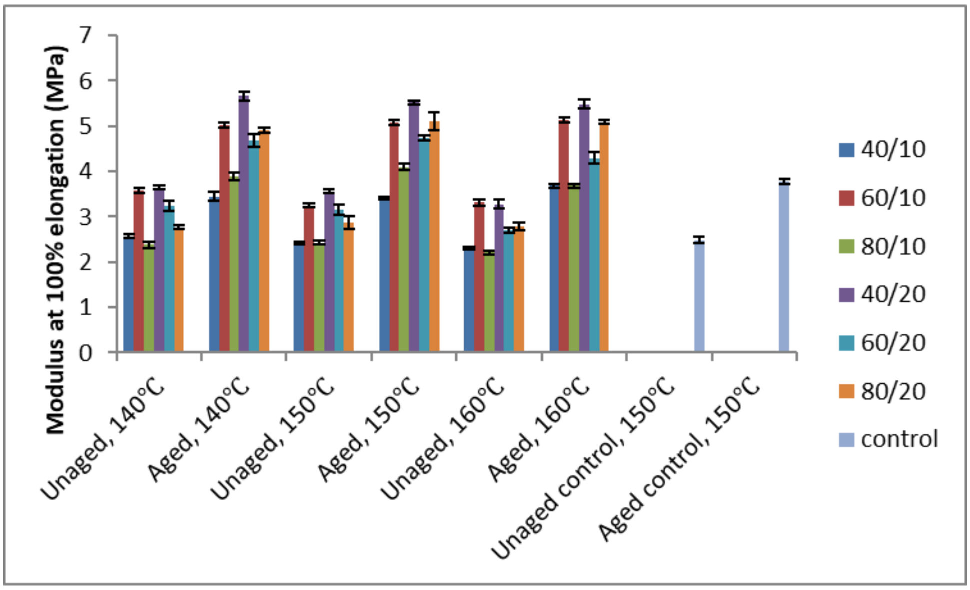

Figure 7a: Modulus at 100% elongation of the rubber vulcanizates before and after aging

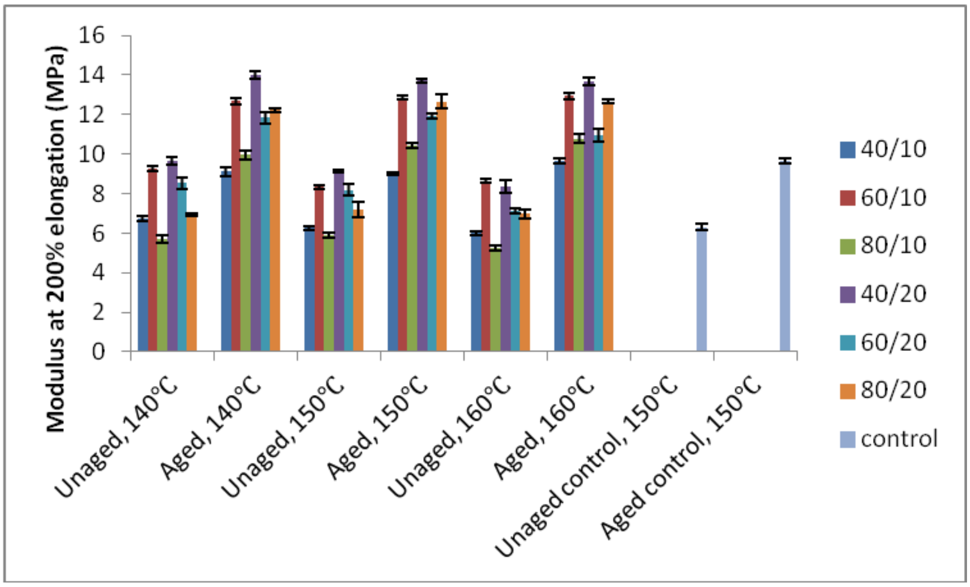

Figure 7b: Modulus at 200% elongation of the rubber vulcanizates before and after aging

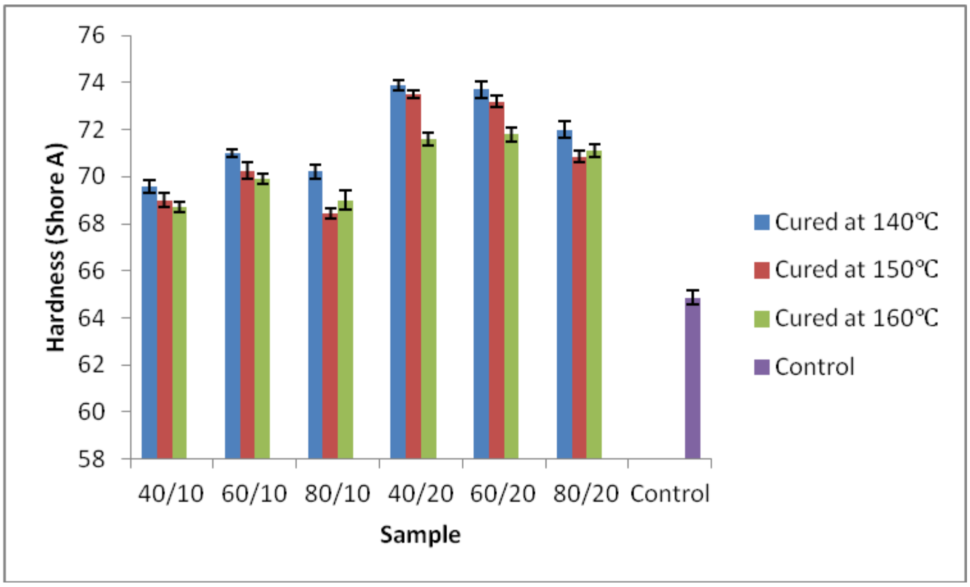

Figure 8: Hardness of samples at different mesh size of GTR in rubber vulcanizate cured at 140°C, 150°C and 160°C curing temperatures

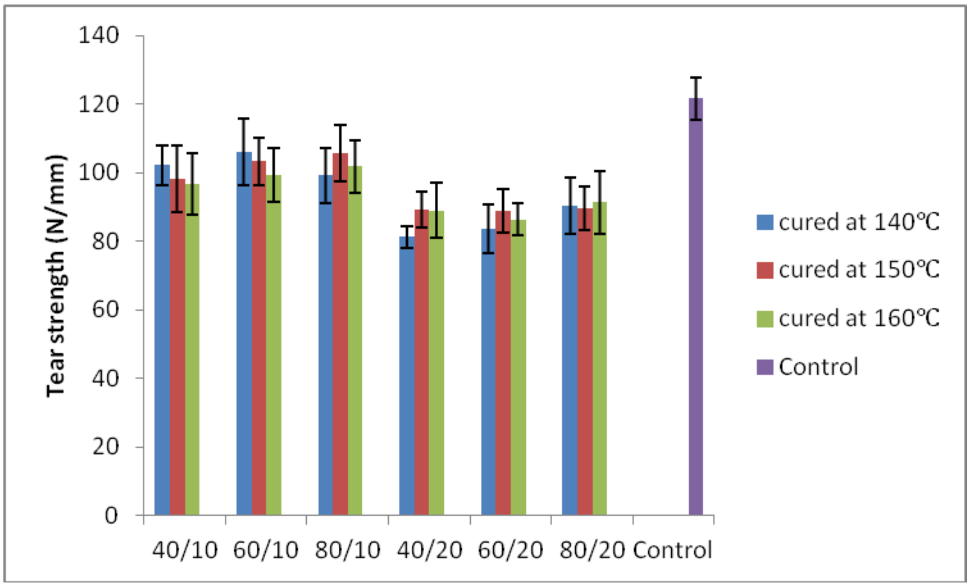

Figure 9: Tear strength of samples at different mesh size of GTR in rubber vulcanizate cured at 140°C, 150°C and 160°C curing temperatures

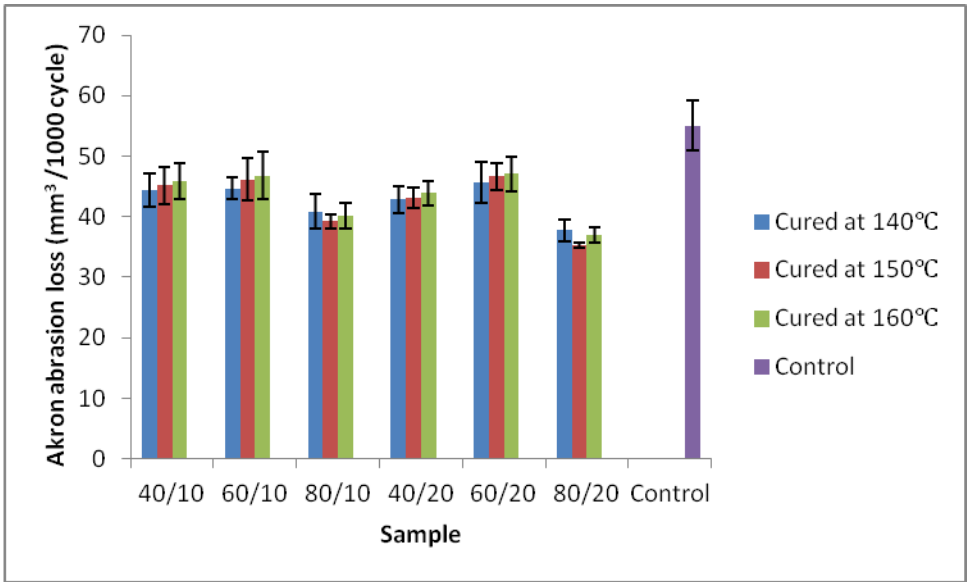

Figure 10: Akron abrasion loss of samples at different mesh size of GTR in rubber vulcanizate cured at 140°C, 150°C and 160°C curing temperatures

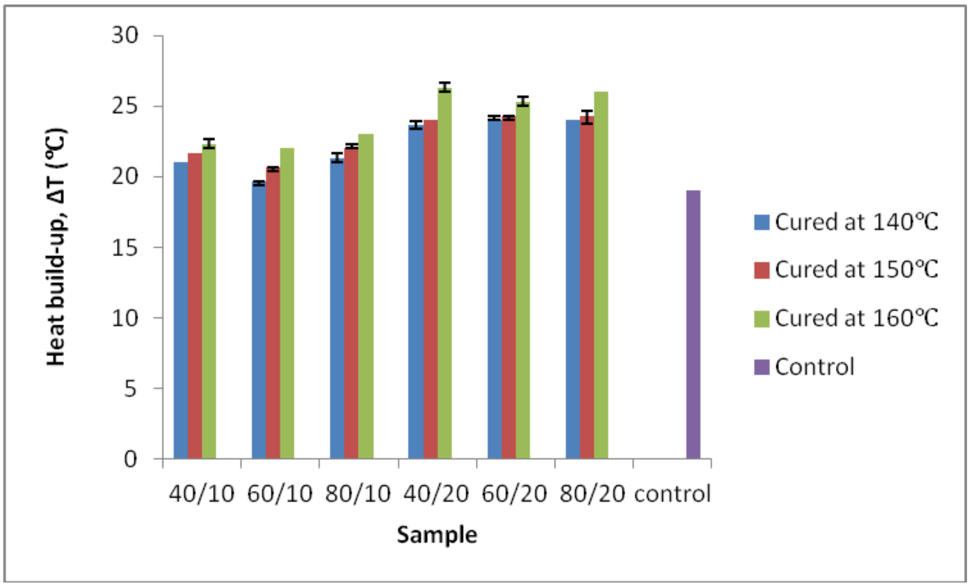

Figure 11: Heat build-up of samples at different mesh size of GTR in rubber vulcanizate cured at 140°C, 150°C and 160°C curing temperatures

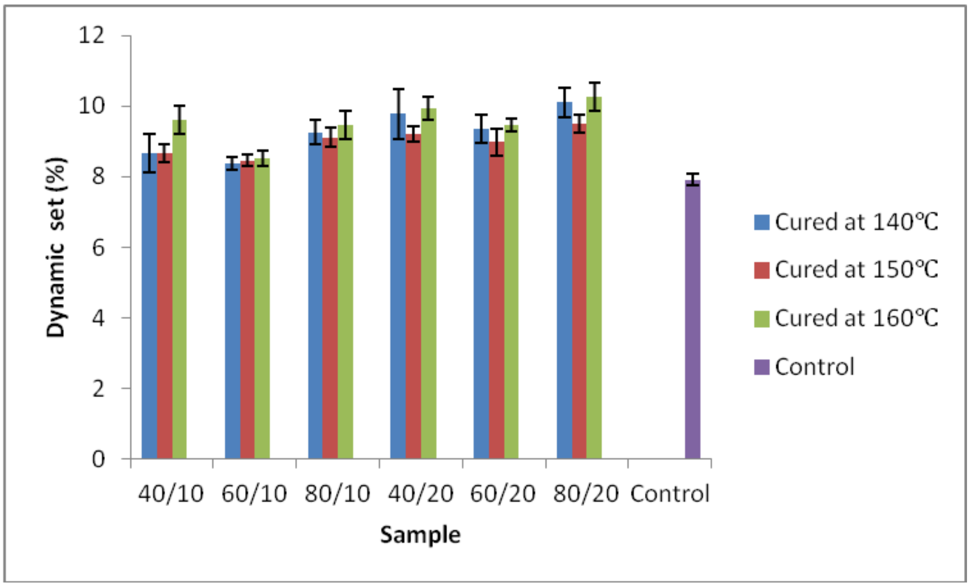

Figure 12: Dynamic set of samples at different mesh size of GTR in rubber vulcanizate cured at 140°C, 150°C and 160°C curing temperatures

4. Conclusions

The characterizations of the GTR show that it was mechanically ground at ambient temperature and composed of mainly natural rubber and styrene butadiene rubber together with the compounding additives. The rheograhs suggest that the particle sizes do not influence the cure rate much but higher cure temperature resulted in a higher cure rate and lower cure time. The viscosity of the rubber compounds was higher with a higher amount of GTR in the rubber suggesting more difficulty in processing. While the samples containing 40 mesh size and 60 mesh size GTR showed similar viscosity at 10 phr and 20 phr incorporation GTR, the samples containing 80 mesh size GTR exhibited higher viscosity. The addition of GTR in the rubber resulted in reduced mechanical properties of the vulcanizate. Increasing the curing temperature did not result in any significant improvement. In all, the samples cured at 150℃ showed better comprehensive combinations of mechanical properties compared to those cured at 140℃ and 160℃. Heat built-up increased as the cure temperature increased. It is suggested that higher cure temperature resulted in vulcanizates of weaker cross-links. While we continue to explore the possible options to improve the properties of tire tread compounds containing GTR, it is recommended that the rubber compounds are cured at 150℃ instead of at higher or lower temperatures.

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Conflict of Interest

We declare no conflict regarding the publication of the study.

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