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EFFECT OF GROUND TIRE RUBBER (GTR) PARTICLE CHARACTERISTICS ON SOLID TIRE TREAD COMPOUNDS PROPERTIES

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Abstract: Researchers are looking at the possibility of incorporating ground tire rubber (GTR) in composite materials. This study evaluates the effect of increasing the initial mixing temperature from 60°C to 100°C of solid tire tread compound filled with various loading (10 and 20 phr) and particles sizes (40–mesh, 60–mesh size and 80–mesh) of GTR on the properties of rubber compounds and vulcanizates, including cure, viscoelastic and mechanical behavior. A two–step mixing technique was employed. The results indicate that the Mooney viscosity of compounds containing 40–mesh and 60–mesh GTR increased as the mixing temperature rose. The compound containing 80 mesh size GTR showed decreased viscosity as the mixing temperature increased. Results from mechanical testing suggest improvement on the mechanical properties of the solid tire tread rubber vulcanizates containing 40–mesh size and 60–mesh size GTR with increased mixing temperature, while those of the vulcanizates containing 80–mesh size GTR reduced with increased mixing temperature.

Keywords: Tire tread, Recycling, Ground tire rubber, Mixing temperature, Properties

1. INTRODUCTION

Mixing method affects the properties of rubber vulcanizates by influencing the dispersion and distribution of materials in the uncured rubber matrix. Some of the parameters that could be controlled during mixing are rotor speed, mixing time, mixing temperature and fill factor of internal mixer. These factors affect the viscosity and shear stress of uncured rubber compound in the mixer which will in turn affect the properties of the rubber vulcanizate. It has been reported that variations in the rotor speed of internal mixer affects the properties of rubber vulcanizate [1]. Kuanusont et al. [2] established that by modifying the mixing method, the mechanical, rheological, dynamic mechanical, thermal properties as well as morphology and gel content of vinyl acetate copolymer/natural rubber thermoplastic vulcanizates were modified. It is important to evaluate the possibility of modifying the mixing methods of rubber-based composites containing ground tire rubber of different particles characteristics in order to improve their properties. The global production car tires and other rubber products in 2017 was reported to be about 4.94 million tonnes and 2.70 million tonnes respectively [3]. The generation of tire waste from end-of-life tires and other rubber goods impacts the environment adversely. It was reported that the total amount of end-of-life tire around the world as of 2019 was 30.9 million tons [4]. Presently, it is estimated that about 1 billion waste tires are generated annually and by 2030 the annual generation of waste tire will be 1.2 billion [5]. The rubber in waste tires and other rubber goods are in vulcanized state and do not degrade upon disposal because of the cross-linked polymer chains. Rubber waste is often improperly disposed of, and stockpiles of end-of-life tires pose a significant fire hazard. Landfill disposal of waste tires are not permitted in most countries. Researchers and industries are exploring the various options for the management, re-use and recycling of end-of-life tires. End-of-life tires are widely repurposed as an alternative fuel, especially in cement kilns and power plants [5]. They also play a role in civil engineering, where shredded tires are mixed into asphalt and bitumen to enhance road construction materials [5]. Progress have been made in the grinding of end-of-life tires into powder as the technology for ambient wet and dry grinding, and cryogenic grinding are now being utilized commercially by recycling companies around the globe. Researchers are exploring the possibilities of adding ground tire rubbers in concrete and polymer-based composites to reduce cost, remove waste from the environment. However, the addition of

ground tire rubber imparts negatively on the properties of the composites. It has been reported to cause decrease in strength and elongation of the vulcanizate and increase in viscosity of the rubber compound [6-9]. Researchers are investigating the possibilities of improving the properties of vulcanized rubbers containing ground tire rubber. Our research interest in this work is to investigate the effect of particles characteristics of GTR on the properties of solid tire tread compound containing GTR and mixed at two different mixing temperatures

2. MATERIALS AND METHODS

■ Materials

Ground tire rubber (GTR) particles of 40, 60, and 80 mesh sizes were procured from the Rubber Technology Research Centre, Mahidol University, Thailand. The base elastomers used were natural rubber (STR 20), sourced from L.C.E.H Bangkok Co. Ltd., and butadiene rubber (BR 150), and obtained from Thai Synthetic Rubber Co. Ltd. Additional compounding ingredients included:

- Antioxidants: N-(1,3-dimethylbutyl)-N'-phenyl-p-phenylenediamine (6PPD) from Eastman Chemical Switzerland LLC and poly(1,2-dihydro-2,2,4-trimethyl-quinoline) (TMQ) from Monflex PTE Ltd.
- Activators and fillers: Zinc oxide (ZnO) from Thai-Lysaght Co. Ltd., stearic acid from Asia Chem Co. Ltd., and carbon black (N330) from Birla Carbon Thailand.
- Plasticizers and processing aids: Aromatic oil from P.S.P. Specialties Public Co. Ltd.
- Accelerators and curatives: N-tert-butyl-2-benzothiazyl sulphonamide (TBBS) from Ningbo Actmix Rubber Chemicals Co. Ltd., and sulphur from The Siam Chemical Public Co. Ltd.
- The vulcanization formulation employed in this study follows the previously published design reported in [10].

■ Characterizations of the GTR

Samples of the GTR of various mesh sizes were mounted on a stub and sputter-coated. The morphological features of GTR particles were examined using a field emission scanning electron microscope (FE-SEM, Hitachi, Japan). Prior to imaging, the samples were mounted on metal stubs and sputter-coated with platinum using a JEC-300FC fine coater (JEOL, USA). SEM imaging was conducted at an accelerating voltage of 10 kV and a magnification of 1500 \times .

Thermogravimetric analysis (TGA) was carried out using a Netzsch TG 209 F1 Libra instrument (Germany), and the thermal degradation behavior was previously reported in [9]. The TGA profile revealed GTR composition comprising approximately 42% natural rubber, 15% synthetic rubber (SBR and BR), 5.6% carbon black, 8.6% volatile matter, and minor components.

The chemical structure of the GTR was further analyzed using a Bruker FTIR spectrometer (USA), operating in the spectral range of 4000-340 cm^{-1} at room temperature (22 °C) to confirm the presence of characteristic functional groups.

■ Mixing of the Various Samples of Tire Tread Compound

Two distinct mixing methods were employed in the preparation of the samples. The first method, referred to as Mix 1, utilized a two-step mixing procedure. Initially, a 500 ml laboratory internal mixer (Brabender Plasticoder Lab Station, Germany) was used at 60°C, with a fill factor of 0.78 and a rotor speed of 40 rpm, following the mixing sequence previously reported [10]. After the compound was discharged and allowed to cool to room temperature, it underwent a second mixing stage on a two-roll mill at 30°C for 5 minutes to ensure uniformity.

The second method, designated Mix 2, also involved two mixing stages. The first stage used the same internal mixer, fill factor, and rotor speed, but with a higher starting temperature of 100°C, and followed the same previously reported procedure [10]. The compound was removed from the mixer after 6 minutes, without the addition of curatives during this stage. In the second stage, mixing was carried out on a two-roll mill at 30°C for 6 minutes. For Mix 2, the curatives (TBBS and sulphur) were added at the third minute during this step and thoroughly blended into the compound. All rubber compounds were stored for 24 hours before undergoing testing and further processing.

■ Measurements of Mooney Viscosity and Cure Behaviour

The Mooney viscosity (ML1+4 at 100°C) of each rubber compound was measured using a Mooney viscometer (GOTECH, Taiwan), in compliance with the ISO 289-1:2015 standard [11]. Around 25 g of uncured rubber was tested at 100°C for 4 minutes, and the reported value represents the average of three specimens per compound.

Cure characteristics were evaluated using a moving die rheometer (MDR-01, CGM Technology, Thailand) at 150°C, in accordance with the ASTM D2084-2019 standard [12]. The resulting

rheograph provided data for scorch time (t_{s1}), optimum cure time (t_{c90}), maximum torque (MH), and minimum torque (ML). The cure rate index (CRI) was calculated using equation (1) [13].

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

■ Production and Testing of the Rubber Vulcanizates

Rubber sheet samples with a thickness of 2 mm were vulcanized using a hydraulic compression press (Wabash MPI, USA) at 150 °C under a pressure of 20 tons-force. The cure time for each sample corresponded to the t_{c90} value obtained from rheometric analysis. After vulcanization, the rubber samples were kept at room temperature for a minimum of 24 hours before testing [9, 14].

Tensile specimens in a dumbbell shape were prepared and categorized into aged and unaged groups. Tensile testing was carried out at room temperature (23 °C) using a universal testing machine (Instron 5566, USA) at a strain rate of 500 mm/min, in accordance with the ASTM D412-16 (2021) standard [15], with elongation monitored using an extensometer. The results were averaged from five repeated tests per sample. In order to assess aging resistance, specimens were placed in an air-circulating oven (Gotech GT-7017-EL, Taiwan) at 100 °C for 22 hours, then cooled at room temperature (23 °C) for 24 hours [16], followed by tensile testing. Aging resistance was calculated as the percentage retention of tensile strength after aging using Equation (2) [17]. Tear strength was evaluated according to ASTM D624-00 (2020) [18], at a strain rate of 500 mm/min, with five tests averaged for each sample

3. RESULTS AND DISCUSSIONS

■ Properties of the GTR

Figure 1 (a-c) shows SEM images of 40, 60, and 80 mesh GTR, revealing irregular shapes and rough surfaces, characteristic of mechanical grinding at ambient temperature [19, 20]. White stains indicate foreign materials like ZnO, stearic acid, or sulfur from the original tire compound [21].



Figure 1(a-c). SEM micrographs of the GTR used for the work

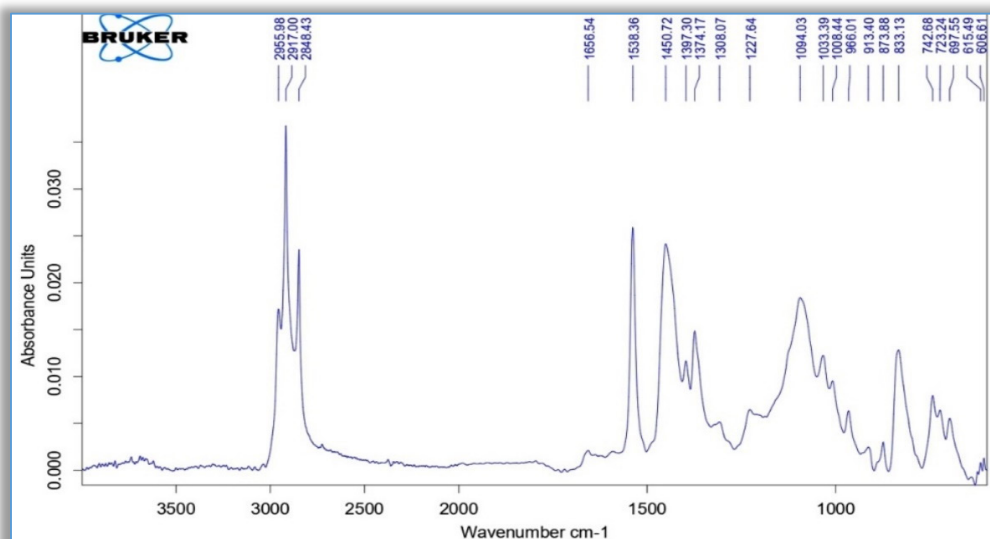


Figure 2. FTIR spectra of the GTR

The 40 mesh GTR has the least exposed surface area, with impurities sticking to the rubber. The 60 mesh GTR shows the best surface exposure, with well-dispersed impurities. In the 80 mesh GTR, finer particles result in impurities being fully separated and clustered. Figure 2 shows the FTIR spectra of the GTR. The absorption peak at 2848.43 cm^{-1} corresponds to C-H stretching from isoprene, while peaks at 2955.98 and 2917 cm^{-1} indicate C-H elongation in the aromatic styrene unit. The 1656.64 cm^{-1} peak represents C=C stretching, and 1538.36 cm^{-1} corresponds to CH₂ and

CH₃ vibrations. Peaks at 1450.72, 1374.17, and 833.13 cm⁻¹ indicate --CH₂- deformation, C-H deformation of CH₂ and =C-H, and 966.01 cm⁻¹ represents the elongation of unsaturation in the aromatic ring [22, 23, 24]. The other absorption bands are from the functional groups for the compounding additives. These absorption bands confirm GTR consists of natural rubber and styrene-butadiene rubber.

Mooney Viscosity

Table 1 presents the Mooney viscosities of the rubber compounds. As expected, GTR-containing compounds exhibited higher viscosity than the control sample due to the cross-links in GTR restricting chain mobility [25] and the additional carbon black from GTR. When the initial mixing temperature increased from 60°C to 100°C, viscosity rose in compounds with 10 and 20 phr of 40 and 60 mesh GTR but decreased in those with 80 mesh GTR, especially at 20 phr. Higher viscosity can hinder additive distribution but enhances shear stress for better dispersion. The viscosity increase at higher temperatures suggests partial cross-linking from residual sulphur and accelerator in GTR, while the decrease in finer 80 mesh GTR may result from degradation, depolymerization, and oil release, improving flexibility and reducing viscosity [26, 27]. Coarser GTR particles remain solid and swollen at high temperatures, maintaining viscosity, whereas finer particles tend to depolymerize, lowering viscosity [27].

Table 1. Mooney viscosity of the rubber compounds

Sample	Control	40/10		60/10		80/10		40/20		60/20		80/20	
		Mix 1	Mix 2	Mix 1	Mix 2	Mix 1	Mix 2	Mix 1	Mix 2	Mix 1	Mix 2	Mix 1	Mix 2
MU	58.87	63.99	78.52	65.71	72.90	77.14	78.14	69.47	86.68	69.83	81.69	86.51	79.25
Std. dev.	0.26	0.43	0.26	0.53	0.10	0.42	0.62	0.64	0.30	0.22	0.23	0.89	0.30

Cure Behavior

Figure 3(a-b) shows the cure curves for the rubber compounds with 10 and 20 phr GTR, where “A” represents “mix 1” and “B” represents “mix 2.” Cure parameters are in Table 2.

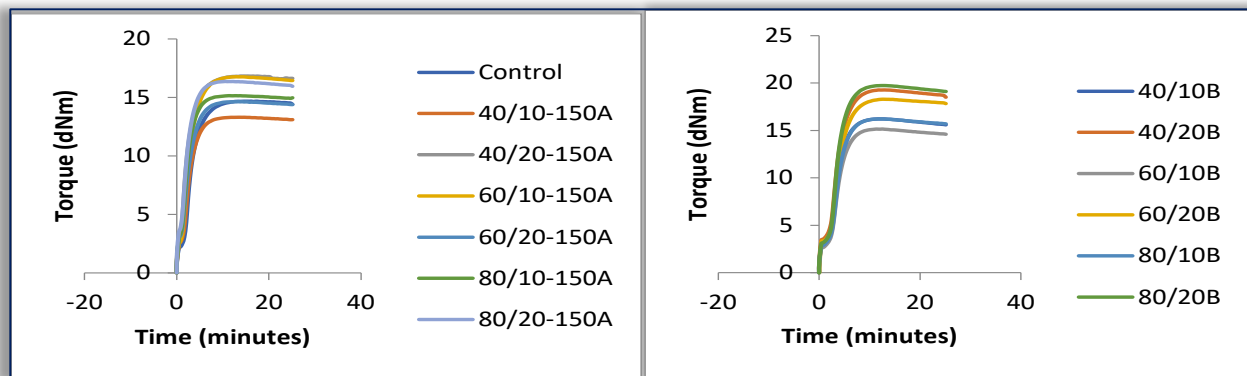


Figure 3. (a). Cure curves of Mix 1 samples; (b). Cure curves of Mix 2 samples

Table 2. Cure parameters from MDR tests for Mix 1 and Mix 2

Cure Parameters at 150°C for Mix 1 (mixing at 60°C and 40 rpm)							
Parameter	Control	40/10A	40/20A	60/10A	60/20A	80/10A	80/20A
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 ±.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 150°C for Mix 2 (mixing at 100°C and 40 rpm)							
	40/10B	40/20B	60/10B	60/20B	80/10B	80/20B	
Maximum torque, M _H , (dNm)	16.23 ±0.16	19.29 ±0.33	15.16 ±0.24	18.32 ±0.34	16.21 ±0.30	19.72 ±0.05	
Minimum torque, M _L , (dNm)	2.96 ±0.01	3.54 ±0.10	2.76 ±0.04	3.34 ±0.05	2.89 ±0.25	3.19 ±0.04	
M _H – M _L , (dNm)	13.26 ±0.15	15.74 ±0.24	12.49 ±0.22	14.95 ±0.27	13.32 ±0.28	16.53 (0.08)	
Scorch time, ts ₁ , (min)	2.11 ±0.02	2.06 ±0.04	2.21 ±0.01	2.22 ±0.03	2.18 ±0.01	2.10 ±0.01	
Optimal cure time, tc ₉₀ , (min)	6.36 ±0.14	6.68 ±0.12	6.44 ±0.12	6.88 ±0.01	6.41 ±0.08	6.44 ±0.14	
CRI (min ⁻¹)	23.54 ±0.73	23.76 ±0.52	23.69 ±0.65	21.41 ±0.62	23.65 ±0.42	23.02 ±0.78	

Minimum torque (M_L) increased with higher mixing temperature for the samples containing 40 and 60 mesh GTR but decreased for those containing 80 mesh GTR, aligning with Mooney viscosity trends. Higher M_L indicates increased viscosity and shear stress. Both scorch time (t_{s1}) and cure time (t_{c90}) increased with mixing temperature. Shorter t_{s1} and t_{c90} of the samples mixed at lower temperatures may have resulted from shear heating due to higher bulk viscosity of the samples mixed at lower temperature, increasing thermal history. Samples mixed at 60°C had lower t_{c90} and higher cure rate, likely due to accelerator migration from GTR to the rubber matrix [28]. During mixing at higher temperatures, the accelerators and sulfur in GTR may have been consumed in cross-linking reactions, hence, slowing the cure rate and increasing the t_{c90} of the samples mixed at 100°C. Samples mixed at initial temperatures of 100°C showed t_{c90} like the control as shown in Table 5, suggesting only added sulfur and accelerator (added after high temperature mixing at lower temperature) contributed to vulcanization.

■ Properties of the Vulcanizate

Figure 4(a) and 4(b) show the tensile strength of the samples before and after thermal aging, respectively, while Figure 5 illustrates the tensile strength retention. As seen in Figure 4(a), increasing the initial mixing temperature from 60°C to 100°C resulted in a slight improvement in tensile strength. Among all the compounds, the sample containing 20 phr of 60 mesh size GTR showed the highest improvement (about 8.32%) in tensile strength. The SEM image of the 60-mesh size GTR shown in Figure 1b indicates higher surface area of the GTR compared with others, hence the improvement in the tensile strength of the sample containing 60 mesh size GTR due to better interaction of the GTR, filler and the rubber matrix. However, the compounds mixed at higher temperatures exhibited slightly reduced aging resistance, as shown in Figure 5. This reduction may be attributed to partial degradation of the GTR caused by the elevated mixing temperature. It is an indication that rubber compounds containing GTR should not be mixed at temperatures much higher than 100°C. The elongation at break shown in Figure 6 followed the same trend as tensile strength and the samples containing 60 mesh size GTR showed the highest improvement due to mixing at higher temperature.

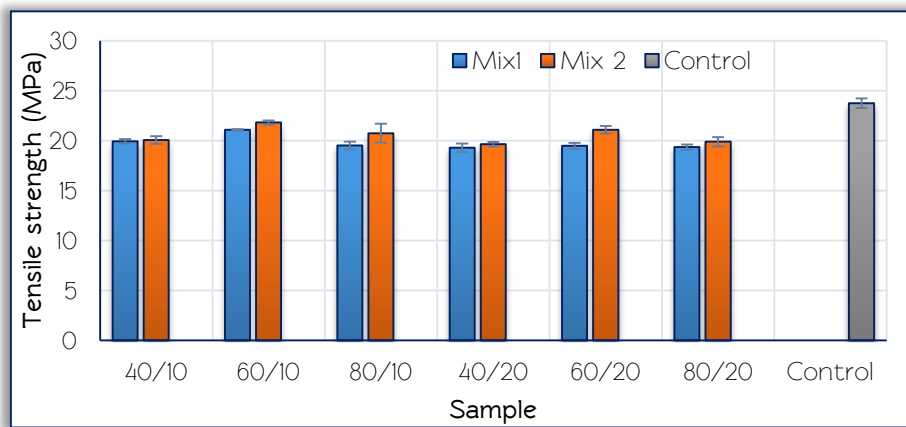


Figure 4(a). Tensile strength of the vulcanizates before ageing

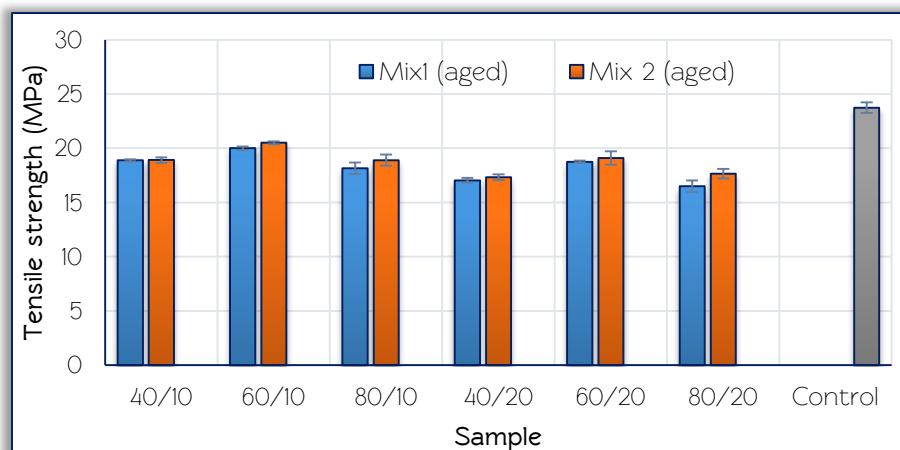


Figure 4(b) Tensile strength of the vulcanizates after ageing

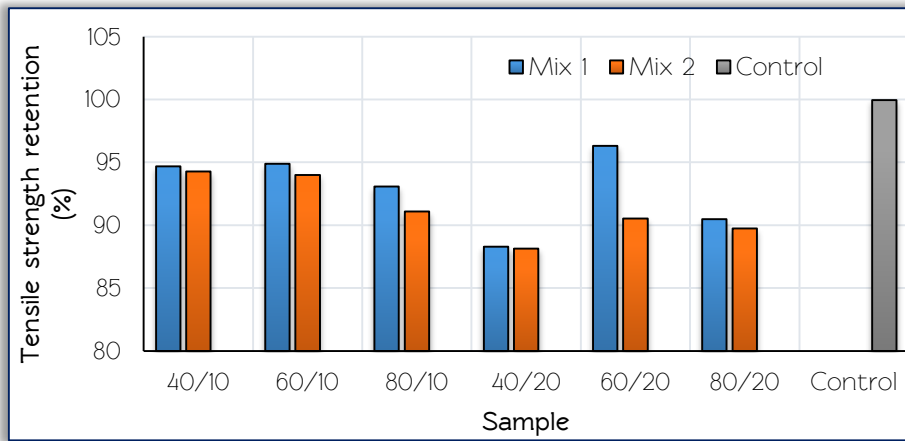


Figure 5. Tensile retention

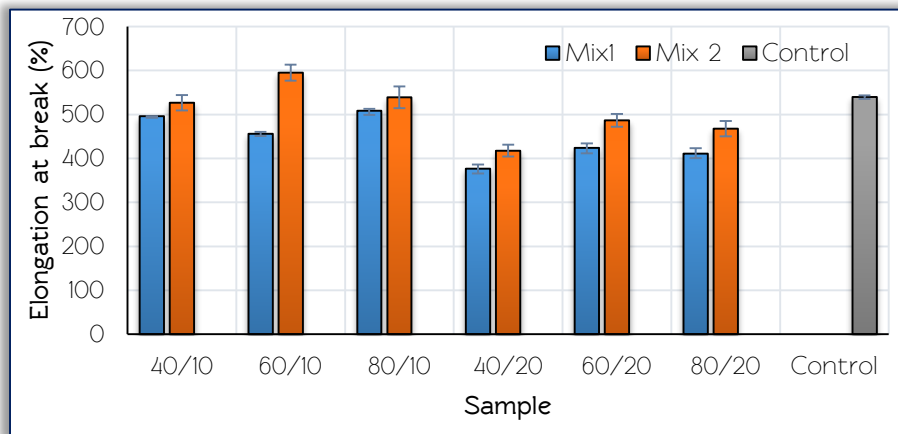


Figure 6. Elongation at break

The moduli at 300% elongation are shown in Figure 7. The increase in GTR loading means the increased CB loading acting as reinforcing filler. Thus, the modulus is increased as we increased GTR loading. The effect of higher elongation showed in the slightly lower moduli of the compounds containing 40 mesh size and 60 mesh size GTR while those containing 80 mesh size GTR exhibited slightly higher modulus because and this might be due to poor dispersion of the GTR agglomerates. The trend is more visible in the tear strength shown in Figure 8. The decrease in tear strength at high GTR loading might be due to the poor dispersion of GTR acting as defects. Also, poor interfacial adhesion between matrix and GTR surface might create flaws. The effect of improved filler-rubber interaction resulting from mixing at higher temperature can be seen in the improved tear strength of the samples containing 40 mesh size and 60 mesh size GTR, while the samples containing 80 mesh size GTR showed reduced tear strength compared to those mixed at lower temperature.

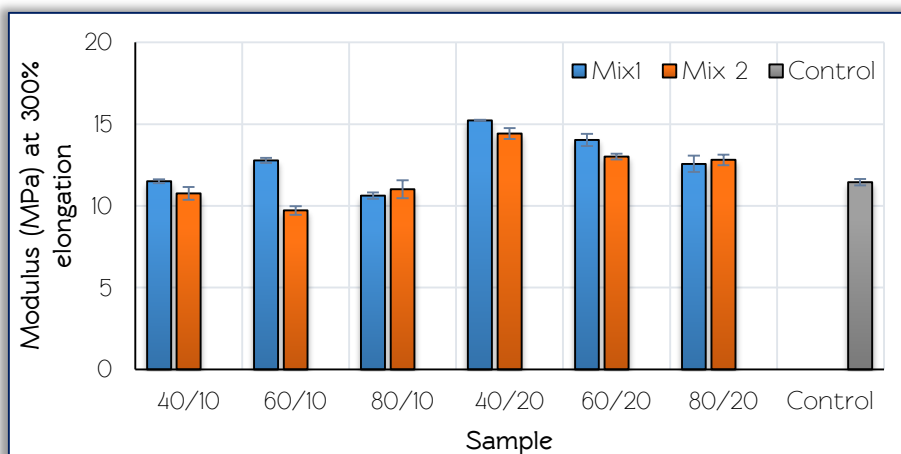


Figure 7. Modulus at 300% elongation

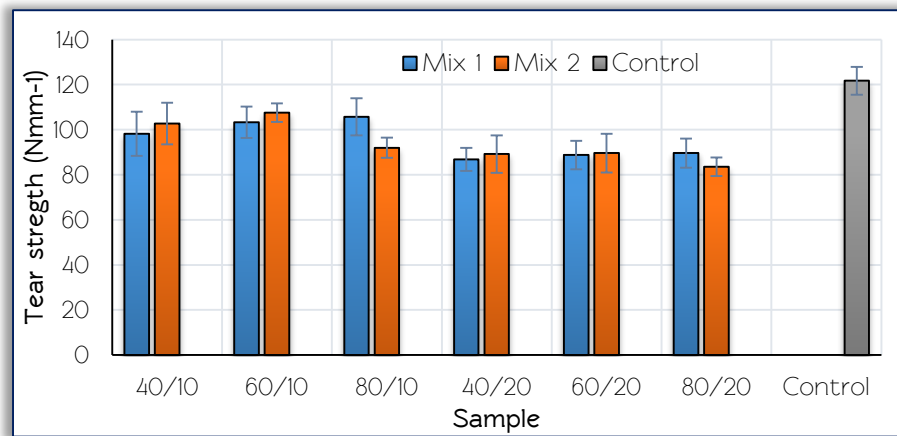


Figure 8. Tear strength of the vulcanizates

4. CONCLUSIONS

In this work, effects of GTR particles characteristics on the properties of tire tread compound containing GTR and mixed at initial temperatures of 60°C and 100°C were reported. While the increase in mixing temperature resulted in increased viscosity of the samples containing 40 mesh size and 60 mesh GTR, the opposite is the case for the samples containing 80 mesh size GTR. The results show that the samples containing 40 mesh size and 60 mesh GTR and mixed at higher temperature exhibited improved tensile strength, elongation and tear strength. This shows correlation between the rheological and mechanical properties.

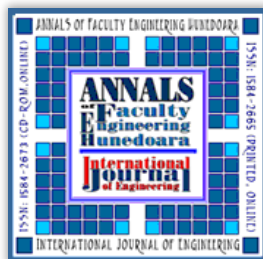
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