

ORIGINAL RESEARCH ARTICLE

The Effect of Ground Rubber Tire (GRT) Filler Particle Size on the Morphological, Thermal and Mechanical Properties of Waste High-Density Polyethylene (rHDPE)

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ABSTRACT

This research investigates morphological, thermal and mechanical properties of ground rubber tire (GRT) filled waste high-density polyethylene (rHDPE), showing the effect of variable particle sizes. Samples were prepared using a two-roll mill machine, followed by compression moulding. Different blends of variable GRT mean particle sizes and a constant concentration of 150 wt % were prepared (150 μm , 212 μm , 300 μm , and 425 μm). The properties of the composites were analysed using field emission scanning electron microscopy (FESEM), dynamic mechanical analysis (DMA), tensile and flexural tests, respectively. The SEM result shows that Ground Rubber is not evenly distributed throughout the matrix (HDPE), resulting in a composite with low mechanical strength and a non-homogeneous morphology. This is due to the reduction in defects observed upon increasing the filler size to 212 μm , leading to improved interfacial adhesion with the HDPE matrix. Further increase to 300 μm gave a more homogenous micrograph, but the addition of GTR particles of 425 μm led to a rough surface, and thermal behaviour at different sizes of the GGTR HDPE matrix, modulus decreases, attributed to the material softening caused by the soft rubber particles which result in lower rigidity except for sample made with 150 μm . Incorporation of 300 μm particles GTR results in higher rigidity, which similarly gave more homogenous micrograph with uniform dispersion, result also shows that, increasing the GTR particles causes decline in the flexural stress and modulus, having similar trend with the tensile modulus elongation at break also shows a decline with addition of the different particle sizes of the filler, with the exception of sample 300 μm .

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INTRODUCTION

With the increase in human population, manufacturing industries globally have increased their use of plastic for various reasons. Over the years, there has been a tremendous increase in the use of vehicles, largely due to transportation, which raises tire production. This worldwide scrap tire occupies huge amounts of landfill area globally. In other countries, including Nigeria, a scrap tire site records over 100 million tires, weighing approximately 850,000 tons of scrap thrown away on the land. Furthermore, more than two-thirds of the billions of tires used worldwide every year are discarded in the permitted or unauthorised waste areas (Simon and Varga, 2023).

Stacked waste tires attract disease-carrying rodents, making the land useless, and release a lot of harmful substances to the atmosphere as they decay over time. Waste tire scraps catch fire because of their size and the

combustibility of tire materials. Most of the waste tire fires could last up to several months, as millions of tires piled together. There must be a proper way to get rid of these millions of waste tires and develop a good technique to overcome this global issue (Zedler Burger, Wang and Formela, 2022).

The polymer waste represents a considerable portion of all waste materials. With the quick improvement of the vehicle business, waste tires contribute to the expanded polymer waste volume. A polymer mix like elastic thermoplastic mixes when elastic-rich, creates delicate thermoplastic elastomers, while plastic-rich mixes produce elastic-hardened thermoplastics. Rubber-hardened thermoplastics that display adaptable and high-elasticity properties will be utilised as economical alternatives to standard plastic materials (Fazli and Rodrigue, 2024).

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Composite materials are widely used in various industries such as construction, automotive, aerospace and packaging due to their desirable properties such as high strength, light-weight, and cost-effectiveness. Polymer composites, in particular, have gained significant attention as they offer the potential of using recycled materials, which are cost-effective and eco-friendly. High-density polyethylene (HDPE) is a popular polymer matrix for composite materials due to its excellent mechanical and thermal properties (Egodage and Walpalage, 2009).

Ground rubber, often in the form of crumb rubber or ground tire rubber (GTR) and high-density polyethylene (HDPE) are two materials that are sometimes combined to create composite materials or to modify other materials. They are combined for various applications, including road construction, concrete mixing, and even to create new rubber-like materials. The quantity of GTR is increased within the polymer composition, and its mechanical properties decrease. Better substrates are introduced to extend the interference between the polymer and GTR to induce better mechanical properties.

The thermal and morphological properties of a composite material play a pivotal role in determining its suitability for various applications. Thermal properties such as thermal conductivity, thermal diffusivity and specific heat capacity are important parameter to consider as they affect the heat transfer characteristic of the material also the morphological properties such as surface morphology, microstructure and particle size distribution are also important as they determine the mechanical and physical properties of the composite material (Rajalingam, and Baker, 2021).

Lukasz Piszczyk *et al.* (2017) researched the likelihood of using polyurethane (PU) with GTR to supply compression-absorbing buoys and floating trays. Also stated is that incorporating GTR into the polyurethane increases the static mechanical properties and thermal stability. They studied the acoustic absorption of the blend of PU/GTR, which significantly increases the insulation properties of the blended foam (Rahmani, Adamian, Hosseini-Sianaki, 2022).

Marín-Genescà *et al.* (2020) reported that due to the lack of adhesive between two phases, the blend's mechanical characteristics are weakened. It is because of GTR's large particle size, crosslinking and surface properties which resist the molecular entanglement with polymers. Numerous efforts have been made to make them homogeneous and to produce a composition with better properties (Marín-Genescà *et al.*, 2020).

Ali Fazil and Denis Rodrigue (2024) reported that larger GTR particles increase the possibilities of larger voids, and tiny particles of GTR increase small cracks. It introduced the 20% by weight GTR (200 μm), which has decreased the endurance by 25% (from 24% to 18%). But on acid treatment (sulfonitric), the durability decreased by 13%. The acid treatment increases the

surface roughness by which the interaction of GTR with the matrix phase increases (Fazli and Rodrigue 2024).

The problem of air pollution caused by wasted tires, with chemicals released and heavy metals leached into the environment, has become an environmental issue. These chemicals are carcinogenic and mutagenic, which cause cancer and gene mutations. The leaching of ground tires also affects the soil around the old tires; if the tires are moved from that place, the soil is still a toxin. So the toxins in the soil are transferred to the groundwater, and the water becomes harmful for living things when they come in contact with water (Favakeh, Bazgir, Karbasi, 2023). The hollow portion of the tires will contain rainwater, and if the tires are left open to the atmosphere and the tires are undisturbed for a long period. It will create a breeding ground for mosquitoes. Additionally, the combustibility of tires is a problem. If the tires are burned, the chemicals are released into the surrounding environment, becoming harmful to human health. The landfill problem is associated with the rubber from ground tires (Favakeh, Bazgir, Karbasi, 2023).

The aim of this research is to study the effect of ground rubber tire (GRT) filler particle size on the morphological, thermal and mechanical properties of waste high-density polyethylene (rHDPE) using GTR as a filler in rHDPE to observe its mechanical properties. GTR has outstanding impact strength with elastomer when the composition of rHDPE/GTR is set. This composition improves impact strength, percentage elongation, and the Flexural strength of its product.

MATERIALS AND METHODS

Materials

The polymer used in this project was sourced locally around Ahmadu Bello University, Samaru Campus, Zaria, Kaduna Nigeria. The polymers used were mainly HDPE waste bottles, which were rinsed and dried to remove contaminants. The cleaned HDPE bottles were then crushed using sieve mesh size as the size of filler particle. The sample was stored in a container prior to compounding.

Methodology

Compounding and compression

The composite was compounded using a two-roll mill machine (CPS183), which consists of two cylindrical oppositely rotating rollers with adjustable knobs, which help to increase or decrease the nip of the rollers at a rotational speed and control temperature.

The composites were prepared using the ratios shown in Table 1.

The matrix (HDPE) was melted in the two rolls machine at a temperature of 180 °C, and then the filler was added

with the aid of the rotating heated rolls and mixed together for about 20 minutes. As the materials melt the nip was reduced to make the melt form a band. Then a knife-like

object was used to remove the band from the rolls. This was done on a hydraulic press machine, which makes use of hydraulic pressure.

Table 1: Matrix and reinforcements composition

Samples	Filler size (μ)	Filler concentration (μm)/(g)	HDPE content (g)
A	Control	–	150
B	150	150/15	135
C	212	212/15	135
D	300	300/15	135
E	425	425/15	135

After blending, the band formed was fitted into a mold plate of 240 mm × 120 mm × 4 mm) and wrapped with aluminium foil paper to which silicon oil was applied for easy removal of the sample after cooling. The mold was then mounted on the bottom plate with an oversized pressure valve tightened and pressure built-up using the handle, the top and bottom plates of hydraulic press was pressed against each other with the sample in-between the plates under pressure of 10 MPa. The sample was pressed for about 15 mins at a temperature of 220 °C, and then the presser was removed and placed for cooling. After cooling, the samples were removed from the aluminium foil paper and cut into different required dimensions for mechanical, thermal and morphological analysis.

2.3 Characterisation

The following tests were carried out on the composites

Morphological Analysis

The ground rubber-filled HDPE composite materials with varying rubber particle size content were studied using a field emission scanning electron microscope (FESEM, Joel MT, model JSM-7600F) to assess the interfacial adhesion and smooth cross section of all the specimens.

Thermal Analysis

The thermal behaviour of the composite material was evaluated using Dynamic Mechanical Analysis (DMA), which applies a sinusoidal force and measures the sample's response at a given temperature. The samples were studied at: Frequency/ frequencies: 2 Hz, 5 Hz and 10 Hz, Amplitude: 60 μm, Heating rate: 6 k/min and End temperature: 120 °C.

Mechanical Test

Tensile Strength

The tensile strength of the composite samples depends on the strength of the materials, length of the material and the material matrix interaction. This test was used to determine the tensile modulus, percentage elongation and tensile strength of the materials. The samples were cut into dumbbell shape according to the ASTM D638-98 standard. The required dimension of each sample used was 100m×30mm×3mm. The test was carried out using a Universal Materials Testing Machine (Model, TM2101-T7 10KN). The machine provides a mechanism for applying different measurable loads to the sample with a device to measure the change in the length of the sample.

The sample was attached to the machine through a clamp grip. The actual procedure is to apply the stretching load to the sample starting from a zero value to an ultimate value when the sample fractures.

$$\text{Tensile strength} = \frac{P}{A} \dots\dots\dots (1)$$

Where;

P = Breaking load in (N),

A= Cross-sectional area of sample in (mm²)

Tensile strain is the ratio of the extension to the original length. It has no unit.

$$\text{Tensile strain } (\epsilon) = \frac{\text{Extended length}}{\text{Original length}} = \frac{\Delta l}{l} \dots\dots (2)$$

Percentage elongation (elongation at break) is the ratio of extension of the original length multiplied by 100.

$$\text{Elongation at break } (\%) = \frac{\Delta l}{l} \times 100 \dots\dots\dots (3)$$

Tensile (Young) modulus is the measure of the ability of a material to withstand changes in length when under lengthwise tension or compression. It is also equal to the longitudinal stress divided by strain.

$$\text{Young modulus } (\gamma) = \frac{\text{Stress}}{\text{Strain}} \dots\dots\dots (4)$$

Flexural Analysis

The test was carried out using the Enerpac Universal Testing Machine (100KV) and measured under a three-point bending approach according to ASTM D790. The samples were cut into rectangular bars with the dimensions of 60mm × 30mm × 4mm. The was applied along a sharp line perpendicular to the length of the bar and at a point midway along the gauge length of the support. The load at the point of fracture was recorded from which the cross-breaking strength was determined by using standard formulae. The flexural strength and modulus were calculated using the following equation;

$$\text{Flexural strength} = \frac{3FL}{2bd^2} \dots\dots\dots (5)$$

$$\text{Flexural modulus} = \frac{FL^3}{4bd^3 D} \dots\dots\dots (6)$$

Where;

F = force (N), L = span length (mm), b =width (mm), d = thickness(mm), D= deflection (mm)

RESULTS AND DISCUSSION

3.1 Morphological Characterization

SEM micrograph was used to determine the effect of different particle sizes of Ground Rubber on the developed HDPE composite, as illustrated in Figure 1(a) above. The Ground Rubber was poorly dispersed within the matrix (HDPE), which might result in improper stress transfer along the interface and formation of cracks. Hence, this might limit the interfacial adhesion within the

developed composite, resulting in a reduction in mechanical strength. Increasing the GTR particle sizes shown in Figure 1(b) led to the agglomeration of weakly dispersed clusters and heterogenous morphology promoting void formation, which was attributed to incompatibility and high interfacial tension between the matrix and the filler. This led to interfacial voids, GTR pull-out from the HDPE matrix (Fazeli, Keley and Biazar, 2018). Incompatibility in the sample due to the poor interfacial adhesion might cause catastrophic failure.

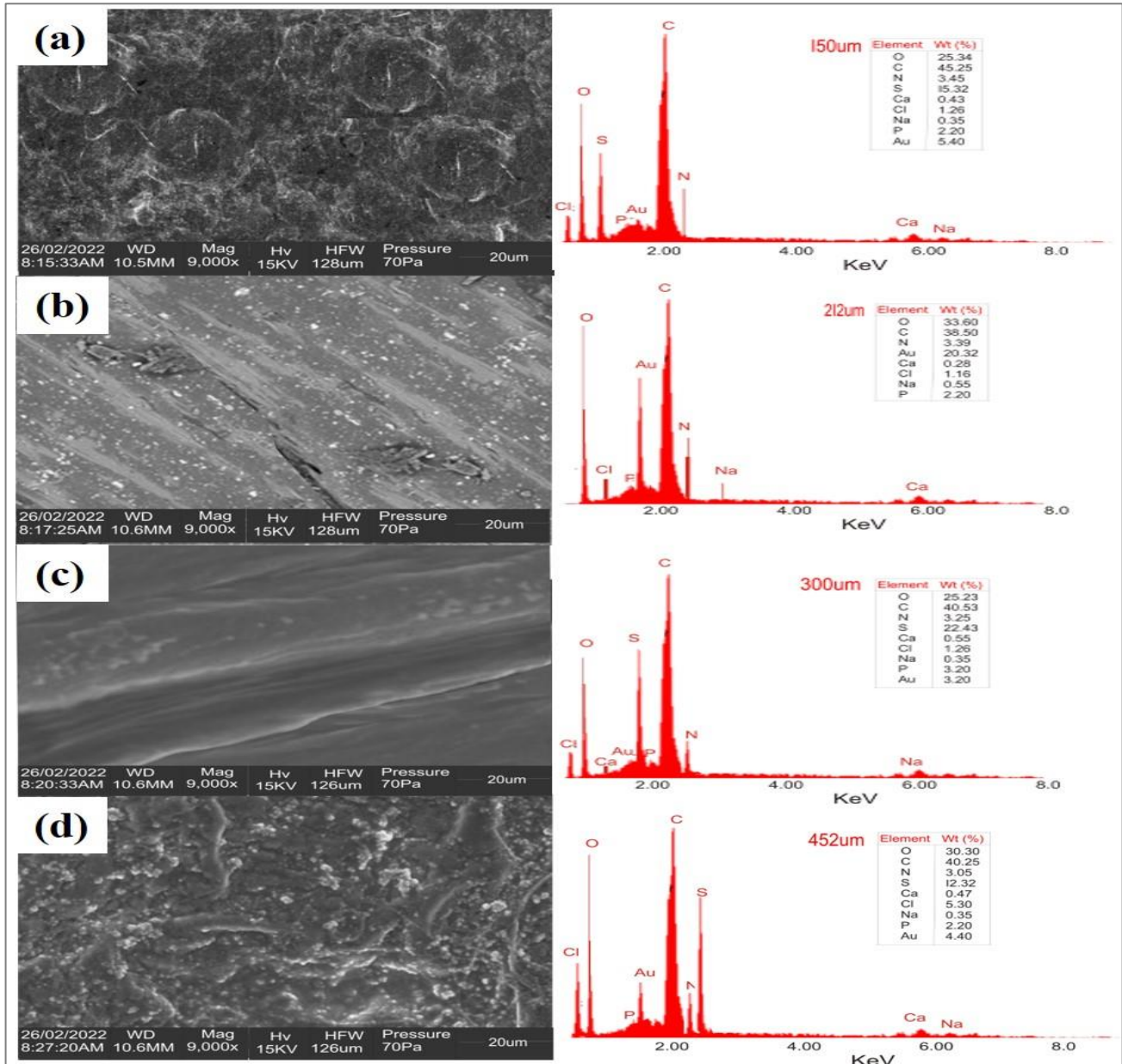


Figure 1: FESEM image of developed HDPE-filled Ground Rubber (GTR) at (a) 150µm, (b) 212µm, (c) 300µm, (d) 425µm

However, increasing the filler size from 150 µm to 212 µm improves the interfacial adhesion with the HDPE matrix, leading to a more homogeneous morphology and a reduction in defects like cracks, voids, agglomeration, and cavities. Further increase in the filler concentration (from 212µm to 300µm) gave a more homogeneous micrograph and a rough fractured surface, which suggests a possible effect of proper blend and uniform distribution of the filler. Proper blend suggests better interaction and proper

dispersion of the GTR fillers in the matrix. Generally, an improved interfacial stress transfer is expected due to the difference in morphology. However, further addition of GTR particles up to 425 µm led to a rough surface, which causes interfacial gaps between the matrix and the filler. Comparing the surfaces, no particle agglomeration was seen in Figure 1(c) with fewer voids. Therefore, it is expected that the developed composite with fewer voids might give better mechanical properties due to more

effective stress transfer and better interfacial quality (Fazli *et. al.*, 2022).

Thermal Characterisation

The stiffness of the polymer blends with respect to interfacial bonding and phase morphology can be understood from the storage modulus curves (Ali and Rodrigue, 2021). The maximum energy stored by the material shows how rigid the material could be, as illustrated in Figure 2, which shows the effect of different particle sizes of GRT on the HDPE/GRT blends. The control, which is 150 g of pure HDPE, had a higher storage modulus which is as a result of the semi-crystalline

nature of the HDPE structure (Ali and Rodrigue, 2021), which also yields high stiffness. As shown in Figure 2 below, as the different particle sizes of the GRT filler were introduced into the rigid HDPE matrix, the storage modulus decreases except for the sample made with 150 μm , attributed to the material softening caused by the soft rubber particles, which result in lower rigidity. Hence, incorporation of 300 μm particles of GTR results in higher rigidity, ascribed to better stress transfer between the phases as a result of better interaction between the matrix and the GTR. As seen in Figure 3, the sample with 300 μm gave a proper blend which reduces the interfacial tension between the matrix and the GTR, causing a higher storage modulus and better interfacial interaction

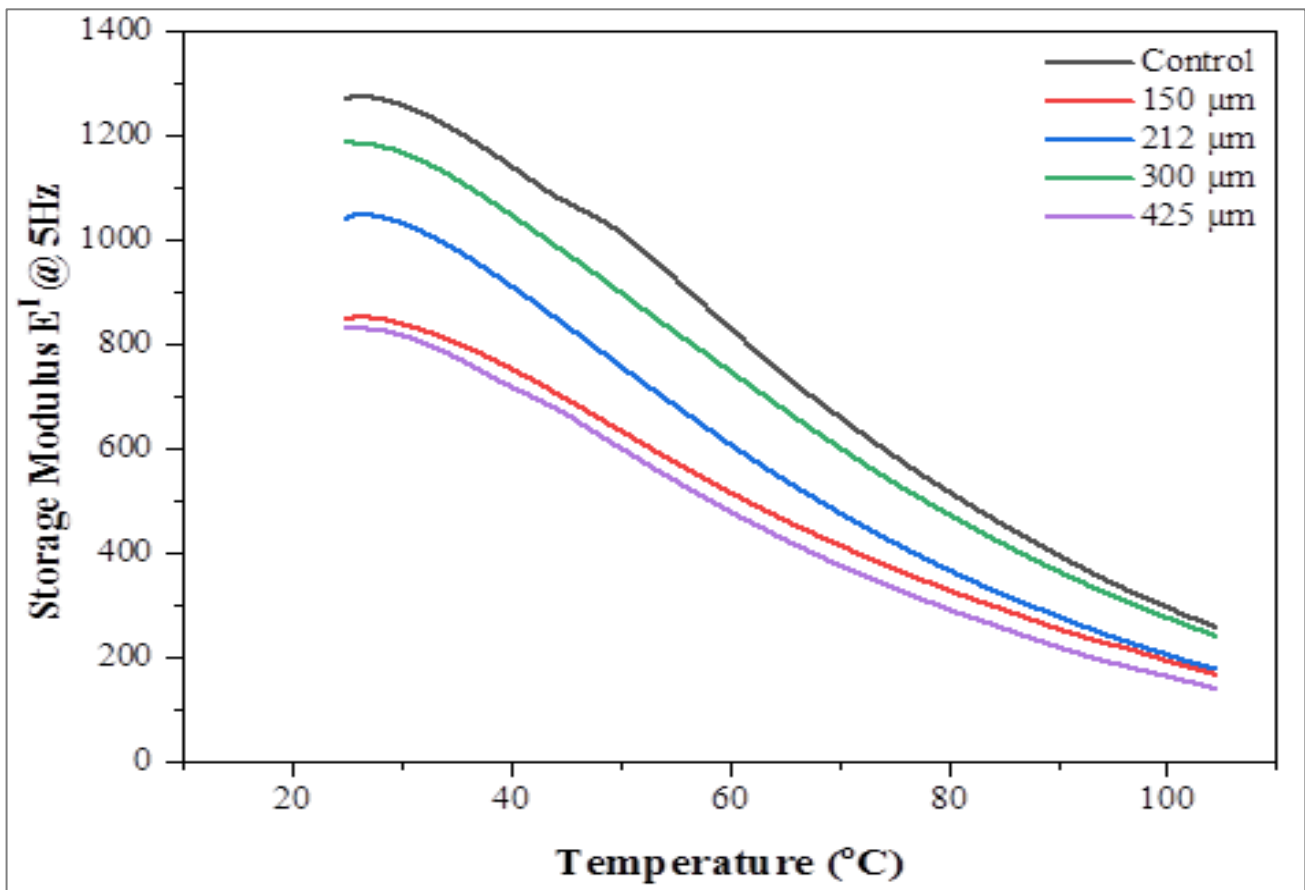


Figure 2: The storage modulus of the unreinforced and reinforced GTR/HDPE polymer composite as a function of temperature.

The tangent ($\tan \delta$) of the developed samples with variation in the GTR particle is illustrated in Figure 3. Generally, it is expected that damping is affected by friction resulting from the sliding effect between the filler and matrix and also the quality of the blend interface (Zhao *et.al.*, 2019). As expected, the increase in the particle size of GTR particles led to an increase in the tangent of delta since the GTR particles enhance the viscoelastic property of the composite. Also, the rise in the loss modulus is attributed to the complex crosslink density and the presence of carbon black, which reduces or prevents mobility within the polymer chain. Low peak intensity is observed for reinforced samples, and this indicates improved compound elasticity. Hence, lower energy is

required for molecular chain motion as the samples move close to the rubber state from the glassy state (Ali and Rodrigue, 2021)

Mechanical Analysis

Tensile Test

The inclusion of GTR particles in the matrix causes a reduction in the tensile properties attributed to the constraint in the stress transfer and the weak interfacial adhesion between the filler and the matrix. Figure 4 shows the tensile strength and tensile modulus of the unreinforced and reinforced GTR/HDPE polymer composite. In Figure 4, the addition of GTR particles reduced the tensile strength of the pure HDPE matrix. For instance, when 150 μm and 212 μm of GTR particles

were incorporated into the matrix, a decline was observed when the filler was added. The decline in the tensile strength was by 28% and 31% respectively. The inclusion of 212 μm particles of GTR) causes a higher reduction in the tensile strength. This could be attributed to more agglomeration, as seen in Figure 4 and weak interfacial adhesion between the matrix and the filler, causing easy

initiation of a crack in the sample and possible premature failure. The samples made with 300 μm and 425 μm gave the best tensile strength with 21.4 MPa and 21.5 MPa, respectively. Similarly, the tensile modulus declines with the addition of the 150 μm GTR particles from 0.79 MPa to 0.71 MPa and further reduction was observed with increase in the particle size (see Figure 4).

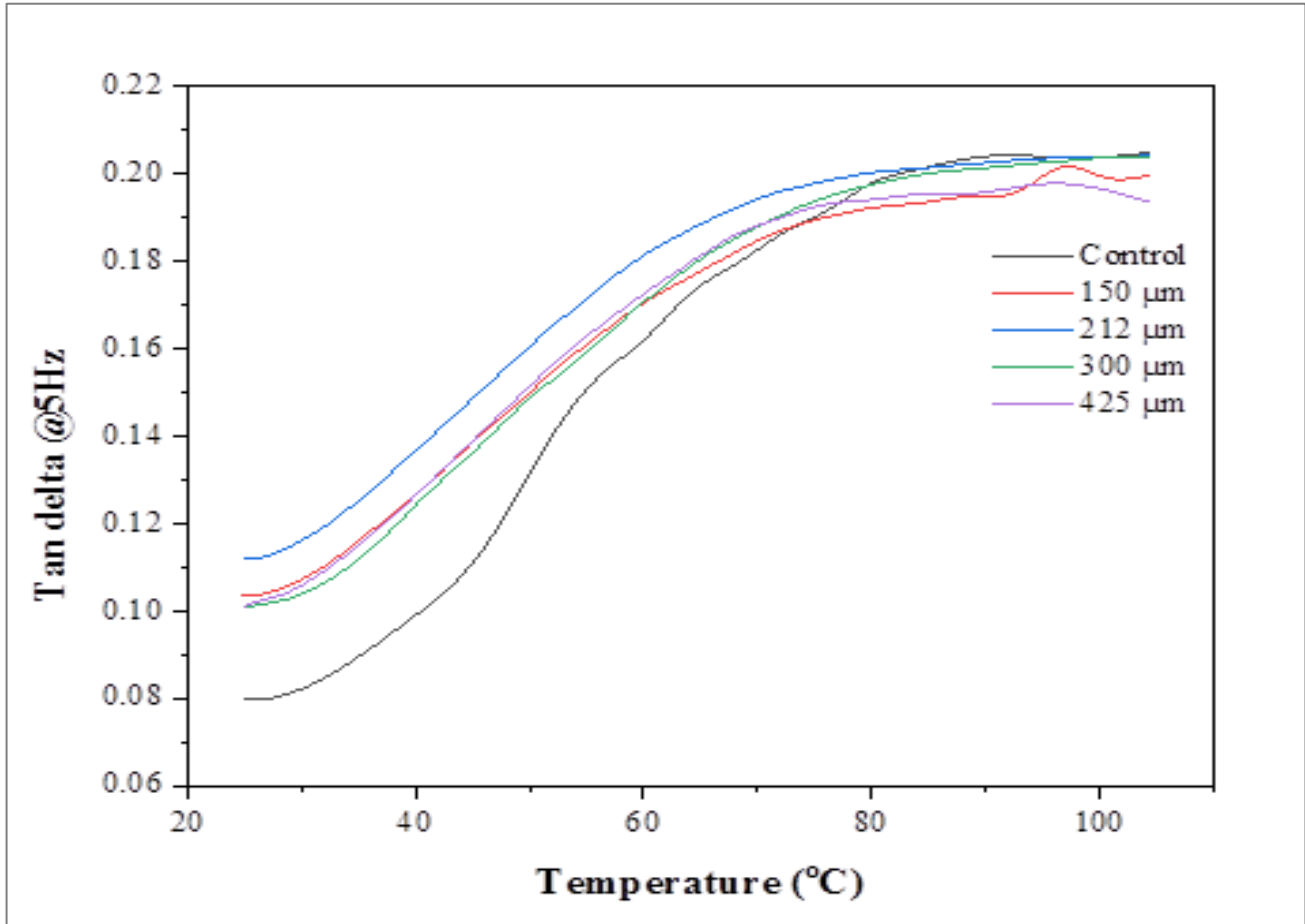


Figure 3: Tan delta of the unreinforced and reinforced GTR/HDPE polymer composite as a function of temperature.

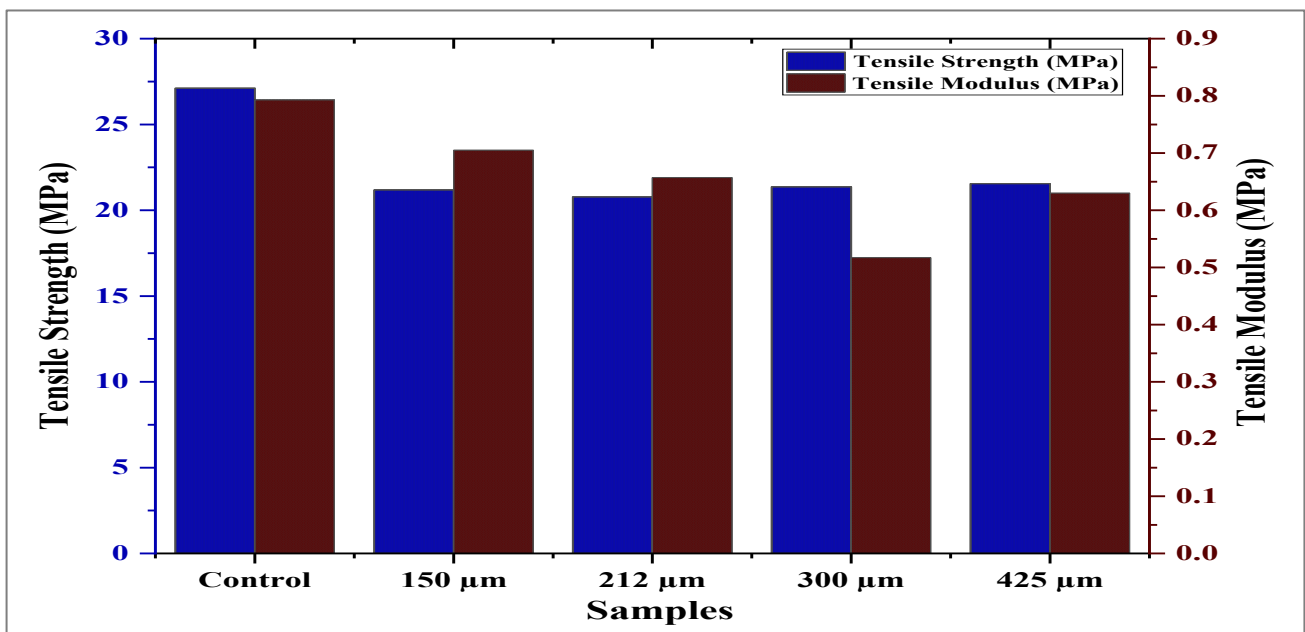


Figure 4: Tensile strength and tensile modulus of the unreinforced and reinforced GTR/HDPE polymer composite

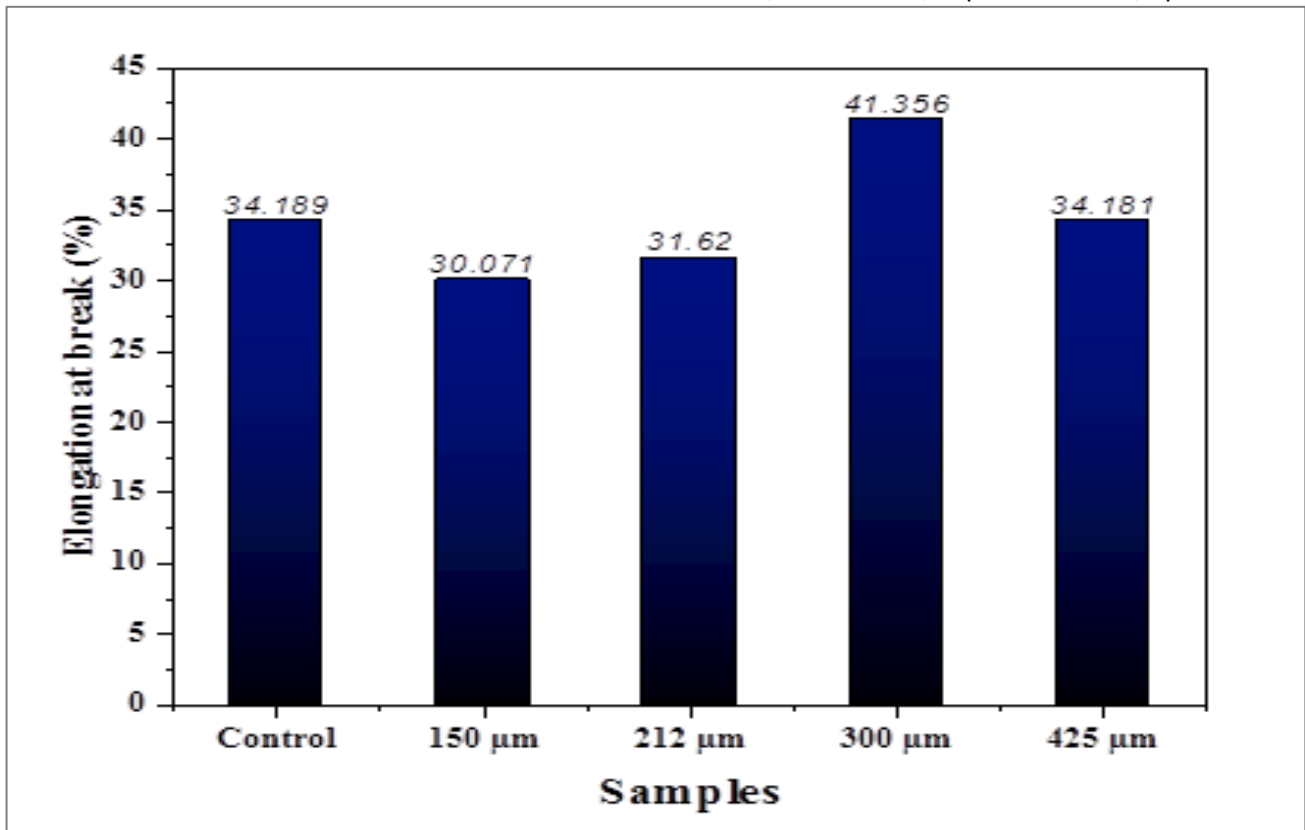


Figure 5: Elongation at break of the unreinforced and reinforced GTR/HDPE polymer composite

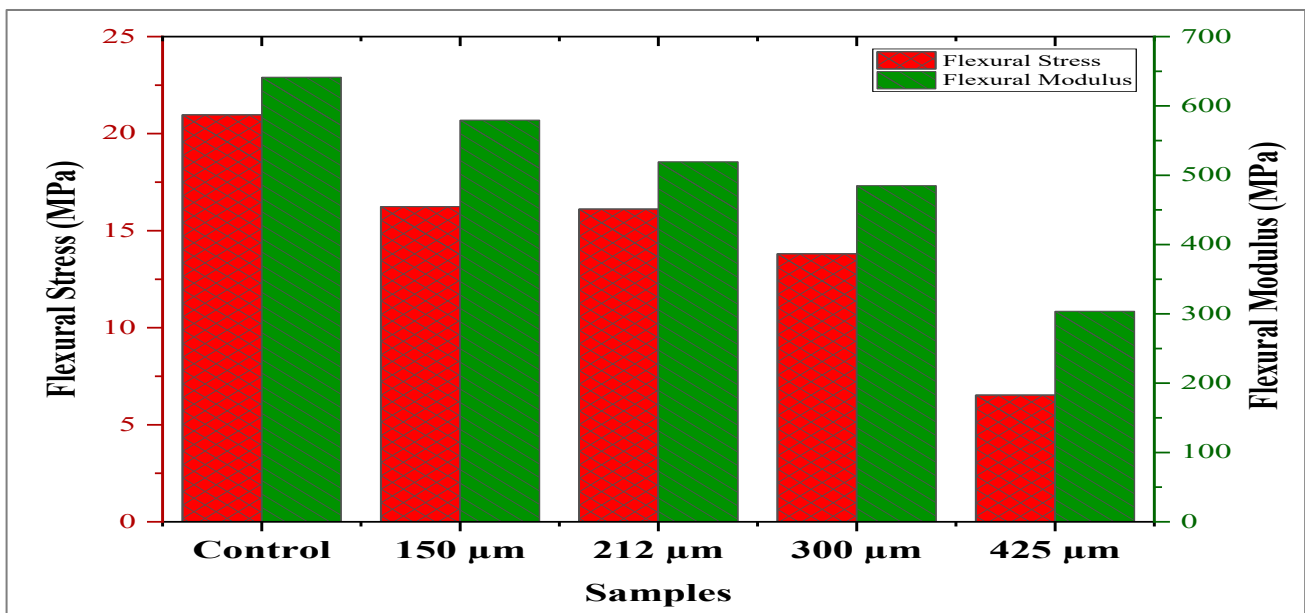


Figure 6: Flexural stress and Flexural modulus of the unreinforced and reinforced GRT/HDPE polymer composite

The effect of GTR particles on the elongation at break is an important parameter used to show the homogeneity and compatibility of the GTR particle/HDPE blend. Figure 5 illustrates the elongation at break of the produced GTR/HDPE polymer. The addition of the filler at different particle size causes decline in the elongation at break also, the incorporation of 150 μm of the particles reduced the elongation at break from 34.1% to 30.1% the result shows that using higher particle size of GTR particles gave much higher elongation at break but increasing the particles from 300 μm to 425 μm resulted

in the decline in the elongation at break from 41.4% to 34.2%. The high elongation at break seen in samples produced with higher particle size could be attributed to high affinity, better bond, and good compatibility between the blend, which reduces the stress concentration point. It is expected that the presence of the GTR particles in the matrix might serve as a point of crack initiation, which could hinder the flow or mobility of the thermoplastic molecules. Hence, the elongation at break reported in this study showed a similar trend to the values reported by Wang et al. (2018). The authors reported that the

elongation at break was around 50% and it was stated that high elongation at break causes good interfacial adhesion between the blend, making initiation of cracks difficult.

Flexural Stress and Modulus Analysis

The flexural stress and modulus are shown in Figure 6. As the GRT particles increase continuously, the flexural stress and modulus decline, following a similar trend to the tensile modulus. The result obtained from this study is similar to that of Ali and Denis (2021). The addition of the GRT particles causes a reduction in the flexural modulus by more than 30%. The flexural modulus dropped from 641 MPa to 579 MPa when 150 µm GRT particles were first introduced, resulting in an 11% decline. Similar reduction can be seen for samples loaded with 212 µm, 300 µm, and 425 µm of the GRT particles, which decline by 23%, 32%, and 11% respectively. This decrease can be attributed to the presence of voids in the developed polymer blends. However, the developed sample with the largest particles gave the least flexural modulus attributed to the poor interfacial interaction between the filler and HDPE polymer molecules as seen in the SEM micrograph (Figure 1.) Hence, it could be concluded that the interfacial interaction and dispersion of the filler strongly influences the mechanical properties and the particle size of the GRT filler controls these.

CONCLUSION

In conclusion, a thermoplastic-elastomer blend was prepared from ground tire rubber (GTR) and high-density polyethylene (HDPE) using a two-roll mill and a compression moulding machine, and its properties were examined in detail. The samples were produced by melt-mixing HDPE/GRT with varying particle sizes (150 µm, 212 µm, 300 µm, and 425 µm). The results show poor dispersion and defects upon addition of high GRT particles, resulting in poor mechanical properties due to their crosslinking. On the other hand, smaller particle sizes of the GRT (150 µm) with fewer voids gave good melting properties, higher crystallinity, as well as good tensile properties of the mixture. This is attributed to their higher specific surface area. This point can further be confirmed from the SEM Micrographs. The resulting composite with fewer voids gives better mechanical properties due to the homogeneity in particle dispersion. During the Dynamic Mechanical Analysis (DMA), it was observed that the incorporation of higher particle-sized GRT (300 µm) gave higher rigidity of the resulting composite. The loss tangent was deduced from the DMA result, and it implied that when the size of the GRT particles increases, the loss modulus increases and vice-versa. Also, the tensile strength and tensile modulus of the developed polymer composite increased according to the amount GRT particle sizes in the HDPE, 300 µm and 425 µm had higher tensile strength of the among the resulting composites, this however is not applicable to the flexural modulus because, increase in the reinforcement particles led to decrease in the flexural modulus as well as

the flexural stress for its application purpose it can be used in an automotive industries.

DECLARATION OF CONFLICTING INTERESTS

I declared that there is no potential conflict of interest with respect to the research authorship and publication of this article.

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