

The Mechanical and Physical Properties of 3D Printing Filament made from Recycled Polypropylene and Ground Tyre Rubber Treated with Alkali

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ABSTRACT

When molten, used vehicle tyres are unable to decompose or be recycled. Despite global efforts to find new uses for these materials, many worn tyres are still dumped in landfills. Therefore, this study proposes using ground tyre rubber (GTR) as a fill material for recycled polypropylene 3D printing filament. The filament composite's physical and mechanical properties will be assessed in this investigation. GTR is expected to give the filament elastic characteristics, which could lead to rubber-like filaments. This study filled recycled polypropylene (rPP) polymer matrix composites with GTR to make filament. The mechanical and physical properties of a 3D-printed specimen made from rPP and GTR filament with varying compositions were analysed. Compared to pure rPP, rPP/GTR samples with 3 wt% GTR had a maximum tensile strength of 716.76 MPa. The flexural test findings showed that rPP/GTR with 3 wt% GTR had the highest flexural strength at 80.53 MPa, followed by rPP/1 wt% GTR at 65.38 MPa. In physical tests, the rPP/GTR at 5 wt% GTR had the highest water absorption at 5.41 %, and the wt% of GTR connected directly with water absorption. This study has shown that affordable, environmentally friendly rPP/GTR filaments can be developed with less amount of GTR content (3 wt%) and used for

3D printing applications, helping to lessen the impact of plastic and waste while having valuable mechanical and physical properties that are comparable to those of the pure polypropylene material produced.

Keywords: 3D printing filament, ground tyre rubber, mechanical properties, physical properties, recycled polypropylene, waste management

ARTICLE INFO

Article history:

Received: 16 August 2023

Accepted: 09 May 2024

Published: 14 June 2024

DOI: <https://doi.org/10.47836/pjst.32.S2.10>

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INTRODUCTION

Ground tyre rubber (GTR) is a form of recycled material composed of tyres that have been shredded and ground into fine particles. GTR has been utilised in an expanding number of applications, including construction, roads, and sporting surfaces. In addition, using GTR in these applications reduces the number of tyres that wind up in landfills or the environment, thereby promoting sustainable waste management (Fazli et al., 2020). As interest in sustainability increases, GTR will likely become more widespread.

On the other hand, recycled polypropylene (rPP) is a plastic made from post-consumer and post-industrial waste. It is a thermoplastic polymer used in a variety of products, including food packaging, automotive components, and domestic goods. Recycled polypropylene is an environmentally preferable alternative to virgin plastic because it reduces waste sent to landfills and conserves natural resources. In the coming years, the demand for rPP is projected to increase significantly due to the world's ongoing emphasis on sustainability and waste reduction (Pandey et al., 2023).

Utilising recycled materials to produce 3D printing filament is a manufacturing strategy that is gathering popularity in the 3D printing industry. Filament for 3D printing is the material utilised by 3D printers to produce three-dimensional objects. Historically, 3D printing filament has been manufactured from non-biodegradable virgin materials such as ABS or PP, contributing to the growing plastic waste problem. 3D printing filament can be manufactured environmentally using recycled materials such as PP bottles or industrial waste (Zander et al., 2019). Compared to the production of virgin filament, the procedure conserves resources, reduces waste, and has a smaller carbon footprint.

It is currently impossible to 3D print with natural rubber, ethylene propylene diene monomer (EPDM) rubber, or other rubber materials that do not readily liquify or transform into a cured state. Depending on the ultimate applications, this poses limitations for 3D-printable rubber products (Drossel et al., 2020). At the moment, thermoplastic polyurethane (TPU) and thermoplastic elastomer (TPE) are the most effective rubber-like materials for 3D printing (Leon-Calero et al., 2021). TPE, for example, is a rubber-like filament used to make flexible and durable objects. It is resistant to stress and impact, unlike PLA and ABS. Its qualities allow it to be employed in 3D printing automobile parts, toys, and medical supplies, to mention a few applications. Nevertheless, TPE filaments exhibit certain drawbacks. First, they are not recyclable. Second, they are not user-friendly for beginners, as printer settings must be precise to achieve the desired results (Musa et al., 2022). In addition, TPE is more temperature-sensitive than other materials, and almost every printed model requires a rework procedure.

Thus, this study aims to develop a new filament composite for 3D printing applications incorporating rPP and GTR as fillers. This study intends to evaluate the mechanical and physical properties of the completed filament composite. The usage of GTR in this study is expected to impart elastic qualities to the created filament, providing a new option for

the creation of rubber-based filaments while also assisting in the utilisation of GTR in new applications.

LITERATURE REVIEW

Rapid growth in the automotive industry generates an increasing quantity of waste rubber, especially from worn-out tyres. The natural and synthetic rubbers in used tyres can be used as strengthening materials in composite manufacture. Incorporating used tyres into virgin matrices reduces the final product's cost and lowers the quantity of virgin materials needed. Ground tyre rubber (GTR) is a substance that is derived from the process of recycling and repurposing waste tyres. The GTR recycling process occurs due to its positive environmental and economic impacts. Alkadi et al. (2019) and Tri et al. (2022) stated that the production of GTR is environmentally friendly and economically advantageous, as it eliminates some of the operations required to recycle natural rubber.

The utilisation of GTR as a filler in diverse fundamental materials also contributes to reducing final product costs. According to Alkadi et al. (2019) and Nguyen et al. (2022), it has been observed that GTR additions have improved characteristics such as lightweight composition, improved thermal and acoustic capabilities, and resistance to booth ageing and severe weather conditions. Rapidly, it became evident that there was a need to assess and ensure the quality of GTR. The two primary quality attributes of tyre rubber (GTR) that are of utmost importance are the particle size range, also known as particle size distribution, and the level of contamination.

According to Alkadi et al. (2019), a correlation exists between the amount of filler in a matrix and the mechanical characteristics of composite materials, including tensile strength, compression strength, and abrasion resistance. Furthermore, the interplay between polymer molecules and the surface of additives can substantially influence the composite materials' mechanical properties. In order to obtain the appropriate composite for a specific application, it is imperative to take into account the composition, properties (such as particle size), and processing (including manufacturing method and pre-treatment) of tyre rubber GTR when it is used as a filler in the rubber matrix.

According to previous research by Kociuszko et al. (2022), highly filled PP/GTR compounds can be mass-produced using a fine portion of tyre rubber GTR with an ordinary particle size of 400 μm as the filler. Standard injection moulding machines can produce thermoplastic compounds when rubber powder is adequately prepared. The PP/GTR90 and PP/GTR90-P compositions have a positive cohesion despite excessive filling. Additionally, Hernandez et al. (2017) introduced a GTR/PP composite material and investigated the impact of sulfuric acid treatment on its mechanical and thermal properties. The study found that exposing the rubber particles to an acidic treatment enhanced their surface roughness and porosity, improving the mechanical characteristics of GTR/PP composites.

GTR can be a filler in the polymer matrix to create composite materials suitable for printing ink using the direct-printing method. The findings indicate that 3D-printed samples exhibit similar tensile strength to moulded samples. Additionally, surface modification of GTR, such as chemical modification, has been demonstrated to enhance the tensile strength of the 3D-printed samples (Alkadi et al., 2017). Nguyen et al. (2022) also developed GTR-based composite materials by incorporating up to 50 wt% rubber fillers into ABS as the main matrix. The study found that adding rubber from 0 wt% to 50 wt% in the 3D-printed GTR-based composite led to a 260% enhancement in damping capabilities. Additionally, specimens containing larger rubber particles had longer contact lines with the host polymeric matrix.

As a result, the energy dissipation is increased, leading to improved overall damping characteristics of the 3D printed composites. Another work by Laoutid et al. (2021) included GTR into ABS at 15 wt% and 30 wt% to produce filaments suitable for 3D printing with FDM. Using a compatibilizing agent to create composite materials with improved mechanical characteristics is advisable. A compatibilizing agent decreases the interfacial tension between incompatible tyre and polymeric phases, serving as a barrier to hinder the coalescence of GTR particles.

Polypropylene (PP) is categorised as a thermoplastic material. Unlike thermosets, thermoplastics can be heated and reshaped indefinitely (Satya & Sreekanth, 2020). Thermoplastics are lightweight and durable and can be manufactured with high precision. Polypropylene exhibits a notable glass transition temperature or softening point, demonstrating a strong resistance to bending stress, exhibiting minimal water absorption, displaying great electrical resistance, maintaining dimensional stability, possessing high impact strength, and being non-toxic.

Polypropylene (PP) has been employed as the matrix material. Polymers exhibiting low melting, such as PP, are widely employed within the 3D printing industry sector owing to their advantageous characteristics, including reduced weight, cost-effectiveness, and versatile processing capabilities. The wide-ranging uses of 3D-printed polymer products are hindered by their limited mechanical strength and functionality, notwithstanding their geometric complexity. Table 1 demonstrates the benefits and drawbacks of some of the 3D printing filaments currently available and commonly used in FDM-based 3D printing.

Integrating diverse materials to achieve specific mechanical and functional properties presents a viable strategy for addressing mechanical performance and functioning challenges. There has been a notable surge in curiosity over the past few years regarding the advancement of composite materials that can be effectively utilised with currently available printers (Wang et al., 2017). Composite 3D printing has utilised numerous materials, including thermoplastics based on nylon and continuous fibres, such as fibreglass, carbon fibre, high-temperature and high-strength fibreglass, and Kevlar materials. Fibreglass with

Table 1

Existing 3D printing filament advantages and drawbacks (Flynt, 2020)

Filament Material	Advantage	Disadvantage
Polylactic acid (PLA)	<ol style="list-style-type: none"> 1. Easy to use. 2. Biodegradable and made from sustainable sources. 3. Inexpensive. 4. Do not produce fumes while printing. 5. Does not warp. 	<ol style="list-style-type: none"> 1. Poor mechanical characteristics. 2. Low heat resistance 3. Tend to deform easily. 4. Naturally, it disintegrates over time. 5. Absorbs moisture easily.
Acrylonitrile Butadiene (ABS)	<ol style="list-style-type: none"> 1. Superior toughness. 2. Excellent impact resistance. 3. Chemical stability. 4. Good thermal stability. 5. Can be finished via an acetone vapour bath. 6. Inexpensive. 	<ol style="list-style-type: none"> 1. Non-biodegradable. 2. Highly prone to warping and stringing. 3. Gives off toxic fumes while printing. 4. Very irritating and noxious.
Nylon	<ol style="list-style-type: none"> 1. Very strong and flexible. 2. Thermally and chemically stable. 3. Abrasion resistant. 4. Takes up dyes easily. 	<ol style="list-style-type: none"> 1. Prints at very high temperatures. 2. Prone to warping and stringing. 3. Degrades under UV radiation. 4. Absorbs moisture easily.
Thermoplastic Polyurethane (TPU)	<ol style="list-style-type: none"> 1. Rubber-like flexibility. 2. Excellent layer-to-layer adhesion. 3. Impact resistant. 4. Resilient against solvents and oils. 	<ol style="list-style-type: none"> 1. Difficult to handle. 2. Poor bridging performance. 3. Cannot be smoothed or polished. 4. Prone to blobs and stringing.
Polycarbonate (PC)	<ol style="list-style-type: none"> 1. Excellent strength and impact resistance. 2. Flexible. 3. High tensile strength. 4. Heat-stable. 	<ol style="list-style-type: none"> 1. It requires a very high temperature for printing. 2. Prone to abrasion. 3. Resistance to scratches. 4. Difficult to work with.

high-temperature and high-strength properties has exceptional heat resistance, rendering it well-suited for various applications such as 3D-printed thermoforms, thermoset moulds, and welding fixtures. Consequently, this investigation aims to develop a new filament composite using rPP and GTR as reinforcements. This investigation aims to assess the physical and mechanical properties of the completed filament composite. The use of GTR in this study is expected to contribute elastic properties to the produced filament and may offer an alternative for producing rubber-based filaments.

MATERIALS AND METHODS

Raw Materials Preparation

The GTR particles, produced by mechanical pulverising tyre rubber in an ambient environment, were obtained from the waste processing industry in Selangor, Malaysia and used as received. The recycled rubber particles exhibited a particle size distribution ranging from 10 to 600 μm as determined by the Malvern particle size analyser. Prior to that, the

GTR powder was alkaline-treated using 6 wt% sodium hydroxide (NaOH) in water (Ismail et al., 2021). This treatment process is necessary to modify the surface properties of the rubber particles, which can improve their compatibility with other materials and enhance their performance in various applications.

The GTR was stirred evenly in 6 wt% NaOH solution to ensure it was mixed thoroughly and later soaked for 40 minutes to ensure that all the rubber particles were in contact with the solution (Nuzaimah et al., 2020). Afterwards, the GTR was washed and rinsed with distilled water to eliminate any leftover NaOH solution and other impurities and dried for 24 hours at 60°C using an oven. The recycled polypropylene (rPP) used in this study was post-manufacturer recycled polymer obtained from San Miguel Plastic Melaka in granulated form. Figure 1 depicts this study's overall process flow for raw material preparation.

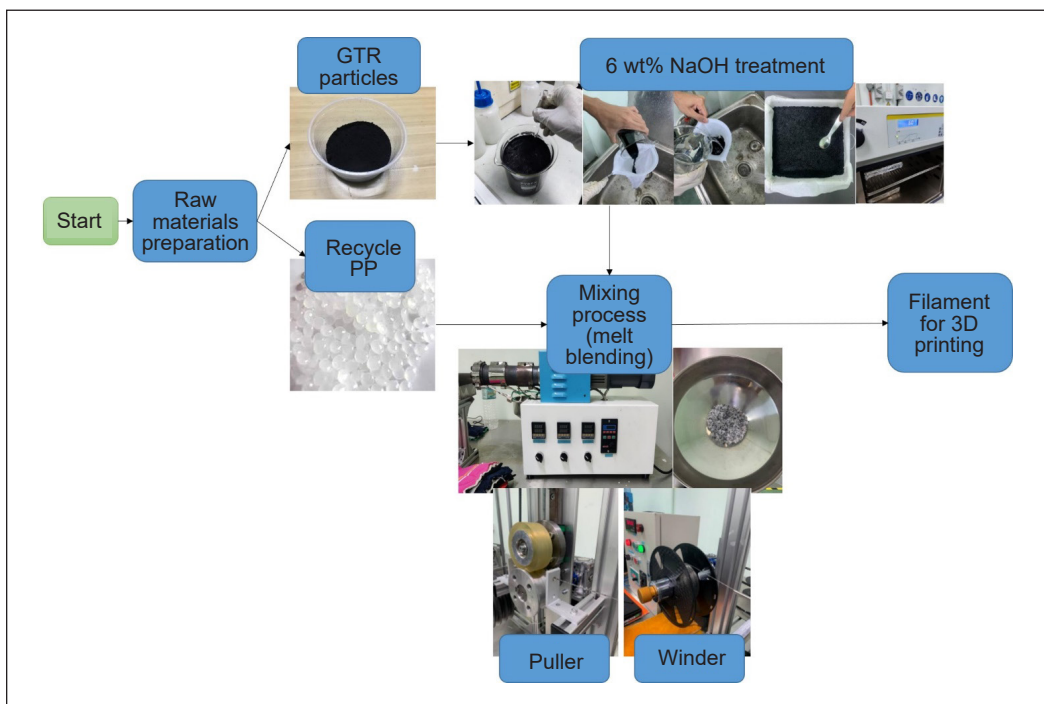


Figure 1. Overall process flow of raw material preparation in this study

Filament Production

The rPP and GTR were compounded by melt blending at various compositions, as shown in Table 2, using a single screw extruder (Figure 2). The study utilised GTR content of 1%, 3%, and 5%, with a 0% GTR sample as the control. The low weight percentage of GTR utilised in this study is determined by the researcher's assessment of the performance of rPP filament in combination with a natural fibre blend deemed more appropriate and of superior quality with reduced fibre content. Hence, the weight percentage content of GTR is considered appropriate

for the first study at lower levels to assess its compatibility with rPP and its potential usage as filament material. The barrel and nozzle zones of the extruder were pre-heated to 170°C and 180°C, respectively, prior to the feeding material process. The speed of the screw and side feeder was set to 18-20 rpm. Table 3 displays the overall extrusion process parameter. The filament was then cooled in a water bath and was coiled up and spooled on the spooler to maintain the acceptable filament diameter ranges.



Figure 2. Single screw extruder machine used in this study

Specimens Printing with 3D Printer

Samples were 3D printed using an FDM 3D printer utilising the produced filaments. The required (.stl) files for printing were created using the open-source Ultimaker Cura 4.8.0 software. The printed samples were the typical Type 1 ASTM D638 tensile test specimens and ASTM D790 rectangular shape. The printing process parameters used, which include a printing strategy of [0, 90] degrees and an infill percentage of 100 %, are shown in Table 4. The nozzle and bed of the printer printed the filament at 180–220°C and 85°C, respectively. The molten filament was fed via a 1 mm nozzle with a 0.32 mm layer thickness at 20 mm per minute.

Table 2
Various compositions of mixed rPP and GTR

	rPP (g)	GTR (g)	wt % of GTR
A	200	0	0
B	198	2	1
C	194	6	3
D	190	10	5

Table 3
Filament extrusion processing parameters

Parameter	Value
Barrel Temperature (°C)	170
Die/ Nozzle Temperature (°C)	180
Die/ Nozzle diameter (mm)	1.75
Screw extrusion speed (rpm)	18–20
Speed of Filament pulling roller (mm/s)	300
Speed of Filament winding roller (mm/s)	200

Table 4
3D printing process parameter

Parameters	Value
Printing temperature (°C)	190
Printing temperature, initial layer (°C)	200
Build plate/ bed temperature (°C)	85
Build plate temperature, initial layer (°C)	95
Infill pattern	Lines
Infill flow (%)	110
Layer height (mm)	0.3
Line width (mm)	0.38
Top and bottom layers (layers)	2
Print speed (mm/s)	25
Initial layer speed (mm/s)	15
Build plate adhesion type	Brim

Mechanical Testing

Tensile tests were performed at ambient temperature in accordance with the ASTM D638 standard, utilising a 500N load cell and a strain rate of 10 mm/min on a Shimadzu Universal Tensile machine. A minimum of five dog bone specimens, each having a thickness of 3 mm, were utilised for every formulation. The averaged values of the tensile strength (σ_Y), tensile modulus (E), and yield strength (ϵ_b) were presented alongside their corresponding standard deviations.

The flexural tests were conducted using a Shimadzu Universal Tensile machine equipped with a 50N load cell, following the guidelines in ASTM D790 at ambient temperature. The experiment involved testing rectangular specimens of 60×12.7 mm². Each formulation was tested five times using a three-point bending mode with a span length of 60 mm. The specimens were tested at a speed of 2 mm/min.

Physical Testing

A water absorption test was also carried out to monitor the capacity of produced filaments to absorb moisture from their environment. A water absorption test was carried out according to ASTM D570 standards, with 3 samples for each parameter submerged in water for 24 hours, and the average value was calculated. Water absorption is expressed as an increase in weight per cent or % weight gain of a plastic specimen, as shown in Equation 1.

$$\text{Increase in weight, \%} = (\text{wet weight} - \text{conditioned weight} / \text{conditioned weight}) \times 100 \quad (1)$$

RESULTS AND DISCUSSION

Mechanical Properties

Tensile Properties. Figure 3 illustrates the variations in the tensile strength, yield strength, and elastic modulus of rPP/ GTR composites with respect to the percentage of GTR content. Incorporating GTR enhanced the tensile strength, yield strength, and elastic modulus of rPP compared to a control sample consisting of only rPP. For instance, increasing the GTR content by 1% and 3% increased the tensile strength by 528.19 N/m² and 716.76 N/m², respectively, compared to the tensile strength of pure rPP, which was 498.64 N/m². The rising trend relates to tensile strength properties, yield strength, and elastic modulus value. At 3 wt% GTR, the sample exhibited the highest values for tensile strength, yield strength, and elastic modulus; at 5 wt% GTR, the tensile properties exhibited a reducing trend.

The reduced tensile strength, yield strength, and elastic modulus properties observed in the presence of 5 wt% of GTR can be attributed to increased agglomeration and particle-matrix interaction caused by a higher quantity of GTR. It led to stress concentration points and weak interfacial adhesion, ultimately increasing the likelihood of crack

initiation and premature failure due to easier crack propagation (Fazli et al., 2020). In agreement with Shaker et al. (2019) and Wang et al. (2018) for recycled PE/GTR, it is evident that less GTR content contributes to increased tensile and yield strengths as well as elastic modulus. At lower GTR content, the effect of filler on tensile properties was found to be more significant, whereas at higher GTR content, poor interaction was the primary factor regulating tensile properties (Fazli et al., 2021). The optimal specific surface area of the filler promoted better interfacial stress transfer and interaction between GTR and rPP, making a quantity that was not excessive the most important factor for producing filament with good tensile strength.

Flexural Properties. Figure 4 depicts the flexural characteristics. The flexural strength of the material increased to 65.38 MPa and 80.53 MPa, respectively, when the GTR content was increased by 1 wt% and 3 wt%, respectively, in comparison to the flexural strength of pure rPP, which was 57.83 MPa. Additionally, the flexural modulus values improved to 1070.63 MPa and 1557.82 MPa, respectively, compared to the flexural modulus characteristics of pure rPP, which was 887.40 MPa. On the other hand, when the GTR concentration is about 5%, both flexural properties decline. This study showed a strong relationship between the mechanical properties of the filler dispersion (GTR) and the interfacial interaction, which is controlled by the concentration of the GTR. In addition, the flexural characteristics of the material are diminished when a significant quantity of GTR is utilised. This finding is consistent with the results for the previously tensile properties. Once more, the enhanced contact between GTR particles and thermoplastic molecules is responsible for the higher flexural characteristics, reducing structural flaws (Fazli et al., 2023).

An additional factor contributing to a reduction in flexural strength with increasing weight per cent GTR is the deterioration of the polymer matrix. In addition to disrupting the polymer matrix's homogeneity and integrity, introducing GTR particles may also introduce a

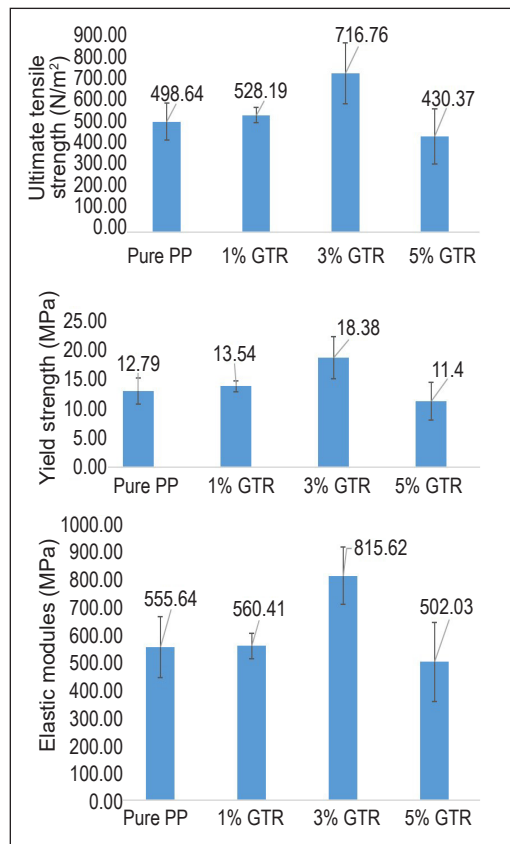


Figure 3. Tensile properties result

vulnerable point into the filament, rendering it more prone to deformation and stress-induced failure (Celestino & Aboelkheir, 2022). Furthermore, including rubber particles could potentially disrupt the intermolecular forces between the chains of polymers, resulting in a degradation of the material’s overall strength (Allegra et al., 2008). Consequently, the flexural strength may be diminished due to the compromised resistance of the polymer molecules to deformation.

Physical Properties

Water Absorption Properties. Figure 5 depicts the water absorption behaviour of the rPP/GTR filament generated. The GTR

addition boosted the water absorption characteristics of the filament in general. The presence of GTR particles, which increases the composite’s hydrophilic behaviour, and the presence of filler particles in the matrix, which can cause voids at the interface and increase the capacity of water molecules to penetrate the composite via capillary transport, are the two main causes of water absorption in composites (Mohammed et al., 2022). Both mechanisms were expected to become less active when the phases were suitably compatible. It suggested that water molecule diffusion through microscopic gaps between polymer macromolecules is the primary mechanism of water absorption inside composites.

CONCLUSION

Using recycled rubber particles, also called GTR, in a recycled thermoplastic matrix (rPP) improved mechanical qualities, even though a lower percentage of GTR was used. The presence of a significant amount of filler material led to a decrease in the mechanical properties of the composite material. It is primarily attributed to insufficient interfacial adhesion and inadequate contact between the cross-linked rubber particles and the thermoplastic chains. This study involved

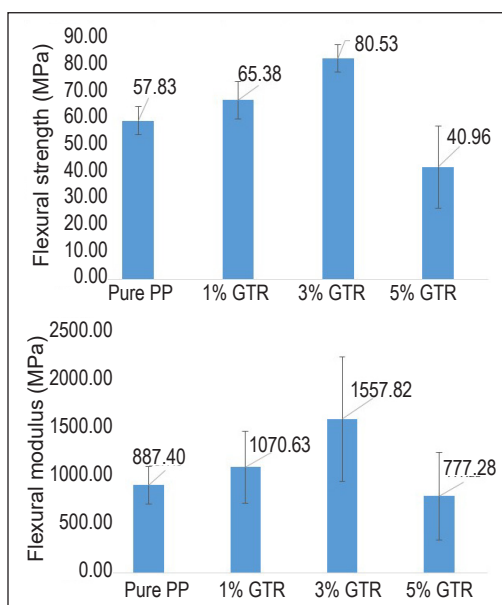


Figure 4. Flexural properties

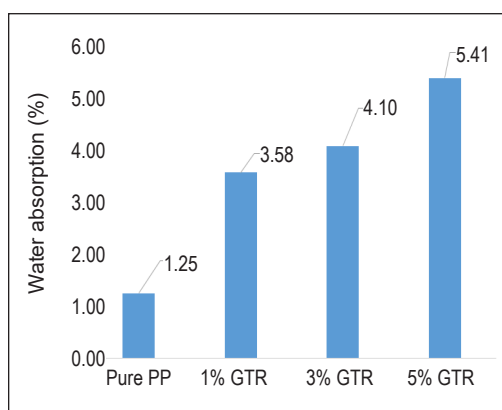


Figure 5. Percentage of water absorption in various compositions of rPP/ GTR and pure rPP

the production of samples by a 3D printing process, utilising a filament composed of a combination of recycled rubber particles (GTR) and recycled thermoplastic matrix (rPP). The samples were prepared with varying concentrations of GTR, specifically 1, 3, and 5 wt% GTR. The study's findings confirm that including GTR at a concentration of 3 wt% led to enhancements in tensile and flexural properties. Nevertheless, it was shown that the sample exhibited a higher water absorption rate with an increased quantity of GTR.

ACKNOWLEDGEMENT

The research conducted in this study received financial support from the Ministry of Higher Education Malaysia and an internal grant from Universiti Teknikal Malaysia Melaka (PJP/2020/FTKMP/PP/S01737).

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