

The Effect of Fibre Loading on the Thermal, Physical, and Mechanical Properties of Waste Tire Rubber-Reinforced Polylactic Acid (PLA) Composites

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ABSTRACT

This study investigates the effects of incorporating waste tyre rubber (WTR) fibres on the mechanical, morphological, thermal, and physical properties of polylactic acid (PLA) composites. WTR fibres, sourced from end-of-life tyres, were introduced into the PLA matrix at weight fractions of 2.5%, 5%, and 7.5% using a single-screw extrusion method to produce composite filaments. Mechanical testing, conducted in accordance with ASTM D638, showed a consistent improvement in tensile performance as WTR content increased. The 7.5% WTR composite achieved the highest tensile stress and Young's modulus, measured at 26.43 MPa and 1822.41 MPa, respectively. Morphological analysis via Scanning Electron Microscopy (SEM) revealed that higher fibre loading enhanced fibre-matrix adhesion, reduced voids, and increased fibre breakage, all of which are indicative of effective stress transfer under load. In contrast, the 2.5% composite exhibited noticeable voids and signs of poor interfacial bonding. Thermal and physical evaluations using Thermogravimetric Analysis (TGA), Differential Scanning Calorimetry (DSC), and surface texture analysis indicated that the inclusion of rubber fibres improved thermal stability and water resistance, likely due to the fibres' inherent heat resistance. Slight increases in surface roughness were also observed, which may influence the composite's surface characteristics and adhesion properties. Notably, these enhancements in mechanical and thermal performance were achieved without compromising the ductility of the PLA matrix. Overall, the 7.5% WTR composite demonstrated the most balanced and enhanced properties, highlighting its potential as a sustainable and cost-effective material for structural applications, while also contributing to environmentally responsible waste tyre management.

Keywords: Waste tyre rubber (WTR); polylactic acid (PLA) composites; Scanning Electron Microscopy (SEM); tensile strength; morphological analysis; profilometer analysis

INTRODUCTION

The global rise in waste tyre accumulation poses a significant environmental challenge, driven by increased demand for automotive tyres and inefficient waste management practices. Traditional disposal methods, such as landfilling and incineration, are not only environmentally hazardous but also economically and spatially unsustainable. Moreover, they contain toxic substances such as heavy metals and polycyclic aromatic hydrocarbons (PAHs) that can leach into soil and water or volatilize into the atmosphere, threatening ecosystems and public health (Gomes et al. 2021; Zhang et al. 2023; Graça et al. 2022). These concerns underscore the urgent need for sustainable recycling strategies, including the upcycling of waste tyres into functional materials like ground tyre rubber (GTR), which has been successfully applied in asphalt, construction materials, and polymer composites due to its flexibility, strength, and shock resistance (Tri et al. 2022).

Polylactic acid (PLA) is a leading biodegradable thermoplastic derived from renewable resources such as corn, wheat, and sugarcane. It exhibits good mechanical strength and printability, making it widely used in additive manufacturing, especially in fused deposition modelling (FDM) (Petchwattana et al. 2019). PLA's low processing temperature, between 160°C and 220°C, allows for easier extrusion and shaping during 3D printing (Lau et al. 2023a). However, its inherent limitations, such as brittleness, low impact strength, and limited heat resistance, restrict its application in more demanding engineering environments. Recent developments have explored compounding PLA with natural or recycled fibres to overcome these drawbacks (Rios-Soberanis, Carrillo-Varela, & Terán-Contreras, 2013). Chemical treatments of reinforcing fibres and careful control of raw material conditions have further enhanced the performance of PLA-based composites, making them more viable for structural and functional uses (Petchwattana, Naknaen & Boonkerd 2019; Rios-Soberanis, Carrillo-Varela & Terán-Contreras 2013; Tri, Nguyen & Tran 2022).

Incorporating waste tyre rubber (WTR) Fibres into PLA have emerged as a promising strategy to enhance both mechanical and thermal properties while addressing environmental concerns (Qasem, Mat Piah & Alhadadi 2023). WTR serves as a cost-effective, sustainable filler that improves the tensile strength, impact resistance, and flexibility of PLA composites. The heat-resistant nature of rubber fibres also contributes to improved thermal stability, while increased surface roughness from the addition of fibres may influence adhesion properties in printed parts (Qasem, Mat Piah & Alhadadi 2023; Ramarad et al. 2015; Ou et al. 2021). Research has shown that WTR-PLA

composites exhibit stronger interfacial bonding, better stress transfer, and reduced void content at optimal fibre loading levels. However, excessive fibre content can negatively affect dispersion and adhesion, compromising structural integrity. Therefore, determining the optimal fibre-to-matrix ratio is critical for maintaining desirable mechanical performance and printability in 3D-printed components (Rios-Soberanis, Carrillo-Varela & Terán-Contreras 2013; Xu et al. 2023; Nawafleh et al. 2021).

Despite the benefits, comprehensive studies on how varying waste tyre rubber (WTR) fibre loadings affect the 3D-printing behaviour, thermal performance, and structural properties of PLA composites remain limited. One recent study demonstrated that blending PLA with 10 wt% recycled tyre fibre, along with a chain extender, markedly improved mechanical and thermal properties, reflecting enhanced interfacial bonding and stress transfer during extrusion processes (Qasem, Mat Piah & Alhadadi 2023). Moreover, a study on PLA-ground tyre rubber (GTR) blends peroxide-vulcanised showed that while moderate GTR addition enhanced flexibility and toughness, excessive loading impeded dispersion and weakened the matrix–fibre interface (Candau et al. 2021). Polymer matrix composites using rubber fillers have similarly reported that high rubber content, particularly above ~30 wt%, can degrade thermal stability and mechanical integrity due to poor interfacial adhesion (Liu et al. 2020). Collectively, these findings support our systematic analysis of varied WTR loadings in PLA composites, emphasising the optimisation of printability, mechanical behaviour, and thermal characteristics to leverage WTR as an effective reinforcement agent in high-performance 3D-printed PLA materials, offering both environmental and functional advantages.

METHODOLOGY

The development of waste rubber tyre fibre-reinforced PLA filament via FDM involves four key phases: fibre treatment, extrusion, printing, and testing. Fibre treatment using NaOH and silane enhances surface roughness and interfacial bonding. This dual treatment enhances the mechanical and bonding properties of the fibres when used in composite materials (Mohd Ghaztar, Nik Ibrahim, & Romli, 2022). The treated fibres are blended with PLA during extrusion, followed by 3D printing of specimens. Figure 1 shows the Waste Tyre Fibre, and Figure 2 shows the Polylactic acid (PLA) pellet.



FIGURE 1.
Waste Tyre Fibre



FIGURE 2. Polylactic
acid (PLA) pellet

Unsieved waste tyre rubbers (100–500 μm) and PLA pellets were used to prepare composite materials. The rubber particles were treated with alkali by immersion in a 6 wt% NaOH solution prepared from 30 g of NaOH in 500 ml of distilled water for 3 hours. Additional solutions included APS, methanol, and acetic acid. Figure 3 shows the NaOH in pellet form before dissolving in distilled water. Figure 4 shows the condition of waste tyre rubbers immersed in the NaOH solution.



FIGURE 3. NaOH



FIGURE 4. Waste tyre rubber
in NaOH solution

After alkaline treatment, waste tyre fibres were rinsed with running water to remove residues, then oven-dried at 60 °C for 6 hours to ensure complete moisture removal. Figure 5 shows the fibre in the oven during the drying process.



FIGURE 5. Waste tyre rubber in the oven

After alkaline treatment, waste tyre fibres were treated with a 3.0 wt% silane solution (60% methanol, 40% water),

with acetic acid added to adjust the pH to 3.5. Post-treatment, fibres were rinsed and oven-dried at 60 °C for 24 hours to ensure complete moisture removal. This improved interface boosts the mechanical strength, durability, and thermal stability of 3D-printed composites and significantly reduces the risk of fibre pull-out during mechanical stress (Lv et al. 2015). Table 1 presents the parameters for each weight percentage of silane solution, distilled water, and methanol, and Figure 6 shows the pH level.

TABLE 1. Parameters of each solution

Parameter	Amount of Silane (ml)	Amount of Distilled Water (ml)	Amount of Methanol (ml)
3.0% Silane	9	116.4	174.6

The waste tyre fibre and PLA were prepared using the law-of-mixtures formula, as shown in Table 2. The composition of the composites was determined by calculating the elemental weights using the composite composition formula (Callister & Rethwisch, 2020). By incorporating the fibre and PLA densities, the data reflected the precise weight of a 2.5 wt% composite in 1000g.

TABLE 2. Weight of each material

Samples	Weight of Composite (g)	Weight of Fibre (g)	Weight of PLA (g)
2.5%	1000	22.1	977.1
5%	1000	44.2	955.3
7.5%	1000	66.3	933.2

The waste tyre fibre is thoroughly mixed with PLA in a beaker. Subsequently, the mixture will be melt-blended during filament extrusion. Figure 6 illustrates the waste tyre fibre and PLA in a beaker.

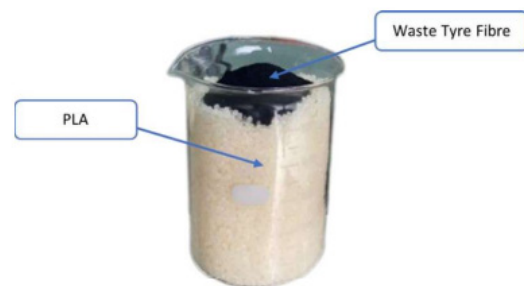


FIGURE 6. Waste tyre fibre with PLA pellet

The filament extrusion process, used for tensile testing samples, employs a single-screw extruder to blend waste

tyre fibre with PLA, producing 1.75 mm composite filaments at 360 rpm and 160 °C. A jig ensures a consistent diameter while maintaining a pulling speed of 30 rpm. Laser measurements monitor filament diameter, and extrusion parameters are regulated via a control panel (Mohd Shukri et al. 2025). Figure 7 illustrates the single extruder employed in the extrusion of the composite filament.

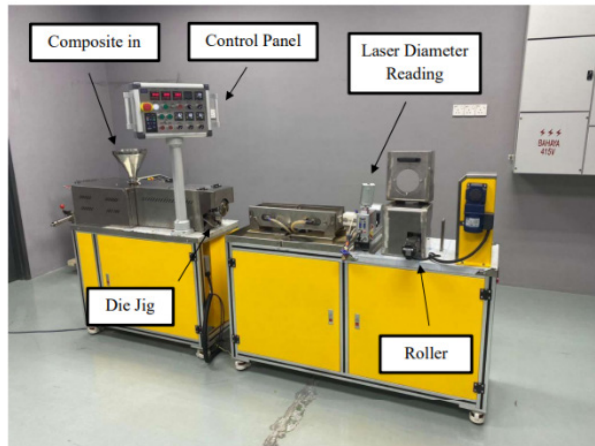


FIGURE 7. Single Screw Extruder

TENSILE TEST

For tensile-strength testing, filaments were 3D-printed with 100% infill and a linear infill pattern to maximise part density and strength. Printing parameters included a nozzle temperature of 170 °C, a bed temperature of 80 °C, and a nozzle speed of 50 mm/s. These settings optimised layer adhesion and surface finish for PLA-based composites (Jamadi et al. 2021; Hsueh et al. 2021). Figure 8 shows the schematic of 3D printing of the composite filament.

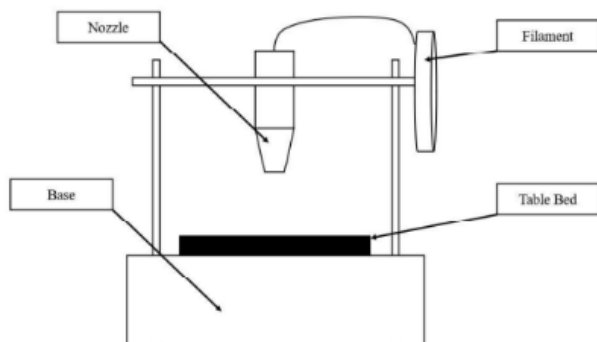


FIGURE 8. Schematic diagram of 3D printing (FDM)

Table 3 lists the tensile-strength test specimens, the applicable testing standard, and the mechanical testing equipment used.

TABLE 3. Tensile strength testing, associated specimen standards, and equipment used for evaluation.

Testing	Specimen standard	Testing
Tensile	ASTM D638 standard 	 UTM Instron 8872

ASTM = American Society for Testing and Materials
UTM=Universal Testing Machine

The tensile test is performed to evaluate the mechanical properties of the composite material, providing essential data on Young's Modulus and tensile strength. The test will follow ASTM D638 and will be conducted using three specimens. The tensile strength of the composite can be calculated using the designated stress formula (1). The designated stress formula is currently printed as:

$$\sigma = \frac{F}{A} \quad (1)$$

where σ is a tensile strength (P_a), F is a maximum force (N), and A is the area of the cross section (m^2).

Tensile properties were evaluated using the Instron 8872 Universal Testing Machine with a 5 kN load cell and a crosshead speed of 1 mm/min. Different jigs and configurations were used for tensile, flexural, and impact tests to ensure accurate alignment and loading conditions

SEM OBSERVATION

Scanning Electron Microscopy (SEM) analyses surface morphology and microstructural features of PLA/waste tyre fibre composites, revealing fibre dispersion, interfacial bonding, and treatment effects on adhesion and defects (Goldstein et al. 2018; Nguyen et al. 2022; Akram et al. 2021; Ilyas et al. 2020). Figure 9 shows that the HITACHI and the model SU5000 utilise a Scanning Electron Microscope (SEM), a specialised apparatus that produces images of a specimen by scanning with a focused electron beam.



FIGURE 9. Scanning Electron Microscope HITACHI - SU5000

THERMAL TESTING

Thermal testing is essential for evaluating material properties under varying conditions, with Differential Scanning Calorimetry (DSC) and Thermogravimetric Analysis (TGA) among the most widely used techniques. DSC measures heat flow during thermal transitions such as melting and glass transition, while TGA monitors mass changes to assess thermal stability, decomposition, and volatile content. Together, they provide a comprehensive thermal profile of materials. TGA is especially valuable in composite analysis, as it can identify distinct mass-loss stages related to polymer degradation, fibre breakdown, and additive content. By analysing samples with different additive levels (e.g., 2.5%, 5%, 7.5%), TGA helps determine the impact of composition on thermal performance. It also aids in estimating filler content and evaluating the compatibility of composite components, offering insights critical to predicting material behaviour in thermally demanding applications.

Meanwhile, Differential Scanning Calorimetry (DSC) is a key thermal analysis technique that measures the heat flow into or out of a material during controlled heating, cooling, or isothermal conditions. It identifies critical thermal transitions, including the glass transition temperature (T_g), melting point (T_m), crystallisation temperature (T_c), and oxidative events. These transitions provide valuable insights into a material's thermal history, phase behaviour, degree of crystallinity, and interaction with additives. In composite materials, the T_g indicates the transition from a rigid, glassy state to a more flexible, rubbery phase. At the same time, T_m and T_c relate to the material's purity and structural organisation. When analysing samples with varying additive concentrations (e.g., 2.5%, 5%, and 7.5%), changes in T_g or T_m observed in DSC thermograms can reflect the level of compatibility, miscibility, or potential cross-linking between the base polymer and additives with factors critical to optimising material performance (Rahman et al. 2017; Gregorová et al. 2011). Figure 10 shows the Thermogravimetric Analyser

(TGA) machine, and Figure 11 shows the Differential Scanning Calorimetry (DSC) machine.



FIGURE 10. Thermogravimetric Analyser (TGA) machine



FIGURE 11. Differential Scanning Calorimetry (DSC) machine

PHYSICAL TESTING

Profilometer analysis is a widely used technique to assess surface roughness and topography by measuring parameters that include factors like average roughness (R_a), root mean square roughness (R_q), peak-to-valley height (R_z), and other profile characteristics that characterise the material finish, which is determined in large part by this research. Contact and non-contact profilometers are the two primary varieties. This is achieved using either contact (stylus-based) or non-contact (optical/laser) profilometers, each suited for specific material types and surface sensitivities. Surface texture directly influences interfacial bonding in composites, including filler–matrix interaction and interlayer adhesion, which are critical to mechanical performance. This analysis is useful for evaluating surface modifications in materials treated at varying concentrations (e.g., 2.5%, 5%, and 7.5%). Changes in surface roughness reveal treatment effects: smoother surfaces suggest polishing or refinement, while increased roughness indicates greater surface area, enhancing mechanical interlocking and interfacial adhesion, both vital for structural performance.

RESULT DAN DISCUSSION

TENSILE TESTING

Tensile testing is used to evaluate a material's hardness, elastic modulus, and ultimate tensile strength (Xu et al. 2023). This method involves subjecting the material to a uniaxial force until fracture occurs (Xu et al. 2023). During the test, tensile stress and strain are recorded to analyse the material's mechanical response. Specifically, the tensile stress and tensile strain at the point of maximum load are

determined. The results are presented as the average tensile stress at maximum load, along with the corresponding standard deviation for each composite ratio. This research on the tensile properties of PLA composites containing 2.5% WTR fibre was evaluated using tensile testing in accordance with ASTM D638. Additionally, PLA composites with varying fibre loadings of 2.5%, 5%, and 7.5% were also tested following the same standard. Calculation manual for Tensile Data Test Result:

- Cross-sectional area (mm^2) = width $\times$ thickness = $13 \text{ mm} \times 3 \text{ mm} = 39 \text{ mm}^2$ (Specimen).
- Maximum Load (N) = Tensile Stress at Max. Load (MPa) $\times$ Cross-sectional area (mm^2)
- Load at break (N) = $\frac{\text{Tensile Stress at Break (MPa)}}{\text{Tensile Stress at Max. Load (MPa)}} \times \text{Maximum Load (N)}$
- Young Modulus (E-Modulus) = $\frac{\text{Tensile Stress at Max. Load (MPa)}}{\text{Tensile Strain at Max. Load (mm/mm)}}$

TABLE 4. Tensile Data Results

Property	Sample 2.5%	Sample 5%	Sample 7.5%
Maximum Load (N)	601.84	858	1030.58
Tensile Stress at Maximum Load (MPa)	15.43	22.0	26.425
Tensile Strain at Maximum Load (mm/mm)	0.0135	0.0145	0.0145
Tensile Strain at break (mm/mm)	0.0140	0.0145	0.0146
Tensile Stress at break (MPa)	10.5	13.5	24.52
Young Modulus (E-Modulus) (MPa)	1143.84	1549.3	1822.41

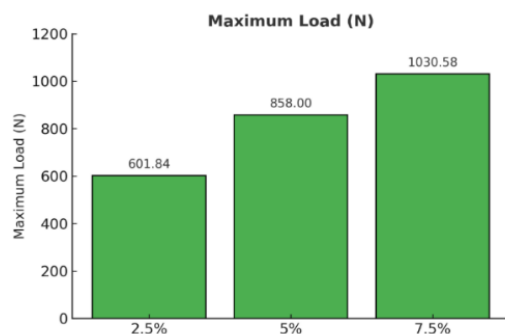


FIGURE 12. Bar Chart of Maximum Load (N)

As illustrated in Figure 12, the maximum load capacity of PLA composites increases with higher waste tyre rubber (WTR) fibre content. The composites with 2.5%, 5%, and 7.5% WTR fibre loadings recorded maximum loads of 601.84 N, 858.00 N, and 1030.58 N, respectively, indicating a consistent improvement in load-bearing capability. This enhancement is attributed to the reinforcing effect of WTR fibres, which improve the distribution of strain energy within the PLA matrix (Bijarimi et al. 2021). Enhanced stiffness and fibre-matrix bonding, particularly through NaOH and silane treatments, improve stress transfer and mechanical strength in PLA/WTR composites, supporting higher loads and suitability for high-performance applications (Fekete et al. 2021). These findings confirm that incorporating higher WTR fibre content enhances the composite's strength, making it more suitable for high-performance applications.

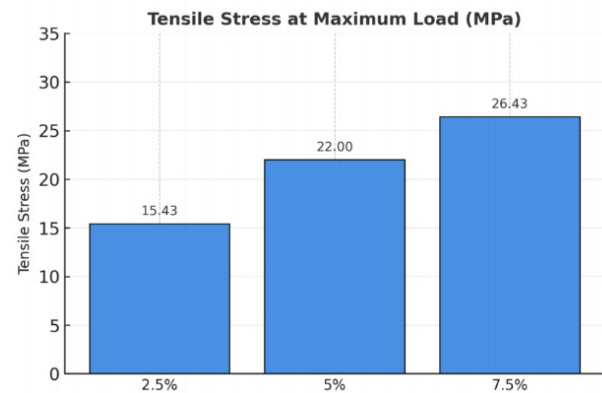


FIGURE 13. Bar Chart of Tensile Stress at Maximum Load (MPa)

As shown in Figure 13, the tensile stress at maximum load increased with higher WTR fibre content, mirroring the trend observed in maximum load values. Composites with 2.5%, 5%, and 7.5% fibre content exhibited tensile stresses of 15.43 MPa, 22.00 MPa, and 26.43 MPa, respectively, indicating enhanced resistance to stress before permanent deformation. Improved tensile stress in PLA/WTR fibre composites aligns with findings by Bijarimi et al. (2021) and Lau et al. (2023), highlighting enhanced stress transfer, fibre alignment, and bonding. Chemical treatments further strengthen fibre matrix interactions, promoting effective load distribution and improved performance in resin transfer molding applications. Overall, the data indicates that at 5% and 7.5% fibre loadings, the synergistic effects of surface treatment and filler content markedly improve the mechanical performance of PLA-WTR composites.

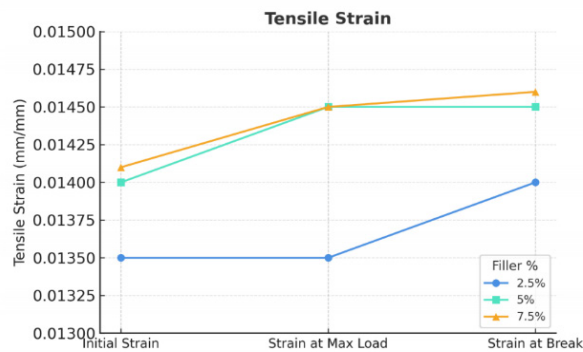


FIGURE 14. Line Chart of Tensile Strain

Figure 14 illustrates the tensile strain behaviour of PLA-WTR composites at three key points: initial strain, strain at maximum load, and fracture. The composite with 7.5% WTR fibre loading exhibited the highest strain across all stages, reaching 0.0146 mm/mm, indicating superior energy absorption and ductility before failure. In contrast, the 2.5% fibre-loaded sample showed lower strain values, reflecting reduced flexibility. These findings support Lau et al. (2023) and Fekete et al. (2021), indicating that improved fibre dispersion and interfacial bonding enhance ductility and stress distribution. Despite minor strain variation, increased fibre content maintains flexibility while promoting ductile fracture behaviour and efficient energy dissipation under loading conditions. Overall, the findings confirm that increased WTR fibre loading not only strengthens PLA composites but also maintains adequate strain capacity, making them suitable for flexible, load-bearing applications.

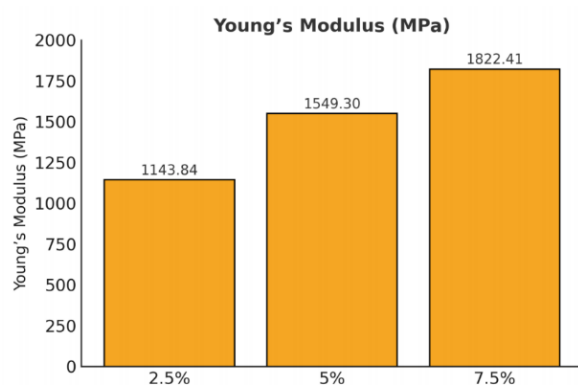


FIGURE 15. Bar Chart of Young's Modulus (E) (MPa)

Figure 15 presents a bar chart demonstrating that Young's Modulus increases with higher WTR fibre loading, indicating greater stiffness in the PLA composites. The modulus increases from 1143.84 MPa at 2.5% fibre content to 1549.30 MPa at 5%, then to 1822.41 MPa at 7.5%.

Increased fibre content restricts polymer chain mobility, enhancing stiffness and structural density without substantially increasing weight. This aligns with Palanisamy et al. (2021), who reported that effective fibre dispersion and bonding improve resistance to deformation, boosting structural performance under tensile and compressive loads. Therefore, for applications requiring high structural integrity and resistance to bending, flexing, or deformation under applied force, the 7.5% PLA-WTR composite offers the best performance among the tested samples, delivering the highest stiffness and load-bearing capacity within the studied range.

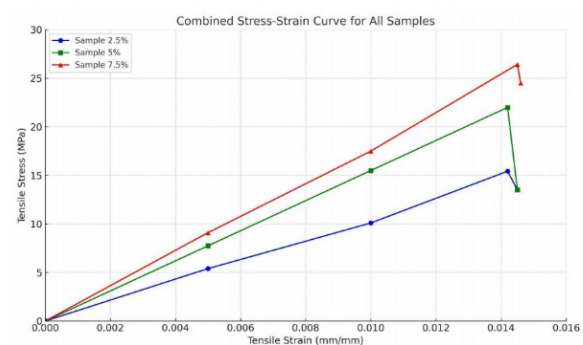


FIGURE 16. Line Chart of Combined Stress-Strain Curve for All Samples

Figure 16 illustrates the stress-strain behaviour of PLA composites reinforced with 2.5%, 5%, and 7.5% waste tyre rubber (WTR), revealing a clear trend of improved tensile performance with increasing fibre content. The 2.5% WTR composite exhibited the lowest tensile strength (15.43 MPa), which decreased to 10.5 MPa at failure, with a strain of 0.0142 mm/mm, suggesting weak interfacial bonding and limited energy absorption. Consistent with Sreenivasan et al. (2019), improved tensile strength at 5% WTR loading reflects enhanced fibre dispersion, reduced porosity, and stronger fibre-matrix coupling. Similar findings by Fekete et al. (2021) underscore the role of interfacial optimization in boosting tensile performance in PLA-rubber composites. The 7.5% WTR composite exhibited the best mechanical performance, with a tensile strength of 26.43 MPa, break stress of 24.52 MPa, and strain of 0.0146 mm/mm. Its steep linear curve indicates a high Young's Modulus and greater structural stiffness. A slight post-peak drop in stress suggests strong toughness and effective energy dissipation. These findings support Palanisamy et al. (2021), who linked improved stiffness to reduced chain mobility and enhanced filler compatibility. Overall, as shown by Lau et al. (2023), the 7.5% WTR composite presents an optimal balance of strength and flexibility, making it suitable for structural and flexible applications.

MORPHOLOGICAL ANALYSIS (SEM OBSERVATIONS)

Prior to SEM analysis, fractured specimens were trimmed to approximately 10 mm in length to ensure compatibility with the chamber. To facilitate practical imaging, samples were coated to allow adhesive mounting of non-conductive materials without interference. Interfacial adhesion and bonding were examined in tensile-tested composites containing 2.5%, 5%, and 7.5% fibre content. Figure 17 presents the corresponding SEM specimens.

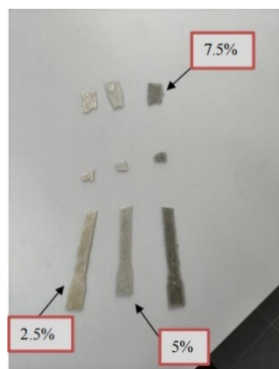


FIGURE 17. Specimen 2.5%, 5% and 7.5%

Samples were gold-coated using the Quorum SC7620 Sputter Coater under vacuum to improve surface conductivity and prevent electron charging during SEM imaging. Argon plasma sputtering, lasting 15 to 30 seconds depending on surface features, enabled clear visualisation of fracture surfaces and fibre-matrix interfaces. Figure 18 illustrates the sputter coater used.

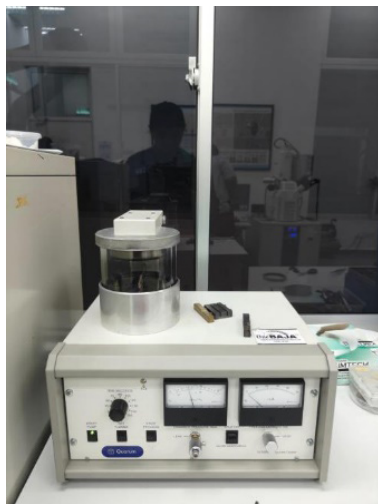


FIGURE 18. Quorum SC7620 Sputter Coater

After gold coating, the specimens were mounted on the holder and placed in the chamber, which was evacuated to achieve the required vacuum. Imaging was conducted at an accelerating voltage of 10 kV and a working distance of 36 mm, with the settings adjusted for optimal resolution and magnification. The electron beam scanned the sample surface over several minutes, generating high-resolution grayscale images. These images facilitated detailed examination of fracture features, including voids, fibre pull-out, fibre breakage, and matrix-fibre debonding. Figure 19 shows the specimens that were prepared for placement inside the Hitachi SU5000 Scanning Electron Microscope (SEM).

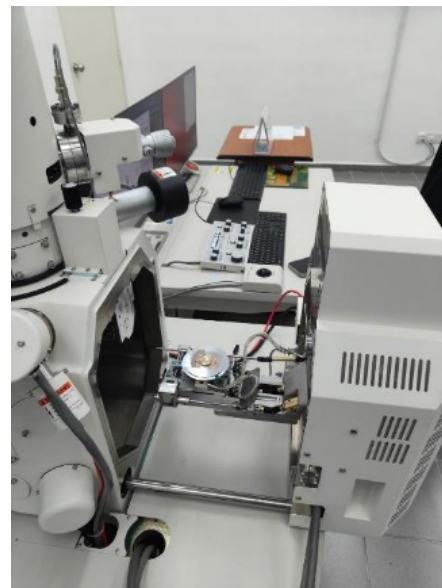


FIGURE 19. Specimens are prepared to place inside the Hitachi SU5000 Scanning Electron Microscope (SEM)

SAMPLE WITH PURE PLA

The pure PLA demonstrated that voids within the composite show unsatisfactory reinforcement, principally due to the absence of fibre surface modification. To optimise interfacial adhesion between the fibre and the PLA matrix, chemical treatments involving sodium hydroxide or silanes are commonly applied. These findings align with those of Alao et al. (2021), who reported the impact of Alkali and Silane Treatment on PLA Composites' Performance, and with Lin et al. (2021), who stated in their report on The Effect of Alkali Treatment on Properties of Dopamine Modification of Bamboo Fibre/Poly(lactic Acid) Composites. These voids compromise the material's structural integrity and overall performance; implementing an appropriate fibre treatment remains critical in composite fabrication. Figures 20 and 21 present the corresponding SEM results.

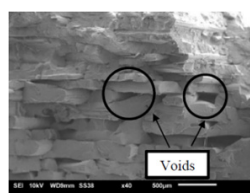


FIGURE 20. Sample pure (30 × Magnification)

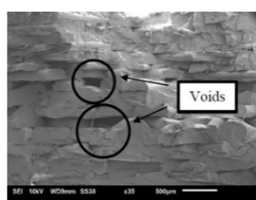


FIGURE 21. Sample pure (50 × Magnification)

SAMPLE WITH 2.5% FIBRE LOADING

Analysis at 500× magnification, as shown in Figures 22 and 23, revealed significant microstructural defects in the 2.5% WTR composite. SEM micrographs showed a high void content across fracture surfaces, indicative of poor matrix fibre wetting caused by inadequate fibre dispersion or insufficient chemical treatment. Evident fibre pullout fractures indicate weak interfacial adhesion, resulting in ineffective stress transfer and compromised load sharing between the PLA matrix and fibres. Additional matrix fibre debonding was observed, with gaps and cavities surrounding some fibres, further indicating weak bonding and separation under tensile deformation. These microstructural features correlate with the sample's low tensile strength, consistent with findings by Sreenivasan et al. (2019), who attributed premature failure to insufficient chemical treatment and under-bonding. The mechanical behaviour of the 2.5% composite aligns with expectations, exhibiting the lowest tensile strength and stiffness among the tested samples.

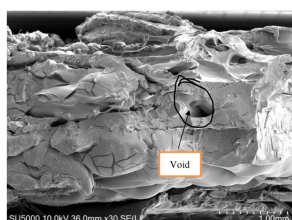


FIGURE 22. Sample 2.5% (30 × magnification)

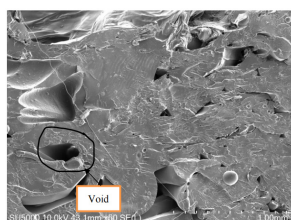


FIGURE 23. Sample 2.5% (50 × magnification)

SAMPLE WITH 5% FIBRE LOADING

The 5% WTR composite demonstrated a more compact and interconnected fracture morphology than composites with lower filler content. Reduced fibre pull-out and well-embedded fibres, with minimal voids, indicate improved fibre-matrix adhesion and dispersion. Evidence of fibre breakage suggests effective stress transfer, likely enhanced by surface treatments. Fekete et al. (2021) noted that silane treatment strengthens interfacial bonding through chemical bridging with PLA, thereby reducing load-transfer failures. These microstructural features correlate with the observed

improvement in tensile performance. Figures 24 and 25 present the corresponding SEM results.

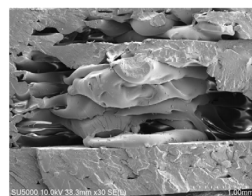


FIGURE 24. Sample 5% (30 × Magnification)

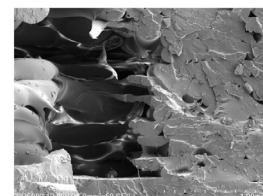


FIGURE 25. Sample 5% (50 × Magnification)

SAMPLE WITH 7.5% FIBRE LOADING

The 7.5% WTR specimen exhibited the most uniform and cohesive fracture surface among all samples. Evidence of fibre breakage indicated that the matrix–fibre bond was sufficiently strong to transfer applied loads effectively into the fibres. Minimal fibre pull-out or matrix–fibre debonding was observed, suggesting robust interfacial adhesion. SEM micrographs revealed a rough, fibrous morphology characteristic of ductile behaviour, with only a few small voids, primarily in high-stress areas. The strong interfacial bonding in this sample reflects improved composite strength, attributed to efficient transfer between components. The increased WTR content, combined with appropriate surface treatment, enhanced both chemical bonding and mechanical interlocking at the PLA matrix interface. These findings align with those of Palanisamy et al. (2021), who reported improvements in tensile strength and stiffness at an optimal filler volume, resulting from enhanced bonding dynamics. Figures 26 and 27 present the corresponding SEM micrographs supporting these observations.

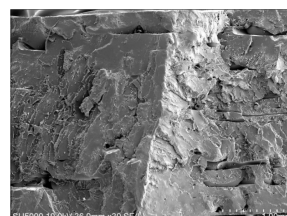


FIGURE 26. Sample 7.5% (30 × Magnification)

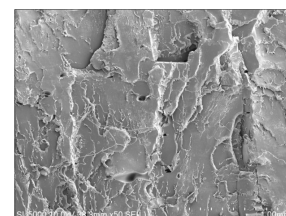


FIGURE 27. Sample 7.5% (50 × Magnification)

THERMOGRAVIMETRIC ANALYSIS (TGA)

Thermogravimetric analysis (TGA) was employed to assess the thermal stability of PLA composites containing waste tyre rubber (WTR). The TGA curves (Figure 28) revealed that all samples, including pure PLA and PLA/WTR blends, exhibited a single central degradation stage, consistent with

PLA's typical thermal behaviour. Pure PLA degraded rapidly, beginning at approximately 300°C and reaching nearly complete decomposition by 390°C. The 2.5% WTR blend showed a slightly broader degradation range, suggesting a marginal improvement in thermal stability due to initial matrix-rubber interactions. Increasing WTR content to 5% and 7.5% shifted the degradation onset to 310-320°C and extended thermal decomposition to 430°C. This improvement is attributed to rubber's insulating and char-forming properties, which slow down the degradation process. The process in which a material, instead of fully combusting or vaporising during thermal degradation, partially decomposes to form a solid, carbon-rich residue. These findings, consistent with prior studies by Mathew et al. (2005) and Causin et al. (2006), confirm that WTR enhances PLA's thermal stability. The 7.5% composite showed the most significant resistance, supporting its suitability for applications requiring greater thermal endurance.

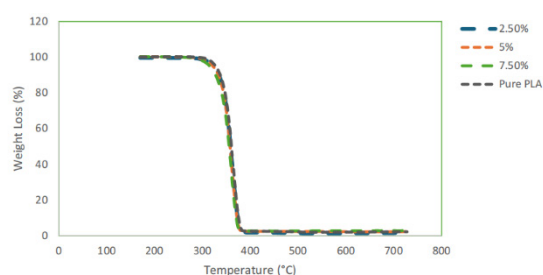


FIGURE 28. Thermogravimetric Curves of PLA / Waste Tyre Rubber Composite

DIFFERENTIAL SCANNING CALORIMETRY (DSC)

Differential Scanning Calorimetry (DSC) was used to evaluate the thermal behaviour of pure PLA and PLA composites with 2.5%, 5.0%, and 7.5% WTR. DSC provides key data on the glass transition temperature (T_g), melting temperature (T_m), and crystalline phase, informing processability and thermal performance in composite manufacturing (Figure 29).

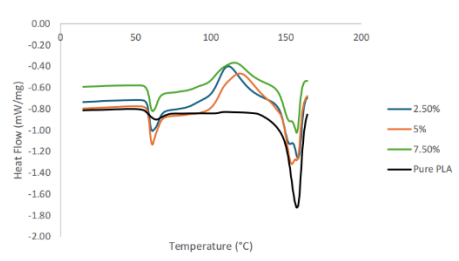


FIGURE 29. Differential Scanning Calorimetry (DSC)

TABLE 4. Differential Scanning Calorimetry (DSC) Data

Parameter (PLA + Waste Tyre Rubber)	T_g (Glass Transition Temperature) °C	T_m (Melting Temperature) °C
2.5%	58.3	157.8
5%	58.3	153.8
7.5%	57.7	157.1
Pure PLA	58.4	157.3

Differential Scanning Calorimetry (DSC) analysis revealed that the glass transition temperature (T_g) remained relatively stable across all PLA/WTR composite samples. Pure PLA exhibited a T_g of 58.4°C, while the 2.5% and 5.0% WTR blends showed a marginal decrease to 58.3°C. The 7.5% WTR composite exhibited a slight further reduction to 57.7°C. These minimal changes suggest that the amorphous region of PLA is largely unaffected by the incorporation of WTR, even at higher filler contents. The consistent T_g values indicate that the molecular mobility of the PLA matrix remains intact, implying no significant disruption of the polymer chain structure.

However, the minor T_g reduction at 7.5% WTR may suggest a subtle plasticising effect, where rubber particles slightly enhance PLA chain mobility. Overall, the thermal softening behaviour across the composites remains comparable to that of pure PLA. In contrast, the melting temperature (T_m) showed greater variation. Pure PLA exhibited a T_m of 157.3°C, consistent with values reported for semicrystalline PLA. A slight increase to 157.8°C in the 2.5% WTR sample may be attributed to heterogeneous nucleation, where dispersed rubber particles promote more ordered crystalline formation. However, the 5.0% WTR composite showed a notable decrease in T_m to 153.8°C, suggesting interference in PLA crystallisation, potentially due to poor compatibility or dispersion. In this case, rubber particles may hinder chain alignment, resulting in a weaker crystalline phase. This reduction in T_m could adversely affect the composite's thermal resistance and structural stability at elevated temperatures.

The melting temperature (T_m) of the 7.5% WTR composite, recorded at 157.1°C, closely resembled that of pure PLA, indicating improved interaction and dispersion between WTR and the PLA matrix at higher filler content. This may suggest enhanced interfacial adhesion or compatibility at this concentration, potentially supporting rather than disrupting PLA crystallisation. Additionally, rubber particles may act as thermal stabilisers, buffer heat and delay degradation. The recovery of T_m at 7.5% indicates that the adverse effects observed at 5% WTR are mitigated or reversed, thereby improving thermal stability. DSC thermograms for all samples displayed a single, pronounced endothermic peak, characteristic of

semicrystalline PLA melting. The absence of additional thermal transitions suggests neither phase separation nor the formation of secondary crystalline structures. These results confirm that PLA remained the dominant continuous matrix, with WTR functioning as a dispersed filler phase, without significantly altering the composite's crystalline behaviour.

Based on the DSC analysis, the addition of waste tire rubber (WTR) influences the thermal behaviour of PLA composites, with the degree of impact varying by concentration. The 5% WTR blend demonstrated the least favourable thermal performance, with a noticeable decrease in melting temperature (T_m) without a corresponding increase in glass transition temperature (T_g) or other thermal parameters. This suggests potential disruption of PLA's crystalline network due to suboptimal dispersion or interfacial contact at this concentration. In contrast, both the 2.5% and 7.5% WTR composites displayed more promising results. The 2.5% blend showed a slight increase in T_m , likely due to a nucleating effect that enhances crystallinity. The 7.5% WTR composite preserved T_g and T_m values close to those of pure PLA, indicating strong phase compatibility and thermal stability, despite the higher recycled content.

This formulation effectively balances thermal integrity with sustainability, making it the most favourable option among the tested composites. The recovery of melting behaviour and minimal variation in T_g support its suitability for thermally demanding and environmentally conscious applications. Thus, PLA with 7.5% WTR is recommended as the optimal formulation based on thermal performance.

PROFILOMETER ANALYSIS

The roughness of PLA composites incorporating waste tyre rubber at different concentrations (2.5%, 5%, and 7.5%) was evaluated using profilometer analysis. Surface roughness plays a crucial role in determining the quality, functionality, and performance of polymer composites. Table 5 summarises the key roughness parameters, presenting average values from three measurements for each sample.

1. R_a (μm) – Arithmetic average of surface roughness.
2. R_z (μm) – Average maximum height of the profile.
3. Roughness parameter defined by JIS standards, often used in engineering surface characterisation.

TABLE 5. Average values of each sample

Parameter	R_a (μm)	R_z (μm)	R_{zjis} (μm)
Pure PLA	1.53	10.17	6.23
2.5%WTR+ PLA	3.13	13.27	9.30
5%WTR + PLA	5.33	32.67	16.47
7.5%WTR + PLA	2.97	15.17	7.77

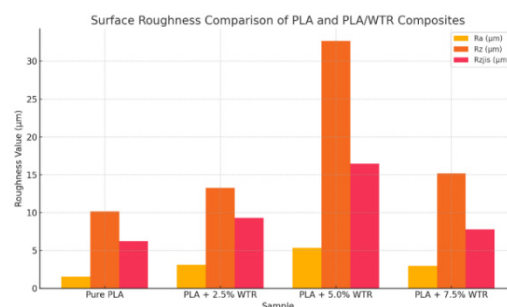


FIGURE 30. Surface roughness of each sample

Figure 30 shows that pure PLA demonstrated the lowest surface roughness among all samples, with R_a , R_z , and R_{zjis} values of 1.53 μm , 10.17 μm , and 6.23 μm , respectively. This reflects a smooth, uniform surface characteristic of a homogeneous, filler-free thermoplastic, making pure PLA suitable for applications demanding high surface quality, low friction, and aesthetic precision. However, because it is derived from virgin bioplastics, its contribution to waste reduction and circular-economy efforts is limited. In contrast, incorporating waste tyre rubber (WTR) into the PLA matrix offers a more sustainable alternative by repurposing non-biodegradable rubber waste, aligning with environmentally conscious material development.

At 2.5% waste tyre rubber (WTR) content, surface roughness increased moderately ($R_a = 3.13$ μm , $R_z = 13.27$ μm , $R_{zjis} = 9.30$ μm), suggesting good dispersion and compatibility of rubber particles within the PLA matrix. This blend maintains adequate surface quality while enhancing sustainability, making it suitable for semi-precision applications where both environmental considerations and functional surface characteristics are important.

At 5% WTR content, the composite exhibited the highest surface roughness ($R_a = 5.33$ μm , $R_z = 32.67$ μm , $R_{zjis} = 16.47$ μm), indicating significant surface irregularities, likely due to poor dispersion or agglomeration of rubber particles. Such surface defects may compromise mechanical performance and visual quality, limiting suitability for applications requiring smooth finishes. However, increased roughness could be advantageous in

contexts where enhanced surface adhesion or mechanical interlocking is desired.

At 7.5% WTR content, surface roughness decreased ($R_a = 2.97 \mu\text{m}$, $R_z = 15.17 \mu\text{m}$, $R_{zjs} = 7.77 \mu\text{m}$), indicating improved rubber dispersion and enhanced matrix uniformity. This reduction is likely due to a plasticising effect at higher rubber concentrations, which facilitates better melt flow and more even distribution during processing. The dominant rubber phase may also reduce the brittleness of the PLA matrix, resulting in a smoother, more ductile surface. These characteristics make the composite suitable for applications that require an improved surface finish, such as biocompatible parts or decorative elements.

The 7.5% WTR composite strikes an effective balance between environmental sustainability and surface performance, exhibiting surface characteristics more comparable to pure PLA than to those of the rougher 5% blend. While pure PLA offers the highest surface smoothness, the 7.5% WTR formulation emerges as the most favourable among the composites, combining acceptable surface quality with the environmental advantage of incorporating recycled rubber waste. This composition is recommended as the optimal formulation, offering a practical and sustainable alternative for industries seeking eco-friendly materials without significantly compromising functional or aesthetic performance.

CONCLUSION

This study comprehensively examined the mechanical, morphological, thermal, and surface characteristics of polylactic acid (PLA) composites reinforced with waste tyre rubber (WTR) at concentrations of 2.5%, 5%, and 7.5%. Tensile testing indicated that mechanical performance improved with increased WTR content, as evidenced by the rise in tensile stress from 15.43 MPa to 26.43 MPa and Young's modulus from 1143.84 MPa to 1822.41 MPa. The enhanced load-bearing capacity and stiffness observed in the 7.5% composite suggest that WTR functions effectively as a reinforcing filler, promoting improved stress transfer through enhanced fibre-matrix interaction.

Scanning electron microscopy (SEM) further validated these findings, revealing significant microstructural enhancements as WTR content increased. The 2.5% composite exhibited voids, fibre pull-out, and weak interfacial adhesion, whereas the 5% blend showed improved fibre embedding and fewer voids. Notably, the 7.5% composite exhibited a cohesive fracture surface with minimal defects and strong bonding, which supports its superior tensile performance. Thermogravimetric analysis

(TGA) confirmed improved thermal stability, with the 7.5% blend exhibiting a delayed degradation onset and a longer decomposition range to 430°C, due to WTR's insulating and char-forming properties.

Differential Scanning Calorimetry (DSC) revealed minimal variation in glass transition temperatures across all composites, indicating retained molecular mobility. While the 5% composite showed a lower melting temperature, the 7.5% blend maintained a T_m close to that of pure PLA, indicating better crystallinity and phase compatibility. Profilometer analysis showed that although surface roughness peaked at 5% WTR, the 7.5% formulation regained smoothness, likely due to improved dispersion and plasticising effect. Overall, the 7.5% WTR composite emerged as the most favourable formulation, balancing mechanical performance, thermal stability, surface quality, and environmental sustainability.

In conclusion, the 7.5% WTR reinforced PLA composite demonstrated the most balanced performance across all evaluated parameters: mechanical strength, thermal stability, morphological integrity, and surface quality, while offering environmental benefits through recycled content. Its superior tensile properties, enhanced thermal resistance, and improved surface smoothness make it a promising, sustainable alternative to pure PLA for applications requiring both functional performance and eco-conscious material choices.

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DECLARATION OF COMPETING INTEREST

None.

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