Borneo Engineering & Advanced Multidisciplinary International Journal (BEAM)
Volume 5, Issue 1, May 2026, Pages 1-7

Effect of Silane Concentration on the Mechanical Properties of Waste Tyre Fibre-Reinforced PLA Filaments for FDM

Full Paper

Article history
Received
31 January 2026
Received in revised form
31 March 2026
Accepted
13 April 2026
Published online
5 May 2026

Hussein Md Zan[1]*, Sinatu Sadiah Shapie[2], Rahim Ishak[3]

[1]Department of Mechanical Engineering, Politeknik Port Dickson, KM 14, Jalan Pantai 96400 Si Rusa, Port Dickson, Negeri Sembilan, Malaysia
[2]Department of Civil Engineering, Politeknik Port Dickson, KM 14, Jalan Pantai 96400 Si Rusa, Port Dickson, Negeri Sembilan, Malaysia
[3]Department of Civil Engineering, Politeknik Mukah, KM 7.5, Jalan Oya, 96400 Mukah, Sarawak, Malaysia

*Corresponding author: hussein@ppd.edu.my
Please provide an **official organisation email** of the corresponding author

Abstract

This research produces waste rubber tyre fibre-reinforced polylactic acid (PLA) filament via fused deposition modelling (FDM). Demand for sustainable materials has prompted the exploration of new ways to utilize waste materials in composites, given growing environmental concerns. Due to high aspect ratio and unique composition, waste tyre fibres, derived from discarded automotive tyres, possess inherent reinforcing properties. However, the characteristics such as compatibility with PLA, a biodegradable polymer, and the overall enhancement of composite properties via chemical treatment remain unexplored. The samples were fabricated from filaments produced by a single-screw extruder and then printed via Fused Deposition Modelling (FDM) according to standard dimensions for mechanical testing. The treatment's effect on mechanical properties, including tensile and flexural strengths, was systematically evaluated. Results indicate that the chemical treatment significantly alters the interfacial interactions between waste tyre fibres and the PLA matrix, leading to a notable enhancement in the mechanical performance of the composites. The data indicate that a 3% silane treatment combined with NaOH yielded the best performance. This project highlights the potential of chemical treatments to enhance composite performance and provides valuable insights into the development of sustainable composites using waste tyre fibres and PLA. The conclusion of this project outlines a path toward environmentally and economically feasible composite materials, with implications for the utilization of waste materials in engineering appliances.

Keywords: Polylactic Acid (PLA) filament, Fused Deposition Modelling (FDM), silane treatment

1. Introduction

The inadequate disposal of waste vehicle tyres (WT) poses a serious environmental hazard and exacerbates global pollution due to their non-biodegradability and a decomposition period of up to 80 years. Encountering has attracted considerable attention in sustainable recycling approaches, particularly in recycling WT fibres as reinforcement materials in composite engineering (Hejna et al., 2020). Recycling offers a sustainable approach to environmental and resource management challenges; it focuses on reducing waste while recovering materials for reuse.

Polylactic acid (PLA) is a biodegradable thermoplastic polymer commonly used in additive manufacturing. Their application is gaining acceptance due to their low melting point, good processability, and reduced environmental impact. PLA enables effective extrusion and is more user-friendly than traditional polymers like ABS, with a melting range of 160°C to 220°C (Fekete et al., 2021 & Lau et al., 2023). When combined with recycled WT fillers, PLA provides both material sustainability and ease of fabrication. Making it a strong candidate for embryonic eco-friendly composites suitable for 3D printing.

The mechanical performance of the material is important towards responsible application of composite materials. Mechanical properties such as strength, ductility, fatigue resistance, and toughness need to be optimized for robust applications (Haryati et al., 2021). Waste tyre rubber exhibits properties such as flexibility, elasticity, and shock absorption. Conversely, owing to the hydrophobic nature, chemical treatments are required to increase adhesion to the PLA matrix. Surface modification methods using Ground Tire Rubber (GTR) significantly enhance interfacial bonding and overall mechanical integrity of the composite (Araujo-Morera et al., 2021).

PLA-WT composites offer additional features, including reduced weight, improved thermal stability, enhanced acoustic insulation, and excellent weather resistance and durability, making them suitable for long-term use across diverse environments (Nguyen et al., 2022). These improvements contribute to good performance and to sustainable products by decreasing dependence on original materials and encouraging the reuse of waste in high-value applications.

Eventually, natural and synthetic fibres are used to reinforce PLA, thereby enhancing its mechanical properties, including stiffness, tensile strength, and dimensional stability. The fibre content, type, and dispersion method can affect the degree of PLA reinforcement (Kawade & Narve, 2017 & Li et al., 2020). The composition of synthetic and natural fibre reinforcements is vital in expanding opportunities for the sustainability of PLA-based materials through various industries. Table 1: The common polymers used in 3D printing.

Table 1: Normally used thermoplastic polymer (Tian et al., 2022)

Matrix	Characteristics			
Polymer	Density (g/cm^3)	Tensile Strength (MPa)	Tensile Modulus (GPa)	Flexural Modulus (GPa)
PLA	1.26	42.6	2.25	2.4
ABS	1.05	50	2.10	2.0

Improperly disposed of tyres end up in landfill sites and release toxic contaminants into the environment and waterways due to the rapid accumulation of waste tyres (Moasas et al., 2022). Refer to Zhong & Zhang (2022): substituting PVA fibres into Engineered Geopolymer Composites (EGC) is necessary as a sustainable, cost-effective solution to preserve structural performance while reducing environmental impact. Fig. 1 shows the utilisation of waste tyres in China in 2021.

Intended for use as sustainable reinforcement for advanced concrete, recycled tyre polymer fibres (RTPF) exhibit high traction in the construction sector. RTPF is well- suited to high-performance and eco-friendly infrastructure projects owing to its enhanced tensile and flexural strength, durability, impact resistance, and crack resistance (Chen et al., 2023).

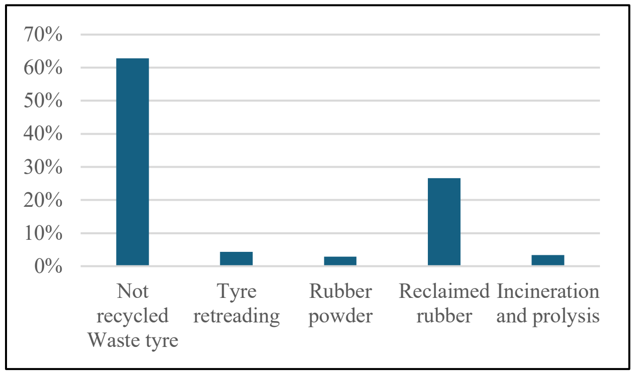


Fig. 1: Waste tyre utilization (Zhong & Zhang, 2022)

To advance together quality and fabrication efficiency, modern 3D printing methods enable the detailed manufacture of fibre-reinforced PLA composites. Combining treated WT fibres with PLA enhances load transfer, minimises defects, and yields stronger, beyond reliable composite materials (Araujo-Morera et al., 2021; Hejna et al., 2020 & Fekete et al., 2021).

This research aims to evaluate the mechanical properties of PLA-WT fibre composites subjected to three chemical treatments. PLA and treated WT fibres are combined via extrusion, and the resulting composite is 3D-printed into test samples. Mechanical property assessments include tensile and flexural strength, measured using a Universal Testing Machine (UTM). The outcomes provide insights into the delivery of concepts for optimising fibre treatment and composite formulation. This reveals the potential of waste tyre fibre composites for sustainable, high-performance additive manufacturing (Lau et al., 2023).

2. Methodology

Preparing the sampling testing is the second main stage of the development method. There are two phases: material preparation and material testing.

2.1. Materials Preparation

The development of waste rubber tyre fibre-reinforced polylactic acid (PLA) filament through fused deposition modelling (FDM) comprises four stages: fibre treatment, extrusion, printing, and testing. In the fibre treatment stage, waste tyre fibres are treated with sodium hydroxide (NaOH) and silane solutions to enhance mechanical properties. The second stage involves blending PLA with waste tyre fibre via single- or twin-screw extrusion. In the last stage, a 3D printer is used to fabricate the sample according to the required design.

The first stage reveals that unsieved waste tyre fibres and polylactic acid (PLA) bits remain as cast-off during the formulation of the materials using various solutions, including sodium hydroxide (NaOH), APS (aminopropyltriethoxysilane), methanol, and acetic acid. Random waste tyre sizes ranging from 100 to 500 µm were exposed to alkali treatment in this experimental setup. The 6% NaOH volume was estimated at 30 mL from 500 mL of distilled water, assuming 30 g of NaOH is prepared in the pellet. The treatment process involved immersing waste tyre fibre in a 6 wt% sodium hydroxide (NaOH) solution for 3 hours. Fig. 2 shows the condition of waste tyre fibres immersed in the NaOH solution.

Fig. 2: Waste tyre fibre in NaOH solution

The waste tyre fibres were then thoroughly washed and rinsed with seriatim water to remove any residual alkali solution. Subsequently, the fibres were gently dried in an oven at 60 °C for 6 hours to confirm complete removal of moisture. Fig. 3 shows the fibre in the oven during drying.

Fig. 3: Waste tyre fibre in the oven

Next, the silane coupling agent procedure ensued. A resolution comprising 1.0 wt%, 2.0 wt%, and 3.0 wt% APS (aminopropyltriethoxysilane), liquefied in a combination of 60% methanol and 40% distilled water (300 ml of each solution), was prepared for this treatment. To adjust the pH to 3.5-4, around 10 mL of acetic acid was additional. To ensure thorough mixing, the mixture was stirred for 10 minutes. Table 2 presents the parameters for each weight percentage of silane solution, distilled water, and methanol.

Table 2: Parameters of each solution

Parameter	Amount of Silane (ml)	Amount of Distilled Water (ml)	Amount of Methanol
1.0% Silane	3	118.8	178.2
2.0% Silane	6	117.6	176.4
3.0% Silane	9	116.4	174.6

The waste tyre fibres were rinsed with distilled water for approximately 30 minutes after silane treatment. Later, dried for 24 hours at 60 °C in an oven to ensure that all moisture is removed from the fibres.

The waste tyre fibre and PLA were combined using a weight-based mixture, as shown in Table 3. The arrangement of the composites was established by determining the elemental weights utilizing the composite composition formulation (Callister & Rethwisch, 2020). By incorporating the fibre and PLA densities, the data corresponded to a detailed weight of 1000 g for a 2.5 wt% composite.

Table 3: Weight of each material

Samples	Weight of Composite (g)	Weight of Fibre (g) 2.5%	Weight of PLA (g)
All Samples	1000	22.1	977.1

The tyre fibre waste is absolutely blended with PLA in a beaker. Then, the mixture will be melt-blended throughout the filament extrusion process. Fig. 4 illustrates the waste tyre fibre and PLA in a beaker.

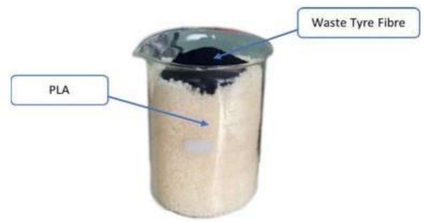


Fig. 4: Waste tyre fibre with PLA pellet

2.2. Testing Materials

The second stage involved filament extrusion, which was employed during manufacturing to produce continuous thermoplastic filament for 3D printing (FDM) used for mechanical testing (tensile and flexural strength). A single-screw extruder produces composite filaments comprising waste tyre fibre and PLA polymer. The filaments were extruded at a constant speed of 360 rpm, resulting in a fixed diameter of 1.75 mm as the extruded filament passed through a jig, with the laser-diameter indicating the filament diameter. The temperature of the heating single-screw extruder is maintained at 160°C from the funnel to the die zone. The rotational speed of the filament feed at the end of the machine is set to 30 rpm. Fig. 5 illustrates the single- screw extruder used in the extrusion of the composite filament.

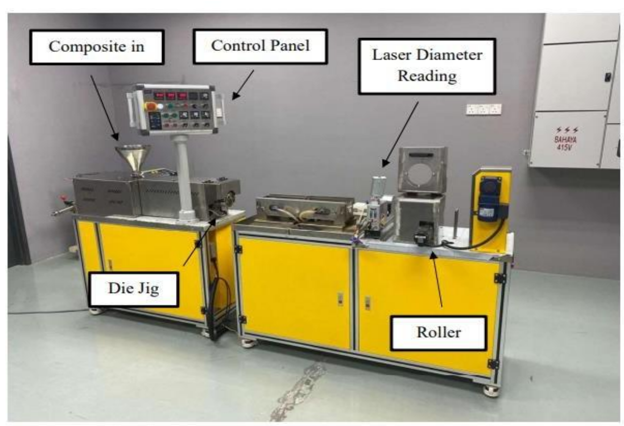


Fig. 5: Single screw extruder

The last stage involved extruding the filament to assess tensile and flexural test samples using a 3D printer. The 3D printer parameters were set to nozzle temperature 170°C, as the composite is suitable at this temperature. Since PLA polymer does not require high temperatures, the bed temperature was kept at 80°C. Printing speed affected the performance of the printed samples, and the nozzle speed was set to 50 mm/s. Fig. 6 shows a schematic of 3D printing the composite filament.

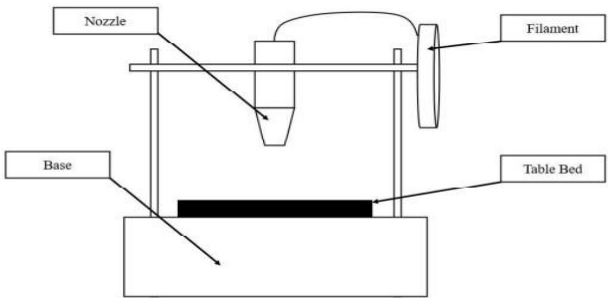


Fig. 6: Schematic diagram of 3D printing (FDM)

Table 4 shows the mechanical testing results (tensile strength, flexural strength, and impact resistance), the specimen standard, and the testing machine.

Table 4: Mechanical testing (tensile strength, flexural strength, impact resistance), specimen standard and machine used for testing

Testing	Specimen Standard	Testing
Tensile	ASTM D638 Standard 	UTM 
Flexural	ASTM D790 Standard 	

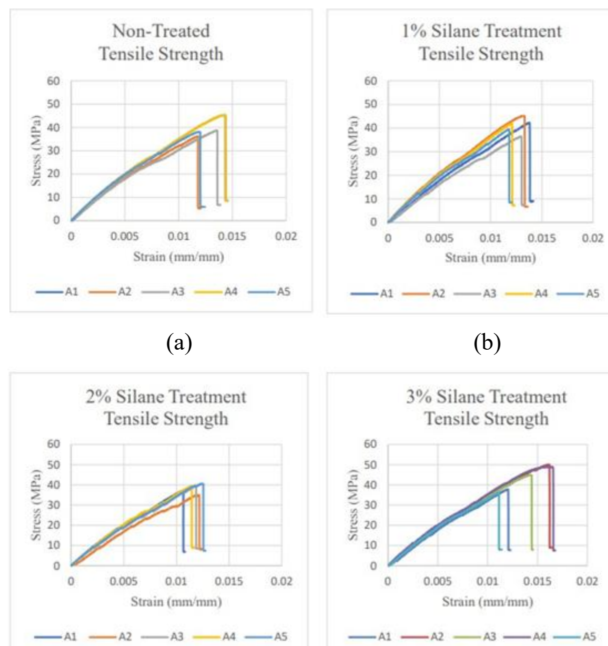
ASTM = American Society for Testing and Materials
UTM=Universal Testing Machine

3. Results and Discussion

The report investigates the mechanical properties of waste tyre fibres following chemical treatment, including tensile and flexural strength. These treatments comprised sodium hydroxide (an alkaline solution) and silane at modifying concentrations (1%, 2%, and 3%). The objective was to assess how these processes are integrated into composite materials and influence the fibres' ability to withstand mechanical stress. The outcomes contribute to high-performance composites utilizing recycled tyre material, thereby enhancing sustainability.

Compared with earlier research by Haryati et al. (2021), the current figures indicate a stable increase in tensile strength for preserved fibres relative to untreated fibres. This tendency proposes that chemical treatments considerably improve the material's resistance to tensile stress. The improvement is attributed to improved interfacial bonding with fibre surface properties developing during the chemical modifications. Consistent tensile performance results reinforce the efficiency of the applied treatment methods.

Tensile testing was conducted on five specimens per treatment set, with standard values derived to further precision to justify sample variability. As shown in Fig. 7, Non-treated waste tyre fibres exhibit an average maximum tensile strength of 39.02 MPa. Treatment with 1% silane in alkaline mixtures yielded an average tensile strength of 41.04 MPa across five specimens, compared with 31.00 MPa in untreated specimens, demonstrating improved mechanical performance. However, the 2% silane treatment with NaOH exhibited the lowest tensile strength (38.35 MPa), indicating an adverse effect at that concentration. In comparison, the 3% silane treatment with NaOH delivered the highest tensile strength, revealing enhanced fibre–matrix compatibility and effective surface reformation. These outcomes emphasize that most treated samples exceeded the 40 MPa limit, assisting the inference that appropriate chemical treatment extensively improves the tensile properties of waste tyre fibres (Haryati et al., 2021 & Bijarimi et al., 2019).



(a) (b)

(c) (d)

Fig. 7: Tensile strength graph for (a) non-treated, (b) 1% silane, (c) 2% silane and (d) 3% silane

Table 5 designates that the 3% silane-treated waste tyre fibre with NaOH shows the lowest Young's modulus (E-modulus) at 3334.09 MPa, signifying improved flexibility and a greater capability to experience deformation under stress without substantial material failure. In contrast, the untreated fibre produced a stiffer, less deformable composite with the highest E-modulus (3353.09 MPa). These differences in stiffness are critical, as Young's modulus absolutely affects the material's deformation behavior. In this way, it affects both its resistance to plastic deformation and its hardening near crack tips.

Table 5: Tensile data result

Parameter	Young Modulus (MPa)	Maximum Tensile Strength (MPa)	Tensile Strain (mm/mm)
Non-Treated	3353.09	39.02	2.078
1.0% Silane + NaOH	3381.84	41.04	2.111
2.0% Silane + NaOH	3485.97	38.35	1.922
3.0% Silane + NaOH	3334.09	43.55	2.301

Dhinesh et al. (2021) emphasized that mixing composite materials increases the final product's mechanical strength and overall performance. The incorporation of materials such as polylactic acid (PLA) and treated waste tyre fibres increases mechanical properties, involving flexural strength. These properties are significantly influenced by factors such as surface hardness, porosity, and surface roughness (Ra), which affect the interfacial bonding within the composite. Kumar et al. (2020) reported that chemical treatment plays a key role in forming a robust matrix that efficiently combines PLA and tyre waste fibres, by improving the composite's structural integrity.

In the meantime, the average maximum flexural strength was determined in accordance with ASTM D790 by testing five specimens for each parameter. The result displayed in Fig. 8 indicates that the untreated fibre's average maximum flexural strength is 26.47 MPa. The flexural strength of waste tyre fibre treated with 1% silane and alkaline treatment is indicated by the average tensile strength of the five specimens, which resulting is 34.21 MPa. The average flexural strength for the 2% silane treatment with NaOH is 28.28 MPa, the lowest among the treated samples. Notably, the 3% NaOH-silane-treated composite has the highest average flexural strength at 34.94 MPa, indicating excellent compatibility.

Table 6 indicates that the Young's modulus is consistent with the findings on tensile strength. The lowest Young's modulus was for the 3% silane treatment. By contrast, the highest Young's modulus was stated with the 1% silane treatment, demonstrating the composite's stiffness. As shown by the Young's modulus results, the degree of silane treatment has a noticeable effect on the material's tensile strength and overall stiffness.

Tábi et al. (2021) showed that using Fibre-Reinforced PLA filaments yields comparable strength and stiffness to that achieved by lowering material density. This approach integrates functional or mechanical performance with improved material efficiency by enabling lighter components (Chaouadi & Gérard, 2021).

Treating waste tyre fibres notably improves the mechanical properties of PLA composites compared to untreated fibres and pure polymer. Haryati et al. (2021) found that increased strength in treated fibres results in a more durable, stronger composite material. Specifically, the three-point bending tests revealed that the 3% silane treatment achieved the highest flexural strength, indicating superior functioning under bending loads. This outcome emphasizes the efficiency of a specific treatment mixture in improving the composite's structural properties.

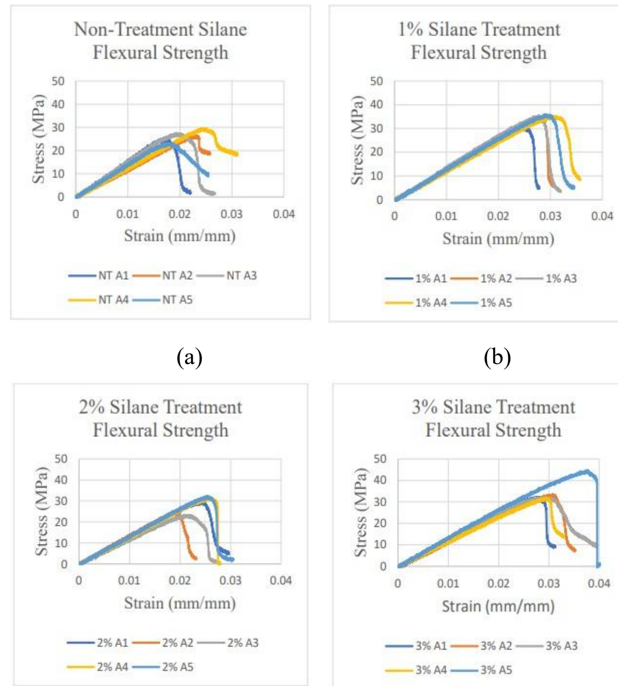


Fig. 8: Flexural strength graph for (a) non-treated, (b) 1% silane, (c) 2% silane and (d) 3% silane

Table 6: Flexural data result

Parameter	Young Modulus (MPa)	Flexural Strength (MPa)	Maximum Load (kN)
Non-Treated	1408.35	26.47	56.23
1.0% Silane + NaOH	1295.34	34.21	72.68
2.0% Silane + NaOH	1298.98	28.28	60.07
3.0% Silane + NaOH	1235.4	34.94	74.23

In contrast, the 2% silane treatment consistently yielded the least promising results across all mechanical tests. This lower performance results in reduced mechanical interfacial strength bonding between the fibre and matrix. The limited compatibility of the 2% treatment concentration for PLA and waste tyre fibre underscores the significance of selecting appropriate chemical treatment concentrations to confirm effective reinforcement in composite development (Gomes et al., 2022).

The 3% silane treatment improves interfacial adhesion between the PLA matrix and the reinforcing fibres. Strengthening this bond significantly contributes to the composite's overall mechanical performance and structural integrity. The outcomes reveal that fibre treatments are essential for manufacturing high-performance, sustainable composite materials, particularly for applications requiring high strength and durability.

Consistent results across tensile and flexural tests confirmed the excellent performance of the 3% silane treatment, highlighting its reliability in the composite mixture. In comparison, the lower performance at the 2% treatment may be attributable to manufacturing contradictions. These effects of fibre waviness or void formation during processing underscore the need for precise processing conditions in composite fabrication (Song et al., 2022).

4. Conclusions and Recommendations

The preliminary results indicate that a comprehensive analysis of the provided data indicates that silane treatment significantly influences the mechanical properties of waste tyre fibre-PLA composites. Remarkably, the 3% silane treatment steadily yields the highest mechanical properties, highlighting the importance of improving matrix bonding. Overall, the treated fibres exhibit improved mechanical properties relative to untreated fibres. The study highlights the crucial role of silane concentration in enhancing composite strength, thereby ensuring accurate and reliable outcomes.

Meanwhile, recycling tyre waste in filament production offers substantial environmental benefits by reducing landfilling while minimizing pollution associated with tyre disposal. It promotes environmentally responsible consumption by converting non-biodegradable waste into valuable composite materials. This leverage not only preserves natural resources but also correspondingly decreases the carbon footprint of manufacturing processes. Subsequently, within the circular economy, it will help strengthen environmental responsibility in material innovation across industries such as 3D printing and construction.

The subsequent project proposals employ maleic anhydride-grafted polypropylene (MAPP) as a compatibilizer to improve interfacial adhesion between waste rubber tyre fibres and polylactic acid (PLA), thereby enhancing composite filaments. The PLA/MAPP/rubber mixture is extruded into filament and fabricated via Fused Deposition Modelling (FDM). The mechanical properties of the in-print parts will be assessed to evaluate the efficiency of MAPP in developing composite performance.

Author Contributions: The study was conducted successfully with contributions from all authors.

Conflicts of Interest: The authors declare no conflict of interest.

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