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RESEARCH ARTICLE

CHARACTERIZATION OF PLA-BASED HYBRID COMPOSITES: MECHANICAL AND MORPHOLOGICAL PROPERTIES

Batrisyia Norhazlin[1], Mohd Adrinata Shaharuzaman[1,2,*], Nadlene Razali[1,2]

[1]*Faculty of Mechanical Technology and Engineering (FTKM), Universiti Teknikal Malaysia Melaka, Hang Tuah Jaya, 76100, Durian Tunggal, Melaka, Malaysia.*

[2]*Centre for Advanced Research on Energy (CARe), Universiti Teknikal Malaysia Melaka, Hang Tuah Jaya, 76100, Durian Tunggal, Melaka, Malaysia.*

Abstract. Sustainable materials and growing environmental concerns have contributed to the development of natural fiber-reinforced polymer composites. Sugar palm fiber (SPF) is renewable, biodegradable, and eco-friendly while waste tyre rubber (WTR) is also useful for improving composite properties. This research focuses on creating poly(lactic acid) as a matrix material for these green composites (PLA) filaments reinforced with SPF and WTR for 3D printing. WTR and SPF were treated with 6 % NaOH and 3 % silane to improve interfacial adhesion with 97.5%PLA:2.5%SPF/WTR. Three different fiber loadings were evaluated 75%SPF:25%WTR, 50%SPF:50%WTR, and 25%SPF:75%WTR. The fabricated filaments from a twin-screw extruder were used to 3D print tensile ASTM D638, and flexural ASTM D790 test specimens with infill densities of 50 %, 70 %, and 100 %. The 75%SPF:25%WTR composite exhibited the best mechanical properties, with a tensile strength of 37.89 MPa and a flexural strength of 54.52 MPa. Consistent performance was observed across infill densities of 50 %, 70 %, and 100 % for 75%SPF:25%WTR highlighting the optimal mechanical characteristics at this fiber loading. Additionally, scanning electron microscopy (SEM) analysis confirmed that the 75%SPF:25%WTR of this combination resulted in superior tensile strength. Enhanced performance is attributed to the improved interfacial adhesion between the treated fibers and the PLA matrix, as well as the uniform dispersion of the fibers within the composite. According to these findings, sugar palm and waste tyre rubber hybrid composites are highly sustainable and high-performance alternatives to petroleum-based plastics.

Keywords: 3D printing, hybrid composites, mechanical properties, morphology, poly (lactic acid).

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***Corresponding author: adrinata@utem.edu.my**

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1. INTRODUCTION

Growing concerns about environmental sustainability and increased awareness of environmental issues have led to a greater focus on developing biodegradable materials, especially in the field of composites. This shift highlights the significance of using polymers derived from renewable sources that are typically biodegradable [1]. Poly(lactic acid) (PLA), a biopolymer derived from renewable resources such as corn starch and sugarcane, has emerged as a promising matrix for composite materials due to its biodegradability and lower environmental impact compared to conventional petroleum-based polymers [2]. In addition to being low in energy consumption and releasing low greenhouse gases during production, PLA is an excellent material for 3D printing [3]. Aside from that, PLA itself has poor toughness, and has some limitations when it comes to commercial applications. This problem can be solved by blending PLA with various natural fibers to enhance mechanical and degradability properties [4]. This study focuses on the comparative analysis of PLA-based hybrid composites reinforced with Sugar Palm Fiber (SPF) and Waste Tyre Rubber (WTR) two materials that not only enhance mechanical properties but also contribute to sustainability.

Sugar palm fiber, a natural fiber known for its high strength and durability, offers significant advantages when used as a reinforcement in composites. It is abundant, renewable, and provides lightweight. Additionally, the use of SPF helps in reducing agricultural waste, aligning with sustainable practices. WTR, on the other hand, is a recycled material that addresses the growing issue of tire waste, which poses environmental hazards [5]. By incorporating WTR into PLA composites, the overall toughness will be improved making these composites suitable for various applications, including automotive components [6].

The mechanical properties of these hybrid composites, particularly tensile and flexural strengths are critical for assessing their performance [7]. Morphological studies often conducted using scanning electron microscopy (SEM) reveal the interfacial bonding characteristics and the distribution of fibers within the composite, which are essential for understanding how these materials behave under stress [8].

In precise, the exploration of PLA-based hybrid composites reinforced with SPF and WTR not only highlights the mechanical advantages of combining natural and recycled materials but also emphasizes the importance of developing sustainable, biodegradable alternatives in material science. The incorporation of natural fibers not only helps reduce the cost of PLA products but also enhances their competitiveness in commercial applications. This research aims to provide insights into the mechanical properties and morphological characteristics of these innovative composites, contributing to the ongoing efforts in creating environmentally friendly materials.

2. MATERIALS AND METHODS

Poly(lactic acid) (PLA) was used as the matrix material for the hybrid composites. Sugar Palm Fiber (SPF) and Waste Tyre Rubber (WTR) were used as the reinforcing agents. The SPF and WTR were obtained from local sources in Malaysia.

2.1 Fiber Treatment

Surface treatment is crucial for optimizing the interfacial properties of natural and synthetic fibers. In this study SPF and WTR were treated with 6 % sodium hydroxide (NaOH) for 3 hours and dried at 60 ºC for 24 hours to improve interfacial bonding. SPF were treated with NaOH to remove hemicellulose, lignin, and other impurities that could hinder fiber-matrix adhesion. The treated fibers were then rinsed with distilled water and dried in an oven at 60 °C for 24 hours.

Following the NaOH treatment, the SPF and WTR were treated with 3 wt% silane solution for 3 hours to promote chemical bonding between the fibers and the PLA matrix. After treatment, the fibers were rinsed thoroughly with distilled water to remove any residual chemicals and then dried in an oven at 60 °C for 72 hours to ensure complete moisture removal.

2.2 Composites Preparation

The PLA pellets were dried in an oven at 60 °C for 24 hours prior to compounding~~.~~ The dried PLA pellets were then mixed with the treated SPF and WTR fibers in a twin-screw extruder to produce the hybrid composite filaments in size 1.75 mm. The composition of the hybrid composites is shown in Table 1. To ensure proper mixing and dispersion of the composites fibers within the PLA matrix, the extruder was operated at a temperature profile as shown in Table 2 and the resulting filaments were cooled and pelletized for further processing characterization. Based on the composites 97.5 % PLA: 2.5 % SPF/WTR ratios, the amount of PLA is larger, and the melting temperature of PLA is used as setting temperature. The melting point of polylactic acid (PLA) typically ranges from 150°C to 180 °C. The exact melting point can vary based on the specific formulation and additives present in the PLA filament, such as plasticizers or pigments, which can influence its thermal properties.

Table 1: Compositions of hybrid composites

PLA	**Hybrid composites**			
97.5 %	2.5 %			
	SPF, wt%	SPF, g	WTR, wt%	WTR, g
975g	75	18.75	25	6.25
	50	12.5	50	12.5
	25	6.25	75	18.75

Table 2: Parameter of extrusion

Composites	**Setting Temperature (°C)**	**Screw speed (rpm)**
PLA/75%SPF:25%WTR	174.3	20.0
PLA/50%SPF:50%WTR	168.4	20.0
PLA/25%SPF:75%WTR	164.3	18.0

2.3 3D Printing

The prepared filaments were utilized for 3D printing using a Fused Deposition Modelling (FDM) printer (Ender 3). Next, there were several parameters that needed to be considered such as temperature of nozzle, temperature of bed and percentage of infill density. The nozzle temperature was set at 180 °C as follow the manufactured safety sheet data and the bed temperature was 80 °C considering that PLA polymer did not require high temperature [9]. The speed of printing also affects the performance of printed samples. In this printing process, the speed of nozzle is 60 mm/s while travel printing is 80 mm/s. Standard test specimens for tensile and flexural testing were produced according to ASTM D638 and ASTM D790 standards, respectively. Three different infill densities were used during the printing process as shown in Table 3.

Table 3: Infill density of hybrid composites

Composites	**Infill density**		
75%SPF:25%WTR 50%SPF:50%WTR 25%SPF:75%WTR	50 %	70 %	100 %

2.4 Sample Characterization

In this section, we focus on the characterization of PLA-based hybrid composites reinforced with Sugar Palm Fiber (SPF) and Waste Tyre Rubber (WTR), emphasizing both mechanical testing and morphological properties. The mechanical properties of the composites were evaluated through tensile and flexural testing, which provided insights into their strength, stiffness, and overall performance under load. Additionally, morphological studies were conducted using Scanning Electron Microscopy (SEM) to examine the interfacial bonding between the fibers and the PLA matrix, as well as the dispersion of the reinforcing materials within the composite. This comprehensive characterization approach allows for a better understanding of how the incorporation of SPF and WTR influences the mechanical behavior and structural integrity of the hybrid composites.

2.4.1 Mechanical Testing

For this research, the testing was carried out by following the ASTM D638 standard and five samples have been taken for each parameter to find the results. By using this standard testing, the crosshead speed is 1 mm/min with a load cell of 5 kN. The tensile properties of composites were determined using the Universal Testing Machine model Instron 887, manufactured in Norwood, Massachusetts, United States with 5 sample for each parameter to find average result. The tensile strength of the single fiber can be calculated using Equation

$$\sigma = \frac{F}{A} \tag{1}$$

where, σ is the tensile strength of the fiber (Pa), F is the maximum force at break (N), and A is the area of the cross section (m^2).

The flexural strength was assessed using a Universal Testing Machine model Instron 5585 manufactured in Norwood, Massachusetts, United States. The sample size is 100 × 16 × 5 mm following the ASTM D790 standard with a span length of 80 mm (Figure 1). Using a three-point bending setup with five samples of the specimen for each parameter to find average. The flexural strength of composite materials can be calculated using the stress formula for three-point bending as stated.

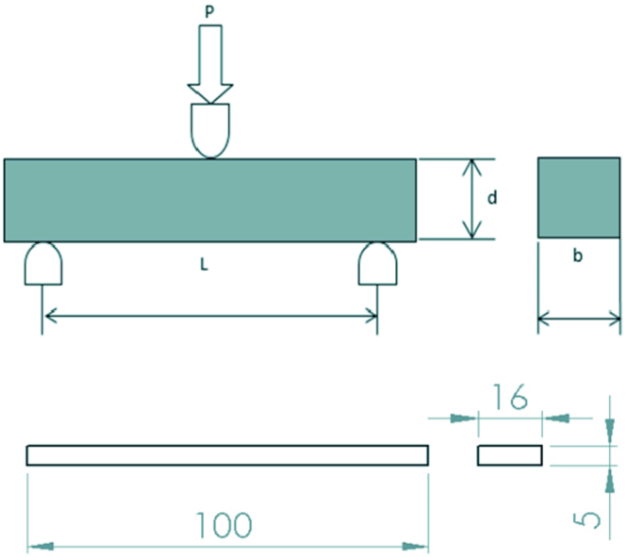

Figure 1: ASTM D790 illustration measurement

$$\sigma = \frac{3PL}{2bd^2} \quad (2)$$

where σ is flexural strength (Pa), P is maximum force (N), L is the support span (mm), b is the width of the specimen (mm), and d is the depth of the specimen (mm).

2.4.2 Morphological Analysis

For this research, morphological studies were performed in detail on the fracture surface of the tensile test sample using a SEM. Platinum coating is used for this research. As a coating material in electron microscopy, platinum is commonly used due to its high conductivity and ability to enhance contrast. By adding a second layer, the sample can be protected from damage during imaging with electron or ion beams, charging effects are reduced, and the images will be of higher quality. The five different samples taken from the tensile specimen were tested. The samples were coated with platinum to get a better result of resolution as it offers good electrical conductivity. The micrograph was obtained by using a JSM6010PLUS/LV SEM (Jeol Ltd., Tokyo, Japan).

3. RESULTS AND DISCUSSION

3.1 Mechanical properties

The mechanical properties of the PLA-based hybrid composites reinforced with Sugar Palm Fiber (SPF) and Waste Tyre Rubber (WTR) were evaluated through tensile and flexural testing. The results indicated that the incorporation of hybrid composites significantly influenced the tensile strength and flexural strength of the composites.

3.1.1 Tensile strength

The tensile strength of the PLA composites varied depending on the fiber loading ratios. In Figure 2, pure PLA demonstrates as the parameter flexural strength among all tested samples [10]. The incorporation of WTR into PLA has been shown to enhance its tensile strength with 43.55 MPa. From previous studies that exposing the rubber particles to an acidic treatment enhanced their surface roughness and porosity, improving the mechanical characteristics and has better energy dissipation [11]. The addition of SPF into PLA tends to decrease tensile strength 21.9 MPa. In the hybrid blends Composites with a 75 % SPF and 25 % WTR ratio exhibited the highest tensile strength, reaching approximately 37.89 MPa. Conversely, the 25 % SPF and 75 % WTR composite showed a decrease in tensile strength, indicating that while WTR contributes to toughness, excessive rubber content may compromise the load-bearing capacity of the composite. However, all blends consistently exhibit higher tensile strength compared to PLA/SPF composites alone. Notably, the PLA/WTR composite demonstrates the most significant improvement in tensile strength when compared to pure PLA, indicating that the inclusion of WTR effectively enhances the mechanical performance of the composite material. According to a study, composites made with 75 % SPF and 25 % WTR showed better tensile strength than other composites, which may plateau or decline due to issues like poor interfacial adhesion or excessive rubber content, resulting in less cohesiveness. Theoretically, WTR can strengthen the structural integrity of composites by improving energy dissipation and flexibility, which can improve performance in bending loads. However, in hybrid composites too much rubber may introduce weaknesses over optimal levels, implying that a balance must be struck to maximize performance.

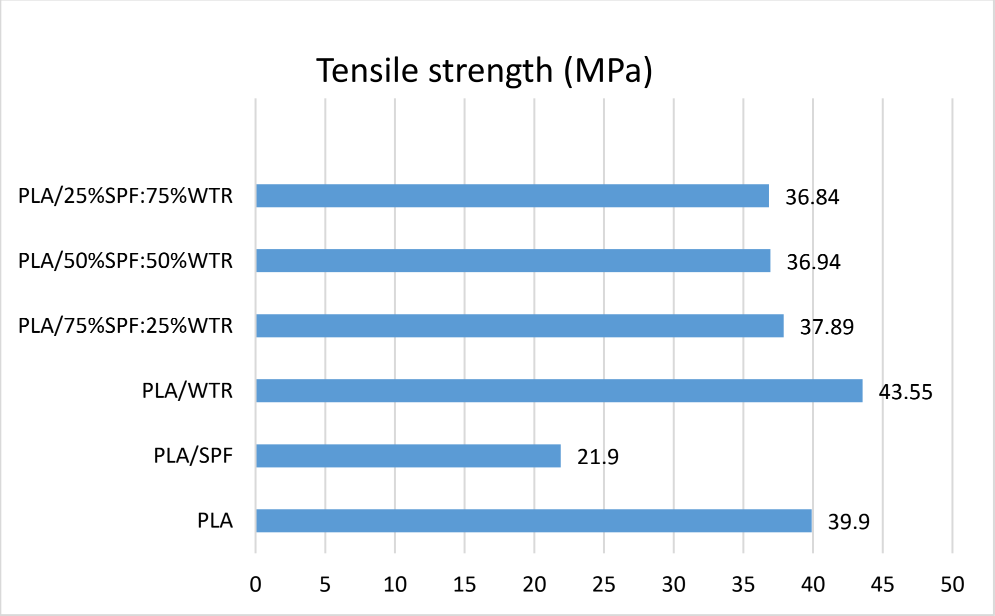

Figure 2: Tensile properties of hybrid fiber reinforced PLA composites

3.1.2 Flexural strength

The mechanical properties of PLA-based composites exhibit complex interactions based on the specific combinations and ratios of additives. In Figure 3, pure PLA demonstrates as the parameter flexural strength among all tested samples 52.5 MPa [10]. Both PLA/SPF and PLA/WTR composites individually show lower flexural strength compared to pure PLA with 36.5 MPa and 34.94 MPa. The flexural strength of hybrid results mirrored those of the tensile tests, the PLA blend containing 75 % SPF and 25 % WTR exhibits the highest flexural strength 54.52 MPa, surpassing that of pure PLA.

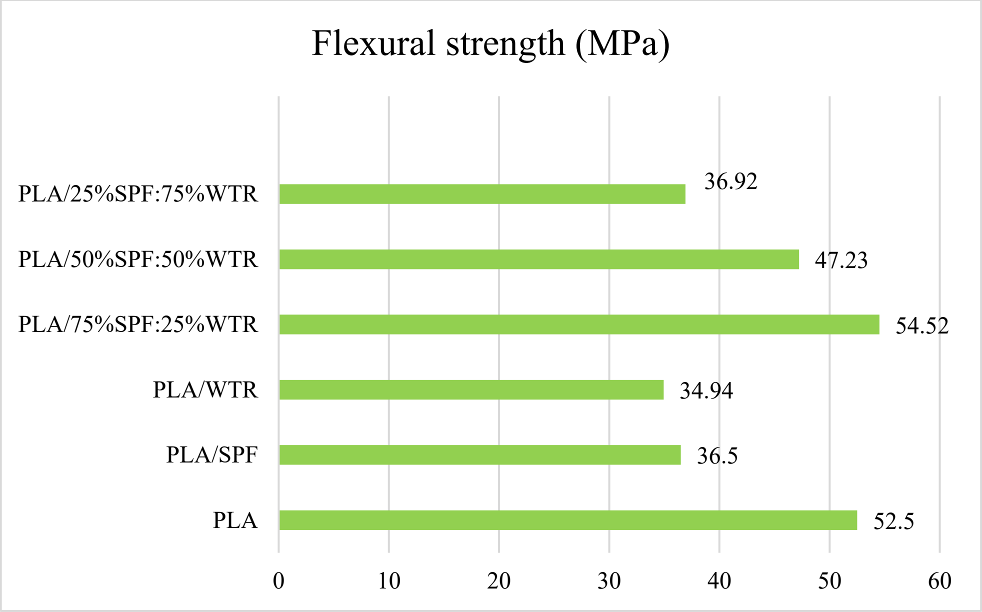

Figure 3: Flexural properties of Hybrid fiber reinforced PLA composites

The incorporation of SPF not only improved the load-bearing capacity but also contributed to the overall stiffness of the composite. As the proportion of WTR increases in the blends, a corresponding decrease in flexural strength is observed from 54.52 MPa, 47.32 MPa and 36.92 MPa. In contrast, composites with higher rubber content exhibited lower flexural strength, indicating that although WTR improves tensile strength, excessive use may weaken the overall structural integrity.

The effect of fiber loading on the mechanical properties of PLA-based hybrid composites was significant. As the fiber content increased, the tensile and flexural strengths of the composites generally improved, up to a certain point. For instance, composites with a higher percentage of SPF demonstrated enhanced mechanical properties due to the effective load transfer from the matrix to the fibers. This behaviour is consistent with the findings of previous studies, which indicated that the mechanical properties of natural fiber-reinforced composites improve with increased fiber loading, as the fibers provide additional reinforcement [12].

However, it was observed that beyond an optimal fiber loading, the mechanical properties began to decline. This phenomenon can be attributed to several factors, including fiber agglomeration and inadequate wetting of the fibers by the PLA matrix. For example, in the study by Sharma et al (2023), it was reported that increasing fiber loading beyond 30 wt% resulted in diminished flexural strength due to poor interfacial adhesion and increased fiber-fiber interactions which hindered stress transfer, indicating that there is a critical balance to be maintained in fiber loading to optimize mechanical properties [12].

3.1.3 Infill density

Infill density also played a crucial role in determining the mechanical properties of the printed PLA-based hybrid composites [13]. The results indicated that increasing the infill density from 50 %, 70 % and 100 % led to a marked overall improvement in both tensile and flexural strengths. This enhancement can be attributed to the increased material volume within the printed structure, which provides greater resistance to deformation and failure under load.

The relationship between infill density and mechanical properties is supported by the findings of Tanveer et al. (2022), who noted that higher infill densities contribute to improved stiffness and strength due to the increased amount of material resisting applied loads [13]. Conversely, lower infill densities resulted in reduced mechanical properties, likely due to the presence of voids and less material to distribute the applied stresses effectively. The findings align with the general consensus in the literature that optimizing infill density is essential for achieving desired mechanical performance in 3D-printed composites [14].

The Figure 4 displays a bar chart comparing the tensile strength of three different PLA composite blends at varying infill percentages 50 %, 70 %, and 100 %. For the 75%SPF:25%WTR blend, the tensile strength values are relatively consistent across infill percentages: 38.84 MPa at 50 % infill, 38.37 MPa at 70 % infill, and 37.89 MPa at 100 % infill. The 50%SPF:50%WTR blend shows an increasing trend with infill percentage 26.05 MPa at 50 %, 35.17 MPa at 70 %, and 36.94 MPa at 100 %. The 25%SPF:75%WTR blend also demonstrates an increase in tensile strength with infill percentage, but with a more pronounced effect, 29.47 MPa at 50 %, 34.79 MPa at 70 %, and 36.84 MPa at 100 %. As the infill density increases, the distribution and interaction of the rubber particles within the PLA matrix may lead to a less uniform stress distribution. This can result in increased voids or defects at higher infill levels, which act as stress concentrators and reduce overall strength.

Notably, the 75%SPF:25%WTR blend maintains the highest tensile strength across all infill percentages, while the other two blends show more variability. The 50 %SPF:50%WTR and 25%SPF:75%WTR blends exhibit lower tensile strengths at lower infill percentages but approach similar values to the 75%SPF:25%WTR blend at 100 % infill. This suggests that the ratio of SPF/WTR and the infill percentage both play significant roles in determining the tensile strength of these PLA composites.

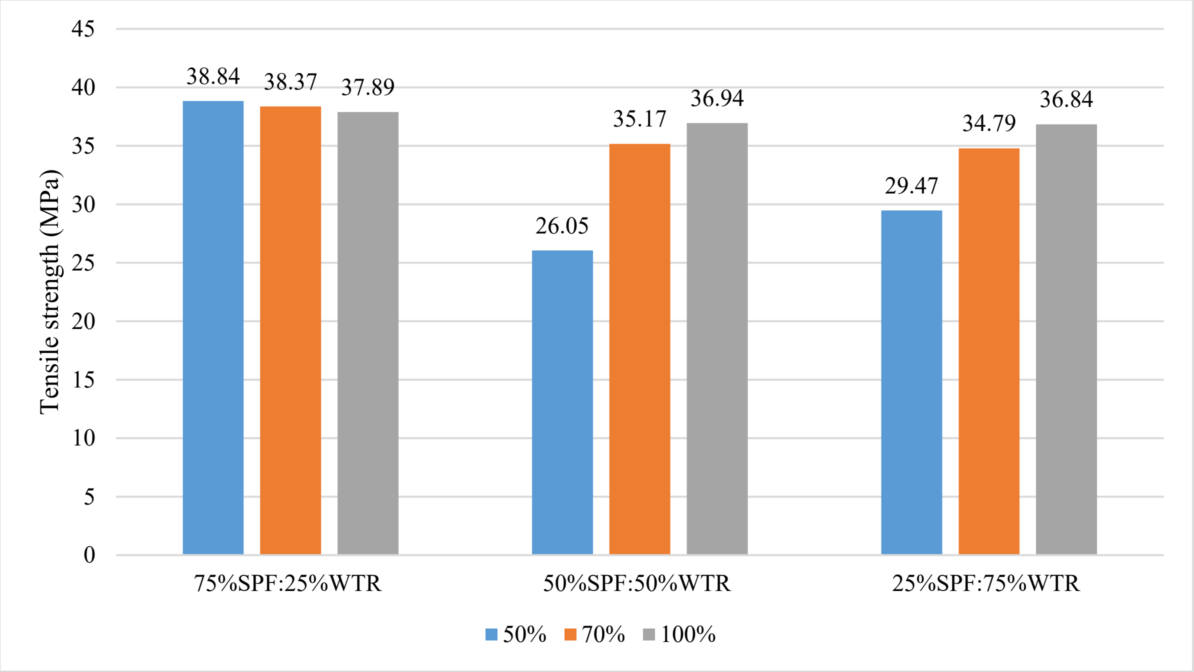

Figure 4: Effect of infill density on tensile properies of hybrid fibre reinforced PLA composites

Figure 5 displays a bar graph illustrating flexural strength measurements across different material compositions and densities. The graph compares three composites ratios of SPF and WTR at 75 %:25 %, 50 %:50 %, and 25 %:50 % respectively. For each mixture, the flexural strength is measured at three density levels: 50 %, 70 %, and 100 %.

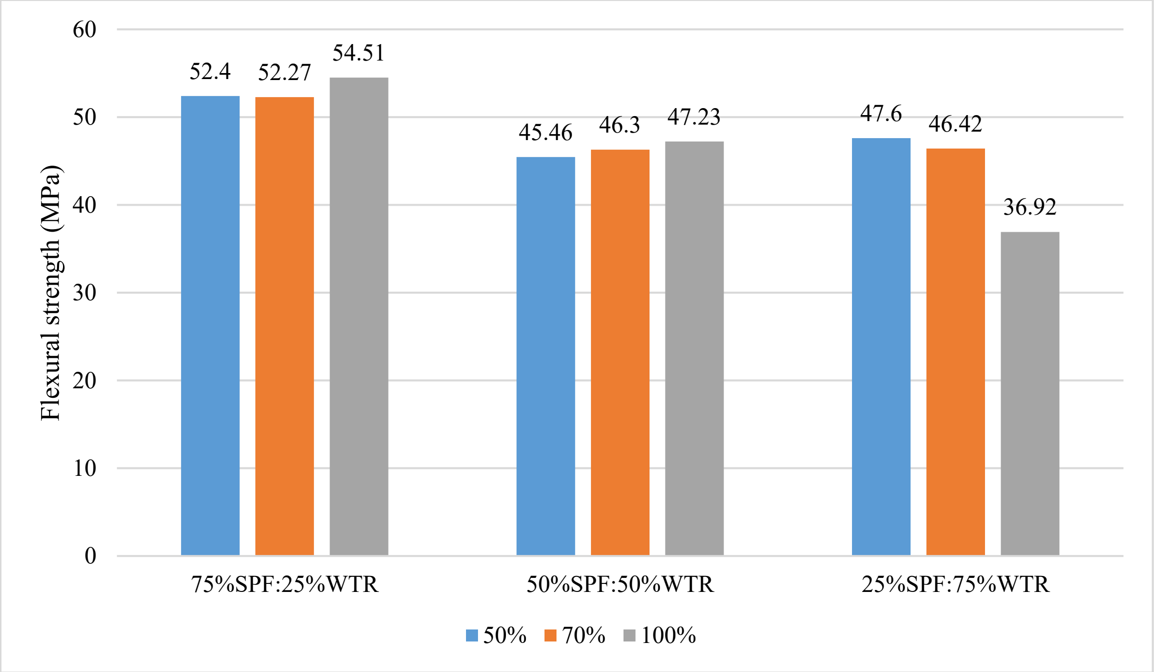

Figure 5: Effect of infill density on flexural properies of hybrid fibre reinforced PLA composites

The 75%SPF:25%WTR mixture shows consistently high flexural strength across all densities, ranging from 52.4 to 54.51 MPa. The strength increases slightly with density, peaking at 100 %. The 50%SPF:50%WTR mixture demonstrates lower overall strength compared to the first mixture, with values from 45.46 to 47.23 MPa. It also shows a gradual increase in strength as density increases. Interestingly, the 25%SPF:75%WTR mixture exhibits a unique pattern. At 50 % and 70 % densities, its strength is 47.6 and 46.42 MPa respectively seems comparable to the 50%SPF:50%WTR mixture. However, at 100 % density, there's a significant decrease in strength to 36.92 MPa, due to issues like poor interfacial adhesion or excessive rubber content, resulting in less cohesiveness. The overall results suggest that higher proportions of SPF generally lead to greater flexural strength.

3.2 Morphological studies

Morphological analysis was conducted using Scanning Electron Microscopy (SEM) to investigate the interfacial adhesion after enduring the tensile test between the hybrid fibers and the PLA matrix. The treated SPF and WTR were well-dispersed within the PLA matrix, which is critical for achieving optimal mechanical properties. The uniform distribution of fibers minimizes voids and enhances load transfer efficiency between the matrix and the fibers [15]. The analysis clearly indicates the tensile properties of the composites, as illustrated in the accompanying Figures 6(a), 6(b) and 6(c).

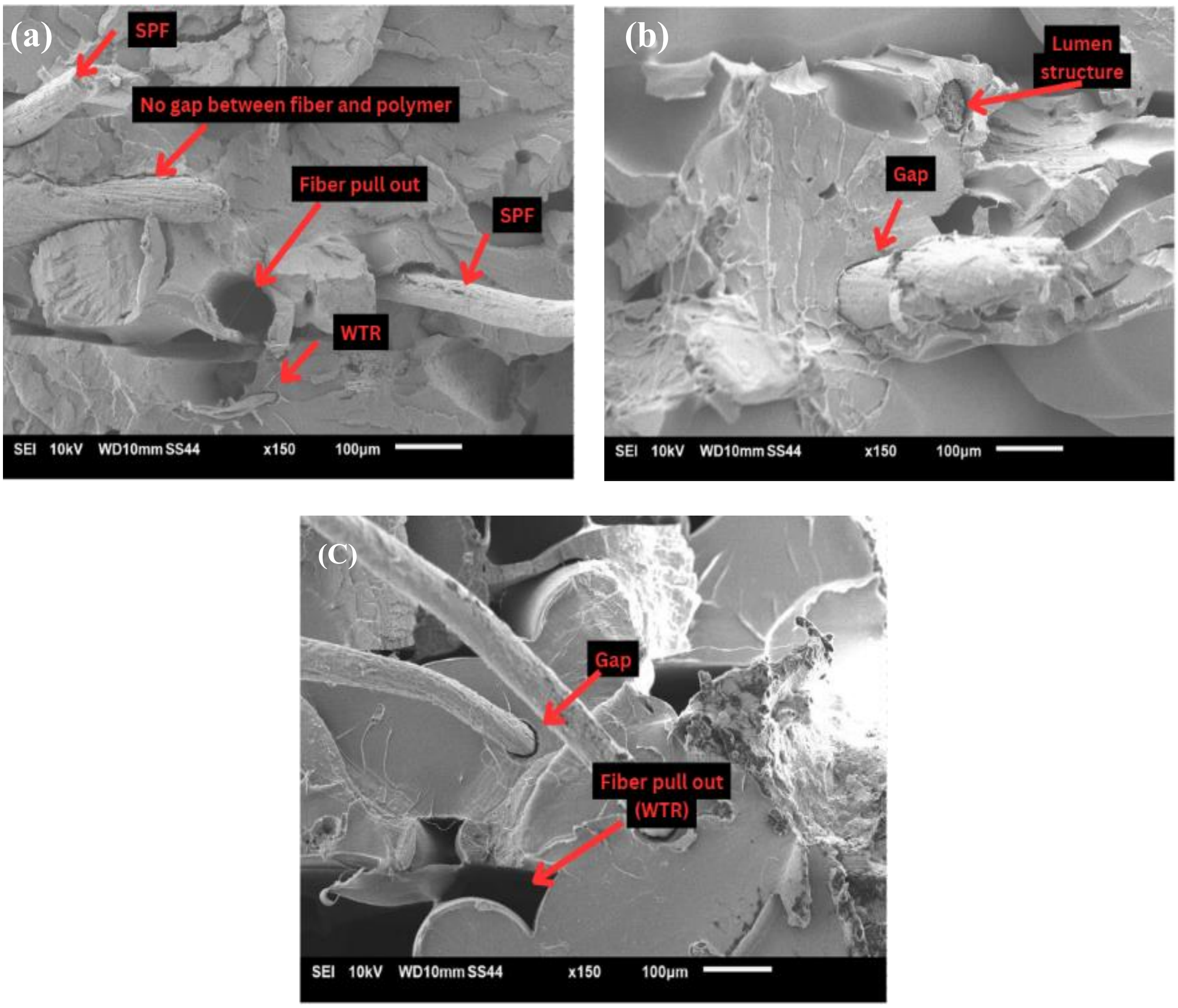

Figure 6: SEM micrographs of (a) PLA/75%SPF:25%WTR, (b) PLA/50%SPF:50%WTR and (c) PLA/25%SPF:75%WTR

Sample in Figure 6(a) demonstrates superior strength compared to the other composites, which can be attributed to the enhanced interfacial bonding between the fibers and the PLA matrix. This is evidenced by the minimal fiber pull-out observed in the fracture surfaces of Sample in Figure 6(a), in contrast to Samples Figure 6(b) and Figure 6(c) and these results support the tensile and flexural results, in which PLA/75%SPF:25%WTR had the best tensile and flexural properties. The effective chemical treatments with sodium hydroxide (NaOH) and silane have significantly improved the compatibility between the hydrophilic fibers and the hydrophobic PLA, resulting in enhanced mechanical performance [16].

In comparison, Sample Figure 6(b) exhibits some fiber pull-out, although it shows fewer gaps than Sample Figure 6(c). During the test, the fibers were easily pulled out due to gaps around the fibers, which indicated weak interfacial bonding between PLA and fibers [17]. Notably, Sample Figure 6(c) displays severe fiber pull-out, characterized by the presence of holes and flake-like shapes on the composite surface. This phenomenon can be attributed to the impurities present in fibers, which lead to weak interfacial bonding between the fibers and the polymer matrix [18]. Consequently, when load is applied, the fibers in Sample Figure 6(c) cannot effectively withstand the stress and detach from the matrix. To improve the performance of WTR in these composites, it is essential to enhance the chemical treatment processes. The current results indicate that the interfacial bonding in composites containing WTR is insufficient, which adversely affects their mechanical properties. Further optimization of the treatment methods may lead to better adhesion and overall performance of the hybrid composites.

4. CONCLUSIONS

The present study investigated the potential of developing sustainable and biodegradable PLA-based hybrid composites reinforced with Sugar Palm Fiber (SPF) and Waste Tyre Rubber (WTR). The incorporation of these natural and recycled materials aimed to enhance the mechanical properties and contribute to environmental sustainability by reducing waste and promoting the use of renewable resources. The comparative analysis of the hybrid composites with varying fiber loading ratios 75%SPF:25%WTR, 50%SPF:50%WTR, and 25%SPF:75%WTR revealed that the optimal combination for tensile strength was the 75%SPF:25% WTR composite 37.89 MPa, mirror to the 75%SPF:25%WTR composite exhibited the highest flexural strength 54.51 MPa. These findings highlight the importance of balancing the stiffness provided by the natural fibers and the toughness contributed by the rubber to achieve desired mechanical performance. The morphological studies conducted through Scanning Electron Microscopy (SEM) validated the positive impact of the chemical treatments of 6 % NaOH and 3 % silane on the interfacial adhesion between the fibers and the PLA matrix. The treated fibers exhibited uniform dispersion within the matrix and enhanced compatibility, which facilitated improved load transfer and contributed to the overall mechanical performance of the composites. These findings underscore the importance of effective fiber treatment in optimizing the properties of PLA-based hybrid composites, ultimately leading to more robust and sustainable material solutions for various applications. The results of this study demonstrate the potential of PLA-based hybrid composites reinforced with SPF and WTR as sustainable alternatives to conventional petroleum-based materials. By leveraging the advantages of natural fibers and recycled rubber, these composites offer enhanced mechanical performance while contributing to environmental sustainability through the use of renewable resources and waste reduction in material science and engineering.

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Author Contributions

Batrisyia Norhazlin: Writing – original draft, Methodology, Software, Data curation, Formal Analysis Investigation. Nadlene Razali: Conceptualization, Methodology, Validation, Proofreading. Mohd Adrinata Shaharuzaman: Methodology, Writing – Review & Editing.

Disclosure of Conflict of Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Compliance with Ethical Standards

The work is compliant with ethical standards.

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