

Article

Effect of Plasma Surface Treatment and Hybrid Fibers on Polypropylene Composites

Pablo Mazón-Ortiz ^{1,2} , Gabriel Mazón-Ortiz ^{3,4} , Luis Quishpe-Quishpe ^{3,4} , Bryan Rosero-Ortiz ⁴ 
and Cristina E. Almeida-Naranjo ^{5,*} 

- ¹ Departamento de Ingeniería Civil, Construcciones, E.T.S de Ingenieros de Caminos, Canales y Puertos, Universidad Politécnica de Madrid, C/Profesor Aranguren, 3, 28040 Madrid, Spain; pablo.mazon.ortiz@upm.es
- ² Facultad de Ingeniería en Ciencias de la Tierra (FICT), Escuela Superior Politécnica del Litoral, ESPOL, Campus Gustavo Galindo Km 30.5 Vía Perimetral, Guayaquil 090112, Guayas, Ecuador
- ³ Grupo de Investigación en Biomimética Amazónica y Nanotecnología (GIBAN), Universidad Regional Amazónica Ikiam, Vía Muyuna—Alto Tena km 7, Tena 150150, Napo, Ecuador; gabriel.mazon@ikiam.edu.ec (G.M.-O.); luis.quishpe@ikiam.edu.ec (L.Q.-Q.)
- ⁴ Universidad Regional Amazónica Ikiam, Vía Muyuna—Alto Tena km 7, Tena 150150, Napo, Ecuador; bryan.rosero@ikiam.edu.ec
- ⁵ Grupo de Biodiversidad Medio Ambiente y Salud (BIOMAS), Facultad de Ingeniería y Ciencias Aplicadas, Universidad de Las Américas, Redondel del Ciclista Antigua Vía a Nayón, Quito 170124, Pichincha, Ecuador
- * Correspondence: cristina.almeida@udla.edu.ec

Abstract

Thermoplastic hybrid composites reinforced with flax and glass fibers offer a sustainable, high-performance alternative for structural applications by balancing stiffness and energy absorption. This study investigated the impact of low-pressure plasma treatment on the thermal, mechanical, and microstructural properties of two polypropylene-based laminate configurations, PFGFP (polypropylene–flax–glass–flax–polypropylene) and PFGGFP (polypropylene–flax–glass–glass–flax–polypropylene), to optimize fiber–matrix interfacial adhesion. Materials were characterized using differential scanning calorimetry (DSC), thermogravimetric analysis (TGA), tensile testing, and scanning electron microscopy (SEM). The plasma treatment significantly enhanced the lignocellulosic fibers’ surface energy, reducing the flax contact angle from 93.5° to 56.1°. DSC analysis revealed a matrix crystallinity of 35.41%, while TGA confirmed flax thermal stability up to 250 °C. The PFGFP configuration exhibited superior mechanical performance (Tensile strength = 61.69 MPa; Young’s modulus = 518.62 MPa), attributed to its symmetric architecture and efficient fiber impregnation. Conversely, PFGGFP showed reduced strength and microstructural voids due to incomplete wetting in dense reinforcement regions. These findings conclude that the synergy between plasma surface modification and optimized laminate architecture is critical for the design of high-performance sustainable composites, providing an objective basis for improving interfacial compatibility in hybrid systems.



Academic Editor: Kadir Bilisik

Received: 15 January 2026

Revised: 13 February 2026

Accepted: 16 February 2026

Published: 20 February 2026

Copyright: © 2026 by the authors.

Licensee MDPI, Basel, Switzerland.

This article is an open access article distributed under the terms and conditions of the [Creative Commons Attribution \(CC BY\)](https://creativecommons.org/licenses/by/4.0/) license.

Keywords: polypropylene composites; flax fiber; plasma surface treatment; interfacial adhesion

1. Introduction

The global demand for high-performance composite materials has experienced a transformative shift towards sustainability and functional integration. Beyond traditional structural benefits such as high specific strength and corrosion resistance, modern composite design increasingly focuses on biomimetic architectures. By emulating the hierarchical

structures found in natural materials like wood and bone [1,2], researchers can tailor microstructural interfaces to optimize mechanical response. However, translating these naturally optimized interfacial mechanisms into synthetic thermoplastic systems remains a major scientific and technological challenge.

The synergistic performance of composites is fundamentally governed by the efficiency of the interface between their distinct constituents. In natural systems, such as the cellulose–lignin complex in wood or the hydroxyapatite–collagen arrangement in teeth, the exceptional mechanical properties arise from a highly engineered interfacial bonding [3–5]. Emulating this chemical and physical compatibility in synthetic thermoplastic composites remains a significant challenge. While the incorporation of lignocellulosic fibers offers environmental benefits and high specific stiffness, the inherent incompatibility between the hydrophilic nature of plant fibers and the non-polar, hydrophobic character of polymer matrices like polypropylene (PP) often leads to poor stress transfer and premature failure [6,7]. Consequently, surface modification strategies are required to bridge this interfacial gap, moving beyond simple material combination toward true structural integration [7].

In fibrous composites, the strategic exploitation of anisotropy allows for the precise tailoring of mechanical properties along principal load paths. While the reinforcement phase, the discontinuous phase provides the primary load-bearing capacity, its efficiency is intrinsically linked to the morphological and chemical compatibility with the continuous matrix phase [8–10]. In hybrid systems combining natural fibers like flax with technical reinforcements like glass, this anisotropy is further complicated by the divergent surface energies of the constituents [11]. The disparate nature of these reinforcements necessitates an optimized laminate architecture to prevent premature delamination or stress concentration at the interfaces [12]. Consequently, the development of advanced hybrid laminates requires not only a focus on fiber orientation but also a fundamental modification of the fiber–matrix boundary to ensure a cohesive anisotropic response under external loads [13].

Within the framework of sustainable materials design, lignocellulosic reinforcements—particularly flax fibers—have emerged as a compelling alternative to traditional glass fibers. Flax offers a favorable ecological footprint characterized by low density, widespread availability, and biodegradability, alongside competitive specific mechanical properties (≈ 1200 MPa strength, $\approx 2\%$ strain at failure, ≈ 60 GPa modulus) [14–16]. Despite these advantages, the industrial adoption of flax in high-performance structural applications is often hindered by its inherent hydrophilic nature and significant batch-to-batch variability [17]. In Europe, where flax and hemp dominate the natural fiber market due to their established textile infrastructure, current research is pivoting towards hybridizing these bio-fibers with technical reinforcements [18,19]. This hybridization aims to mitigate the environmental-mechanical trade-off, though it introduces new complexities regarding interfacial compatibility that must be addressed through targeted surface modifications [18].

The structural performance of fiber-reinforced composites is fundamentally governed by the efficiency of stress transfer across the fiber–matrix interface [20]. To optimize this boundary, different surface modifications have been explored, with physical pretreatments proving particularly effective in enhancing wettability and mechanical interlocking without compromising the fiber's bulk chemical integrity [21–24]. Among these, cold plasma treatment has emerged as a high-efficiency, dry-process alternative to traditional chemical etching. Previous studies using helium, nitrogen, and sulfur hexafluoride (SF_6) plasmas have demonstrated significant increments (up to 25%) in tensile strength and elastic modulus by inducing surface functionalization and reducing fiber polarity [25–31]. However, while the benefits of plasma on individual fiber–matrix adhesion are well-documented, the synergistic effect of combining plasma-treated natural fibers with technical reinforcements in specific laminate architectures remains under-explored. Recent advances in hybridization

suggest that placing technical fibers as skin layers can mitigate environmental degradation [32,33], yet the microstructural implications of stacking sequences in plasma-activated systems require further investigation to ensure industrial reliability [34–36].

Considering these advancements, the present study aims to contribute to the development of sustainable hybrid composites by analyzing the influence of plasma surface treatment on flax–PP interfacial adhesion and its interaction with distinct laminate architectures. Specifically, this work evaluates the synergistic effect of Argon low-pressure plasma (LPP) and stacking sequence on the wettability, thermal stability, and mechanical performance of the resulting composites. By correlating the thermodynamic surface modifications with the macroscopic rheological limitations observed during compression molding, this study seeks to define the processing–structure–property relationships necessary to optimize high-performance hybrid thermoplastics

2. Materials and Methods

2.1. Materials

The materials employed in this study were selected based on their commercial availability and suitability for thermoplastics composite manufacturing. A thermoplastic PP matrix was combined with natural and synthetic fibrous reinforcements.

The matrix phase consisted of a commercial-grade semicrystalline thermoplastic PP (PP089Y1E, batch AK-2210101, ISPLEN®, Repsol, Madrid, Spain). This polymer is characterized by a linear structure and a melt flow index of 31 g/10 min (2.16 kg/230 °C), ensuring optimal processability and fiber impregnation during compression molding. The reinforcement system comprised a unidirectional flax fabric (Bio-tex, 275 g/m², Easy Composites Ltd., Stoke-on-Trent, UK) and an E-glass fiber fabric (Roving 300, 295 g/m², commercial fiber). Flax fibers were used to investigate the effect of surface modification on the lignocellulosic–polymeric interface, whereas glass fibers were employed as received to provide structural stiffness and balance the mechanical response of the hybrid laminates.

2.2. Plasma Treatment of Flax Fibers

To enhance the flax–PP compatibility, the flax fabric was subjected to LPP treatment using a Harrick Plasma Cleaner (model PDC-002, Harrick Plasma, Ithaca, NY, USA; 220 V, 50 Hz) with argon as the process gas. Prior to plasma ignition, the chamber was evacuated using the connected vacuum pump and subsequently backfilled with argon to stabilize the working pressure at 300 mTorr. Although optical emission spectroscopy (OES) was not performed, the system operates within the low-pressure range required by the manufacturer (≤ 200 mTorr ultimate pressure capability), and we therefore refer to the process as argon low-pressure plasma while acknowledging that trace residual gases cannot be fully excluded. The chamber pressure was stabilized at 300 mTorr, followed by plasma exposure at 30 W for 60 s per side of the fabric. To treat both sides, the fabric was flipped between the two exposures, which involved brief ambient-air contact during sample handling prior to the subsequent evacuation and plasma exposure. This vacuum-based technique was selected over atmospheric plasma to prevent thermal/structural degradation of the natural fibers [13].

2.3. Composite Manufacturing

Hybrid composites were manufactured using a two-step compression molding process. The formulation for all samples was maintained at a constant mass ratio of 20 wt.% reinforcement and 80 wt.% PP matrix in order to balance stiffness, toughness, and processability, following trends reported for PP/flax/glass hybrid composites [11].

2.3.1. Preparation of PP Films

PP films were first produced as a semi-finished product using a $200 \times 200 \times 1.3$ mm steel mold charged with 46.8 g of PP pellets. Compression molding was performed in a Fontune TP 400 press, following a thermal cycle consisting of heating from 18 °C to 190 °C over 14 min, an isothermal stage of 14 min at 40 kN, and a controlled cooling period of 12 min.

2.3.2. Laminate Consolidation and Stacking Sequence

PP films and reinforcements were cut to mold dimensions. Individual fiber layers had average masses of 11.6 g (flax) and 11.8 g (glass). Laminate consolidation was performed at a maximum temperature of 195 °C and a compressive force of 50 kN, following the established heating and cooling ramps. The PFGFP laminate (PP–Flax–Glass–Flax–PP) represents a symmetric hybrid architecture expected to favor homogeneous impregnation and balanced through-thickness stress transfer. In contrast, the PFGGFP laminate (PP–Flax–Glass–Glass–Flax–PP) intentionally increases reinforcement packing density by adding an additional glass layer in the core, creating a more stringent impregnation scenario. This design enables evaluating whether plasma-induced improvements in flax/PP wettability translate into mechanical benefits when the laminate approaches the rheological and consolidation limits of molten PP during compression molding. Tensile specimens were subsequently machined from different regions of the manufactured laminate plates to capture intra-plate variability and support repeatability assessment at the specimen level.

2.4. Characterization of Materials

2.4.1. Wettability and Contact Angle Analysis

Interfacial interaction between the PP matrix and the reinforcements was evaluated using the sessile drop method in a Krüss DSA10 HT system (20000609), equipped with a high-temperature furnace, an optical monitoring camera, and a specialized illumination system. Flax and E-glass fiber samples were placed on a heated platform, where PP pellets were deposited onto the fabric surfaces. The system was heated at a controlled rate of 5 °C/min until reaching an isothermal state between 180 °C and 200 °C, the range in which the polymer reaches its molten state and establishes contact with the reinforcements.

Wettability was assessed under three specific conditions: as-received E-glass fiber, untreated flax fabric, and plasma-treated flax fabric. The contact angle was measured and quantified using DSA1 software (Krüss GmbH, DSA1 v1.90, Hamburg, Germany) to determine the effective surface energy and identify the hydrophilic or hydrophobic nature of the substrates. This analysis provided a critical baseline to evaluate the effectiveness of surface modifications on interfacial adhesion and stress transfer. Contact angle values are reported as mean $\pm$ standard deviation from repeated sessile-drop measurements under identical thermal conditions.

2.4.2. Thermal Analysis

Thermal transitions of the PP matrix were analyzed by DSC using a Mettler Toledo DSC822. A 9.53 mg sample was placed in a 40 μ L aluminum crucible with a 50 μ m perforated lid and heated at a rate of 10 °C/min under a nitrogen atmosphere (80 mL/min). This analysis determined the melting temperature (T_m), enthalpy of fusion, and degree of crystallinity to define the optimal processing window.

Moreover, thermal stability of the flax fibers was evaluated by TGA using a Perkin Elmer Pyris1 system. Samples were heated from 30 °C to 400 °C at 10 °C/min under an inert atmosphere. This analysis identified the moisture desorption stage (~100 °C) and the onset of lignocellulosic degradation (~250 °C), defining safe processing limits for composite manufacturing. DSC and TGA are reported as representative screening measurements to

define the processing window (PP melting/crystallinity and flax thermal stability) and were not intended for statistical comparison; replicate counts are not reported due to limited traceability of archived run logs.

2.5. Mechanical and Morphological Characterization

2.5.1. Tensile Testing

Uniaxial tensile tests were conducted following the ASTM D3039 standard to determine the mechanical properties of the neat PP and the hybrid composites. The specimens were tested using a Microtest EM/2FR universal testing machine equipped with a 20 kN load cell at a constant crosshead speed of 10 mm/min. The stress–strain curves were used to determine the ultimate tensile strength ($\sigma_{\max}$), Young's modulus (E) calculated from the linear region, and tensile toughness, estimated as the area under the curve until fracture. A total of $n = 17$ specimens were tested for neat PP, $n = 14$ for PFGFP, and $n = 13$ for PFGGFP. Reported values correspond to mean $\pm$ standard deviation.

2.5.2. Scanning Electron Microscopy (SEM)

Fracture surface morphology was analyzed using a Philips XL-30 scanning electron microscope (Eindhoven, The Netherlands) operated at an acceleration potential of 10–15 kV. Samples were extracted from the failure zones of the tensile-tested specimens to evaluate fiber–matrix adhesion, impregnation quality, and internal cohesion. Prior to observation, specimens were sputter-coated with a thin gold/palladium layer to ensure electrical conductivity and prevent charging effects under the electron beam.

Figure 1 presents a schematic overview of the sequential steps involved in the composite manufacturing process.

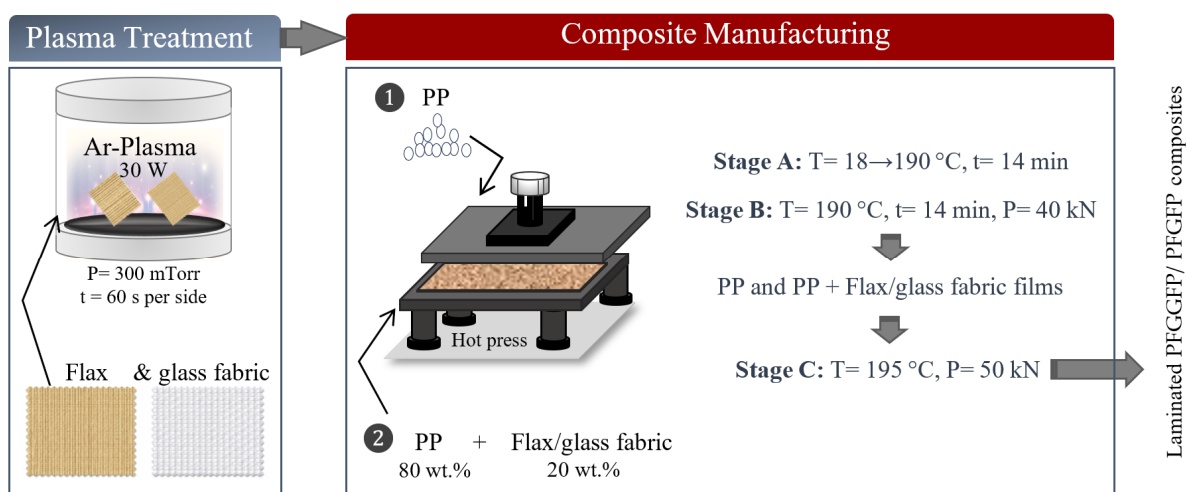


Figure 1. Schematic sequence of the composite manufacturing process.

3. Results

3.1. Contact Angle and Surface Wettability

The results of the contact angle measurements for the different reinforcements are presented in Figure 2. The E-glass fiber showed an average contact angle (θ) of $67.2^{\circ} \pm 9.6^{\circ}$. The untreated flax fabric exhibited the highest contact angle at $93.5^{\circ} \pm 8.2^{\circ}$, indicating limited wetting of molten PP on the fiber surface (reduced spreading and interfacial contact area). According to the Young–Dupré relationship, a higher θ corresponds to a lower work of adhesion, which is typically associated with weaker interfacial interactions and less efficient stress transfer. Following the LPP treatment, the contact angle of the flax fabric

decreased to $56.1^\circ \pm 6.2^\circ$, representing a 40% reduction in the measured angle compared to its untreated state.

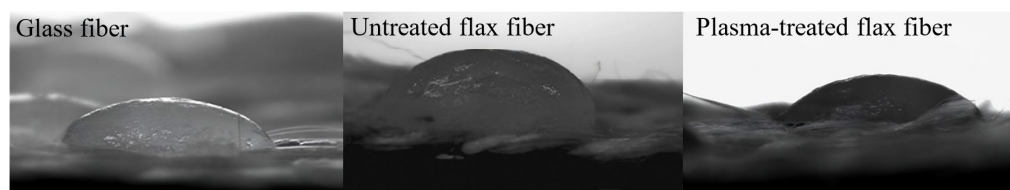


Figure 2. Contact angle measurement between molten polypropylene and the reinforcements.

3.2. Thermal Analysis (DSC and TGA)

The DSC thermogram of the PP matrix is presented in Figure 3a. A minor heat flow deviation was observed in the 60–70 °C range. A prominent endothermic peak was identified between 110 °C and 180 °C, with a melting temperature (T_m) peak at 165 °C. The enthalpy of fusion (ΔH_m) calculated from the area under the curve was 73.30 J/g, resulting in a degree of crystallinity (X_c) of 35.41%.

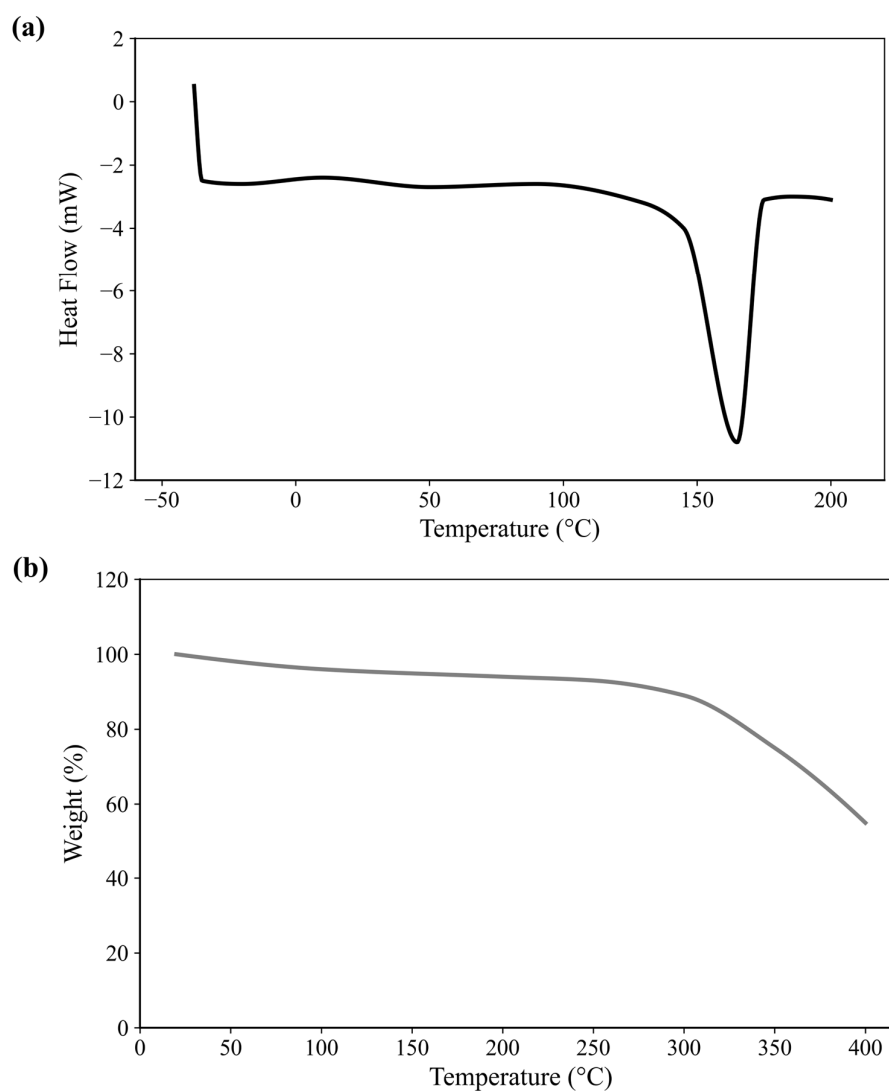


Figure 3. Thermal behavior of the composite constituents. (a) DSC curve of PP, showing the melting transition, and (b) TGA of flax fiber, illustrating initial moisture evaporation and thermal degradation at elevated temperatures (>250 °C).

Regarding the flax fiber, the TGA curve (Figure 3b) shows two distinct mass loss stages. The first stage occurs at approximately 100 °C, showing a slight decrease associated with moisture desorption. The second and more significant degradation stage begins at 250 °C, where a sharp drop in mass is recorded.

3.3. Mechanical Properties

The tensile properties of the neat PP and the hybrid composites (PFGFP and PFGGFP) are summarized in Figure 4. Neat PP exhibited the lowest tensile strength, with a value of 24.47 MPa. In contrast, the PFGFP laminate achieved the highest tensile strength (61.69 MPa), while the PFGGFP configuration showed an intermediate value of 45.15 MPa.

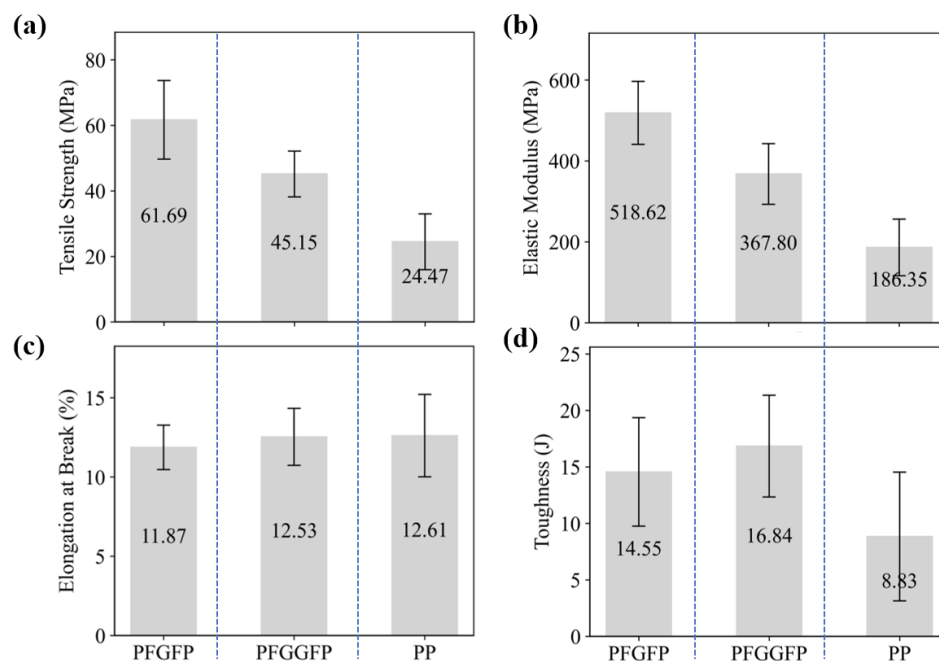


Figure 4. Mechanical performance of the evaluated configurations. (a) Tensile strength, (b) Young Modulus, (c) Elongations at break, and (d) Toughness. The comparison illustrates the effect of laminate stacking sequence and reinforcement type on stiffness, strength, and impact-related energy absorption.

A significant increase in stiffness was observed with fiber reinforcement. The Young's modulus increased from 186.35 MPa for neat PP to 518.62 MPa for PFGFP and 367.80 MPa for PFGGFP. Despite the differences in strength and stiffness, all materials exhibited comparable ductility. Neat PP showed an elongation at break of 12.61%, followed by PFGGFP (12.53%) and PFGFP (11.87%).

Regarding energy absorption, neat PP exhibited the lowest tensile toughness (8.83 J). Both hybrid composites showed a substantial improvement, with values of 14.55 J for PFGFP and a maximum toughness of 16.84 J for the PFGGFP configuration.

The comparative analysis presented in Table 1 places the mechanical performance of the present hybrid laminates within the broader context of fiber-reinforced polypropylene systems reported in the literature. It is important to note that direct comparisons must be interpreted cautiously, as different studies report distinct strength metrics (e.g., yield strength, ultimate tensile strength, or axial strength) and employ diverse processing routes and reinforcement architectures.

Table 1. Comparative mechanical performance of fiber-reinforced polypropylene composites reported in the literature and present study.

Composite System	Reinforcement Architecture/Processing	Tensile Strength (MPa)	Strength Type	Young's Modulus (GPa)	Elongation at Break (%)	References
Neat PP	Compression-molded laminate	24.47	$\sigma_{\max}$	0.186	12.61	Present study
PFGFP	Hybrid laminate (PP–Flax–Glass–Flax–PP)	61.69	$\sigma_{\max}$	0.519	11.87	Present study
PFGGFP	Hybrid laminate (PP–Flax–Glass–Glass–Flax–PP)	45.15	$\sigma_{\max}$	0.368	12.53	Present study
PP + Glass fiber (G40)	Extrusion + compression molding	25.8	Yield strength	0.738	7.1	[11]
PP + Flax fiber (F40)	Extrusion + compression molding	19.1	Yield strength	0.473	7.4	
PP + Glass/Flax (G30F10)	Hybrid composite	25.5	Yield strength	0.629	9	
PP + Glass/Flax (G10F30)	Hybrid composite	24.2	Yield strength	0.589	7.2	
PP + Flax fabric (untreated)	Laminated composite	66.84	UTS	1.88	3.28	[28]
PP + Flax fabric (plasma treated)	Laminated composite	71.7	UTS	2.42	2.3	
UD Flax/PP (filament wound)	Unidirectional laminate	251–321	Axial strength	27–29	-	[20]
PP/PB + S-Glass + Flax hybrid composite	-	26.54	$\sigma_{\max}$	1.38	4.21	[37]

Compared with extrusion-based hybrid systems such as those reported by Ghasemzadeh-Barvarz et al. [11], the laminated PFGFP configuration developed in the present study exhibits superior ultimate tensile strength despite a lower overall modulus, which can be attributed to improved load transfer efficiency achieved through plasma-enhanced interfacial compatibility and controlled laminate stacking [25–31]. In contrast, unidirectional filament-wound flax/PP laminates reported by Malkapuram et al. [20] demonstrate substantially higher axial strength and modulus due to fiber alignment and high fiber volume fractions, highlighting the strong influence of reinforcement orientation on mechanical response [13].

When compared to plasma-treated flax fabric laminates reported by Leone et al. [28], the present results show competitive tensile strength values while incorporating a hybrid glass–flax architecture, demonstrating that hybridization can balance stiffness and damage tolerance. Additionally, recent hybrid thermoplastic systems incorporating glass and flax fibers [37] generally exhibit lower tensile strength than the optimized PFGFP laminate presented here, reinforcing the importance of architecture design and interfacial engineering [7,12].

Overall, Table 1 confirms that the mechanical performance achieved in this study is consistent with, and in several cases competitive with, previously reported fiber-reinforced polypropylene composites, while emphasizing that laminate architecture and interfacial optimization play a more decisive role than reinforcement type alone.

3.4. Morphological Analysis (SEM)

The microstructural characteristics of the fractured surfaces for PFGGFP and PFGFP laminated composites are shown in Figure 5. Low-magnification SEM micrographs confirm the sequential layer arrangement in both architectures. Higher-magnification views are provided to highlight local interfacial damage features; panels C and D are shown at different magnifications and therefore the comparison is qualitative and based on the scale bars and representative regions. Under these conditions, representative regions of the PFGGFP configuration reveal voids, fiber pull-out cavities, and partially detached reinforcement bundles, suggesting locally incomplete impregnation in regions with higher

reinforcement density. In contrast, the PFGFP laminate shows flax fibers well embedded within the polypropylene matrix, with a continuous and cohesive fiber–matrix interface, with no obvious interfacial voids or delamination at the observed scale.

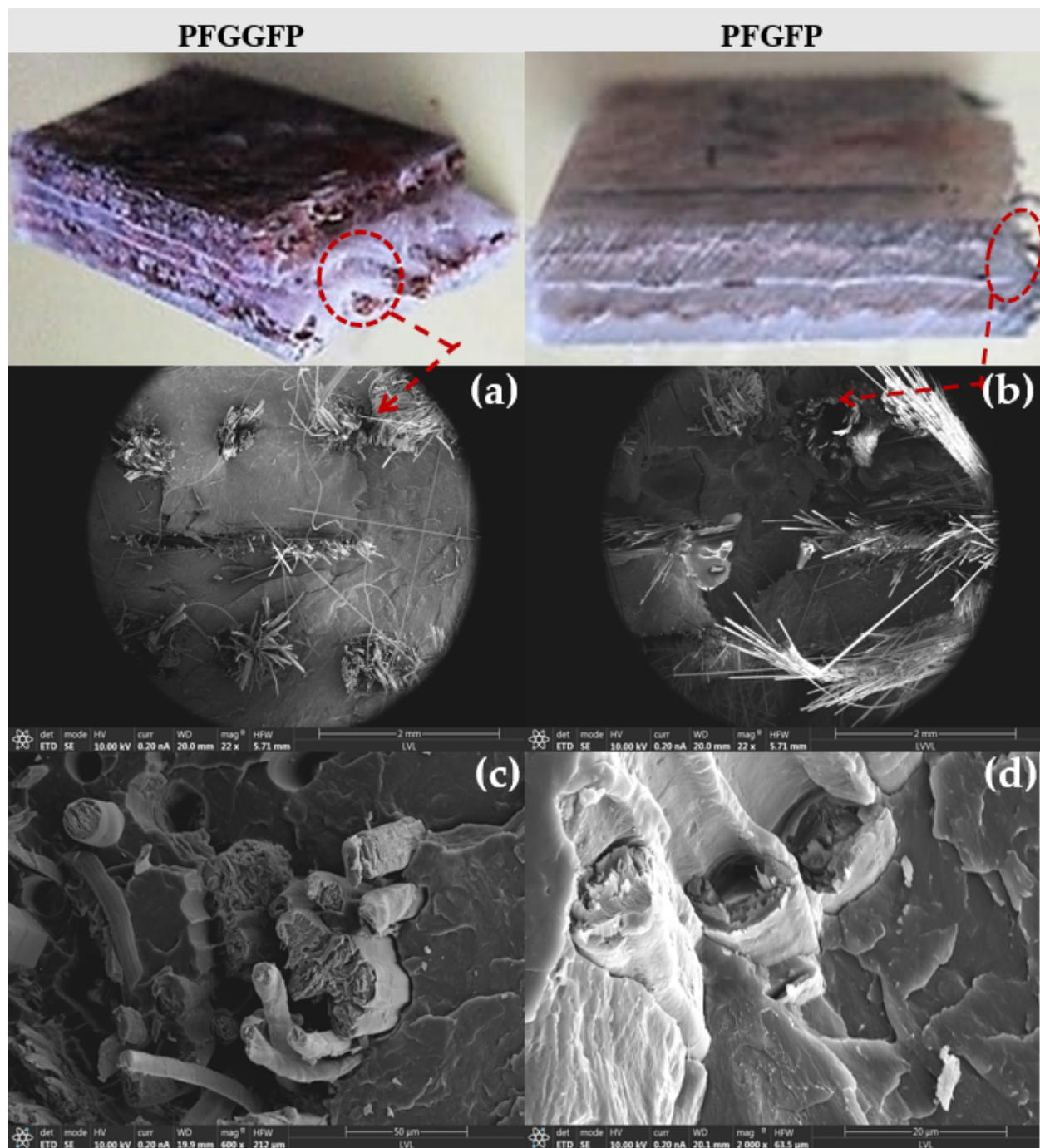


Figure 5. SEM images of the fractured surfaces of the composites at different magnifications, showing the laminate architecture and interfacial features. (a) PFGGFP at 22× magnification; (b) PFGFP at 22× magnification; (c) PFGGFP at 600× magnification; and (d) PFGFP at 2000× magnification.

4. Discussion

The substantial reduction in the contact angle of flax fibers ($\Delta\theta = 37.4^\circ$) confirms a thermodynamic shift from a hydrophobic to a hydrophilic state (Figure 2), which is critical for wetting. Although FTIR and XPS were not performed in this study, previous studies have shown that FTIR analysis of Argon plasma-treated lignocellulosic fibers typically

reveals limited chemical functionalization. Therefore, the enhanced wettability is mainly attributed to plasma-induced physical surface (surface cleaning and Ar^+ ion bombardment/physical etching) rather than to bulk chemical modifications. At the same time, since the fabric was treated on both sides (60 s per side), a brief handling interval between steps and the presence of trace residual gases ($\text{O}_2/\text{H}_2\text{O}$) may enable limited post-plasma oxidation confined to the outermost surface; thus, minor oxygen incorporation cannot be ruled out and would require XPS to be conclusively assessed. As demonstrated by Bapat et al. [13] on similar lignocellulosic fibers, Argon plasma induces specific nanotexturing through Ar^+ ion sputtering. This physical roughening increases the effective surface area, shifting the wetting regime towards the Wenzel state, thereby enhancing the surface free energy available for mechanical interlocking without compromising the bulk chemical structure. Furthermore, the selected parameters (30 W, 300 mTorr) fall within the “soft processing window” identified by Huh et al. [38], effectively avoiding the excessive etching that can degrade fiber tensile strength by up to 25% in natural fibers like jute.

As a direct consequence of this surface activation, plasma-treated flax fibers exhibit enhanced compatibility with the polypropylene matrix specifically during the early stages of composite consolidation. Improved wetting promotes more efficient polymer infiltration into the fiber bundles under compression molding conditions, facilitating interfacial continuity prior to laminate densification. In contrast, untreated flax reinforcements typically hinder thermoplastic flow at the fiber–matrix interface, limiting effective stress transfer [12,28]. Accordingly, the improved mechanical performance observed for the PFGFP configuration can be attributed not only to laminate symmetry, but also to the processing-stage advantages enabled by plasma-treated flax fibers, which mitigate the intrinsic incompatibility between lignocellulosic reinforcements and non-polar polypropylene matrices [12].

The divergent mechanical response between PFGFP (61.69 MPa) and PFGGFP (45.15 MPa) highlights the critical role of laminate architecture in thermoplastic impregnation (Figure 4a). Although quantitative porosity tests were not conducted, the presence of voids in the PFGGFP configuration is confirmed qualitatively via SEM (Figure 5c). This configuration, containing a double layer of glass fiber, evidenced a higher occurrence of interfacial debonding, attributed not only to structural complexity but also to a reinforcement volume that exceeded the matrix’s wetting capacity under the specific processing conditions employed. As noted by Rahman et al., [12] increasing the fiber packing density in laminate architectures often restricts matrix flow, leading to higher void content; these voids act as stress initiators that significantly reduce overall energy absorption and strength. Similarly, Ghasemzadeh-Barvarz et al. [11] reported that in hybrid PP/flax composites, poor impregnation within natural fiber bundles acts as a critical site for crack initiation. Consequently, while the plasma treatment successfully lowered the thermodynamic barrier for wetting, the PFGGFP architecture exceeded the rheological processing window of the matrix. These findings underscore the critical importance of optimizing processing parameters specifically pressure, temperature, and dwell time to guarantee homogeneous impregnation and efficient load transfer in high-density hybrid configurations [38]. The rheological limitations discussed herein are inferred from impregnation behavior and laminate morphology rather than direct rheometric measurements.

SEM observations further elucidate the dominant failure mechanisms associated with each laminate architecture. In the PFGGFP configuration, the fracture surfaces reveal extensive fiber pull-out cavities and partially detached reinforcement bundles, indicating that crack propagation preferentially occurred along poorly bonded interfaces rather than through the polymer matrix. This failure mode reflects inefficient stress redistribution under tensile loading and explains the premature loss of load-bearing capacity observed

for this configuration [11,12]. In contrast, the PFGFP laminate exhibits flax fibers uniformly embedded within the polypropylene matrix, with fracture paths frequently crossing the matrix and fiber surfaces, suggesting a more integrated interfacial response. This cohesive failure morphology supports the superior tensile strength of PFGFP and indicates more effective stress transfer across the interface [28].

This lack of homogeneous impregnation had a direct impact on stiffness. The PFGFP configuration achieved a Young's modulus of 518.62 MPa, representing a distinct improvement over the neat PP (186.35 MPa) (Figure 4b). Ideally, hybridization with glass fibers should linearly enhance stiffness. Ghasemzadeh-Barvarz et al. [11] demonstrated that replacing flax with glass fibers in a PP matrix consistently increases the tensile modulus (e.g., from 473 MPa for flax composites to 738 MPa for glass composites) due to the superior rigidity of the synthetic reinforcement. However, in this study, the PFGGFP configuration—despite containing a higher volume fraction of stiff glass fibers—exhibited a reduced modulus of 367.80 MPa. This deviation from the expected trend confirms that the voids and dry spots identified in the core reduced the effective cross-sectional area available for stress transfer, rendering the additional glass layers structurally inefficient under elastic loading [12].

Regarding toughness, the PFGGFP configuration achieved an average energy absorption of 16.84 J, compared to 14.55 J for PFGFP (Figure 4d). However, considering the standard deviations, no statistically significant difference was observed between the two hybrid configurations, although both significantly outperformed neat PP (8.83 J). This result is noteworthy because PFGGFP maintained high energy absorption despite its reduced tensile strength and stiffness. As noted by Rahman et al., [12] mechanisms such as fiber pull-out and frictional sliding in poorly bonded interfaces can compensate for the loss of structural integrity by dissipating energy. In the PFGGFP laminate, the extensive interfacial debonding observed (Figure 5c) likely acted as an energy dissipation mechanism [39], allowing the material to match the toughness of the superior PFGFP laminate despite the presence of macroscopic defects.

The thermal analysis validates the selected processing window (195 °C), as the degradation onset of flax fibers (~250 °C) remains well above the molding temperature (Figure 3a,b). Although the extended dwell time of 14 min approaches the thermal sensitivity threshold of hemicellulose—typically reported around 180 °C under sustained exposure [40]—the consolidated laminates exhibited no visible charring or discoloration. This stability suggests that the molten semicrystalline PP matrix ($T_m = 165$ °C) effectively acted as a thermal barrier, encapsulating the fibers and limiting oxidative degradation during the isothermal hold [6,40].

Beyond thermal stability considerations, thermal expansion behavior is also an important parameter for fiber-reinforced thermoplastic composites, particularly for applications involving dimensional stability and thermal cycling. The coefficient of linear thermal expansion (CTE) of polymers and polymer composite materials is governed by intrinsic matrix properties, reinforcement content, fiber orientation, and interfacial quality, as systematically reviewed in the literature [41].

Experimental studies on composites with varying reinforcement volume fractions have demonstrated that increasing the glass fiber content tends to reduce the effective CTE of the material, reflecting the lower thermal expansion associated with glass fibers relative to polymer matrices [42]. Furthermore, fiber orientation and distribution influence measured CTE values, with systems exhibiting strong alignment or reinforcement directionality showing distinct expansion responses compared to more isotropic arrangements [43].

Although direct CTE measurements were not conducted in the present work, the combined effects of reinforcement type, volume fraction, and laminate architecture provide

a sound physical basis for anticipating distinct thermal expansion behaviors between the PFGFP and PFGGFP configurations. These considerations are consistent with trends reported for fiber-filled thermoplastic systems in the recent literature, where reinforcement characteristics and microstructural organization significantly affect thermal dimensional stability [41–43].

From a degradation-mechanism standpoint, further insight is provided by a detailed analysis of the TGA response of the flax reinforcement. The TGA profile reveals a multistep degradation mechanism characteristic of lignocellulosic reinforcements: an initial minor mass loss below ~ 120 °C associated with moisture desorption, followed by the onset of hemicellulose decomposition at ~ 240 – 270 °C, which defines the upper thermal boundary for defect-free thermoplastic processing due to the risk of volatile release and interfacial porosity formation [44]. The dominant mass-loss event between ~ 340 and 370 °C corresponds to cellulose pyrolysis, indicating the temperature at which the fibrous network irreversibly loses its reinforcing function, while the gradual degradation above 400 °C is attributed to lignin decomposition and the main-chain scission of polypropylene [6]. From a technological perspective, these sequential degradation stages confirm that the selected molding temperature lies safely below the thermal decomposition thresholds of the flax constituents, ensuring homogeneous consolidation without fiber embrittlement or gas-induced voids, while also establishing a practical operational window: continuous service below ~ 120 °C with tolerance to short-term thermal excursions below ~ 200 °C, beyond which durability would be compromised by progressive hemicellulose degradation and loss of fiber–matrix cohesion [40].

Furthermore, the high degree of crystallinity observed (35.41%) indicates that the reinforcements influenced the crystallization kinetics of the PP matrix, suggesting the promotion of interfacial transcrystallinity, a phenomenon widely reported in natural-fiber-reinforced thermoplastics [13,16,45,46]. The plasma-induced nanotexturing increased the effective surface area of the flax fibers, enabling them to act as efficient heterogeneous nucleating agents [6,45,47,48], likely promoting a transcrystalline interphase in which polymer crystallites grow perpendicular to the fiber surface. This microstructural feature is strongly associated with enhanced interfacial shear strength and improved stress transfer, linking the observed thermal behavior directly to the mechanical performance and structural reliability of the composite in real processing and service conditions [48].

5. Conclusions

This study demonstrates that Argon low-pressure plasma treatment is an effective strategy to enhance the interfacial performance of polypropylene/flax/glass hybrid composites, provided that the laminate architecture remains compatible with the rheological processing window of the thermoplastic matrix. Plasma-treated flax fibers exhibited significantly improved wettability, as evidenced by the marked reduction in contact angle, which facilitated polymer infiltration and mechanical interlocking through a predominantly physical surface activation mechanism rather than chemical functionalization. The mechanical response of the composites was found to be strongly architecture-dependent: the symmetric PFGFP configuration achieved the most favorable balance between stiffness, strength, and toughness, confirming the effectiveness of the treated fiber–matrix interface, whereas the PFGGFP laminate suffered a reduction in tensile strength despite its higher glass fiber content due to impregnation limitations associated with increased reinforcement density. Microstructural analysis revealed that, although such impregnation defects reduced load-bearing efficiency, they also contributed to energy dissipation mechanisms that preserved high toughness in the PFGGFP configuration. Thermal analysis confirmed that the selected processing temperature ensured fiber integrity during consolidation, while

the high matrix crystallinity suggests a potential contribution of plasma-induced surface roughness to interfacial transcrystallinity in well-impregnated regions. Overall, the results highlight that overcoming the thermodynamic barrier to wetting through plasma treatment is a necessary but not sufficient condition to maximize composite performance; optimal properties are ultimately governed by the interplay between surface modification and processing-induced flow constraints. Future work will focus on extending the thermal and interfacial characterization through techniques such as X-ray photoelectron spectroscopy and thermal expansion analysis, as well as on optimizing processing parameters for high-density hybrid architectures, to further exploit the potential of plasma-treated natural fiber reinforcements in sustainable thermoplastic composites.

Author Contributions: Conceptualization, P.M.-O. and G.M.-O.; methodology, P.M.-O.; software, P.M.-O.; validation, P.M.-O.; formal analysis, P.M.-O., G.M.-O., L.Q.-Q., B.R.-O. and C.E.A.-N.; investigation, P.M.-O., G.M.-O., L.Q.-Q., B.R.-O. and C.E.A.-N.; data curation, P.M.-O.; writing—original draft preparation, P.M.-O., L.Q.-Q., B.R.-O. and C.E.A.-N.; writing—review and editing, P.M.-O., G.M.-O., L.Q.-Q., B.R.-O. and C.E.A.-N.; visualization, P.M.-O., L.Q.-Q., B.R.-O. and C.E.A.-N.; supervision, L.Q.-Q. and B.R.-O.; project administration, P.M.-O., G.M.-O.; funding acquisition, P.M.-O., G.M.-O., L.Q.-Q., B.R.-O. and C.E.A.-N. All authors have read and agreed to the published version of the manuscript.

Funding: This research was funded by Universidad Regional Amazónica Ikiam, project DIFCSA-001-2024, and by Universidad de Las Américas, grant number 584.A.XVII.25.

Data Availability Statement: The data supporting the findings of this study are available within the article. For further details, please contact the corresponding author.

Acknowledgments: The authors gratefully acknowledge the support provided by the research departments of Universidad Regional Amazónica Ikiam and Universidad de Las Américas, whose institutional backing was essential for the development of this study. In particular, the authors acknowledge the support of the Convocatoria de Proyectos Territorio Ikiam, which enabled the execution of the research activities at Ikiam.

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

References

1. Clyne, T.W.; Hull, D. *An Introduction to Composite Materials*, 3rd ed.; Cambridge University Press: Cambridge, UK, 2019.
2. Hueber, C.; Fischer, G.; Schwingshandl, N.; Schledjewski, R. Production Planning Optimisation for Composite Aerospace Manufacturing. *Int. J. Prod. Res.* **2019**, *57*, 5857–5873. [\[CrossRef\]](#)
3. Askeland, D.R. *Science and Engineering of Materials: SI Edition*; Cengage Learning: Boston, MA, USA, 2016.
4. Smith, W.; Hashemi, J. *Fundamentos de La Ciencia e Ingeniería de Materiales*, 4th ed.; McGraw-Hill Interamericana: México City, México, 2014.
5. Callister, W.D.; Rethwisch, D.G.; Molera, P.; Salán Ballesteros, M.N. *Ciencia e Ingeniería de Los Materiales*; Editorial Reverté: Barcelona, Spain, 2018.
6. Arbelaiz, A.; Fernández, B.; Ramos, J.A.; Mondragon, I. Thermal and Crystallization Studies of Short Flax Fibre Reinforced Polypropylene Matrix Composites: Effect of Treatments. *Thermochim. Acta* **2006**, *440*, 111–121. [\[CrossRef\]](#)
7. Arbelaiz, A.; Fernández, B.; Cantero, G.; Llano-Ponte, R.; Valea, A.; Mondragon, I. Mechanical Properties of Flax Fibre/Polypropylene Composites. Influence of Fibre/Matrix Modification and Glass Fibre Hybridization. *Compos. Part A Appl. Sci. Manuf.* **2005**, *36*, 1637–1644. [\[CrossRef\]](#)
8. Besednjak, A. *Materiales Compuestos*, 1st ed.; UPC: Barcelona, Spain, 2005.
9. AEMAC. *Materiales Compuestos*, 3rd ed.; Reverté: Barcelona, Spain, 2003.
10. Verma, D.; Gope, P.C.; Shandilya, A.; Gupta, A.; Maheshwari, M.K. Coir Fibre Reinforcement and Application in Polymer Composites: A Review. *J. Mater. Environ. Sci.* **2013**, *4*, 263–276.
11. Ghasemzadeh-Barvarz, M.; Duchesne, C.; Rodrigue, D. Mechanical, Water Absorption, and Aging Properties of Polypropylene/Flax/Glass Fiber Hybrid Composites. *J. Compos. Mater.* **2015**, *49*, 3781–3798. [\[CrossRef\]](#)
12. Rahman, M.Z.; Jayaraman, K.; Mace, B.R. Impact Energy Absorption of Flax Fiber-reinforced Polypropylene Composites. *Polym. Compos.* **2018**, *39*, 4165–4175. [\[CrossRef\]](#)

13. Bapat, K.; Kailas, L.; Pask, C.; Kee, T.P.; Russell, S. Study of Hemp Fiber Properties Modified via Long-Duration Low-Pressure Argon and Oxygen Plasma Treatments. *ACS Omega* **2025**, *10*, 61435–61451. [[CrossRef](#)]
14. Bourmaud, A.; Beaugrand, J.; Shah, D.U.; Placet, V.; Baley, C. Towards the Design of High-Performance Plant Fibre Composites. *Prog. Mater. Sci.* **2018**, *97*, 374–408. [[CrossRef](#)]
15. Bourmaud, A.; Morvan, C.; Bouali, A.; Placet, V.; Perré, P.; Baley, C. Relationships between Micro-Fibrillar Angle, Mechanical Properties and Biochemical Composition of Flax Fibers. *Ind. Crops Prod.* **2013**, *44*, 343–351. [[CrossRef](#)]
16. Charlet, K.; Jernot, J.P.; Eve, S.; Gomina, M.; Bréard, J. Multi-Scale Morphological Characterisation of Flax: From the Stem to the Fibrils. *Carbohydr. Polym.* **2010**, *82*, 54–61. [[CrossRef](#)]
17. Baley, C.; Gomina, M.; Breard, J.; Bourmaud, A.; Davies, P. Variability of Mechanical Properties of Flax Fibres for Composite Reinforcement. A Review. *Ind. Crops Prod.* **2020**, *145*, 111984. [[CrossRef](#)]
18. Zhu, J.; Zhu, H.; Njuguna, J.; Abhyankar, H. Recent Development of Flax Fibres and Their Reinforced Composites Based on Different Polymeric Matrices. *Materials* **2013**, *6*, 5171–5198. [[CrossRef](#)] [[PubMed](#)]
19. Margoto, O.H.; Milani, A.S. Comparing Flax Fibre/Biopolymer Woven Composites with Carbon Fibre-Enhanced, Partially Green Alternatives: Mechanical Performance versus Sustainability. *Compos. Part C Open Access* **2025**, *16*, 100547. [[CrossRef](#)]
20. Malkapuram, R.; Kumar, V.; Negi, Y.S. Recent Development in Natural Fiber Reinforced Polypropylene Composites. *J. Reinf. Plast. Compos.* **2009**, *28*, 1169–1189. [[CrossRef](#)]
21. Czirány, T. Special Manufacturing and Characteristics of Basalt Fiber Reinforced Hybrid Polypropylene Composites: Mechanical Properties and Acoustic Emission Study. *Compos. Sci. Technol.* **2006**, *66*, 3210–3220. [[CrossRef](#)]
22. Anbupalani, M.S.; Venkatachalam, C.D.; Rathanasamy, R. Influence of Coupling Agent on Altering the Reinforcing Efficiency of Natural Fibre-Incorporated Polymers—A Review. *J. Reinf. Plast. Compos.* **2020**, *39*, 520–544. [[CrossRef](#)]
23. Mishra, S.; Mohanty, A.K.; Drzal, L.T.; Misra, M.; Hinrichsen, G. A Review on Pineapple Leaf Fibers, Sisal Fibers and Their Biocomposites. *Macromol. Mater. Eng.* **2004**, *289*, 955–974. [[CrossRef](#)]
24. Rong, M.Z.; Zhang, M.Q.; Liu, Y.; Yang, G.C.; Zeng, H.M. The Effect of Fiber Treatment on the Mechanical Properties of Unidirectional Sisal-Reinforced Epoxy Composites. *Compos. Sci. Technol.* **2001**, *61*, 1437–1447. [[CrossRef](#)]
25. Marais, S.; Gouanvé, F.; Bonnesoeur, A.; Grenet, J.; Poncin-Epaillard, F.; Morvan, C.; Métayer, M. Unsaturated Polyester Composites Reinforced with Flax Fibers: Effect of Cold Plasma and Autoclave Treatments on Mechanical and Permeation Properties. *Compos. Part A Appl. Sci. Manuf.* **2005**, *36*, 975–986. [[CrossRef](#)]
26. Marais, S.; Métayer, M.; Labbé, M.; Valleton, J.M.; Alexandre, S.; Saiter, J.M.; Poncin-Epaillard, F. Surface Modification by Low-Pressure Glow Discharge Plasma of an Unsaturated Polyester Resin: Effect on Water Diffusivity and Permeability. *Surf. Coat. Technol.* **1999**, *122*, 247–259. [[CrossRef](#)]
27. Gouanvé, F.; Marais, S.; Saiter, J.M.; Poncin-Epaillard, F.; Métayer, M.; Garda, M.R. Characterization of Composites with Flax Fibers Treated with Cold Plasma: Water Permeation and Thermal Analysis. *Mater. Res. Innov.* **2005**, *9*, 15–17. [[CrossRef](#)]
28. Leone, G.; D'Angelo, G.A.; Russo, P.; Ferraro, P.; Pagliarulo, V. Plasma Treatment Application to Improve Interfacial Adhesion in Polypropylene-Flax Fabric Composite Laminates. *Polym. Compos.* **2022**, *43*, 1787–1798. [[CrossRef](#)]
29. Moradkhani, G.; Profili, J.; Destrieux, A.; Robert, M.; Laroche, G.; Elkoun, S.; Mighri, F.; Vuillaume, P.Y. Plasma-Enhanced Alginate Pre-Treatment of Short Flax Fibers for Improved Thermo-Mechanical Properties of PLA Composites. *J. Compos. Sci.* **2024**, *8*, 106. [[CrossRef](#)]
30. Veiga, A.T.V.; da Silva, Y.F.; Cavalcante, R.C.; Simão, R.A. SF6 Plasma Surface Modification of Flax Fibers for Improved Adhesion in Polypropylene Composites. *Fibers Polym.* **2025**, *26*, 2569–2879. [[CrossRef](#)]
31. Singh, P.; Singari, R.M.; Mishra, R.S.; Bajwa, G.S. A Review on Recent Development on Polymeric Hybrid Composite and Analysis of Their Enhanced Mechanical Performance. *Mater. Today Proc.* **2022**, *56*, 3692–3701. [[CrossRef](#)]
32. Téraube, O.; Agopian, J.-C.; Pucci, M.F.; Liotier, P.-J.; Conchon, P.; Badel, É.; Hajjar-Garreau, S.; Leleu, H.; Baylac, J.-B.; Batisse, N.; et al. Optimization of Interfacial Adhesion and Mechanical Performance of Flax Fiber-Based Eco-Composites through Fiber Fluorination Treatment. *Compos. Part B Eng.* **2025**, *296*, 112228. [[CrossRef](#)]
33. Das, S.C.; La Rosa, A.D.; Goutianos, S.; Grammatikos, S. Glass Fibre Hybridization to Improve Durability of Circular Flax Fibre Reinforced Composites with Recyclable Polymer Matrix Systems. *Compos. Part C* **2024**, *14*, 100491. [[CrossRef](#)]
34. Chandan, V.; Mishra, R.K.; Kolar, V.; Muller, M.; Hrabe, P. Green Hybrid Composites Partially Reinforced with Flax Woven Fabric and Coconut Shell Waste-Based Micro-Fillers. *Ind. Crops Prod.* **2024**, *222*, 119948. [[CrossRef](#)]
35. Gupta, U.S.; Dhamarikar, M.; Dharkar, A.; Chaturvedi, S.; Kumrawat, A.; Giri, N.; Tiwari, S.; Namdeo, R. Plasma Modification of Natural Fiber: A Review. *Mater. Today Proc.* **2021**, *43*, 451–457. [[CrossRef](#)]
36. Sali, A.K.; Ramakrishnan, S.K.; Paduvilan, J.K.; Vackova, T.; Král, R.; Zemenova, P.; Thomas, S.; Spatenka, P. Natural Fiber Thermo-plastic Composites: Exploring the Impact of Plasma Surface Treatment on Viscoelastic and Thermal Behavior. *Mater. Chem. Phys.* **2025**, *338*, 130599. [[CrossRef](#)]

37. Diwahar, P.; Prakalathan, K.; Bhuvana, K.P.; Senthilkumar, K. Examining the Mechanical and Thermal Properties of a Novel Hybrid Thermoplastic Rubber Composite Made with Polypropylene, Polybutadiene, S-Glass Fibre, and Flax Fibre. *Polymers* **2024**, *16*, 3599. [[CrossRef](#)] [[PubMed](#)]
38. Huh, Y.I.; Bismark, M.; Kim, S.; Lee, H.K.; Na, C.U. Effects of Plasma Treatment on Mechanical Properties of Jute Fibers and Their Composites with Polypropylene. *Elastom. Compos.* **2012**, *47*, 310–317. [[CrossRef](#)]
39. Pickering, K.L.; Efendy, M.G.A.; Le, T.M. A Review of Recent Developments in Natural Fibre Composites and Their Mechanical Performance. *Compos. Part A Appl. Sci. Manuf.* **2016**, *83*, 98–112. [[CrossRef](#)]
40. Thomason, J.L.; Rudeiros-Fernández, J.L. Thermal Degradation Behaviour of Natural Fibres at Thermoplastic Composite Processing Temperatures. *Polym. Degrad. Stab.* **2021**, *188*, 109594. [[CrossRef](#)]
41. Khina, A.G.; Bulkatov, D.P.; Storozhuk, I.P.; Sokolov, A.P. Coefficient of Linear Thermal Expansion of Polymers and Polymer Composites: A Comprehensive Review. *Polymers* **2025**, *17*, 3097. [[CrossRef](#)]
42. Zhou, B.Z.; Wang, Y.; Zhao, W.; Wang, Z.; Zhao, Y. Study on Thermal Expansion Coefficient and Absorbing Properties of Fiber Reinforced Resin-Based Absorbing Composites. *Compos. Part C Open Access* **2024**, *14*, 100449. [[CrossRef](#)]
43. Colón Quintana, J.L.; Slattery, L.; Pinkham, J.; Keaton, J.; Lopez-Anido, R.A.; Sharp, K. Effects of Fiber Orientation on the Coefficient of Thermal Expansion of Fiber-Filled Polymer Systems in Large Format Polymer Extrusion-Based Additive Manufacturing. *Materials* **2022**, *15*, 2764. [[CrossRef](#)]
44. Yang, H.; Yan, R.; Chen, H.; Lee, D.H.; Zheng, C. Characteristics of Hemicellulose, Cellulose and Lignin Pyrolysis. *Fuel* **2007**, *86*, 1781–1788. [[CrossRef](#)]
45. Kubiak, N.; Sztorch, B.; Kustos, M.; Frydrych, M.; Pakuła, D.; Jałbrzykowski, M.; Hartmann, T.; Zopp, C.; Kroll, L.; Przekop, R.E. Sustainable PLA Composites Filled with Poaceae Fibers: Thermal, Structural, and Mechanical Properties. *Materials* **2025**, *18*, 3952. [[CrossRef](#)]
46. Yin, S.; Rials, T.G.; Wolcott, M.P. Crystallization Behavior of Polypropylene and Its Effect on Woodfiber Composite Properties. In *Proceedings of the Fifth International Conference on Woodfiber-Plastic Composites, Madison, WI, USA, 26–27 May 1999*; Forest Products Society: Madison, WI, USA, 1999; pp. 139–146.
47. Chen, E.J.H.; Hsiao, B.S. The Effects of Transcrystalline Interphase in Advanced Polymer Composites. *Polym. Eng. Sci.* **1992**, *32*, 280–286. [[CrossRef](#)]
48. Felix, J.M.; Gatenholm, P. Effect of Transcrystalline Morphology on Interfacial Adhesion in Cellulose/Polypropylene Composites. *J. Mater. Sci.* **1994**, *29*, 3043–3049. [[CrossRef](#)]

Disclaimer/Publisher’s Note: The statements, opinions and data contained in all publications are solely those of the individual author(s) and contributor(s) and not of MDPI and/or the editor(s). MDPI and/or the editor(s) disclaim responsibility for any injury to people or property resulting from any ideas, methods, instructions or products referred to in the content.