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Thermo-Mechanical and Structural Properties of Polyester Composites: Effect of Pineapple Leaf and Silk Fiber Hybridization

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Abstract:

Present research explores the production and characteristics of polyester composites strengthened with pineapple leaf and silk fiber hybridization. The composites were prepared using the hand-lay-up method with a 1:1 fiber ratio. The research investigates the impact of different fiber loadings (0, 10, 15, 20, 25, and 30 wt.%) on the thermos-mechanical and structural properties of the composites. The results of the experiments indicate that adding fibers significantly improved the mechanical properties. The highest values for tensile strength (75.4 MPa), tensile modulus (3061 MPa), flexural strength (91 MPa), flexural modulus (1980 MPa), and hardness (72.6 Shore D) were achieved with a 20% fiber loading, suggesting that this composition is optimal for enhancing the strength and stiffness of the composites. Additionally, impact strength peaked at a 25% fiber loading (869 kJ/m^2), indicating that this composition is suitable for applications requiring increased energy absorption. Structural and thermal properties were examined using Fourier-transform infrared spectroscopy and thermogravimetric analysis. Spectroscopic examination confirmed the successful integration of fibers into the composite, while thermogravimetric analysis revealed that composites with a 25% fiber loading displayed superior thermal stability.

Keywords: *Hybrid Polyester Composite; Fiber Loading; Thermo-Mechanical and Structural Properties*

1 Introduction

Natural fiber-reinforced polymer composites (NFRPCs) have attracted growing interest as sustainable alternatives to traditional synthetic composites due to their renewability, biodegradability, and lower environmental impact [1]. Among these, pineapple leaf fiber (PALF) and silk fiber stand out for their excellent mechanical properties, such as tensile strength, toughness, and impact resistance, making them suitable for use in the automotive, construction, and packaging industries [2]. In Bangladesh, utilizing agricultural waste like pineapple leaves and byproducts from the silk industry offers a dual advantage: reducing environmental waste and adding value to local industries. PALF, rich in cellulose, is often discarded despite its potential as a high-performance reinforcement material [3]. Silk fiber, another valuable resource, also holds promise for composite applications. This research investigates the mechanical properties of hybrid polyester composites reinforced with PALF and silk fiber, focusing on their tensile strength, flexural strength, and impact resistance. By exploring these natural fibers, this study seeks to contribute to developing eco-friendly materials that align with global sustainability goals [4].

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Composite materials combine two or more distinct constituents with varying properties to enhance performance. Key components include a matrix that binds the reinforcement and distributes stress, and reinforcement that provides strength and stiffness [5]. Composites offer advantages such as high specific strength, corrosion resistance, and design flexibility, making them suitable for aerospace, automotive, biomedical, and consumer electronics applications. However, challenges include complex manufacturing, limited recyclability, and moisture absorption. They can be classified into polymer matrix composites (PMCs), metal matrix composites (MMCs), and ceramic matrix composites (CMCs), highlighting their versatility in addressing diverse engineering needs while promoting sustainability [6].

Natural Fiber Reinforced Polymer Composites (NFRPCs) utilize lignocellulosic fibers from plants, including bast, leaf, fruit, seed, and stalk. Research on NFRPCs has surged since the 1990s due to their environmental benefits, such as biodegradability and lower emissions [7]. Life cycle assessments indicate they have a reduced environmental impact compared to glass fiber composites, with advantages like lightweight properties and cost-effectiveness. Key benefits include renewability, high strength-to-weight ratio, low density, and versatility with various matrix materials, driving their adoption across multiple industries [8]. Synthetic fibers are engineered materials made from chemical substances through polymerization, where monomers form long polymer chains. This process has transformed the textile and materials industries since the early 20th century. The first synthetic fiber, rayon, was developed in the late 19th century, while true synthetic fibers began with nylon, introduced by Wallace Carothers at DuPont in 1935 [9]. Subsequent developments include high-performance fibers like aramids and carbon fibers, which revolutionized the aerospace and automotive sectors due to their exceptional strength-to-weight ratios. Synthetic fibers are categorized into types such as nylon, known for durability; acrylic, a wool substitute; and aramids, recognized for heat resistance [10]. Natural fibers are renewable and biodegradable, making them suitable for sustainable applications in automotive interiors and construction, despite having lower mechanical properties than synthetic fibers [11]. In contrast, synthetic fibers provide superior strength and stiffness essential for aerospace and automotive sectors but have higher production costs and environmental impacts due to energy-intensive manufacturing [12].

Hybrid composites are more advanced materials that combine two or more types of fibers within a single polymer matrix, enhancing mechanical properties such as strength, stiffness, and toughness [13]. They are categorized into three types: Natural Fiber Hybrid Composites, which blend natural fibers like jute and hemp with synthetic fibers for improved sustainability [14]. Synthetic fiber hybrid composites, which utilize fibers like glass and carbon for high-performance applications; and Hybrid Nanocomposites, which incorporate nanomaterials to enhance mechanical and thermal properties [2].

Present research utilized polyester (PS) as the matrix material, with methyl ethyl ketone peroxide (MEKP) as the hardener. The composite materials were fabricated using the hand lay-up process, incorporating a 1:1 ratio of pineapple leaf fibers and raw silk as reinforcement. Thus, present research advances the field of hybrid natural-synthetic fiber composites by demonstrating the potential of pineapple leaf fiber (PALF) and silk fiber to enhance the mechanical and thermal properties of polyester-based composites. The study provides critical insights into the synergistic effects of combining natural and synthetic fibers, contributing to the development of sustainable, high-performance materials. By addressing key challenges such as optimizing fiber ratios and improving thermo-mechanical properties, the research opens new avenues for the use of hybrid composites in the automotive, construction, and packaging industries, where both performance and environmental sustainability are paramount [15]. The findings also contribute to the broader effort in materials science to promote eco-friendly alternatives without compromising on strength and durability, helping bridge the gap between sustainability and industrial applicability.

2 Methodology

2.1 Materials

This study utilized commercial-grade polyester resin as the matrix material due to its strong mechanical properties, resistance to chemicals, and heat stability. The resin, with a density of 1.2 to 1.5 g/cm³, was commonly used in composite manufacturing for its affordability and compatibility with natural fibers. To reinforce the matrix, PALF and silk fiber were selected, mixed in a 1:1 ratio. PALF, with a density of 1.54 g/cm³, is a sustainable and renewable material known for its impressive strength-to-weight ratio and biodegradability. Its tensile strength ranges from 400 to 627 MPa, making it suitable for eco-friendly composite structures, although its susceptibility to moisture absorption can be a limiting factor. Silk fiber, on the other hand, has a density of 1.36 g/cm³ and a tensile strength between 400 to 700 MPa, providing excellent mechanical reinforcement while being both biodegradable and biocompatible. The high tensile strength of silk enhances the durability of the composite, although processing challenges such as cost and compatibility with synthetic matrices are concerns.

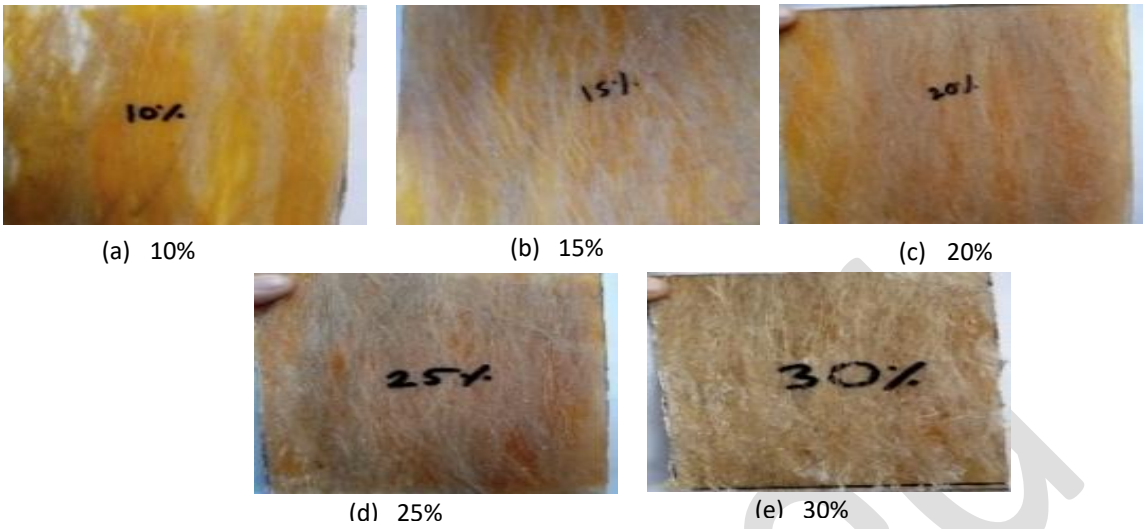
2.2 Fabrication of Composites

During fabrication of PALF-silk fiber-polyester composites following hand lay-up process, polyester resin was mixed with 2% methyl ethyl ketone peroxide (MEKP) hardener to initiate the curing process. The mixture was carefully stirred to prevent the formation of air bubbles, which could negatively affect the mechanical properties of the composite. Six composite samples were fabricated with varying fiber loads of 0, 10, 15, 20, 25, and 30 wt.%. To ensure proper molding, a clean steel plate was positioned on a flat surface, followed by the placement of release film, which was coated with wax to facilitate easy removal of the composite post-curing. A thin initial layer of the polyester matrix was applied to the milot paper, and pre-cut 15 cm long silk fibers were evenly distributed. Another thin layer of the matrix was then applied, followed by the placement of PALF fibers. Once the fibers were fully layered, the remaining matrix mixture was poured to ensure complete wetting. Another piece of milot paper was laid over the structure, and a hand roller was used to lightly press the layers, promoting uniform fiber wetting and adhesion between the silk and PALF layers. A second steel plate was placed over the assembly, and sufficient weight was applied to maintain uniform pressure during the curing process, which lasted 48 hours. After curing, the composite samples were removed from the mold, and excess matrix material was carefully trimmed using a hacksaw. This method ensured strong bonding between the fibers and the matrix, producing high-quality samples for mechanical testing. Figure 1 shows composites with various fiber loadings.

3. Characterization Techniques

3.1 Tensile Test

The tensile test of the composites was conducted to evaluate tensile strength, and Young's modulus, following ASTM D638-01 using an Instron Universal Testing Machine. The dimensions of each sample were measured, and the specimens were loaded with parameters set as per standard. The tensile load was applied until fracture, with stress-strain data recorded in real time. For each fiber load (0, 10, 15, 20, 25, and 30 wt.%) (Figure 2), the samples were tested, ensuring statistical validity for further analysis.



(a) 10% (b) 15% (c) 20%

(d) 25% (e) 30%

Figure 2. Composites with 10, 15, 20, 25 and 30 wt.% fiber loadings

Figure 1. Tensile test samples with different fiber loading

3.2 Flexural Test

The flexural properties of the composite were assessed using a three-point bending test according to ASTM D790-00. The test was performed on an Instron Universal Testing Machine, ensuring the span-to-thickness ratio was 16 times the sample thickness. Samples (Figure 3) were placed on two supports, and load was applied at the midpoint. The machine's crosshead moved at a constant speed until failure, capturing load

Figure 3. Flexural test samples with different fiber loading

and deflection data. Flexural strength, and flexural modulus were calculated for each fiber load (0, 10, 15, 20, 25, 30 wt.%).

3.3 Hardness Test

The hardness of the composite samples was assessed using a Shore D durometer following ASTM D2240-00. This scale is appropriate for polymer composites due to their rigidity. During the test, the durometer, equipped with a specific indenter tip and a calibrated spring, was applied perpendicular to the surface of the composite. The indenter was pressed into the material with a standard force, and the depth of penetration was measured. The Shore D hardness value, ranging from 0 to 100, was recorded for each sample, with higher values indicating greater hardness. Hardness was measured for each fiber load (0, 10, 15, 20, 25, 30 wt.%). The results highlight the composites' surface resistance to deformation, a critical factor for durability in various applications.

3.4 Impact Test

The impact test was conducted to evaluate the toughness of the composite materials, following the ASTM D6110 standard using a pendulum impact testing machine. The specimens were prepared according to the standard, and each was struck by a pendulum hammer to measure the energy absorbed upon impact. The absorbed energy, displayed on a scale attached to the machine, provided critical data on the material's ability to withstand sudden forces. For each fiber load configuration (0, 10, 15, 20, 25, and 30 wt.%), the specimens were tested to ensure reliable data for further analysis.

3.5 Thermogravimetric Analysis (TGA)

Thermogravimetric analysis (TGA) was employed to investigate the thermal stability and decomposition characteristics of the composite materials. The analysis was performed using a thermogravimetric analyzer, where samples of each fiber loading (0, 15, 20, 25, and 30 wt.%) weighing between 10 to 30 mg were placed in a crucible and subjected to a controlled heating rate of 10 °C/min up to a temperature of 500 °C. This method allowed for the continuous monitoring of mass changes as the temperature increased, generating a TGA curve that illustrated the percentage of mass loss in relation to temperature. The resulting data provided insights into the thermal behavior of the composites, including decomposition temperatures and weight loss patterns, which are essential for assessing their suitability for various applications.

3.6 Fourier Transform Infrared Spectroscopy (FTIR)

Fourier Transform Infrared (FTIR) spectroscopy was utilized to identify the chemical functional groups present in the composite materials and to assess the interaction between the matrix and fibers. The analysis was conducted using a SHIMADZU spectrometer, where composite samples were first ground into a fine powder. The powdered samples were then mixed with potassium bromide (KBr) in a ratio of 100:1 (KBr) to form a homogeneous mixture. This mixture was compressed into a pellet using a hand press machine and subsequently placed in the FTIR spectrometer to obtain the infrared spectrum. The absorption peaks in the spectrum were analyzed to identify characteristic functional groups and confirm the successful incorporation of pineapple leaf fibers and silk fibers within the polyester matrix, thus providing valuable information about the chemical bonding and interactions that contribute to the overall properties of the composites.

4 Results and Discussion

4.1 Tensile Test

The tensile properties of the hybrid polyester composite, reinforced with varying amounts of pineapple leaf fiber (PALF) and silk fiber, demonstrated a clear trend influenced by fiber loading. The results (Figure 4 and Figure 5) show that the tensile strength and Young's modulus increased with fiber loading up to 20%, where the composite exhibited its highest mechanical performance. The 20% fiber-loaded composite achieved a tensile strength of 75.4 MPa, a substantial improvement (96.3%) over the pure polyester matrix, which had a tensile strength of 38.4 MPa. This enhancement is attributed to the synergistic effect of combining PALF and silk fiber, promoting effective load transfer between the fibers and the matrix, leading to better tensile performance [16]. Young's modulus (Figure 5) followed a similar trend, peaking at 3061 MPa for the 20% fiber-loaded composite, compared to 878 MPa for the unreinforced matrix. The increase over pure matrix was 248.6%. The increase in stiffness reflects the reinforcing capability of the fibers, improving the composite's resistance to deformation under tensile stress. Beyond 20% fiber loading, however, both tensile strength and Young's modulus showed a decline. At 30% fiber content, the tensile strength dropped to 34.19 MPa, and the modulus decreased to 1732 MPa. This reduction is likely due to fiber agglomeration, poor matrix-fiber bonding, and void formation, which can act as stress concentrators, weakening the composite [17].

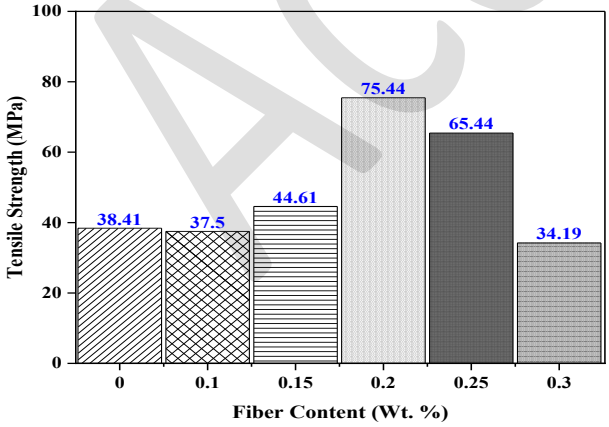


Figure 5. Variation of tensile strength against fiber loading

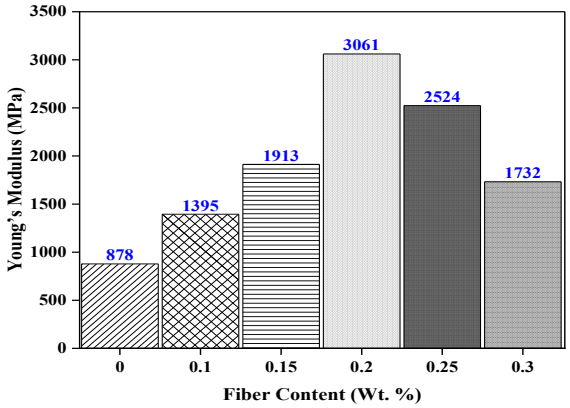


Figure 4. Variation of Young's modulus against fiber loading

4.2 Flexural Test

The flexural strength (Figure 6) and flexural modulus (Figure 7) of the composites increased with fiber content up to 20%, reaching a maximum flexural strength of 91 MPa and a modulus of 1980 MPa. The increase of flexural modulus over pure matrix was 17.7%, while the increase of flexural modulus of 20% fiber reinforced composite over 10% fiber reinforced composite was 60.5%. This improvement reflects effective stress transfer between the polyester matrix and the fibers. However, beyond 20% fiber loading, both flexural strength and modulus declined, with the 30% fiber-loaded composite showing a significant drop to 40 MPa in strength and 1059 MPa in modulus. This decrease is likely due to fiber agglomeration and void formation, which compromised matrix-fiber bonding. These results suggest that 20% fiber content offers the best balance for enhancing flexural properties, while higher fiber loadings reduce mechanical performance due to processing limitations [18].

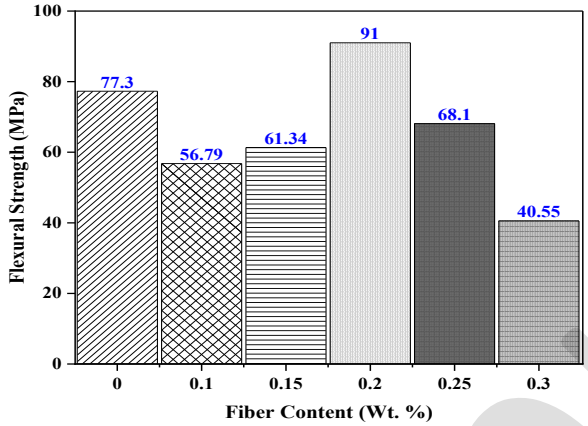


Figure 7. Variations of flexural strength against fiber loading

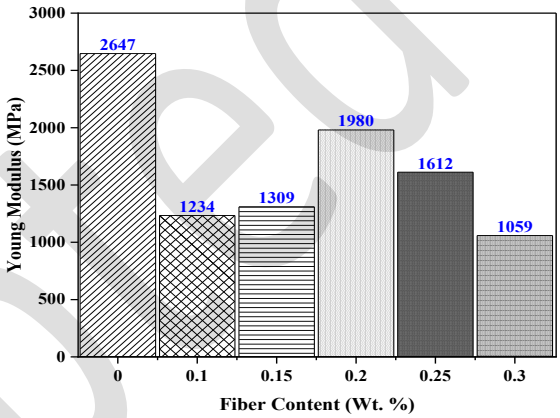


Figure 6. Variations of flexural modulus against fiber loading

4.3 Hardness Test

The hardness of the pineapple leaf fiber (PALF) and silk-reinforced polyester composites was measured using the Shore D scale. As shown in (Figure 8), the hardness of the composites varies with fiber loading, following a distinctive pattern. The highest hardness value of 74 Shore D was observed for the neat polyester (0% fiber loading), while the optimum hardness of 72.6 Shore D was achieved at 20% fiber loading. At lower fiber loadings (below 20%), the reduction in hardness can be attributed to the incomplete fiber dispersion within the polyester matrix. The insufficient fiber distribution results in areas with weaker reinforcement, which compromises the composite's hardness. As fiber loading was increased to 20%, the fibers are better dispersed and well-integrated into the matrix, leading to improved hardness. The increase in fiber content enhances the composite's resistance to indentation by improving the interfacial adhesion and stress transfer between the fibers and the polyester matrix. This optimal fiber loading allows the composite to reach a balance where the fiber distribution and matrix reinforcement are effective in improving the hardness [19]. However, above 20% fiber loading, the hardness began to decrease significantly, with the composite at 30% fiber loading exhibiting a value of 50.2 Shore D. The decline is primarily due to fiber agglomeration and void formation. At higher fiber content, the matrix struggles to fully wet the fibers, leading to reduced fiber-matrix adhesion and the formation of voids. These voids act as weak points within the composite, lowering its resistance to deformation and reducing hardness [20]. In summary, the hardness of the composites improved with increasing fiber loading up to 20%, after which it declines due to fiber agglomeration and void formation. The results indicate that 20% fiber loading offers

the best balance between fiber reinforcement and matrix cohesion, leading to optimal hardness for the composite.

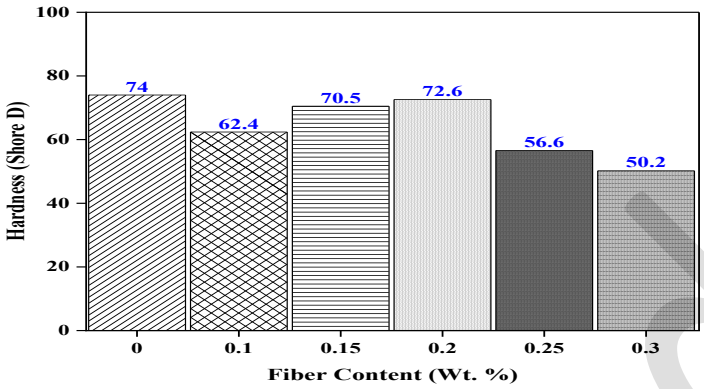


Figure 8. Variation of hardness against fiber loading

4.4 Impact Test

The impact strength of the pineapple leaf fiber (PALF) and silk-reinforced polyester composites was evaluated using an impact test. The results, as shown in (Figure 9), indicate that the impact strength increased with fiber loading up to a certain point before it decreased. The composite with 25% fiber content exhibited the highest impact energy of 869.6 kJ/m², demonstrating its superior ability to absorb energy upon impact. The increase of impact strength of 25% fiber reinforced composite over 10% fiber reinforced composite was 253%. The increase in impact strength can be attributed to the good interfacial bonding between the fibers and the matrix, as well as the effective energy absorption due to the distribution and favorable entanglement of the fibers within the polyester matrix [21]. Up to 25% fiber loading, the fibers were well-distributed and adequately wetted by the matrix, which facilitated energy dissipation through the fibers during impact. However, beyond 25% fiber content, a decline in impact strength was observed. This reduction is likely due to fiber agglomeration and poor wetting at higher fiber loadings, which lead to void formation and reduced interfacial bonding between the fiber and matrix. These voids act as stress concentrators, promoting crack initiation and propagation, thereby reducing the material's ability to absorb impact energy. The findings suggest that fiber-reinforced polyester composites demonstrate enhanced toughness and energy absorption up to an optimal fiber loading of 25%, beyond which the impact strength decreases due to fiber-matrix interface issues [22].

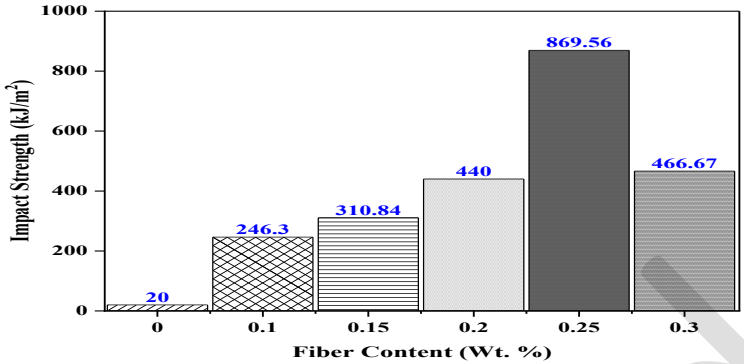


Figure 9. Variation of impact strength against fiber loading

4.5 Thermogravimetric Analysis (TGA) Results

The thermogravimetric analysis (TGA) was conducted to assess the thermal stability of polyester composites reinforced with varying fiber loadings of PALF and silk fiber hybrids. The results (Figure 10) indicate significant changes in the thermal degradation behavior with increasing fiber content. Pure matrix showed the earliest onset of decomposition, starting at 60 °C. In contrast, the 15%, 20%, 25%, and 30% composites began to show more noticeable weight loss around 120°C, demonstrating greater thermal stability. The 20% and 25% composite samples exhibited the highest thermal stability, retaining 99.2% and 99.3% of their initial weight, respectively. The 30% composite showed a slightly lower retention of 98.6%. The 20% composite exhibited the highest weight retention (97.6%), followed by the 25% composite (97.2%). Pure matrix retained the least weight (93.8%) at 300 °C, showing the highest degradation rate. As the composite content increased, the thermal stability improved, with the 20% and 25% composites showing the best balance between weight retention and thermal resistance. The 30% composite, while still stable, demonstrated slightly more rapid weight loss at higher temperatures. The TGA results reveal that composite content significantly impacts the thermal stability of the material [23], [24]. Higher composite percentages (20% and 25%) exhibited superior thermal resistance, retaining more than 97% of their weight at 300 °C. The findings indicate that these composites could be more suitable for applications requiring enhanced thermal performance compared to the 0% and 15% composites.

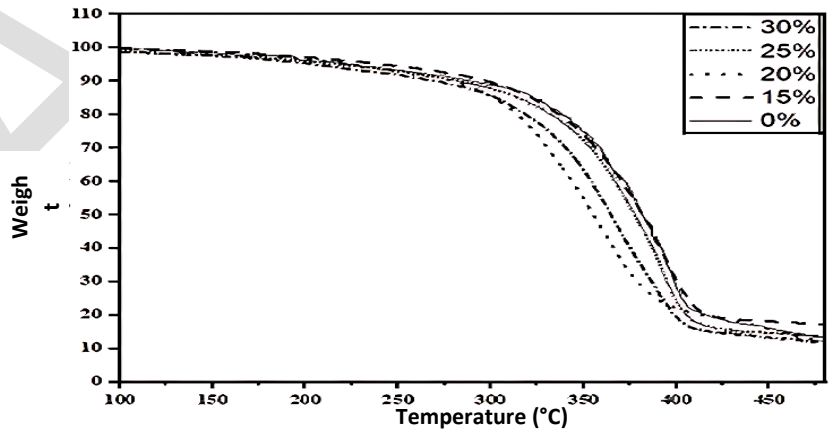


Figure 10. TGA graphs for pure matrix and prepared composites

4.6 FTIR Analysis

The FTIR spectra of the pure polyester matrix (0% fiber) (Figure 11(a)) and the 20% fiber-reinforced polyester composite (Figure 11(b)) demonstrate key differences and similarities that indicate the effect of fiber incorporation on the chemical structure of the composite.

O–H Stretching (~3450 cm^{-1}): In the 0% composite, a moderate peak is observed at 2922.2 cm^{-1}, indicating the presence of residual moisture in the polyester matrix. In contrast, the 20% fiber-reinforced composite shows a more pronounced and broader peak at 3026.6 cm^{-1} and 2929.7 cm^{-1}, corresponding to the O–H stretching vibration. This increase in intensity suggests that the introduction of hydrophilic natural fibers (pineapple leaf fiber and silk fiber) leads to increased moisture absorption. The fibers' hydrophilic nature is confirmed by this heightened O–H presence, which is absent in the neat polyester matrix [25].

C–H Stretching (2950 cm^{-1} and 2870 cm^{-1}): The C–H stretching peaks appear at 2922.2 cm^{-1} in the 0% composite and at 2929.7 cm^{-1} in the 20% composite. The minor shift between these two peaks suggests that the inclusion of fibers has minimal impact on the aliphatic hydrocarbon backbone of the polyester matrix. The consistency of this peak indicates that no significant chemical interaction occurs between the fibers and the polyester's hydrocarbon chains, and the integrity of the matrix structure remains largely unaffected by fiber addition [25].

C=O Stretching (~1725 cm^{-1}): Both composites exhibit a strong peak at 1718.3 cm^{-1}, corresponding to the C=O stretching vibration in the ester linkages of the polyester matrix. The absence of significant shifts in this peak between the 0% and 20% composites suggests that the fibers do not chemically alter the polyester's ester structure. However, a slight broadening in the C=O peak in the 20% composite may indicate physical interactions, such as hydrogen bonding or interfacial adhesion between the fibers and the matrix. This interaction contributes to the enhanced mechanical properties of the fiber-reinforced composite [26].

C–O Stretching (1250 cm^{-1} and 1090 cm^{-1}): The C–O stretching peaks in the 0% composite appear at 1256.1 cm^{-1} and 1118.2 cm^{-1}, characteristic of ester linkages within the polyester matrix. Similarly, in the 20% composite, the C–O stretching peaks remain at 1259.8 cm^{-1} and 1118.2 cm^{-1}, indicating that the ester bonds in the polyester matrix remain unaffected by fiber incorporation. This suggests that the fiber addition does not break or chemically alter the polyester's ester structure, which is essential for the matrix's mechanical integrity [23].

Out-of-plane C–H Bending: Both spectra show peaks at 741.7 cm^{-1} and 700.7 cm^{-1}, corresponding to the out-of-plane C–H bending in the polyester matrix. These peaks remain unchanged between the 0% and 20% composites, further confirming that the fundamental structure of the polyester matrix remains intact with the incorporation of fibers.

The comparative FTIR analysis reveals that the incorporation of 20% natural fibers (PALF and silk fiber) into the polyester matrix does not result in significant chemical modifications of the matrix. However, physical interactions such as hydrogen bonding and increased moisture absorption due to the hydrophilic nature of the fibers are evident. These physical interactions enhance the mechanical properties of the composite without disrupting the chemical integrity of the polyester matrix. The increase in O–H stretching and slight shifts in the aromatic and carbonyl peaks suggest improved interfacial bonding between the fibers and matrix, contributing to the overall performance of the fiber-reinforced composites [26].

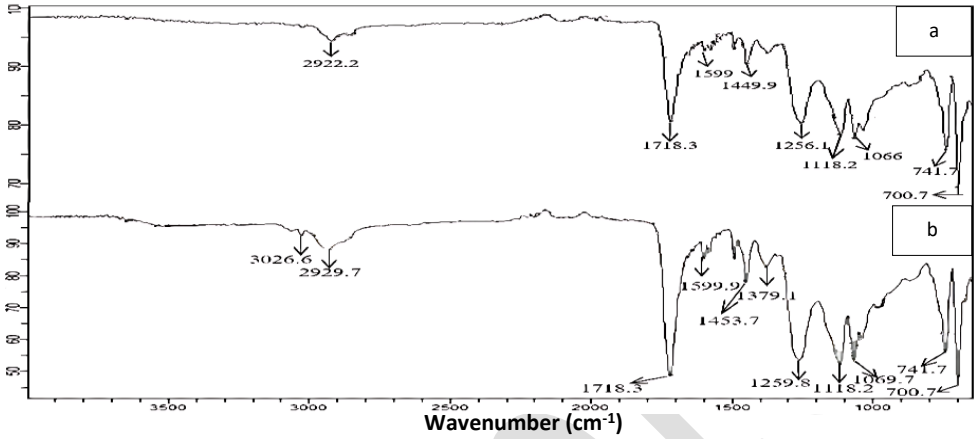


Figure 11. FTIR graphs for (a) 0% and (b) 20% fiber loaded composites

5 Conclusion

Present research examined the effects of varying fiber loading on the mechanical and thermal properties of composites made with pineapple leaf fiber (PALF) and silk fiber reinforced polyester. The results clearly show that fiber loading plays a crucial role in determining the overall performance of the composite materials. Mechanical properties, including tensile strength, flexural strength, and hardness, were found to be optimal at 20% fiber loading. On the other hand, the highest impact strength was achieved at 25% fiber loading. Again, the composite with 25% fiber loading exhibited slightly better thermal stability compared to the other compositions. Thus, 20-25% fiber loading range provides the best balance of mechanical and thermal performance for these composites. In short, present research highlights the synergistic benefits of combining PALF and silk fiber in hybrid composites, offering a pathway for developing eco-friendly, high-performance materials suitable for applications in the automotive, construction, and packaging industries.

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