

Development of *Morus alba* Reinforced Poly-Lactic Acid with Elevated Mechanical and Thermal Properties

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Abstract

This research investigates the mechanical and thermal properties of *Morus alba* combined with polylactic acid in comparison with other natural fibers. The study uses three different fiber and PLA compositions - 20%, 30%, and 40% respectively - to produce composite materials. In addition, another composite with the same fiber volume is treated with a 4% NaOH solution to improve mechanical properties. The composites are processed by twin-screw extrusion, granulation, and injection molding. Tensile strength measurements of raw fibers and NaOH-treated fibers were carried out using a single-fiber tensile test with a gauge length of 40 mm. It was observed that the NaOH surface treatment increases the resistance against tensile loading and exhibited improved properties for raw fiber strands. The diameter of the fibers was measured using optical microscopy. During this research, flexural tests, impact tests, differential scanning calorimetry (DSC), and heat deflection temperature measurements (HDT) were conducted to evaluate the mechanical and thermal properties of the developed composite samples. The results indicate that the mechanical properties of NaOH-treated *Morus alba*-reinforced polylactic acid outperform both virgin PLA samples and untreated *Morus alba* samples.

Keywords

Morus Alba Fiber (MAF), White Mulberry, Polylactic Acid (PLA), Sodium Hydroxide (NaOH) Solution, Single Fiber Tensile Test, Thermal Stability, Optical Microscopy

1. Introduction

Due to increasing ecological concerns, the creation of new materials and prod-

ucts now carefully considers factors such as environmental safety and recyclability. Over the past decade, this shift has sparked a heightened interest in natural fibers, making them an essential part of eco-friendly materials in the automotive, aerospace, and construction industries. Plant fibers like flax, hemp, sisal, and kenaf are now considered cost-effective and environmentally friendly alternatives to glass fibers [1] [2]. Among these fibers, the potential of discarded agricultural waste remains unexplored. Developing such waste into innovative, environmentally friendly products can significantly reduce waste disposal and environmental problems [3]. This study examines discarded agricultural waste, particularly *Morus alba* stems, to investigate their potential as a valuable fiber source. These stems are abundant, low-cost, and commonly used as firewood in southern India.

Morus, a genus of deciduous trees in the Moraceae family, includes mulberries that grow naturally and are cultivated in various temperate regions worldwide. The white mulberry (*Morus alba* L.), a fast-growing tree of small to medium size native to China, is known for its vigorous growth and is sometimes considered weedy. These mulberry plants typically grow tall with a crown height ranging from 1.5 to 1.8 meters, producing 8 - 10 shoots per crown. They are specifically nurtured from well-established saplings aged 2 - 3 months and can be harvested mechanically using tree shaking systems. Initially, their leaves were primarily exported for the silkworm industry, serving as the exclusive food source for the silkworm (*Bombyx mori*). The mulberry tree produces fibrous bark that is used to make paper. Every year, thousands of tons of mulberry stems are being collected for agricultural waste or firewood. This offers greater environmental sustainability when used as a fiber reinforcement for composites [4].

Polylactic acid (PLA) stands out as a rigid thermoplastic of particular interest for its role as a matrix in composite materials. Its significance lies in its remarkable versatility as a biopolymer, notably sourced from renewable crops like corn. PLA lends itself well to various processing methods, including injection molding, blow molding, and film formation [5]. Although PLA's mechanical qualities make it suitable for some industrial applications. Its brittleness makes it not suitable for a wide range of commercial usage. However, by mixing it with other materials, the brittleness can be reduced. Natural fibers can be replaced with synthetic fibers in polymer matrices to improve mechanical performance while promoting environmental friendliness. Natural fiber-based composites are becoming increasingly popular due to their outstanding mechanical and physical properties and environmental sustainability, drawing interest from a wide range of industries [6]-[8].

Natural fibers consist of chemical constituents such as cellulose, hemicellulose, lignin, wax, and moisture. These influence their physio-chemical behavior and mechanical properties in reinforced composites by affecting debonding and void formation. There are more hydrophilic groups in the cellulose structure of raw natural fibers. Interfacial bonding between natural fibers and matrix may be

affected by the incompatibility of the hydrophilic natural fibers with the hydrophobic matrix [9]-[11]. Therefore, it is necessary to alter the natural fibers' surface using chemical treatments such as silane, alkali, etc. Different concentration levels and soaking durations are often used for achieving surface modification during the chemical treatment [12]-[16]. These chemical treatments remove surface impurities from the natural fibers, resulting in the formation of hydroxyl groups. Alkali treatment is proving to be a reliable and cost-effective method of reducing the moisture absorption of natural fibers and lowering their lignin and active hemicellulose content. Surface topography, crystallinity index, and thermal stability improve with varying NaOH concentrations and treatment durations [17].

The impact of fiber content and processing conditions on the characteristics of natural fiber-reinforced composites is very significant. To get the best composite properties, the appropriate fiber composition and manufacturing conditions must be carefully determined [18]. *Morus alba* fiber research is driven by the need for sustainable, high-performance composites that reduce environmental impact and efficiently use natural resources. This study used different fiber percentages to investigate the mechanical and thermal properties of the injection molded bio-composite with a poly-lactic acid as the matrix.

2. Materials

2.1. Fiber Extraction

The *Morus alba* stems (**Figure 1(a)**) are gathered from Rajapalayam, Virudhunagar District, Tamil Nadu, India, and are called regular agricultural waste. Before the retting procedure, the segments of *Morus alba* stems are chopped into lengths ranging from 8 to 10 cm and then cleaned with regular water to eliminate any debris. After washing, the chopped pieces are submerged in water for 15 to 20 days. The fibers (**Figure 1(b)**) could easily be extracted from the node stalk's tip because the fiber layers had become pliable following the period. The collected fiber layers were cleaned with distilled water to remove any pollutants or contaminants from the fiber's surface. To prevent decomposition, the extracted fibers are dried in the sun for three to four days to reduce the moisture



Figure 1. (a) *Morus alba* stems; (b) *Morus alba* fiber.

content. Afterward, they are placed under an airtight cover, which is sealed to protect the fibers from damaging environmental effects [4].

2.2. Matrix Material

The analysis's matrix consisted of pellets of polylactic acid, specifically the type 4043D PLA. The 4043D PLA pellets were purchased from reapworld.de and 123-3D.nl. This PLA has a melting point of 155°C to 160°C. Injection molding was conducted at 190°C to 210°C.

2.3. Chemical Treatment

A 4 wt. % sodium hydroxide (NaOH) solution was obtained from Carlroth.com. The dried fibers were soaked in this solution for 1 hour at room temperature, using a 1:10 ratio of fiber to liquid. After treatment, the fibers were rinsed with water 2 to 3 times to remove excess NaOH, then dried in an oven at 100°C for 6 hours to eliminate moisture and impurities.

2.4. Design of Experiment

The composite materials were prepared using three different proportions of fibers and PLA (**Table 1(a)** and **Table 1(b)**).

Table 1. (a) Composition of raw fiber and PLA; (b) Composition of raw fiber and PLA.

(a)	
Composite Designation	Composition (Vol%)
A1	20% Fiber + 80% PLA (Poly-Lactic Acid)
A2	30% Fiber + 70% PLA (Poly-Lactic Acid)
A3	40% Fiber + 60% PLA (Poly-Lactic Acid)
(b)	
Composite Designation	Composition (Vol%)
A1	20% Treated Fiber + 80% PLA (Poly-Lactic Acid)
A2	30% Treated Fiber + 70% PLA (Poly-Lactic Acid)
A3	40% Treated Fiber + 60% PLA (Poly-Lactic Acid)

2.5. Composite Calculation

Six portions of composites used 4 kg polylactic acid and 2 kg fiber, each weighing 1 kg. The weighing machine is used to measure the fiber weight and PLA percentage accordingly.

$$W_F = \frac{\phi_F \rho_F}{\phi_F \rho_F + \phi_M \rho_M} \quad (2.1)$$

With:

W_F - Weight of the fiber,

ϕ_M - Volume of fiber,

ρ_F - Density of fiber,
 ϕ_V - Volume of the matrix,
 ρ_M - Density of matrix.

2.6. Composite Preparation

Treated and untreated fibers were embedded and melted with poly-lactic acid in a twin-screw extruder from Thermo Scientific at 175°C and 30 rpm. Twin screw extruder samples are crushed into fine granule sizes using a granulator machine. The standard samples are prepared using an injection molding machine at 195°C. Mechanical and thermal properties are evaluated.

2.7. Physical Properties

The chemical composition of natural fibers (**Table 2**) plays a significant role in influencing their mechanical and thermal properties [4].

Measurements of the diameters of both treated and untreated *Morus alba* fiber samples are conducted using optical microscopy. A total of 100 samples, each with a 40 mm gauge length, are analyzed, and measurements are taken at three random locations on each fiber sample [19].

2.8. Mechanical Tests

2.8.1. Single Fiber Tensile Test

Single fiber tensile test is conducted on untreated and NaOH-treated *Morus alba* fiber samples using INSTRON 550R (Zwick Roell) type of UTM. The test is performed at a gauge length of 40mm. For each length, 30 fiber samples are prepared. The samples are subjected to an applied load of 1N at a crosshead speed of 5 mm/min. The average tensile strength of each gauge length is calculated by combining the 30 individual tensile strength values.

2.8.2. Flexural Test

The flexure tests were performed using a Zwick Universal testing machine, according to the DIN EIN ESO 178 3-point test standard. Five standard specimens were prepared for each composition, ranging from 80 mm in length to 10 mm in width and 4 mm in thickness. The test was performed with the following parameters 0.1 MPa preload and 5 mm/min test speed. Flexural strength, flexural modulus, and elongation at break were measured using Test Xpert III testing software.

2.8.3. Charpy Impact Test

A Ray Ran advanced universal pendulum and a DIN EN ISO 179 test standard

Table 2. Chemical compositions of *Morus alba* fiber.

Fiber Name	Cellulose [wt-%]	Hemicellulose [wt-%]	Lignin [wt-%]	Wax [wt-%]	Moisture content [%]	Ash [wt-%]	Density [kg/m ³]
<i>Morus alba</i>	60 - 70	15 - 20	10 - 15	0.56 - 1.5	8 - 12	1 - 3	1.3 - 1.5

from RAY-Ran test Equipment Ltd., Warwickshire, UK, were used to perform the Charpy impact tests with an impact energy of 4 joules.

2.9. Thermal Tests

2.9.1. Differential Scanning Calorimetry (DSC)

The Perkin Elmer DSC 7 from Perkin Elmer LAS GmbH, Rodgau, Germany, which is based on the DIN EN ISO 11375-4 test standard, was used to test the blends for the glass transition temperature (T_g), the melting temperature (T_m), and the recrystallization temperature (T_c). The measurements were carried out from 25°C to 200°C at a temperature rate of 10 K/min while nitrogen gas was flowing at a rate of 50 mL/min.

2.9.2. Heat Deflection Temperature (HDT)

A Zwick Roell HDT/Vicat test device was used for measuring the heat distortion temperature according to DIN EN ISO 75. The sample size for this test was 80 mm × 10 mm × 4 mm thick and 64 mm wide. The test was conducted in a confined chamber using silicone oil as the heating medium. After placing the specimens in the chamber, weights appropriate to their size were applied; the specimen was then submerged in the oil and the temperature gradually increased at a rate of 2 K per minute.

3. Results

3.1. Fiber Diameter

The analysis of *Morus alba* fiber samples indicates that untreated MAFs have a larger diameter than chemically treated CQFs, as the chemical treatment reduces the lignin and hemicellulose content. Specifically, untreated CQFs have an average diameter of 0.132 ± 0.2 mm, whereas treated CQFs have an average diameter of 0.126 ± 0.6 mm.

3.2. Single Fiber Tensile Test

The tensile strength of the MAF sample was calculated using a gauge length of 40 mm as shown in **Table 3**.

The tensile strength of the untreated MAF sample is 260.3 ± 41 (MPa) and the elongation at break is 2.06 ± 1.1 (%). The tensile strength of the 4% NaOH treated MAF sample is 340.1 ± 90 (MPa) and the elongation at break is 2.3 ± 1.4 (%). This is because the fiber's soft surface properties are changed to a rough

Table 3. Single fiber tensile strength, Young's modulus, and strain values of both treated and untreated *Morus alba* fiber values.

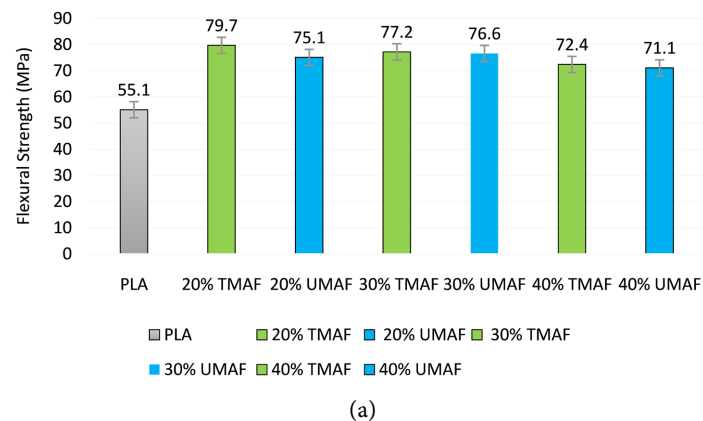
Fiber	Tensile Strength in [MPa]	Young's Modulus in [GPa]	Strain at Break in [%]	Cross-Sectional Area of the Fiber in [mm ²]
Untreated <i>Morus alba</i>	$260.3 \pm 41.x$	10.5 ± 1.1	2.06 ± 1.1	0.0103 ± 0.0025
4% NaOH treated <i>Morus alba</i>	$340.1 \pm 90.x$	15.8 ± 2.4	2.3 ± 1.4	0.0073 ± 0.0040

surface by the alkali treatment, which raises cellulose content and decreases the concentration of hemicellulose and lignin [10]. Treated *Morus alba* fiber has Young's modulus of 15.8 ± 2.4 GPa which is higher than the raw fiber of 10.5 ± 1.1 . Fiber cross-sectional area is reduced for NaOH-treated fibers compared to untreated fibers.

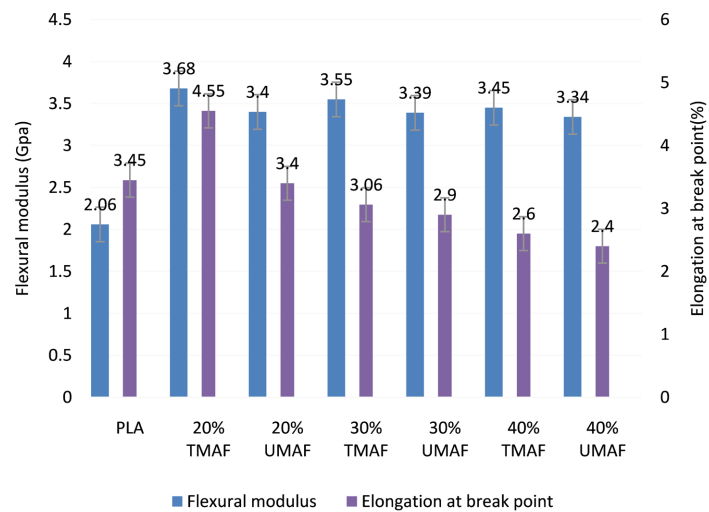
3.3. Flexural Test

The flexural strength of Virgin PLA, treated *Morus alba* fiber composite, and untreated *Morus alba* fiber composite values are shown in **Figure 2(a)**. At weight percentages of 20%, 30%, and 40% of treated *Morus alba* fiber, there were increases in flexural strength of 45%, 40%, and 31%, respectively, compared to PLA. Untreated *Morus alba* exhibits increments of 36%, 39%, and 29% over PLA.

The flexural modulus and elongation at the breakpoint for neat PLA, treated *Morus alba* fiber composite, and untreated *Morus alba* fiber composite is mentioned in **Figure 2(b)**. As the weight percentage of treated samples increases, the



(a)



(b)

Figure 2. (a) Flexural strength of PLA, treated and untreated *Morus alba* fiber composite; (b) The graph shows the difference between flexural modulus and elongation at a breakpoint.

flexural moduli decrease from 8% to 6%. In this instance, 40% of weight percent and 20% of weight percent are increased from 6% to 8% over PLA samples. The elongation at the breakpoint for 20% weight percent treated MAF samples is increased by 32% over PLA. Significantly, 30% and 40% weight percent MAF samples are decreased by 11% and 24% over PLA. The elongation at the breakpoint of untreated samples is decreased over neat PLA samples.

3.4. Impact Strength

The impact strength values of neat PLA, treated *Morus alba* fiber composite, and untreated *Morus alba* composite are mentioned in **Figure 3**. At a 20% weight percentage, the impact strength of *Morus alba* fiber, whether treated or untreated, surpassed that of neat PLA by 56% and 24% respectively. Similarly, at a 30% weight percentage, both treated and untreated *Morus alba* fiber exhibited an increased impact strength of 55% and 14% respectively over neat PLA, while at a 40% weight percentage, the impact strength of treated *Morus alba* fiber composite increased by 23%. However, the impact strength of the untreated *Morus alba* fiber composite at 40% decreased by 15% compared to neat PLA.

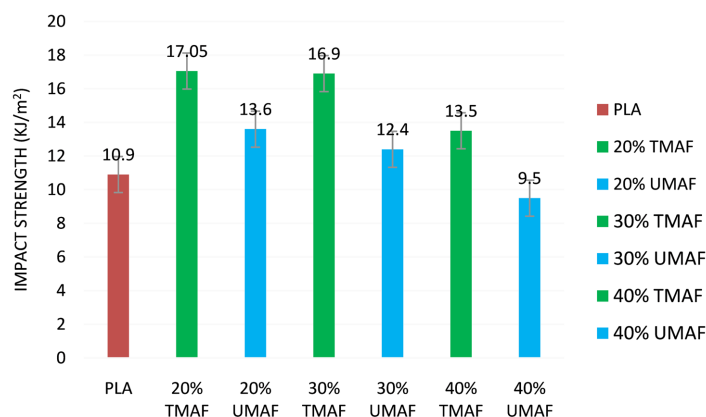


Figure 3. Impact strength of PLA, treated and untreated *Morus alba* fiber composite.

3.5. Differential Scanning Calorimetry

Glass transition temperature (T_g), melting temperature (T_m), and crystallization temperature (T_c) values of virgin PLA treated and untreated *Morus alba* fiber composite are shown in **Figure 4**. The T_g values for treated and untreated *Morus alba* fiber composites range between 53°C and 63°C, whereas PLA exhibits a T_g value of 51.8°C. At a 20% weight percentage, both treated and untreated *Morus alba* fiber composite samples suppressed the T_g of neat PLA by 24% and 18% respectively. Similarly, other samples demonstrated improvements over PLA by 11%, 4%, 5%, and 2%. The T_c ranges from 106.3°C to 113°C for both treated and untreated samples, whereas PLA has a crystalline temperature of 68.6%. The T_c of both 20% and 30% weight percentages of treated and untreated *Morus alba* fiber samples increased by 65%, 59%, 57%, and 56% over

PLA. Meanwhile, at a 40% weight percentage, there was an increase of 52% and 55% over PLA. Consequently, PLA exhibits a melting temperature of 137.4°C, whereas treated and untreated samples have elevated their melting temperature from 172°C to 178°C. As the weight percentage increases, the melting temperature of treated samples rises over PLA by 30%, 28%, and 27%, while untreated samples increase over PLA by 28%, 27%, and 26%.

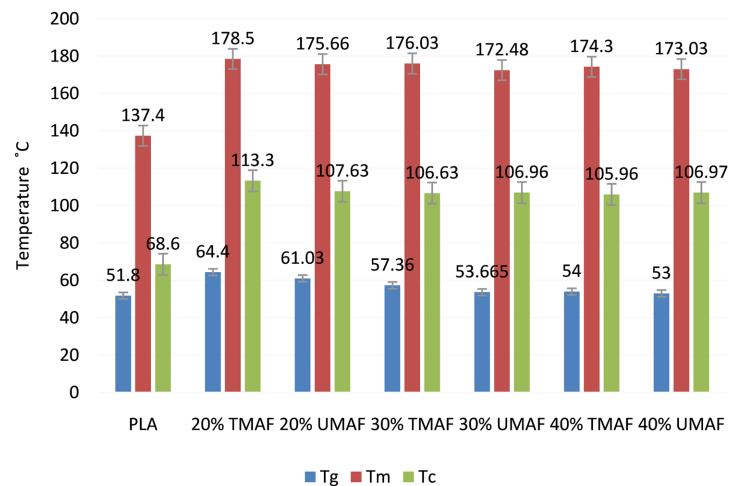


Figure 4. Comparison of Tg, TC, and TM values of different fiber and PLA compositions with PLA.

3.6. Heat Deflection Temperature

The heat deflection values of virgin PLA treated, and untreated *Morus alba* fiber composite samples are shown in **Figure 5**. The material has almost equivalent temperature values to pure PLA and exhibits almost identical thermal stability at high temperatures and under certain loads.

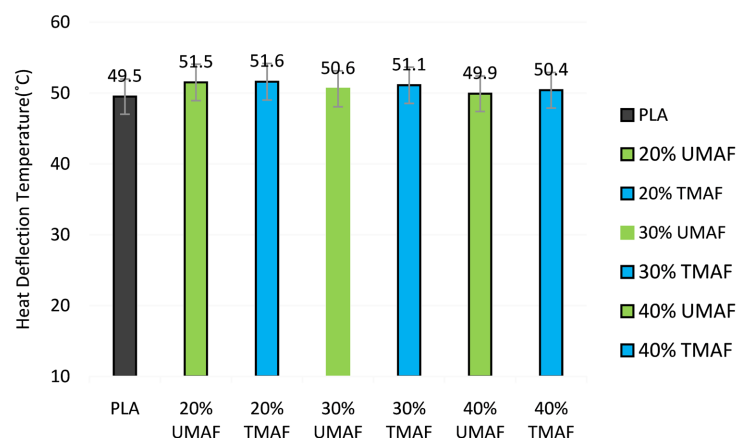


Figure 5. HDT values of PLA, treated and untreated *Morus alba* samples.

4. Discussion of Results

According to the results, the tensile strength of 4% NaOH-treated *Morus alba*

fiber is 340.1 ± 90 (MPa) and thus better than untreated MAF with a tensile strength of 260.3 ± 41 (MPa). This is due to alkali treatment samples having greater resistance against tensile loading and exhibiting higher properties compared to raw fibers. As shown in **Table 4**, the tensile strength of treated *Morus alba* fiber is higher than the tensile strength of jute, *Cissus quadrangularis*, and *Coccinia grandis*.

Table 4. Comparison of Tensile strength with other natural fibers.

S.NO	Fiber	Tensile strength (MPa)	Strain at break in (%)
1.	<i>Morus alba</i>	340.1 ± 90	2.3 ± 1.4
2.	Jute	252 ± 112	2.16 ± 1.5 [20]
3.	<i>Cissus quadrangularis</i>	268.9 ± 51	2.2 ± 1.1 [21]
4.	Sisal	484 ± 135	3.3 ± 1.6 [22]
5.	Kenaf	459.9 ± 46.44	18.8 ± 9.1 [23]
6.	Hemp	353.1 ± 114	3.0 ± 1.5 [24]
7.	<i>Coccinia grandis</i>	273 ± 27.7	2.26 ± 0.28 [17]
8.	Flax	454 ± 40	3 ± 1.5 [25]

Higher values than *Morus alba* fiber, lower values than *Morus alba* fiber.

The alkali treatment improves flexural strength by improving the interfacial bonding between fiber and matrix. However, as the fiber content increases, both treated and untreated MAF samples show a decline in flexural strength due to an improper mixture of fiber matrix of the composite material. Furthermore, the temperature of the injection molding process may cause fiber deterioration, resulting in a drop in the composite's flexural strength. The composite with 20% TMAF content achieved the highest flexural strength. Fiber composites have a higher flexural modulus than pure PLA samples, which average 2.06 GPa. However, fibers that have undergone a chemical treatment have an average modulus of 3.7 GPa. This happened because of the sodium hydroxide's binding properties, which further helped the matrix's capacity to transfer stress. The elongation at the breakpoint is reduced in MAF composite samples compared to PLA samples, with only the 20% TMAF sample exhibiting greater toughness elongation than PLA. The temperature of the injection molding process can cause fiber degradation, resulting in a reduction in the toughness elongation of the composite.

PLA is a brittle polymer, with an average impact strength of 10.94 kJ/m^2 during testing. The addition of *Morus alba* fiber to the PLA matrix resulted in a progressive decrease in impact strength with increasing fiber content, ranging from 13.6 kJ/m^2 at 20% MAF content to 9.5 kJ/m^2 at 40% fiber content, as indicated in **Figure 2**. The stiffening effect of the matrix might be responsible for the lower impact strength found in the tested composites. Inadequate bonding at the interface of composite components leads to poor stress transmission. The highest value for 20% TMAF is 17.05 kJ/m^2 and the lowest for 40% TMAF is 13.5

kJ/m^2 . Chemical treatment with sodium hydroxide improved the adhesion between fiber and matrix. *Morus alba* fiber composites have better mechanical properties than other natural fibers such as *Cissus quadrangularis*, hemp, jute, kenaf, and *Coccinia grandis*, as shown in **Table 5**.

Table 5. Comparison of other natural fiber's mechanical properties and PLA.

S.NO	FIBER	FLEXURAL STRENGTH (MPa)	IMPACT STRENGTH (KJ/m ²)
1.	<i>Morus alba</i> (20% Fiber+ 80% PLA)	79.7	17.05
2.	<i>Cissus quadrangularis</i> (20% Fiber+80% PLA)	60 - 79.3	9.8 - 18.7 [6]
3.	Kenaf (20 Fiber + 80% PLA)	60 - 76.3	4.6 - 8.8 [26]
4.	Hemp (20 Fiber+ 80% PLA)	51.5 - 75.8	7.6 - 13 [27]
5.	Jute (20 Fiber+ 80% PLA)	51.7 - 77.6	6.2 - 8.9 [26]
6.	Sisal (20% Fiber + 80% PLA)	50.4 - 80.7	8.1 - 11.4 [26]
7.	<i>Coccinia grandis</i> (20% Fiber + 80% PLA)	50.3 - 75.6	4 - 9.6 [17]
8.	Flax (20% Fiber + 80% PLA)	55 - 82	7 - 11.6 [28]
9.	Poly-Lactic Acid	2.06	10.94

Higher values than *Morus alba* fiber & PLA, lower values than *Morus alba* fiber & PLA.

In contrast to pure PLA, natural fibers can improve the crystalline temperature of the composite by acting as nucleation sites for polymer crystallization. As more heat is released or absorbed during the crystallization and melting transitions, crystallinity increases, resulting in higher DSC values. This is due to the PLA matrix's low degree of polymerization, strong molecular forces, and restricted chain mobility. Fibers that can support greater temperatures without degradation might affect the composite's overall thermal performance.

It has been observed that both treated and untreated *Morus alba* fiber composite samples have improved thermal properties compared to pure PLA. This helps to retain the material's shape and orientation while applying higher temperatures to the composite material.

5. Conclusions

This research comprehensively studied the mechanical and thermal properties of treated and untreated *Morus alba* fiber-reinforced polylactic acid (PLA) composites, highlighting their potential as sustainable and environmentally friendly alternatives to traditional plastics.

- The tensile strength of 4% NaOH-treated *Morus alba* fibers (TMAF) was significantly higher (340.12 ± 90 MPa) compared to untreated MAF (260.32 ± 41 MPa). This improvement is attributed to the alkali treatment, which increases the fiber's resistance to tensile loading.

- Alkali treatment improved the flexural strength and modulus of the composites. The highest flexural strength was achieved with 20% TMAF content.
- PLA is naturally brittle, with an average impact strength of 10.94 kJ/m². The incorporation of *Morus alba* fiber reduced the impact strength of the composites, with the highest value at 20% TMAF (17.05 kJ/m²) and the lowest at 40% TMAF (13.45 kJ/m²).
- The elongation at break was reduced in MAF composite samples compared to pure PLA samples. Only the 20% TMAF composite exhibited greater toughness elongation than PLA.
- NaOH treatment raised the glass transition temperature (T_g) to between 53°C and 63°C, compared to 51.8°C for pure PLA. The melting temperature of the composites also increased, indicating improved thermal stability.

In conclusion, the study shows that *Morus alba* reinforced PLA composites, especially when treated with NaOH, showed improved mechanical and thermal properties compared to pure PLA and other natural fiber composites. These results highlight the viability of using *Morus alba* as a reinforcing material in the development of sustainable, high-performance composites for various applications.

Future studies will aim to enhance the properties of *Morus alba* fibers by incorporating advanced manufacturing techniques and surface treatments. The goal is to develop high-performance composite materials. This research into improved compatibility and manufacturing processes has the potential to transform the practical uses of these composites across various industries.

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Author Contributions

Girish Kumar Reddy Madda contributed to the data collection, synthesis, and writing of the initial drafts of the manuscript. Jens Schuster helped with the research progress and reviewing the report. The Methodology approach was taken by Yousuf Pasha Shaik and Girish Kumar Reddy Madda. The Supervision of Jens Schuster and Yousuf Pasha Shaik aided in the review of the report. All authors have read and agreed to the published version of the manuscript.

Data Availability Statement

Not Applicable (NA)

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Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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