Macromolecular Materials and Engineering
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Epoxy Composites Reinforced by Alkaline–Acid Treated Yucca Fibers

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This study investigates the impact of chemical treatments on the physico–chemo–mechanical properties of mechanically extracted yucca fibers and the mechanical performance of epoxy-based bio-composites. Initially, yucca fibers are treated with sulfuric acid (H_2SO_4 at 1% and 4%, during 30 min) and sodium hydroxide (NaOH at 3% and 8%, during 120 min), aiming to enhance their properties. Scanning electron microscope (SEM) analysis reveals improved surface quality of fibers and reduced diameters, while fourier transform infrared spectroscopy (FTIR) confirms their functional groups. Yucca-treated fibers exhibit significant tensile strength improvements, with increases of 101.22% (518 MPa) for fibers treated with 3% NaOH and 93.33% (497 MPa) for those treated with 1% sulfuric acid, compared to untreated fibers (257 MPa). Subsequently, bio-composites elaborated via mold casting technique and using these treated fibers demonstrate enhanced mechanical performance. Tensile strength increases by 25% (29.6 MPa) and 45% (34.4 MPa) for composites reinforced with fibers treated with 3% NaOH and 1% sulfuric acid, respectively, compared to untreated fiber composites (23.6 MPa). Similarly, compressive strength improves by 23.35% (79.5 MPa) and 9.85% (89.3 MPa), while bending strength reaches 41.03 MPa (4.3%) and 45.27 MPa (15.1%) for composites reinforced with fibers treated at 3% NaOH and 1% sulfuric acid, respectively.

1. Introduction

Synthetic fibers have long dominated the composite materials industry due to their superior mechanical properties and long-term durability.[1] However, the production of these fibers requires energy-intensive and costly processes, making a significant contribution to environmental pollution.[2] In addition, the difficulty of recycling these fibers and their growing ecological impact have led to an intense search for more sustainable solutions in the reinforcement of composite materials.[3,4]

In recent years, numerous researchers have shown a growing interest in biomass, particularly in exploring natural fibers as an eco-friendly alternative for reinforcing polymer matrices,[5] motivated by the urgent need to reduce environmental impact and promote sustainable materials.[6,7] This growing interest has led to the development of a new generation of composites reinforced with biodegradable fibers known as bio-composites, offering sustainable solutions for a variety of different applications.[8,9] In this context, natural fibers, are sourced from renewable resources such as plants or animals,[10,11] present a number of advantages, especially their biodegradability,[12] low density[13] and good mechanical properties,[14] as well as economic benefits due to their low production cost.[15] In addition, natural fibers have a lower energy demand in production compared to synthetic fibers, contributing to a more sustainable manufacturing process. **Figure 1** presents some additional advantages. In addition, these natural fibers can help reduce carbon footprints while offering thermal and acoustic insulation properties, making them particularly attractive for industrial applications seeking to combine performance and environmental sustainability.[16] The utilization of this type of fiber can be beneficial in several fields, such as the production of ropes in the textile industry,[17] and the manufacture of parts for the automotive, aerospace, and general construction industries in the composite reinforcement field.[18,19]

The natural fiber comes from plants such as banana, sisal, coir, jute, and others known as lingo-cellulosic fibers.[20] They are generally composed of cellulose, lignin, hemicellulose, and other constituents, with each component playing a specific role in the fiber's properties.[21] Nevertheless, cellulose is the principal component of the natural fiber,[17] represents the crystalline

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DOI: 10.1002/mame.202500067

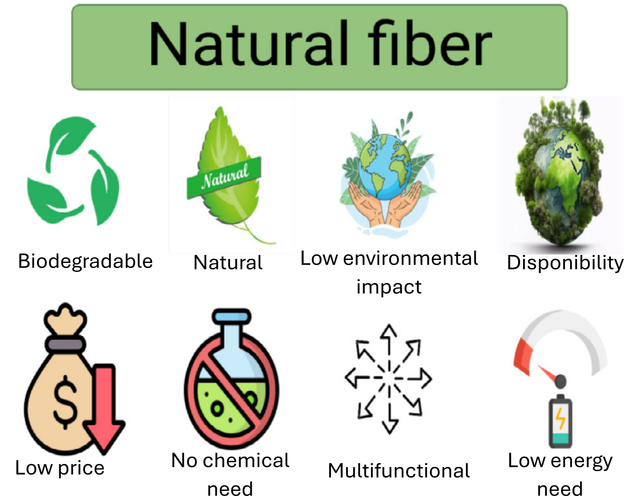

Figure 1. Natural fiber advantages.

phase, and is the element responsible for its tensile strength.[22] Lignin and hemicellulose have a medium content in the bio fiber relative to cellulose, typically ranging between 10% to 25% depending on the fiber type.[23] These two components represent the amorphous phase, they are responsible for the rigidity and the flexibility of the natural fiber respectively.[24]

Primarily, optimal utilization of this natural resource for its intended applications necessitates the prior extraction of the fiber from the plant. Numerous studies have highlighted that a range of extraction techniques exists, which can be broadly categorized into mechanical, chemical, and biological methods.[25] Nevertheless, raw natural fibers inherently comprise lignin, hemicellulose, and surface waxes, which are fundamental structural components of lignocellulosic fibers. While these biopolymers contribute to the mechanical integrity of the fiber, their presence, particularly in excessive amounts, can hinder interfacial adhesion with polymer matrices due to their hydrophobic and crosslinked nature.[26] Consequently, limiting the mechanical performance of bio-composites. However, the improvement of the interfacial compatibility and mechanical properties of bio-fibers and final bio-composites relies essentially on the realization of surface modification treatments after the fiber extraction step. In this context, natural fiber surface treatments were classified into various families of methods, such as physical, biological, and chemical treatments. Chemical surface treatment of natural fibers involves the use of chemical agents, such as acids or alkalis, with the aim of eliminating impurities and modifying the surface structure of the fibers.[27] By increasing their durability as well as their compatibility with polymer matrices. It enhances the mechanical properties of the final bio-composites.[28]

Since these surface treatment categories, a number of studies have examined the chemical treatment's effect on natural fiber proprieties, via the use of acid agents (such as sulfuric acid or acetic acid) or basic agents (such as NaOH or KOH),[29] and can summarize the main phases for studying a chemical treatment on natural fiber in **Figure 2**. However, in literature, treatment with NaOH at 2% demonstrates a ≈10% improvement in the mechanical properties of natural sisal fiber compared to untreated fiber,[30] whereas the same fiber was treated with acetic acid and exhibited an improvement of 14.1% compared with untreated fiber.[31] A second study indicated that pineapple fiber treated with the chemical agent NaOH was stronger than untreated fiber, with an increase of 50%.[32] On the other hand, the yucca fiber is currently undergoing extensive research in the world of natural fibers and composite reinforcement, which is motivated to study the influence of chemical treatments on this type of fiber and to improve their physicochemical and mechanical properties. In the literature, traditionally extracted Iranian yucca fiber was characterized to assess the influence of alkaline treatment using RSM (response surface methodology). The results showed a significant increase in mechanical strength, from 310 to 446 MPa, an improvement of 44%, for a treatment with 4% NaOH for 7.5 h.[33]

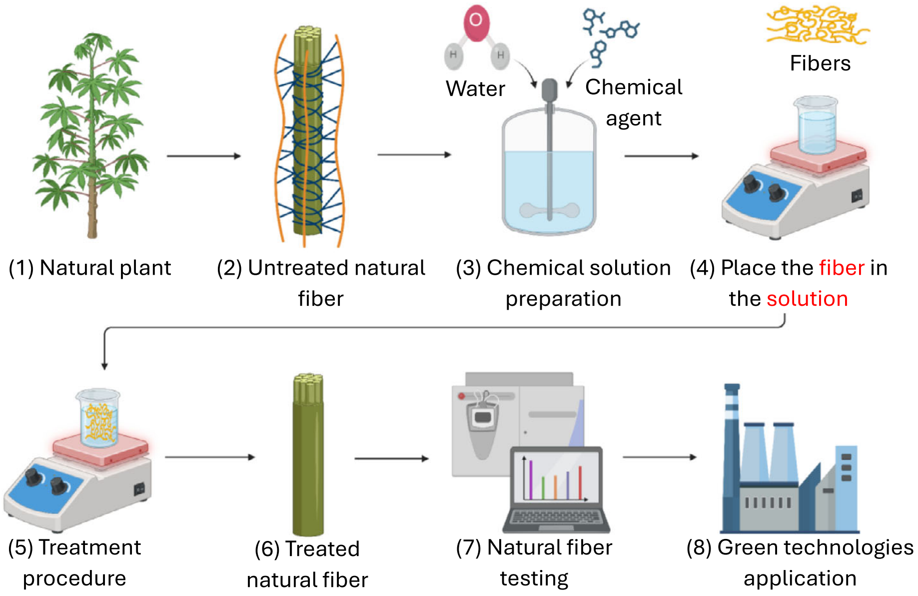

Figure 2. Essentials steps for the chemical treatment of natural fibers.

However, this study investigates the effects of two distinct chemical treatments, sodium hydroxide (NaOH) and sulfuric acid (H_2SO_4), applied at different concentrations to evaluate their impact on the physicochemical and mechanical properties of the fibers, as well as on the performance of the resulting bio-composites. While most existing studies have predominantly focused on alkaline treatments, this work introduces a comparative approach that also examines acid treatment, a less commonly explored technique in this field. By integrating both treatment methods, this study provides novel insights into the optimization of yucca fibers for bio-composite applications.

In the world of materials science, the combination of natural fiber with a polymer leads to the birth of a new material known as bio-composite material. This type of material offers numerous advantages in terms of biodegradability, mechanical and thermal performance as well as production economy.[34–36] The use of raw natural fiber reinforcement shows several limitations, especially in terms of adhesion between the natural fiber and polymer in the bio-composite, which leads to low mechanical performance, as described in the open literature.[37] However, the application of chemical treatment to natural fibers can open a great deal of potential in this area, particularly in the modification of fiber surfaces, which can subsequently enhance their adhesion in the bio-composite, and increase the mechanical performance of the final bio-composite.[38,39] Recent studies have shown that the bio-composite base of treated jute fibers performs perfectly compared to that reinforced using untreated fibers, with a tensile strength of 39.7 MPa compared to 49 MPa, at a 23% improvement ratio.[40] The same observation was noted in a different study of pineapple fiber-reinforced bio-composite, with an advantage for the type of treated fiber-reinforced bio-composite with a strength of 32 MPa, and an enhancement of 25.5%.[41] To the best of the authors' knowledge, yucca fiber has great potential to be treated and employed as a reinforcement of the bio-composite, making it the key motivation for this research.

This study examines the influence of chemical treatments on natural yucca fiber. The primary objective is to extract the yucca fiber from the plant leaves employing a mechanical method, more precisely the defibration method, and then treating the fiber chemically. This investigation evaluates the influence of two chemical treatments on the mechanical, physical, and chemical properties of natural fibers, as well as on their adhesion in the final bio-composite. Based on the hypothesis that chemical treatments enhance the interaction between fibers and the matrix by altering the fiber surface, removing unwanted components, and increasing surface roughness, NaOH and sulfuric acid treatments at different concentrations are applied to the yucca fibers. These treatments are then followed by the fabrication of new bio-composites based on epoxy resin, which are subsequently analyzed. Scanning electron microscopy (SEM), Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), and tensile tests are employed to determine the physicochemical and mechanical proprieties of treated and untreated yucca fiber as well as to evaluate the chemical treatment methods. Subsequently, SEM and mechanical tests including tensile, bending, and compression tests are carried out with the aim to determine the chemical treatment influence on the final bio-composites materials.

2. Experimental Section

2.1. Materials Overview

In this study, various chemical reagents were employed for the pre-treatment, neutralization, and chemical modification of the natural fibers, along with an epoxy-based polymer matrix for composite fabrication. Sodium carbonate (Na_2CO_3, purity 99.7%) and sodium bicarbonate ($NaHCO_3$, E500) were used for the pre-treatment and neutralization steps, both supplied by AdenLabo, Algeria. The chemical treatments were conducted using sodium hydroxide (NaOH) in pellet form with a purity of 99% and sulfuric acid (H_2SO_4) with a concentration of 98%, both obtained from AdenLabo, Algeria. The polymer matrix used for composite manufacturing was a commercial epoxy resin supplied by Genc, Turkey, with a density of 1.3 g cm^{-3}. Moreover, this epoxy resin was a bisphenol A-diglycidyl ether (DGEBA) type resin. This selection of materials ensured a controlled and reproducible methodology for investigating the influence of chemical treatments on fiber properties and composite performance.

2.2. Yucca Leaves and Fiber Preparation

Yucca fibers originated from the leaves of plants belonging to the *yucca treculeana*, which were primarily found in arid and semi-arid regions. These plants were characterized by flat, green leaves measuring ≈50 to 120 cm in length and 3 to 8 cm in width. With an annual production capacity of ≈60 to 80 leaves per plant, yucca represented an economically viable resource with significant potential for fiber extraction.[42] Traditionally, yucca fibers were utilized in rope-making, textiles, and handicrafts due to their durability and strength. In addition, the presence of hydroxyl and carboxyl functional groups in yucca fibers enabled effective interfacial bonding with polymer matrices, making them suitable for bio-composite applications. The yucca fiber studied in this research was recovered from the leaves of the yucca plant that grows in Hadjout region, Algeria, in the geographic coordinates at 36.5122° N, 2.4143° E. In this work, a mechanical extraction method was employed to extract the natural yucca fiber.

2.3. Fiber Extraction Methods

The mechanical extraction method category was represented by the fiberizing method. This technique involved the application of manual force to the leaves of the yucca plant, utilizing a wooden implement to facilitate the processing. As the manual force was exerted, the organic material was progressively separated and displaced, allowing the fibers to be released. The extracted fibers had an average length of ≈25 cm. Once isolated, bio-fibers, with a calculated density of ≈1.45 g cm^{-3}, could easily be released from organic matter, air-dried for 48 h, with the aim to eliminate the moisture and water present on the natural yucca fiber, before the chemical treatment stage.

2.4. Chemical Treatment Process

The chemical treatment on the natural yucca fiber was carried out in a complete process; the treatment underwent several steps as pre-treatment, chemical treatment, and neutralization.

2.4.1. Pre-Treatment Stage

The pre-treatment of the natural fiber aimed to remove organic impurities and surface waxes, as well as increasing the hydrophilicity of the fiber and preparing it for the chemical treatment. However, in this stage, the yucca fiber employed in this study was immersed in a sodium carbonate (Na_2CO_3) solution at 4% concentration for a period of one hour at room temperature. Once the reaction time elapsed, the fibers were removed from the solution, washed with distilled water, and prepared for the next stage

2.4.2. Chemical Treatment Stage

In order to improve the mechanical properties of the natural fiber and its compatibility with the polymer matrix, an alkaline and acid treatment was carried out on the bio-fiber. First, to eliminate the surface impurities and enhance the roughness, the yucca fibers were immersed in a NaOH solution at various concentrations (3% and 8% (w/v)) at room temperature for 120 min (2 h), and then the fibers were carefully washed with distilled water. In another treatment method, with the aim to modify the chemical structure of the fibers and improve their reactivity as well as the mechanical properties, an acid treatment was performed by immersing the yucca fibers in a sulfuric acid (H_2SO_4) solution at various concentrations (1% and 4%) for 30 min at room temperature. Subsequently, meticulous washing with distilled water was performed on the treated yucca fibers.

Adequate neutralization was required to remove excess acid or alkali and stabilize the fibers prior to the analysis phases.

2.4.3. Neutralization Stage

After the alkaline or acid chemical treatment stage, the yucca fibers were subjected to a neutralization stage in order to eliminate the excess chemical reaction and stabilize their pH. This neutralization was carried out by immersing the yucca fibers in a 1.5% (m/v) aqueous sodium bicarbonate ($NaHCO_3$) solution for 45 min at room temperature. The solution was carefully stirred to ensure a homogeneous distribution of the neutralizer on all fiber surfaces. Subsequently, the fibers were rinsed several times with distilled water until the pH of the wash water was close to neutral (pH ≈ 7). Finally, the neutralized yucca fibers were dried in a drying oven at 65 °C for 24 h to eliminate any residual humidity before proceeding to the analysis and characterization phases.

2.5. Natural Fibers Tests

2.5.1. Surfaces Characterization and Morphological Analysis (SEM)

Treated and untreated yucca fibers were subjected to a morphological examination with the purpose of observing the surface quality of each category, and assessing the influence of chemical treatment on the physical characteristics represented by the final diameter of the natural yucca fiber. The diameter was measured using specialized software integrated into the scanning electron microscopy (SEM) machine, with measurements taken in five different zones. To enhance conductivity and obtain high-resolution SEM images, a thin carbon coating was applied to all fiber samples before observation. The analysis was conducted using the JSM-7610Fplus machine under normal conditions.

2.5.2. Functional Group Analysis (FTIR)

Fourier transform infrared spectroscopy (FTIR) was employed to analyze the modifications in the chemical structure of natural yucca fibers before and after the chemical treatments. Untreated fiber samples, as well as alkaline and sulfuric acid-treated fiber samples, were analyzed using an FTIR spectrometer (Shimadzu IRAffinity-1S). FTIR spectra were recorded in the 4000 to 400 cm^{-1} range, with a resolution of 4 cm^{-1}. To enhance spectral accuracy and minimize noise, each spectrum represented the average of 32 consecutive scans.

2.5.3. Crystalline Structure Characterization (X-Ray Diffraction)

In order to determine the crystalline structure (the crystallinity index (CI), and the crystallinity size (D)) of untreated and chemically treated fibers. The yucca fibers, before and after the alkaline and sulfuric acid treatments, were analyzed using an X-ray diffractometer (model PANalytical Empyrean) using CuK-λ radiation ($K\lambda = 0.15\,425$ nm) at 45 kV and 40 mA. The X-ray analysis was performed in an angular range of 5° to 80° (2θ), with a scanning speed of 2° min^{-1}, in order to identify the peak corresponding to crystalline cellulose and amorphous phases.

However, for properly interpreting the results from the X-ray graphs, the equations of Segal Equation (1) and Scherrer Equation (2) were employed in this section of research to determine the crystallinity index (I) and the crystallinity size (CS), respectively.

$$CI = \frac{I_{cr} - I_{am}}{I_{cr}} \times 100 \tag{1}$$

where CI is the crystallinity index, I_{cr} and I_{am} corresponds to the maximum intensity of the crystalline peak and the minimum intensity in the amorphous region respectively.

$$CS = \frac{\mathrm{K}\lambda}{\beta \cos(\theta)} \tag{2}$$

In this context, CS is the crystallinity size, K is Scherrer's shape constant ($K = 0.9$), λ is the X-rays wavelength, β is the peak width at half-height in radians, and θ is the diffraction angle corresponding to the peak I_{cr}.

2.5.4. Mechanical Performance Evaluation

The mechanical properties of yucca fibers, before and after chemical treatments, were assessed in accordance with the ASTM D3822 standard that specified the methods for measuring the tensile strength of a single fiber. Untreated and treated fibers were subjected to tensile tests with the aim to determine their

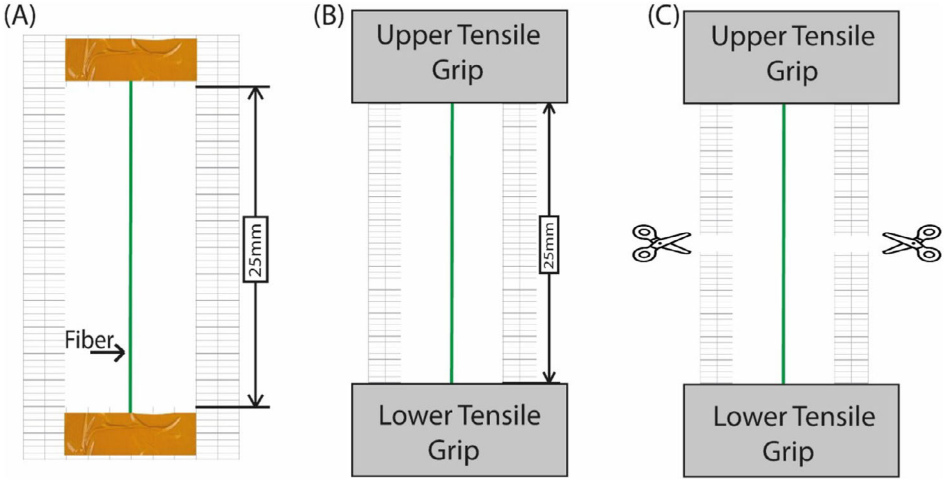

Figure 3. Natural fiber tensile test process, A) Glued the fiber on the sheet, B) Mounted in the machine jaws, C) Cut the sheet extremes, and run the test.

stress at break, elongation at break, and Young's modulus. The tests were conducted using a universal tensile testing machine (zwickiLine, Germany) equipped with a 2.5 N load cell.

The single fiber samples were prepared according to ASTM D3822-01, with a fiber length of 50 mm, glued to a specially prepared sheet, and carefully mounted in the jaws to prevent slippage as shown in **Figure 3**. The strain speed used was 1 mm min^{-1}, the fibers were tested until they broke, and an average number of 10 tests for each fiber category in order to obtain reliable results.

2.6. Biocomposites Preparation and Testing

2.6.1. Samples Preparation

Treated and untreated natural yucca fiber were grinded into a fine powder, and a 100-mesh sieve with an aperture size of ≈150 µm was used to ensure a uniform particle size distribution, allowing only particles of 150 µm or smaller to be retained. The sieved yucca fiber powder (10% by weight) in each category was mixed with epoxy resin (100 g Epoxy, 28 g Hardener) in accordance with the manufacturer's recommendations (Genc, Turkey). A 10 wt.% fiber content was chosen to balance mechanical performance and processability. The resulting mixture was manually mixed, to ensure an even distribution of fibers within the epoxy resin. The mixture was subsequently used to fabricate bio-composite test specimens through the mold casting technique using silicone molds. In this context, each mold was designed for a specific shape in order to produce a normalized specimen in accordance with the ASTM standard. Silicon molds for the production of tensile test specimens according to the ASTM D638 standard, compression test specimens according to the ASTM D695 standard, and bending test specimens according to the ASTM D790 standard were employed in this study.

Prior to mechanical testing, all samples were allowed to cure completely to ensure that the epoxy reached its optimum mechanical properties. Each sample then underwent a rigorous measurement process, during which its thickness and width were assessed at several points to ensure that the dimensions were in accordance with the standards. Only specimens that met the exact dimensions required, without any defects, were selected for testing. **Figure 4** explains the different steps required to elaborate the bio-composite samples for each experimental test.

2.6.2. Fiber Epoxy Adhesion Observation (SEM)

Adhesion of treated and untreated yucca fibers to the epoxy resin was assessed via analysis of the microstructure of the bio-composites. Epoxy resin samples reinforced with yucca fiber samples were examined using a scanning electron microscope (SEM) (model Zeiss GEMINI SEM 300) to observe the fiber–matrix interface and identify any adhesion defects, such as fiber pull out or voids. The SEM observations were also employed to analyze in detail the compatibility and interfacial bonding of the yucca fibers in the epoxy matrix, and to assess the impact of chemical treatments on enhancing the fiber's adhesion to the resin. The examination was performed on the cross-sections of tensile-tested specimens, which were cut into smaller fragments to accommodate the microscope chamber. To enhance electron conductivity and mitigate charging effects, a thin carbon coating was applied to the samples before imaging.

2.6.3. Mechanical Performance of Biocomposite

In order to assess the influence of the chemical treatment of natural fibers on the mechanical properties of bio-composites, as well as to compare the performance of composites reinforced with untreated fibers, those treated with NaOH, and those treated with sulfuric acid. Five (5) samples from each bio-composites category were examined under tensile, compression, and bending tests in accordance with ASTM D638, ASTM D695, and ASTM D790, respectively. The specimens were molded directly into their final shape, without any additional cutting or machining, ensuring that the mechanical properties were evaluated in their as-fabricated state. Tensile tests were carried out using a universal

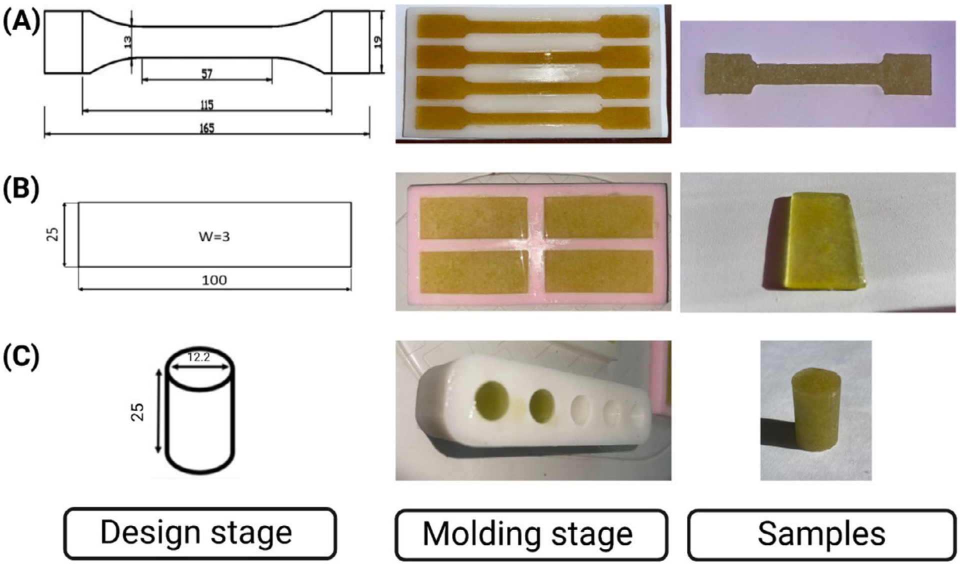

Figure 4. Procedure to elaborate bio-composite samples, A) Bio-composite tensile test samples, B) Bio-composite bending test samples. C) Bio-composite compression test samples (all dimensions in mm).

tensile testing machine (Zwick 8306, Germany) equipped with a 10 kN load cell, at a speed of 2 mm min^{-1}, to measure tensile strength, elongation at break, and determine the Young's modulus. Cylindrical bio-composite specimens measuring 12 mm in diameter and 25 mm in height were tested using a universal testing machine (Zwick 8306, Germany) at a strain rate of 2 mm min^{-1} with the aim of measuring maximum stress and compressive strength. Bending tests were carried out in a three-point bending configuration, with a span of 50 mm, to determine the bending strength and bending modulus of each bio-composite category. **Table** 1 shows the necessary equations used in this study to calculate the different mechanical properties of the bio-composites in each test, as well as the definitions of the symbols used in the equations.

2.7. Nomenclature

Table 2 presents the nomenclature employed in this study to designate the different categories of yucca fibers and bio-composites. Each sample was identified with a specific abbreviation that indicates the type of yucca fiber, the treatments applied to the yucca fibers, and the chemical agent concentrations (%.alkaline or %.acid treatment), as well as the categories of yucca fiber/epoxy bio-composites studied. This classification aimed to simplify the results presentation and to facilitate the comparison between the results of different sample categories obtained from the various experimental analyses performed.

3. Results and Discussion

3.1. Yucca Fiber Properties

3.1.1. Surface Condition Results (SEM)

Scanning electron microscope observations of yucca fibers, shown in **Figures** 5, 6, and 7, reveal marked differences between untreated and chemically treated fibers.

Table 1. Mechanical calculation formulas (all equations expressed in MPa).

Test	Stress	Equation	Symbol definition
Tensile	Tensile strength	$\sigma_t = \frac{F_t}{A}$	F_t: Force applied [N] A: Cross-sectional area [mm^2]
	Young's modulus	$E_t = \frac{\sigma_t}{\epsilon_t}$	σ_t: Tensile stress [MPa] ϵ_t: Tensile strain
Compression	Compressive strength	$\sigma_c = \frac{F_c}{A}$	F_c: Force applied [N] A: Cross-sectional area [mm^2]
Bending	Bending strength	$\sigma_f = \frac{3 \times F_f \times L}{2 \times b \times d^2}$	F_f: Force applied [N] L: Span length [50mm]
	Bending modulus	$E_f = \frac{L^3 \times F_f}{4 \times b \times d^3 \times D}$	b: Sample width [mm] d: Sample thickness [mm] D: Proportional limit point deviation

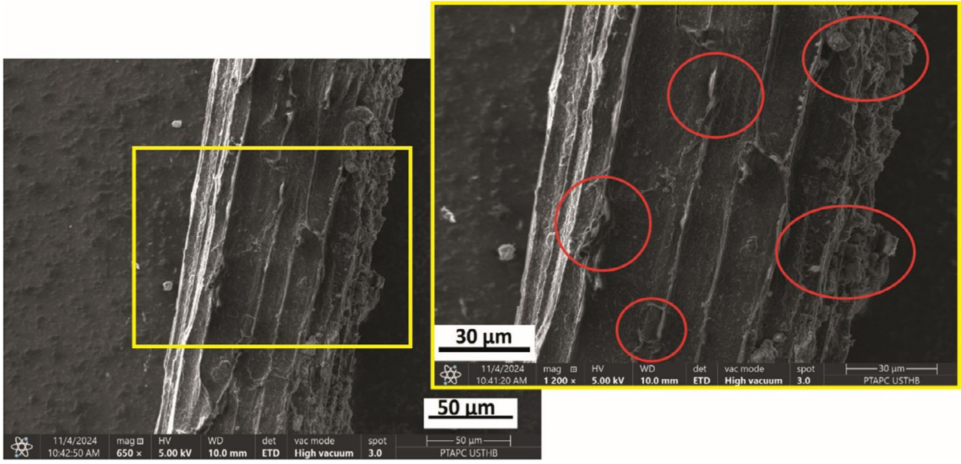

Figure 5. Untreated yucca fiber SEM image.

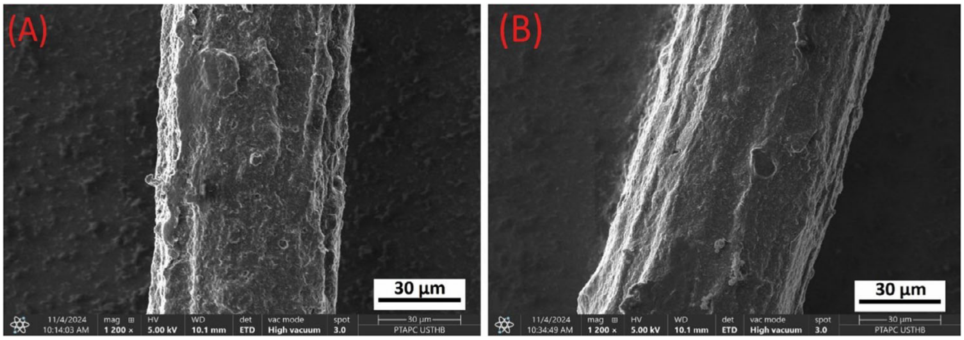

Figure 6. Yucca fiber treated with NaOH, (A) yucca fiber treated at 3%, (B) yucca fiber treated at 8%.

As illustrated in Figure 5, the untreated yucca fiber displayed a relatively smooth surface and covered with various non-cellulosic substances in comparison to the other fiber categories, suggesting the presence of lignin and natural waxes. These components form a protective layer over the cellulose fibrils, potentially reducing adhesion with the polymer matrix. Moreover, the yucca fibers treated with NaOH shown in Figure 6a,b exhibited a surface modified by the alkaline action, which led to partial degradation of these secondary components.[43] In addition, a fine residual layer can be observed on the surface of both categories of fibers treated with NaOH, probably results of incomplete degradation of fiber components, notably lignin and hemicelluloses. This phenomenon can result in a non-uniform film while offering additional adhesion sites with the matrix in composite applications.[44]

In contrast, yucca fibers treated using sulfuric acid which are presented in Figure 7a,b, exhibited a rougher and more eroded surface, often characterized by micro-cracks. The acid treatment effectively dissolves amorphous components, notably lignin and hemicelluloses, exposing the cellulose fibrils and increasing the

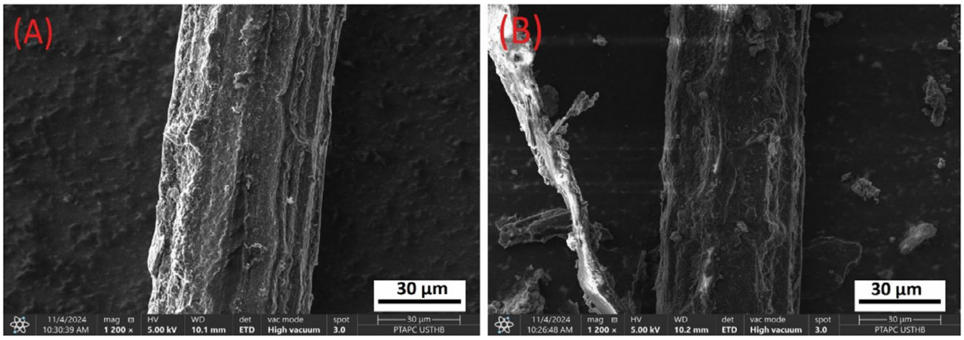

Figure 7. Yucca fiber treated with Sulfuric acid, (A) yucca fiber treated at 1%, (B) yucca fiber treated at 4%.

Table 2. Summary of nomenclature for yucca fiber treatment and bio-composite compositions.

Real designation	Abbreviation
Untreated yucca fiber	UYF
Yucca fiber treated at 3% NaOH	TYFN3
Yucca fiber treated at 8% NaOH	TYFN8
Yucca fiber treated at 1% sulfuric acid	TYFS1
Yucca fiber treated at 4% sulfuric acid	TYFS4
Epoxy reinforced with untreated yucca fiber	EUY
Epoxy reinforced with treated yucca fiber at 3% NaOH	EYN3
Epoxy reinforced with treated yucca fiber at 8% NaOH	EYN8
Epoxy reinforced with treated yucca fiber at 1% sulfuric acid	EYS1
Epoxy reinforced with treated yucca fiber at 4% sulfuric acid	EYS4

surface roughness.[45] This rougher morphology can be beneficial for adhesion.[13] While acid treatment can offer advantages, its excessive application may adversely affect the cellulose structure, leading to increased fiber brittleness.[46] However, yucca fiber treated with 1% sulfuric acid (Figure 7a) shows a relatively intact cellulosic structure, with visible texture, although there are no obvious signs of excessive degradation. As a result, this treatment can offer a good compromise between improving adhesion and preserving the mechanical structure of the fiber.

Figure 7b presents the surface morphology of yucca fiber treated with 4% sulfuric acid. This treatment induces a more severe chemical degradation of the non-cellulosic components, leading to a significantly eroded surface structure. Treatment with this method further exposed the cellulose fibrils, creating an extremely rough surface that could further promote adhesion with a matrix, although it may also make the yucca fibers more fragile.

In another context, the yucca fiber diameter analysis results, for treated and untreated fibers, presented in **Table 3**, demonstrated significant variations depending on the chemical treatments employed. The untreated fibers have an average diameter of ≈110 µm, which served as a reference for the other categories. After treatment with 1% sulfuric acid, the average yucca fibers diameter reduced to 59 µm, while treatment with 4% Sulfuric acid resulted in a further reduction to 51 µm. These results suggest that sulfuric acid degrades the fibers, which reduces their diameter and consequently may lead to an improvement in the specific surface area for composite applications. Conversely, the NaOH treatment also resulted in noticeable changes in the dimensions of the yucca fibers. However, the fibers treated with 3% NaOH have an average diameter of ≈76 µm, and those treated with 8% NaOH have a diameter ≈68 µm. In comparison, these values are inferior to the untreated yucca fibers and superior to those measured for sulfuric acid treatments categories. Nevertheless, alkaline treatment with NaOH leads to a more moderate degradation process, mainly through lignin solubilization and cell structure modification, which may lead to a small decrease in fiber diameter.[47] From a chemical perspective, NaOH induces the cleavage of aryl–ether bonds in lignin, as these bonds are relatively weak and susceptible to hydrolysis by the attack of the hydroxyl group (—OH) from NaOH, which acted as a nucleophile.[48] This bond cleavage resulted in the release of lignin fragments that were soluble in the basic solution.[49] A 3% NaOH concentration provided an adequate amount of OH^- ions to break the ether bonds of lignin. According to the results obtained, the NaOH treatment seems less aggressive than the acid treatment on the physical properties of the yucca fibers.

Table 3. The average diameter of treated and untreated yucca fibers.

Samples type	Average diameter [µm]	Standard deviation [µm]
UYF	110	5.12
TYFN3	76.6	4.37
TYFN8	68.4	3.85
TYFS1	59.6	2.98
TYFS4	51.8	2.63

3.1.2. Functional Group Analysis Results (FTIR)

FTIR was performed to characterize the functional groups present in natural yucca fibers before and after chemical treatment. The results, illustrated in **Figure 8**, provide a detailed assessment of the functional groups and structural modifications resulting from the applied treatments at the peaks of 3363, 2924, 2854, 1743, 1651, 1519, 1249, and 1041 cm^{-1}.

As a result, the FTIR spectra revealed nearly identical peak positions, with only slight variations observed between the untreated and chemically treated fibers at the peak located at 1249 cm^{-1}. However, notable variations in peaks intensities were detected across the spectra, which can be ascribed to the structural changes induced by the chemical treatments applied on the yucca fibers. A broad absorption band ≈3363 cm^{-1} was ascribed to the stretching vibrations of O—H bonds, indicative of the presence of hydroxyl groups within the fiber structure.[50] In another zone, a characteristic double peak at 2924 and 2854 cm^{-1} appeared, corresponding to C—H stretching vibrations, typical of the aliphatic chains present in the cellulose and hemicellulose of yucca fiber.[51,52] According to literature, the absorption band at 1743 cm^{-1} is associated with C═O vibrations bonding the carboxylic acid, and ketone functional groups linked to the lignin macromolecular structure.[53] In addition, for the fibers treated with NaOH and sulfuric acid, a slight decrease in the intensity of the 1743 cm^{-1} peak was observed compared to untreated fibers. While this reduction is not drastic, it suggests a partial degradation of lignin rather than its complete removal. Another C═O stretching vibration was found at 1651 cm^{-1} could be attributed to the presence of aromatic rings associated with the hemicellulose structure.[54] Concerning the 1519 cm^{-1} peak, which corresponded to C═C stretching which is attributed to the deformation vibrations of the aromatic rings of lignin present in the yucca fibers.[55,56] Conversely, a distinct peak at 1249 cm^{-1} is observed exclusively in the FTIR spectrum of untreated fibers. This band is attributed to the asymmetric stretching vibrations of C—O bonds revealing the presence of acetyl groups, potentially attributed to the structural components of lignin.[57] It is important

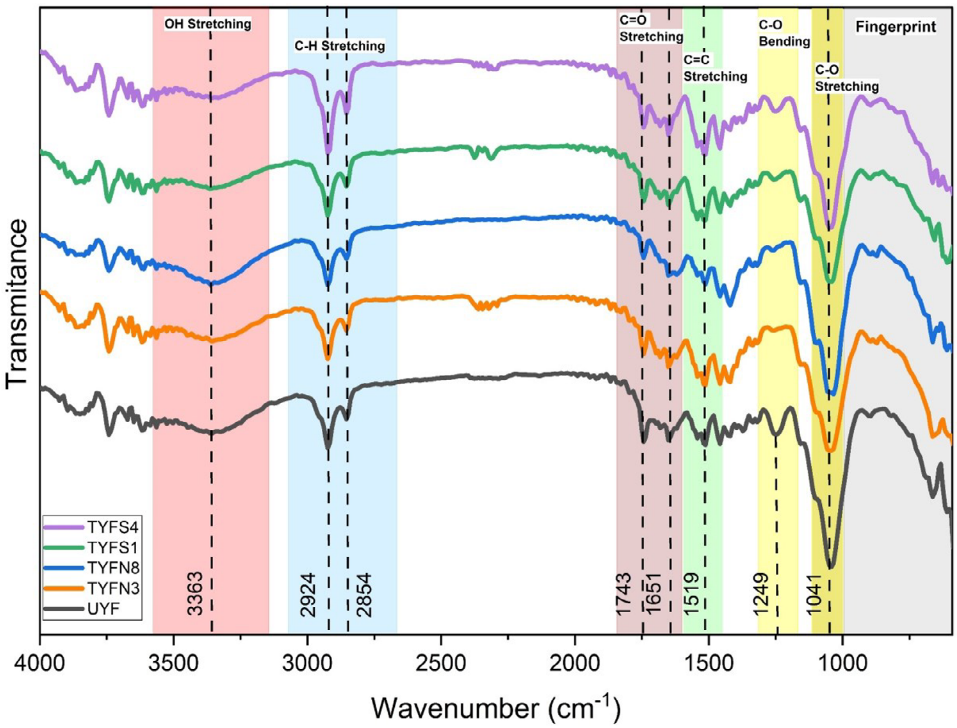

Figure 8. FTIR Spectra of untreated and chemically treated yucca fibers.

to note that acetyl groups can also originate from hemicellulose, particularly in polysaccharide structures. According to the literature, hemicellulose contains acetylated xylans, which contribute to the characteristic absorption in this region.[58,59] Additionally, the lignin present in grass-based fibers, including yucca fibers, was classified as HGS lignin (hydroxyphenyl-guaiacyl-syringyl), reflecting a complex aromatic structure with varying degrees of methoxylation.[60] The absence of this signal in the spectra of treated fibers suggests that these components undergo removal during the chemical treatment process, same remark was observed in another study.[61] The absence of this spectral band suggested a structural modification that enhances fiber–matrix interaction in bio-composites.[62] Specifically, the partial removal of lignin and non-cellulosic components induced by the chemical treatment increases the exposure of cellulose hydroxyl groups. This structural transformation is expected to significantly improve interfacial adhesion between the fiber and the polymer matrix, a key factor in optimizing the mechanical performance of bio-composites. Additionally, an intense peak at 1041 cm^{-1}, present in all spectra, corresponds to the stretching vibrations of C—O bonds of the glucoside structures of the lignin.[63]

3.1.3. X-Ray Diffraction Results

X-ray diffraction analysis was performed to evaluate the effect of alkaline and acid treatments on the crystalline structure of yucca fibers. The phases, crystallinity index (CI), and crystallite size (CS) are shown in **Figure 9** and **Table 4**.

UYF category showed a characteristic diffraction peak at 22.1θ, attributable to crystalline cellulose type I, with a crystallinity index (CI) of 46.4% and a crystal size of 1.3 nm. This pattern was consistent with findings in the literature concerning other natural fibers, such as sisal and jute, in which the crystalline structure of cellulose predominantly exhibits type I characteristics prior to any chemical treatments.[39,64] Treatment with 3% NaOH solution resulted in a modest increase in the relative intensity of the peak at 22.2θ, indicating an enhancement in crystallinity, reaching 52.4%. This increase can be explained by the treatment effect, which partially eliminates amorphous components such as hemicellulose and lignin, producing a more organized cellulose part.[65–67] Interestingly, at a higher concentration of NaOH (8%), the CI was decreased, achieving 47.1% in

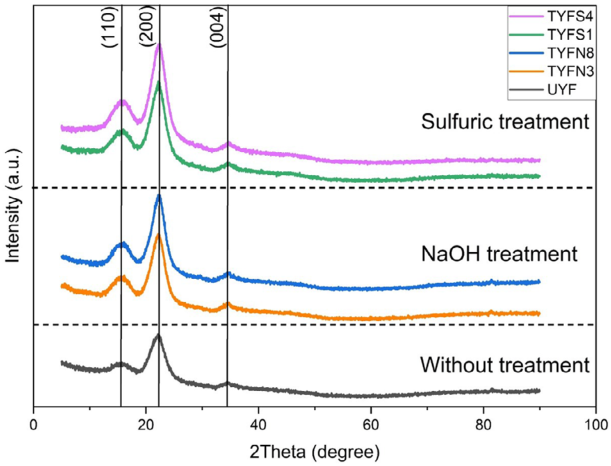

Figure 9. X-ray spectra of treated and untreated yucca fibers.

Table 4. XRD characterization results.

Sample nature	Amorphous peaks [°]	Crystalline Peaks [°]	Crystallinity index [%]	Crystallite size [nm]
UYF	15.6/34.4	22.1	46.4	1.30
TYFN3	16.1/34.7	22.2	52.4	1.47
TYFN8	16/34.8	22.2	47.1	1.41
TYFS1	16/34.8	22.2	55	1.34
TYFS4	15.4/34.6	22.4	51.8	1.33

the TYFN8 category. According to the literature, this decrease is due to the chemical agent concentration, which has attacked the cellulose structure.[68] Furthermore, the higher crystallinity index (CI) at 3% NaOH compared to 8% NaOH can be explained by the selective removal of amorphous components and the potential disruption of crystalline regions at higher alkali concentrations. At 3% NaOH, the treatment efficiently removed non-cellulosic components, enhancing cellulose crystallinity. However, at 8% NaOH, excessive swelling and partial hydrolysis may disrupt ordered crystalline domains, reducing CI. This trend has been documented in previous studies on alkali-treated natural fibers, highlighting the need for an optimal NaOH concentration to enhance crystallinity while preventing cellulose degradation.[69]

Concerning the acid treatments, the application of 1% H_2SO_4 showed a better increase of the crystallinity index at 55% in the TYFS1 type, suggesting that the acid treatment favors the elimination of amorphous fractions while preserving the crystalline phase.[70,71] However, as the concentration of acid increased to 4%, the CI decreased slightly to 51.8% in the TYFS4 category, despite remaining relatively high. This trend can be interpreted by the effect of acid treatment at a high concentration on the crystallinity of natural fiber.[72] This significant decrease in CI indicates an onset of cellulose chain degradation, accompanied by increased amorphization.[73]

3.1.4. Single Yucca Fiber Tensile Properties

Tensile tests were performed on untreated and chemically treated yucca fibers to comprehensively evaluate the impact of chemical treatments on fundamental mechanical properties, namely tensile strength, Young's modulus, and elongation at break. The test findings are summarized in **Table 5**.

As a result, the tensile strength of untreated yucca fiber was ≈257 MPa, which is within the zone of other results achieved previously in the field of natural fibers.[74] However, the application of a chemical treatment may have an influence on the mechanical properties of natural yucca fiber. In particular, the TYFN3 category, treated with 3% NaOH, exhibited a remarkable increase of 101% compared to the untreated fibers, reaching a maximum tensile strength of 519 MPa. This category demonstrates the highest mechanical resistance in this study, mainly due to the treatment method and the low chemical agent concentration,[75] that have been able to remove all the compounds that negatively affect the performance of the natural fiber such as waxes and non-cellulosic parts, and which remain distributed within the interfibrillar regions of the bio-fibers.[64] Afterward, increasing the NaOH concentration to 8% led to a reduction in tensile strength in the TYFN8 category, which nevertheless achieved a strength of 431 MPa. Although this value remained higher than that of untreated fibers, it was notably lower than that of TYFN3. This decrease is due probably to the greater structural deterioration of the fibers induced by the more aggressive attack of NaOH at higher concentrations.[17,76] Furthermore, yucca fibers treated using sulfuric acid (TYFS1 and TYFS4) exhibited tensile strengths of 498 and 260 MPa respectively, with a behavior similar to that of fibers treated with NaOH. Nevertheless, the fibers treated at 4% sulfuric acid (TYFS4) demonstrated significantly lower stress, indicating a more pronounced degradation of the fiber structure under the effect of the acid.[77]

Table 5. Tensile test results of treated and untreated yucca fiber.

Fiber type	Tensile strength [MPa]		Young's modulus [GPa]		Elongation [%]	
	Mean	SD	Mean	SD	Mean	SD
UYF	258	41	6.6	1.56	4.05	0.94
TYFN3	519	46.2	10.6	1.74	5.14	0.6
TYFN8	431	68.6	9.2	1.93	4.79	0.97
TYFS1	498	17.4	12.2	2.31	4.19	0.69
TYFS4	260	24.7	10.3	2.81	2.7	0.79

Additionally, elongation at break, which reflects the fiber's ductility, also varied results according to the treatment. Untreated fibers (UYF) achieved an elongation at break of 4.05%, while fibers treated at 3% NaOH (TYFN3) showed a slightly higher elongation of 5.14%, indicating a slight increase in ductility. However, fibers treated with 8% NaOH (TYFN8) showed an elongation at a break of 4.79%, lower than those treated with 3% NaOH. These results can be explained by the impact of the chemical treatment on the fiber structure, with increasing NaOH concentration, the structural degradation of the fibers intensifies, resulting in a decrease in their ductility.[78] The sulfuric acid treatments (TYFS1 and TYFS4) led to a decreased in elongation at break, with values of 4.19% and 2.7% respectively. This reduction in ductility may be attributed to the excessive stiffening of the yucca fiber, resulting from a more aggressive modification of the fiber's chemical structure.

In another context, the considerable standard deviation in the tensile properties of yucca natural fibers arises from multiple scientific factors. Primarily, the natural variability inherent to these fibers, due to their biological origin, results in structural and compositional heterogeneities, leading to fluctuations in mechanical properties. Additionally, the presence of structural defects, such as microcracks, voids, and fibril misalignment, significantly influences mechanical performance, further contributing to the observed data dispersion.

The selection of NaOH and H_2SO_4 concentrations was determined based on their distinct chemical interactions with the fiber structure and their influence on the mechanical properties of the treated fibers. NaOH was applied at 3% and 8% to induce controlled delignification while preserving cellulose fibril integrity. The 3% concentration allowed for moderate removal of non-cellulosic components, whereas the 8% concentration facilitated more extensive lignin dissolution, without excessive degradation. This controlled delignification can enhance fiber flexibility and

Table 6. Comparison of single natural fiber tensile test results.

Natural fiber	Treatment method			Tensile strength [MPa]		Improvement rate [%]	Ref.
	Method	%	Time [h]	Untreated	Treated		
Yucca	NaOH	3	2	258	519	+101	Present work
Sisal	NaHCO3	10	24	277	661	+139	[81]
Kenaf	NaOH	6	4	105	283	+169	[82]
Date palm	NaOH	6	3	233	366	+57	[83]
Pineapple	NaOH	1	1	630	1560	+148	[84]

tensile strength by exposing cellulose microfibrils while maintaining structural integrity.[79] However, excessive alkali treatment may lead to fibrillation and minor reductions in mechanical performance due to partial degradation of the fiber's cell wall.[80] In contrast, given the strong hydrolytic nature of H_2SO_4, lower concentrations (1% and 4%) were employed to prevent excessive cellulose degradation, which could compromise the mechanical properties of the fibers. The 1% concentration selectively removed hemicellulose and lignin while preserving fiber cohesion. Meanwhile, the 4% concentration resulted in more significant structural alterations. Overall, the difference in selected concentrations between NaOH and H_2SO_4 reflects their distinct mechanisms of action and resulting effects on fiber morphology and mechanical properties.

In a comparison of the tensile strength before and after treatment of yucca fiber studied in this paper with other natural fibers, such as sisal and Pineapple. The strength of untreated yucca fiber (258 MPa) is higher than that of several other natural fibers, such as kenaf fiber (105 MPa) and date palm (233 MPa), and close to the strength of sisal fiber (277 MPa). Conversely, the chemical treatment of yucca fiber with 3% NaOH resulted in a substantial enhancement of its properties, with an improvement rate of 101%, which is notably acceptable in comparison with those reported in existing literature and represented in **Table** 6.

3.2. Epoxy Yucca Fiber Bio-Composites Properties

3.2.1. Adhesion Inspection Results

SEM micrographs of the fracture surfaces of treated and untreated yucca fiber-reinforced bio-composites, after tensile testing, are shown in **Figure** 10. These micrographs offer a comprehensive visualization of the interactions between the fibers and the epoxy matrix, allowing for an assessment of the impact of chemical treatments on the interfacial adhesion.

First, the surface condition of the composites, as observed in all the figures, indicates that the samples underwent the tensile test without any significant slippage or delamination defects. This suggests that the fibers were adequately incorporated into the matrix and that the test conditions were controlled. Another important observation pertains to the orientation of the yucca fiber powder within the epoxy matrix. During the preparation of bio-composites samples, particularly through the casting molding technique, the powder derived from both treated and untreated yucca fibers is unable to assume a uniform orientation within the matrix. In addition, the white patches observed on the bio-composite surface may indicate areas where the surface coating was not uniformly applied. This could be due to an uneven distribution of the coating material during processing, as the surface was not perfectly flat due to the sample being derived from a tensile-tested specimen. The mechanical testing may have caused slight deformations, leading to an irregular surface, which in turn could affect the uniformity of the coating application.

A more detailed comparison of fracture surfaces reveals clear distinctions between untreated and treated fiber-reinforced composites. In particular, the untreated yucca fiber composites (Figure 10A) exhibit fibers being pulled out of the matrix, with visible voids corresponding to the original positions of uprooted fibers. This phenomenon is likely due to the poor cohesion between the matrix and the fibers, causing the applied tensile force to pull the fibers out of place.[85] In contrast, chemical treatments have enhanced the roughness of the yucca fibers, improving cohesion between the fibers and the epoxy matrix. Consequently, the composites in the categories EYN3, EYS1, and EYNS4 represented in Figure 10B,D,E, respectively, show some fibers being interrupted and voids in the matrix, however, they also exhibit cohesive fractures and cut fibers. These failures, occurring at the fiber–matrix interface, suggest improved stress transfer and an overall strengthening of the composite.[86] These findings align with the improved mechanical performance observed in these samples, where homogeneous fiber fracture is evident, rather than fiber pull-out. Such failure modes are often indicative of stronger cohesion in treated composites. Therefore, these observations suggest that chemical treatments have significantly improved the interfacial adhesion and stress transfer between the fibers and the matrix.[87]

Furthermore, the composites reinforced with yucca fibers treated with 8% NaOH, EYN8 category (Figure 10C) show numerous voids, compared to the others categories of treated fibers reinforced bio-composites. These voids indicate poor adhesion and early failure in these areas, characteristic of composites with weak fiber–matrix interaction. The lack of strong fiber–matrix bonding in this category led to matrix cohesive-type failure rather than fiber failure,[88] which may explain the reduced mechanical performance in these composites.

In addition, tensile test results on bio-composites showed an improvement in the mechanical performance of the treated fiber composites, suggesting enhanced interfacial bonding. The reduction in fiber pull-out and the presence of fractured fibers instead of deboned ones, as seen in the SEM images, further support this conclusion. This improvement can be attributed to the chemical treatment, which increased fiber roughness and surface energy, promoting better adhesion with the matrix. Moreover, the increase in tensile strength observed in the treated composites with sulfuric acid (from 23.6 to 34.4 MPa) indicated effective stress transfer from the matrix to the fibers.

According to several previous studies, the use of additives such as coupling agents has demonstrated substantial effects in enhancing the fiber–matrix interaction.[89] Specifically, the incorporation of coupling agents facilitates the dispersion of fibers within the matrix, improving their distribution and reducing agglomeration.[90,91] Furthermore, these agents strengthen the chemical bond between the functional groups of the fibers and

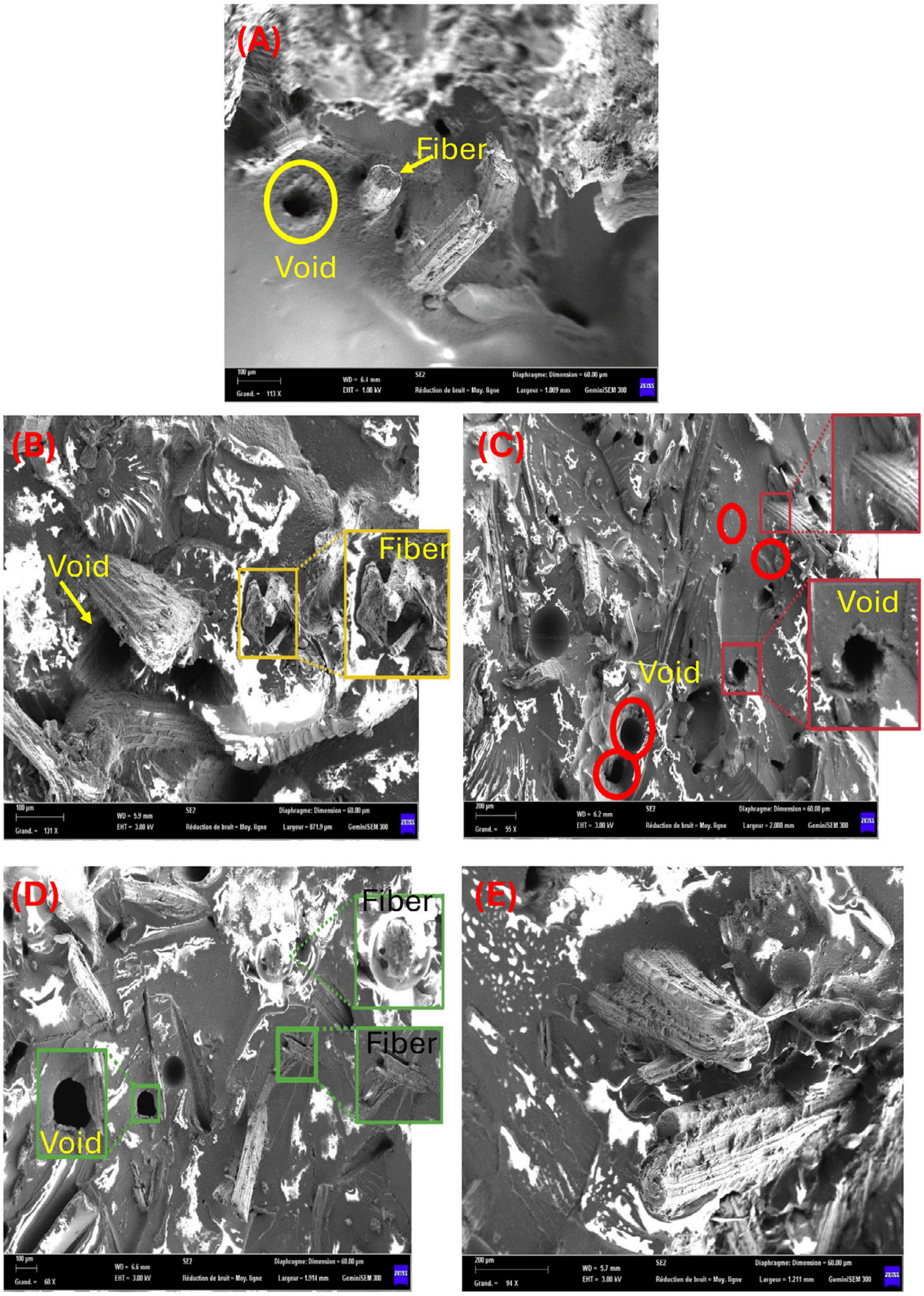

Figure 10. SEM micrographs of yucca fiber–matrix interface in bio-composites. A) SEM micrograph of bio-composites reinforced by untreated yucca. B,C) SEM micrographs of bio-composites reinforced by 3% and 8% NaOH-treated yucca, respectively. D,E) SEM micrographs of bio-composites reinforced by 1% and 4% H2SO4 treated yucca, respectively.

the reactive sites of the epoxy matrix.[92] By promoting better adhesion at the fiber–matrix interface, coupling agents help optimize stress transfer and contribute to the overall mechanical performance of the bio-composite.[93]

3.2.2. Bio-Composites Tensile Properties

Tensile tests carried out on bio-composites reinforced with yucca fibers revealed significant differences depending on the state of the fibers (treated or untreated) as well as the chemical treatment applied. The principal results of the test are presented in **Figure 11** and **Table 7**, assessing the effects of chemical treatments on the mechanical tensile properties of the final bio-composite material.

The results of the untreated yucca fiber (EUY) bio-composite category, which serves as a reference point for comparison with the other categories analyzed in this study, are first presented. The average tensile strength is ≈23.6 MPa, with a Young's modulus of 1598 MPa. However, the incorporation of chemically

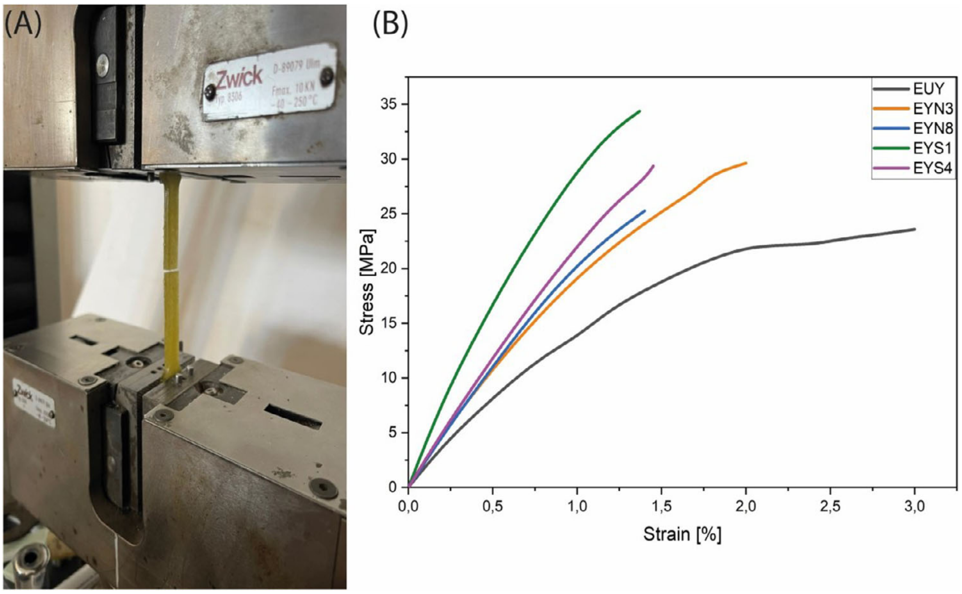

Figure 11. Tensile mechanical performance of biocomposites reinforced with treated and untreated yucca fibers. A) Image of the bio-composite sample after tensile test. B) The tensile stress–strain curve of biocomposites.

treated yucca fibers into the epoxy matrix resulted in notable variations in the properties, which were dependent on the treatment method and the concentration of the chemical agent employed. The EYN3 category demonstrated enhanced tensile strength, recording a value of 29.6 MPa, which represents a 25.6% increase compared to the EUY category. Additionally, Young's modulus was ≈2198 MPa, indicating a favorable modulus for this material. The EYS1 bio-composite category exhibited a 45.8% improvement in tensile strength relative to the EUY category, reaching a tensile strength of 34.37 MPa and a higher Young's modulus of 2637 MPa. The observed increase in these two categories may be attributed to several factors. First, the type of fibers used have previously exhibited superior mechanical strength. Furthermore, the concentration of chemical treatment used effectively eliminates various impurities that impede the adhesion of the natural yucca fiber to the matrix in these categories, while preserving the intrinsic properties of the bio-fiber. As a result, this leads to a more uniform distribution of forces, thereby enhancing the mechanical performance of the final biocomposites.[94,95]

Table 7. Tensile performance of biocomposites reinforced with treated and untreated yucca fibers.

Sample type	Tensile strength [MPa]		Young's modulus [MPa]		Elongation [%]	
	Mean	SD	Mean	SD	Mean	SD
EUY	23.6	2.3	1598	243	3.01	0.75
EYN3	29.6	3.22	2198	183	2	0.21
EYN8	25.2	4.13	2222	66	1.39	0.37
EYS1	34.4	6.61	2637	166	1.3	0.06
EYS4	29.4	2.61	2399	151	1.45	0.19

Despite the relatively modest mechanical properties of the yucca fibers treated chemically (alkaline and acid) at higher concentrations relative to the untreated fibers, the bio-composites reinforced with these treated fibers displayed promising properties. As a result, the EYN8 category exhibited a relatively modest increase in performance in this study, with a 6.78% improvement compared to the EUY category. The properties of the EYN8 bio-composite were ≈25.2 MPa for tensile strength and Young's modulus of 2222 MPa. Additionally, the EYS4 category demonstrated robust mechanical properties, with a tensile strength of 29.4 MPa and Young's modulus of 2399 MPa, indicating an enhancement of ≈24.5% relative to the EUY bio-composite category. The improvement in mechanical properties observed in these two categories compared to the EUY category can be attributed to the effects of chemical treatment, which effectively removes surface impurities from the yucca fibers, thereby enhancing the adhesion between the reinforcement and the matrix.[96,97]

As a result, the EYS1 and EYS4 bio-composite grades exhibited superior mechanical performance in terms of tensile properties. This improvement can primarily be attributed to the effect of sulfuric acid on the chemical and morphology of yucca fibers, as the treatment effectively increase the crystallinity index (CI), which is a key factor in the mechanical properties of the bio-composite reinforcement. It also removed a considerable amount of impurities that hinder the cohesion between the fiber and the matrix and offers a rough fiber surface for better interlocking with the epoxy matrix.[98,99] Consequently, the enhanced interfacial adhesion between the fiber and the matrix contributes to the improved mechanical properties.

In another context, the variability observed in the mechanical properties of the bio-composites presented in Table 7, as indicated by the standard deviations (SD), is an inherent characteristic of materials reinforced with natural fibers. This variability arises from multiple factors, primarily the intrinsic heterogeneity of natural fibers, including variations in chemical

Table 8. Compressive and bending properties of biocomposites reinforced with treated and untreated yucca fibers. (All units are in MPa).

Sample type	Compressive strength		Bending strength		Bending modulus	
	Mean	SD	Mean	SD	Mean	SD
EUY	72.4	0.15	39.3	5.07	1278	278
EYN3	79.5	2.47	41	4.76	1855	108
EYN8	77.4	1.31	40.3	1.85	1546	365
EYS1	89.3	1.25	45.3	4.33	1972	313
EYS4	84.7	6.13	43.1	2.79	1988	201

composition, and microstructural organization, all of which significantly influence mechanical performance.[100] Additionally, the dispersion and orientation of the fibers within the matrix play a crucial role, as uneven distribution or fiber agglomeration can create local stress concentrations, thereby affecting the tensile strength and Young's modulus of the composite.[101,102] Despite these variations, the overall trends observed in this study remain within an acceptable range for natural fiber materials.

3.2.3. Bio-Composites Compressive and Bending Performances

Five samples of bio-composites in each category, reinforced with treated and untreated yucca fibers, were tested for their mechanical properties in compression and bending. The results obtained from these two tests, including compressive strength, bending strength, and bending modulus are listed in **Table 8**, In addition, the compressive and bending stress–strain curves are displayed in **Figure 12**.

Compression tests on bio-composites reinforced with yucca fibers revealed significant differences depending on the chemical treatments employed. Bio-composites reinforced with untreated fibers (EUY) exhibited an average compressive strength of 72.4 MPa, which serves as a reference for assessing the impact of various treatments. The grades of bio-composites reinforced with yucca fibers treated with 3% NaOH (EYN3) and 8% NaOH (EYN8) have strengths of 79.5 and 77.4 MPa respectively, an improvement attributed to the partial removal of lignin and hemicellulose, which increases surface roughness and enhances fiber–epoxy adhesion.[103] Furthermore, the transition to sulfuric acid treatment led to a significant improvement of mechanical performance in terms of compressive strength, as the biocomposite reinforced by yucca fibers treated at 1% sulfuric acid (EYS1) achieving the highest strength, ≈89.3 MPa. This improvement may be explained by selective hydrolysis of the fibers' amorphous regions, resulting in better orientation of the cellulose chains and an increase in the crystallinity index, leading to improved fiber rigidity and increased fiber–epoxy cohesion. However, the EYS4 biocomposites exhibited a compressive strength of 84.7 MPa, lower than that of the EYS1 category and still higher than that of the EUY category. This reduction can be attributed to the more pronounced degradation of the fibers under the effect of a higher concentration of acid, which adversely affected their ability to support compressive loads.

The bending test results indicate that the bio-composite reinforced with untreated yucca fibers (EUY) exhibited relatively low mechanical performance, with a bending strength of 39.3 MPa and a modulus of 1278 MPa. However, the chemically treated bio-composite categories exhibited a clear dependency of their bending performance on the type and concentration of the applied chemical treatments. Notably, composites reinforced with alkali-treated fibers demonstrated significant improvements relative to composites containing untreated fibers (EUY). In particular, the EYN3 bio-composite recorded a bending strength of 41 MPa and a bending modulus of 1855 MPa, this increase is likely a consequence of improved interfacial adhesion between the yucca fiber and the epoxy matrix, facilitated by the effective removal of surface impurities.[104,105] However, the EYN8 category presented slightly lower values than the EYN3 category, at 40.3 MPa for strength and 1546 MPa for modulus. These differ-

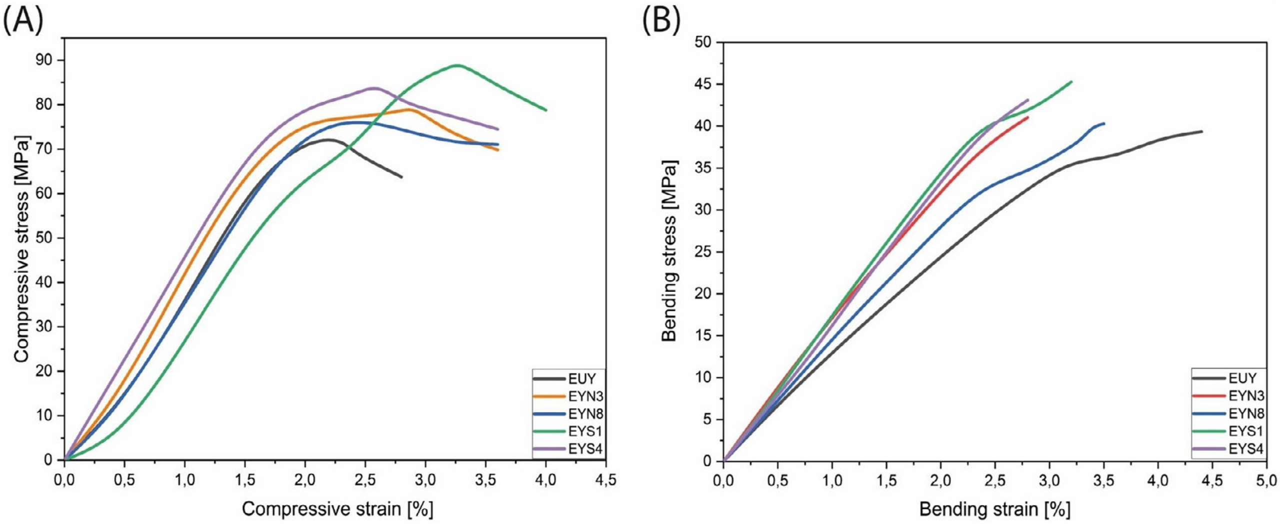

Figure 12. The stress–strain curves determined by the mechanical tests. A) The compressive stress–strain curve of biocomposites. B) The bending stress–strain curve of biocomposites.

ences in mechanical performance could be attributed to the effects of alkaline concentrations, where a high concentration appears to partially alter the cellulose structure of the yucca fibers, reducing their ability to effectively reinforce the matrix.[106] Conversely, bio-composites incorporating sulfuric acid-treated fibers exhibited significantly superior mechanical properties. Samples EYS1 and EYS4 bio-composites achieved bending strengths of 45.3 and 43.1 MPa respectively, accompanied by high bending modulus of 1972 and 1988 MPa. These results can be explained by better chemical activation of the fiber surfaces under the effect of sulfuric acid, leading to the effective elimination of impurities present on the surface, and thus favoring an increased interfacial interaction between the yucca fibers and the epoxy matrix.[107] This improved interaction facilitates more effective stress transmission, enhancing the stiffness and strength of the composites while ensuring that the intrinsic performance of the fibers is maintained. As a result, in contrast to certain alkaline treatment conditions, acid treatment at low concentrations appears to preserve the structural integrity of the fibers while increasing their effectiveness as reinforcing agents.

In a different context, the variability observed in standard deviations (SD) exceeding 10% of the mean can be attributed to the intrinsic characteristics of bio-composites reinforced with natural fiber powder. The random distribution of particles within the epoxy matrix can lead to localized fluctuations in concentration and orientation, thereby affecting the mechanical properties of individual specimens.[108] Nevertheless, these variations fall within the expected range for similar bio-composites and align with trends reported in the literature.[100]

4. Conclusion

Yucca fiber represents an innovative and strategic bio-fiber for the reinforcement of sustainable composite materials, due to its availability, eco-environmental qualities, and intrinsic mechanical properties. This study demonstrated that the chemical treatments, in particular employing NaOH and H_2SO_4, are a reliable and effective approach to improving the physicochemical and mechanical properties of these types of fibers. These treatments enable controlled degradation of amorphous components such as hemicellulose and lignin, leading to an increase in the crystallinity index, and improved interfaciality with polymer matrices. Other main results can be summarized as follows:

- The chemical treatment of natural fibers has the potential to enhance the mechanical properties of yucca fibers, as well as the overall performance of the resulting bio-composite.
- Chemical agent, concentration, and time are the essential factors in the chemical treatment process.
- The treatment of bio-fibers with sulfuric acid represents an innovative approach that has been demonstrated to significantly enhance their properties.
- Fibers treated with 1% H_2SO_4 demonstrated an increase in crystallinity index (CI) to 54%, resulting in a substantial enhancement of their mechanical properties, particularly tensile strength, which increased to 497 MPa. This enhancement translates directly into the performance of bio-composites reinforced with these fibers, where the tensile strength of the composites improved by 45%, reaching 34 MPa.
- Yucca fibers treated with 1% H_2SO_4 and 3% NaOH exhibited significant improvements in mechanical performance, with tensile strength increasing by 93% and 101%, respectively. The improvements demonstrate a balance between the chemical modification required to enhance interfacial bonding and the preservation of the fiber's structural integrity.
- The chemical treatments had a direct and positive impact on the mechanical performance of the bio-composites, significantly enhancing their tensile strength and flexural properties.
- The incorporation of 10 wt.% yucca powder enhanced the uniformity of the bio-composite by promoting homogeneous dispersion within the epoxy matrix, while simultaneously mitigating processing challenges and reducing the risk of particle agglomeration.
- Chemical treatment of the natural fibers significantly improved the compressive strength of the bio-composites, with a peak of 89.29 MPa for the EYS1 category (reinforced with fibers treated at 1% H_2SO_4), an increase of 23.3% compared to untreated samples. Similarly, the EYN3 category (treatment with 3% NaOH) also showed notable improvement, reaching 79.54 MPa.
- The bending strength exhibited significant variation among the samples, reaching a maximum of 45.27 MPa for the EYS1 category, representing a 15.07% increase, while the EYN3 category achieved a strength of 41.03 MPa, reflecting a 4.3% improvement.
- The observed discrepancy between the flexural and tensile moduli in this study was attributed to variations in loading conditions, stress distribution, and failure mechanisms inherent to flexural and tensile testing.

This study demonstrates the importance of controlling processing parameters to maximize the performance of bio-composites while paving the way for future research aimed at exploring more environmentally friendly treatments and optimizing the properties of yucca fibers in various industrial applications. Additionally, further investigations into varying fiber concentrations could provide deeper insights into their effect on bio-composite mechanical properties, enabling the identification of an optimal trade-off between reinforcement efficiency and manufacturing feasibility.

Acknowledgements

M.A.K. and M.G. express their gratitude for the support provided by Kasdi Merbah University in Ouargla under the PRFU project [code: A11N01UN300120220002]. M.B. gratefully acknowledges the support by the Engineering and Physical Sciences Research Council (EPSRC)'s Innovation Launchpad Network+ Researcher in Residence scheme [grant numbers: EP/W037009/1, EP/X528493/1, project: RIR26C230615-6].

Conflict of Interest

The authors declare no conflict of interest.

Data Availability Statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

Keywords

alkaline, epoxy–matrix composites, sulfuric acid, surface treatments, yucca fibers

Received: February 12, 2025
Revised: April 12, 2025
Published online:

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