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Enhancement of Mechanical and Structural Properties of Jute Fabric-Reinforced Polyester Composites via Alkali Treatment

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Abstract

The focus of our research work is on the fabrication and characterization of jute-reinforced composite. The material is manufactured by vacuum contact molding, with stratification conducted according to a well-detailed protocol. This study investigates the effects of alkali treatment on the mechanical and structural properties of jute fabric-reinforced polyester composites. Jute fabrics underwent chemical treatment with sodium hydroxide (5% of NaOH by weight) for five different exposure times (raw, 1, 3, 6, and 24 hours) followed by a pressure contact molding process. In physical characterization process, we focused on determining the reinforcement volume ratio and measuring the density of the composites. To evaluate their mechanical properties, we conducted tensile tests, which help us understand how the materials behave under stress. For structural characterization, we employed several advanced techniques, including X-ray diffraction (XRD) to analyze the crystalline structure, Fourier Transform Infrared spectroscopy (FTIR) to investigate chemical compositions, X-ray tomography for a detailed 3D view of the internal pore structure, and scanning electron microscopy (SEM) to observe the surface morphology. These methods collectively provide a comprehensive understanding of the materials' properties and performance. The results showed that alkali treatment significantly influenced the mechanical properties; i.e., the composites reinforced with fabrics treated for 1 and 3 hours exhibited the highest tensile strength (improvement by 20% and

9%, respectively) compared to composites reinforced with raw fabrics). XRD analysis revealed an increase in crystallinity index after treatment, particularly for an hour. FTIR spectra showed changes in chemical groups after treatment, indicating the removal of certain constituents. Image processing using X-ray tomography offers non-destructive, 3D analysis with high resolution, while conventional methods are simpler and more cost-effective. This technic provides detailed information about the pore structure, including the size, shape, distribution, and connectivity of pores. Conventional methods provide bulk porosity measurements but do not provide detailed information about the pore structure. SEM observations revealed various fracture mechanisms and the treated composites showed improvement in interfacial bonding. Overall, alkali treatment enhanced jute fabric-reinforced polyester composites' mechanical and structural properties, with optimal performance for 1-hour treatment duration.

Keywords

Bio-composites, Alkali treatment, Characterization, Mechanical Properties, X-ray tomography.

I. Introduction

Natural fiber reinforcement of polymers is highly appealing due to their plentiful availability, biodegradability, lower energy consumption in production, and reduced environmental problems compared to synthetic fibers like glass and carbon fibers.^{1,2} However, plant fibers and polymer matrices often exhibit poor compatibility due to their differing natures, necessitating surface treatments to enhance interfacial adhesion³. This incompatibility arises primarily from the hydrophobic nature of most polymer matrices and the hydrophilic nature of plant fibers, which contain a high proportion of cellulose, hemicellulose, and lignin, all of which possess numerous hydroxyl groups that attract water molecules. This inherent hydrophilicity leads to several challenges, such as weak adhesion, reduced mechanical properties, increased moisture absorption, and premature failure of the composite material.^{4,5}

To overcome this morphological issue, surface modification techniques have been widely adopted to alter the fiber surface and improve fiber-matrix interaction. This work highlights the importance of one of these treatments in achieving good compatibility, strong interfacial bonding, and uniform distribution of mechanical properties in plant fiber-reinforced composites.⁶ Both physical (corona, plasma, and steam explosion) and chemical methods (alkali, silane, and acetylation treatments) are employed to reduce fiber hydrophilicity and enhance adhesion with the hydrophobic matrix. These modifications not only improve the structural integrity of composites but also expand their usability across various industrial sectors.^{7,8} Treated plant fiber composites have demonstrated significant potential as sustainable and eco-friendly alternatives to synthetic fiber composites.⁹

In recent years, there has been an increasing trend in the use of composite materials across various industrial applications, including automotive, aviation, space, navy, and even sports.¹⁰ Research activities on these materials are also widespread in laboratories worldwide, aiming to better understand composite structures' behavior through numerous research topics to optimize their use and suitability for industrial applications. Among this class of materials, significant studies have been conducted on composites reinforced with plant-based materials.^{11,12}

Due to industry standards, studying these materials must increasingly account for their environmental impact. Composites reinforced with plant fibers have gained attention recently due to their numerous advantages over mineral or synthetic fibers. Their low-cost, energy-efficient production process, renewability, and recyclability are significant assets for their use.¹³

Continuous research and enhancements in mechanical behavior are being pursued to improve the handling of composite materials in industrial applications.¹⁴

This paper investigates the impact of alkali treatment on the crystallinity and porosity rate in jute/polyester composite materials. By conducting detailed characterization and analysis, the study focuses on fabricating and characterizing jute-reinforced composites using vacuum contact molding. The jute fabrics figure 1(a) are exposed to sodium hydroxide (NaOH) treatment for varying durations, ranging from untreated "Raw" to 24 hours.

The research assesses the link between the structural properties and the mechanical characteristics of the elaborated material. This study includes physical characterization, such as reinforcement volume ratio, and involves conducting tensile tests figure 3. Structural characterization is performed using X-ray diffraction (XRD), Fourier Transform Infrared spectroscopy (FTIR), X-ray tomography figure 4, and scanning electron microscopy (SEM). The Results indicate that alkali treatment significantly enhances the mechanical properties of the composites, with the best performance observed at 1 and 3 hours of treatment duration. This improvement includes increased tensile strength, a higher crystallinity index, reduced porosity, and enhanced interfacial bonding between the jute fabric and polyester matrix, with particular emphasis on changes in crystallinity and porosity.

II. Materials and method

II.1 Materials

II.1.1 Jute fabrics

The reinforcement used in this study is a taffeta configuration of a jute fabric where the weft yarns pass successively above and below the warp yarns. It is produced and commercialized by the jute E.P.E Béjaïa (Algeria) complex. It has a surface density of 380 g/m², and the warp and weft threads are made up of strands with an average thickness of about 750 µm. The slot between two successive yarns is around 1.2 mm, giving them good tearing resistance.

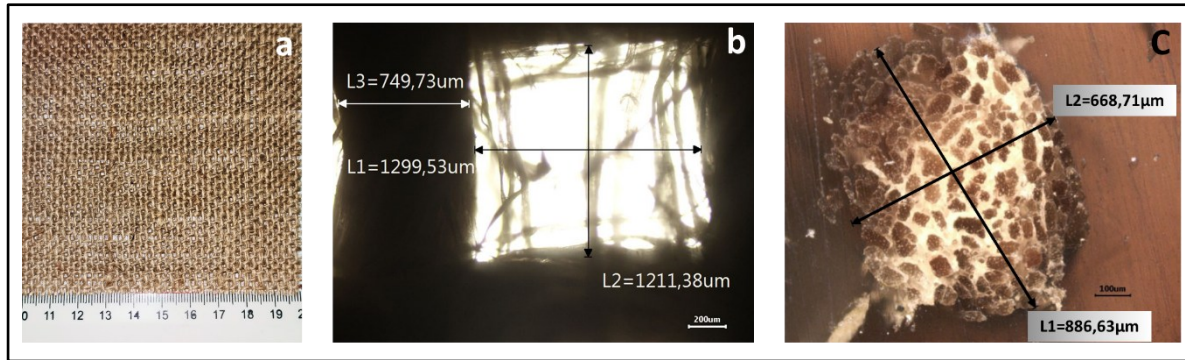


Figure 1. Reinforcement used in composite fabrication (a) Jute fabric, (b) Determination of slot between two successive jute yarns (b) Longitudinal microscopic observation of coated jute yarn

II.1.2 Matrix

The matrix used consisted of a two-component kit, an unsaturated polyester resin of the isophthalic acid type PD6191 supplied by Maghreb Pipe Group of M'sila - Algeria characterized by a density of 1.08g/cm^3 , and 1% of hardener in solution (by volume) of cobalt octoate.

II.2 Method

II.2.1 Alkali-treatment

Good fiber-matrix adhesion is a critical challenge in composite manufacturing.^{15, 16} The hydrophilic nature of plant fibers limits their compatibility with hydrophobic polymer matrices, resulting in weak interfacial bonding, poor mechanical interlocking, and detrimental porosity.¹⁷

To address this limitation, alkaline treatment is employed as an effective and economically viable solution. This chemical modification enhances the fiber-matrix interface by improving surface roughness and promoting better mechanical interlocking, thereby enabling stronger adhesion between inherently incompatible components.¹⁸

First, the jute fabrics underwent a chemical treatment with a 5% sodium hydroxide (NaOH) solution for different durations (raw, 1, 3, 6, and 24 hours). After the treatment, the fabrics were washed several times to remove traces of soda. Subsequently, they were washed with acetic acid to neutralize the Na^+ ions, followed by another wash with water to remove any acid residue. We label our composites as follows:

RC: Raw composite

1TC: 1 hour treated composite

3TC: 3 hours treated composite

6TC: 6 hours treated composite

24TC: 24 hours treated composite

II.2.2 Bio-composite manufacturing process

The jute/polyester composites are manufactured using pressure contact molding.^{19, 20} Each composite consists of eight plies aligned in warp direction [0]_s. Both raw and treated jute fabrics underwent an accelerated drying process at 110°C under 15-bar pressure for 2 minutes. This step serves multiple purposes: It eliminates hygroscopic moisture, improves fibre consolidation by reducing thickness through compression, and minimizes the waviness of the fabric, thereby enhancing the uniformity and quality of the reinforcement.²¹

A succession of plies is arranged out on the shape to be molded so that each fabric is well impregnated with polyester resin before the superposition of the next ply. Debulking is performed using a vacuum pump within a sealed metal enclosure for 30 minutes to eliminate the air inclusions trapped in the composite sheet figure 2.

After the resin reticulation 24h, composite extraction should be effortless due to the radio film covering the inner walls of the mold.

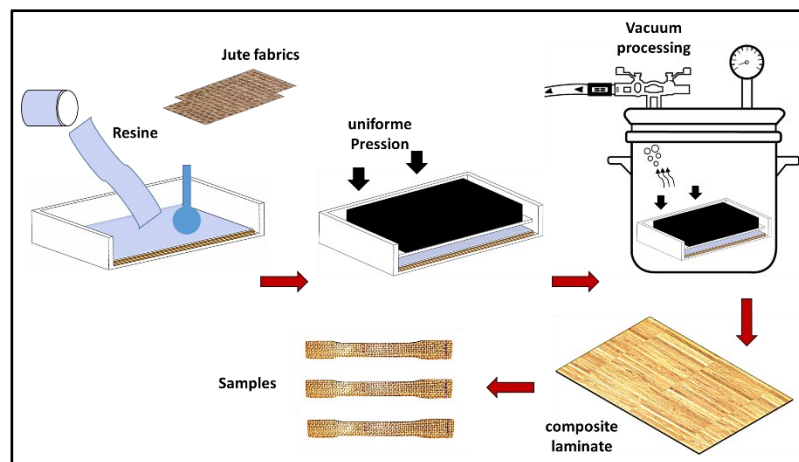


Figure 2. Bio-composite manufacturing process

The polymerization process is then conducted at room temperature for several days before cutting the plate and finishing the specimens by deburring and polishing, bellow is a table summarizing the bio-composite manufacturing process, including the parameters involved and the time required for each step

Table 1. Summary of Bio-Composite manufacturing Steps and Key Parameters

Process step	Parameters involved	Time duration
Alkali treatment	<ul style="list-style-type: none"> • NaOH concentration: 5% • Environment: Room temperature • Neutralization with acetic acid and water washing 	Raw, 1h, 3h, 6h, 24h
Fabrics drying	<ul style="list-style-type: none"> • Temperature: 110°C • Pressure: 15 bar • Purpose: remove moisture and minimize fabrics waviness 	2 minutes
Fabrics lay-up	<ul style="list-style-type: none"> • Number of plies: 8 • Alignment: Warp direction [0]_s 	Time duration of step
Debulking	<ul style="list-style-type: none"> • Vacuum pressure: Sealed metal enclosure • Purpose: Eliminate air bubbles 	30 minutes
Polymerization	Environment: Room temperature	More than 24 hours
Specimen preparation	Tools: Cutting machine, deburring tools, polishing equipment	Time duration of step

III. Characterization

III.1 Structural characterization (XRD)

X-ray diffraction is used to determine the crystal structure of organic reinforcement. When a beam of X-ray photons interacts with a crystalline sample, part of the radiation is diffracted at well-defined angles, whose values are directly related to the distances between atomic or molecular planes.²²

The evolution of the crystalline structure (crystallinity index CI and crystallite size CS) at different chemical treatment times is studied by X-ray diffraction. The tests were conducted on samples mechanically crushed with an electric mill and then manually crushed in a ceramic mortar.

The diffraction analysis is performed using a Bruker diffractometer. The scanning angle is taken between 10 and 90°. The incident monochromatic beam is centered on the K α 1 line of copper ($\lambda = 1.5418\text{\AA}$).

The crystallinity index CI and the average crystallite size CS are calculated using the equations below:

$$CI = (1 - I_{am}/I_{cr}) \times 100 \quad (1)$$

Where: I_{cr} : Major peak representing the crystalline part

I_{am} : Minor peak representing the amorphous part

$$CS = 0,89\lambda/\beta_{cr} \cos(\theta) \quad (2)$$

Where: λ : X-ray wavelength, θ : angle of diffraction
 β_{cr} : width at mid-height of the major peak

III.2 Fourier Transform Infrared spectroscopy FTIR

Transmission spectrum results were obtained from a pellet prepared from jute fabric finely milled with KBr potassium bromide powder in a 1:50 ratio.

The wavenumbers of the (Spectrum Two PerkinElmer) analyzer are between 4000 and 500 cm^{-1} .

III.3 Tensile test

The tensile test is conducted at room temperature using a Zwick Roell Z100 machine, equipped with a 100 KN load cell and an axial extensometer for measuring the longitudinal displacement of the specimens.

The tensile test is performed thrice for each composite type with a crosshead speed of 1 $\text{mm}\cdot\text{min}^{-1}$.

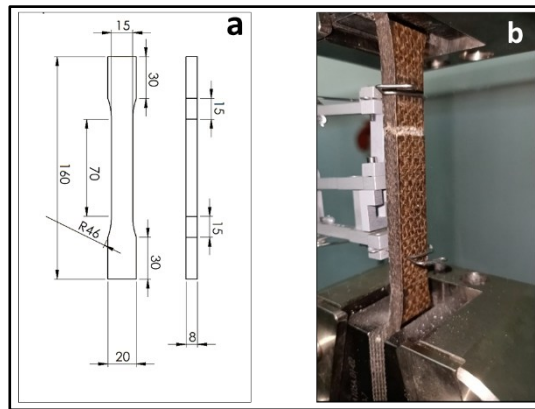


Figure 3. Tensile test sample (a) Specimen geometry (ASTM D 638- 03), (b) Extensometer MTS 634.12F-54

III.4 Physical characterization

III.4.1 Reinforcement volume rate

The fiber volume ratio V_f in composites is determined by the following equation:

$$V_f = 1 - 1/\rho_m(\rho_c - n_p w_f/t) \quad (3)$$

Where: ρ_m : Matrix density = 1.1 g/cm^3

ρ_c : Composite density = 1.29±0.5 g/cm^3

n_p : Number of plies

w_f : Fabric mass (g)

t : Thickness of the composite

$$26\% \leq V_f \leq 31\%$$

NB: ρ_m and ρ_c are calculated using the equation based on Archimedes' thrust.

III.4.2 Density measurement

Density is measured using a precision balance by weighing parallelepipedic specimens of the manufactured composites and a caliper to measure their volumes.

$d_c = \rho_c / \rho_{water}$ where:

ρ_c : mass density of composite

ρ_{water} : mass density of water = 0,997g.cm⁻³ à 25°C

Table 2. Biocomposites density

Composite	RC	1TC	3TC	6TC	24TC
Density (g.cm ⁻³)	1,21	1,31	1,31	1,34	1,29

III.5 X-ray tomography

X-ray tomography is a non-destructive inspection technique that creates cross-sectional images of the scanned zone or object. The Principle is based on acquiring several cross-sections in a single rotation. The two-dimensional images obtained are then stacked to reconstruct a three-dimensional view of the internal structure of the sample.²³

The tomographic observations were carried out with RX Solutions EasyTom Nano tomograph in the MATEIS laboratory of INSA Lyon on specimens that had already been mechanically tested in tensile (ruptured specimens), the scanned zone (X=15 Y=13 Z=6) mm is a little further away from the damaged zone as shown in the figure below (figure 4).

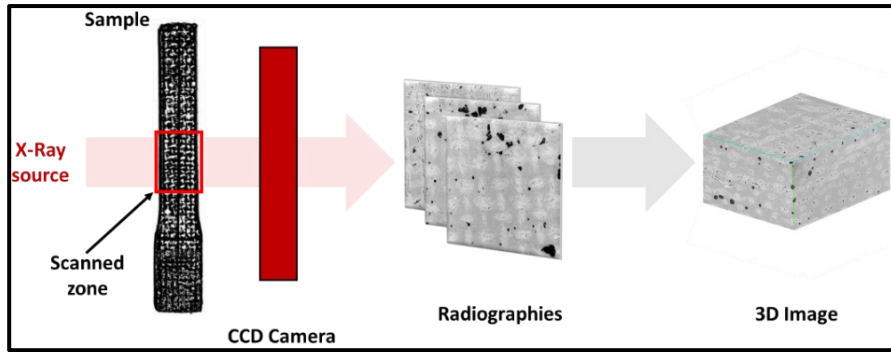


Figure 4. X-ray tomography principle

III.6 Microscopic characterization by scanning electron microscope SEM

To investigate the morphological damage mechanisms in the composite material post-fracture, sections of the fracture surfaces were cut out and analyzed using a JEOL JCM-5000 SEM microscope.

IV. Results and discussion

IV.1 Monotonous tensile test

Many studies focused on mechanical characterization of jute-reinforced composites with thermosetting matrix, which are frequently chosen for their ability to provide structural integrity and strong adhesion.²⁴⁻²⁶

The stress-strain curves presented illustrate the mechanical behavior of jute fabric-reinforced polyester composites subjected to various durations of chemical treatment, ranging from untreated (RC) to 1TC, 3TC, 6TC, and 24TC figure 5.

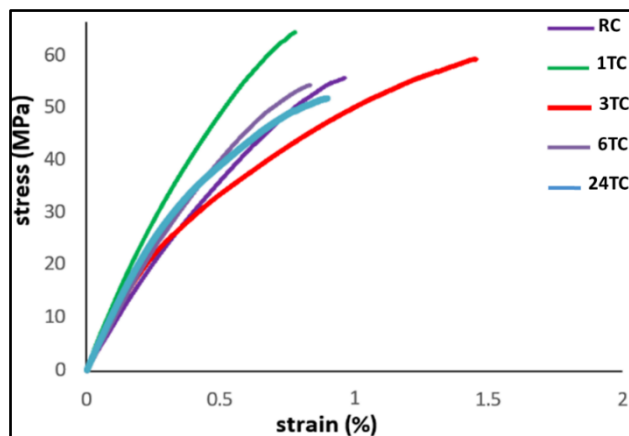


Figure 5. Stress-strain curves of composites at different Alkali treatment exposure times

In the initial linear region of each curve, the stress increases proportionally with strain. This region corresponds to the elastic domain. The slope of this linear portion, which reflects the stiffness or Young's modulus of the composites, varies across the different treatments. According to Saha et al²⁷, a steeper slope indicates a stiffer material. The 1TC sample exhibits the highest slope, indicating superior stiffness, likely due to improved fiber-matrix adhesion and the stiffness of the fibers as enhanced by optimal chemical treatment²⁸.

In contrast, the 6TC and 24TC samples display lower stiffness, suggesting a degradation of the reinforcement structure due to overexposure to chemical treatment. As the curves transition beyond the linear region, they enter the non-linear deformation phase, which signifies the onset of plastic deformation, including irreversible changes such as matrix cracking, fiber debonding, and fiber pull-out. This region also marks the yield point, where the material ceases to behave elastically, a critical threshold in the mechanical performance of composites²⁹.

Among all samples, the 3TC composite shows the highest strain at break, suggesting superior ductility and the ability to absorb more energy before fracture. This increased strain capacity likely results from an optimal balance between fiber integrity and enhanced interfacial bonding due to moderate chemical treatment. Differences in the non-linear behavior between samples can be attributed to variations in fiber alignment, the extent of interfacial debonding, and the contribution of matrix deformation to the overall strain, as discussed by Izaki et al³⁰. The ultimate tensile strength (UTS), which corresponds to the maximum stress each composite can endure before failure, is highest in the 1TC sample, emphasizing the improvement in load-bearing capacity achieved through moderate chemical treatment. Conversely, the 6TC and 24TC samples show reduced UTS, likely due to weak interfacial bonding and fiber degradation. The strain at break, which indicates ductility, is particularly high in the 3TC sample, reinforcing its potential for applications requiring flexibility and impact resistance³¹.

Overall, the results demonstrate that while moderate alkali treatment (1TC–3TC) significantly enhances the mechanical performance of jute-reinforced composites, excessive treatment (6TC and 24TC) may be detrimental, highlighting the importance of optimizing treatment duration for desired properties.

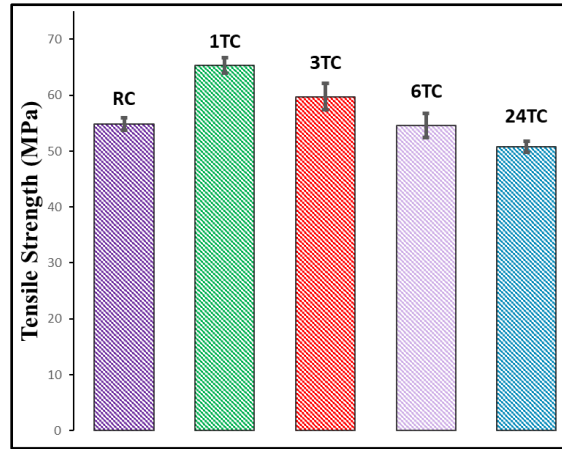


Figure 6. Evolution of the tensile strength of the composites as a function of alkali treatment time

In particular, the mechanical strength of the NaOH-treated jute-reinforced composites for 1h was higher than that of the others, and the strength of the composites reinforced with fabrics treated for 1h and 3h was improved by 20% and 9%, ($65,5 \pm 1.3$ MPa and 59.7 ± 2.3 MPa) respectively, compared to that of the composites reinforced with untreated fabrics (54.8 ± 1.1 MPa). As the studies of Owen et al, Ray D et al indicate that the alkali treatment enhances the mechanical properties of the resulting composite by significantly improving fiber-matrix adhesion, leading to more effective stress transfer and improved performance under tensile loads.^{32, 33}

The strength of the composites reinforced with fabrics exposed to a soda solution for 6 and 24 hours (54.6 ± 2.1 MPa and 50.7 ± 1.0 MPa, respectively) was reduced by 1% and 7% respectively compared to the strength of the composites reinforced with raw fabrics (54.8 ± 1.1 MPa). This decrease is probably due to the extended alkali treatment time, which can remove hemicellulose and lignin and change the native cellulose.^{34,35}

X-ray diffractometry (XRD)

Using the X'Pert HighScore software for diagram analysis, the X-ray diffraction pattern of jute fiber is shown in figure 7(a). It contains two major diffraction peaks at $2\theta = 23^\circ$, which designates the crystalline structure I_{cr} , and the peak at $2\theta = 16^\circ$ indicating the existence of amorphous constituents I_{am} , usually detected in most biofibers.³⁶

Alkali treatment does not change the X-ray diffraction pattern, nor does it cause any

displacement of the crystalline or semi-crystalline planes, but rather an increase in the existing phase peaks, particularly for fibers treated at 1 hour, which have the highest crystallinity index (CI =43.43%).

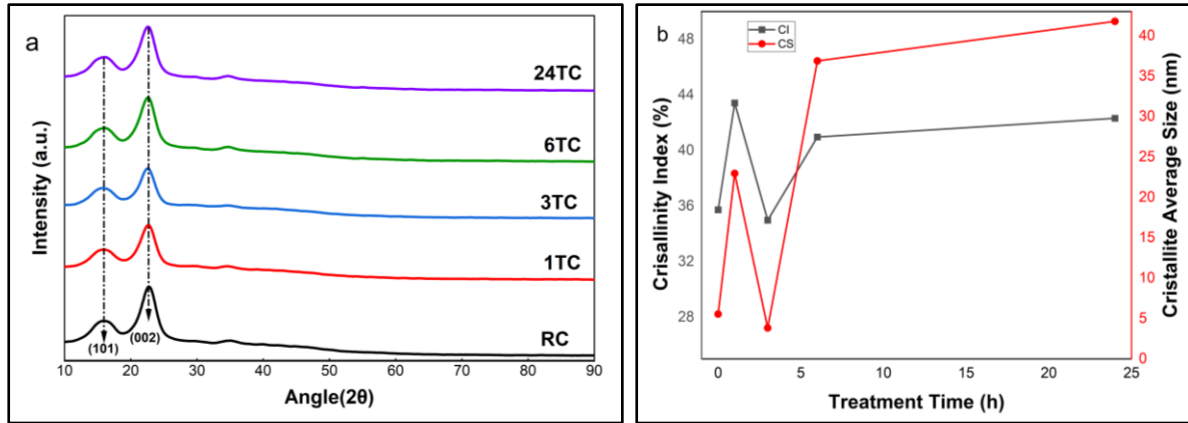


Figure 7. XRD analysis graphs (a) Diffractogram of raw and alkali treated fabrics (b) crystallinity rate and crystallite size

The effect of the caustic solution on the two crystallinity parameters, CI and CS, is shown in figure 7(b). Three significant stages can be distinguished: an increase in the crystallinity index (from 35.73 to 43.42%) and in the average crystallite size (from 5.54 to 22.94 nm) after 1-hour of treatment due to the removal of impurities and waxy constituents from the surface of jute fabric. The second part shows a significant decrease in CI and CS after 3 hours of alkalization due to eliminating the amorphous part constituting our reinforcement.

After 6 hours of alkali treatment, the crystallinity index and crystallite size again increased; the prolonged treatment time led to the reconstruction of new crystalline arrays, which re-increasing the average crystallite size to 36,88 and 41,78 nm in 6 and 24 hours of treatment time, respectively.³⁷

IV.3 FTIR of raw and treated jute fabrics

FTIR spectral data for raw and treated jute fabrics are shown in figure 8. The peak at 3400 cm^{-1} is an O-H stretching vibration recorded only in the untreated jute fabric signal.³⁸ The noticeable peak at 2925 cm^{-1} for raw jute signifies a C-H stretching vibration of cellulose and hemicellulose,³⁹ becoming smaller after prolonged alkaline treatment. The re-emergence of the peak at 2925 cm^{-1} in 24 treated fibres indicates a restoration or alteration of the C-H bonds, which may be caused by the formation of new bonds, a reorientation of existing bonds,

or changes in the physical state of the fiber. This could for example result from the recrystallization of cellulose, leading to a more ordered arrangement of C-H bonds and an intensification of this peak.⁴⁰

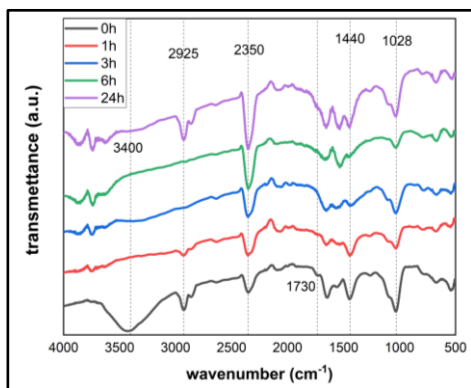


Figure 8. FTIR spectroscopy of jute fabrics

Natural fibers consist mainly of cellulose, hemicellulose, lignin, or protein and generally do not contain nitrile groups in their basic chemical structure. Therefore, the appearance of this peak corresponds to the presence of nitrile groups ($C\equiv N$), which indicates chemical modification after alkalization and perfectly explains the increase in the transmittance peak of around 2350cm^{-1} throughout the treatment. The same applies to raw jute, which is probably pre-treated and undergoes the same chemical modifications.^{41, 42}

The peak recorded at 1735 cm^{-1} in raw jute fabric is no longer present in all treated fabrics; it is signifying a $C=O$ stretching vibration of the carboxylic acid in the hemicellulose,^{43, 44} a significant part of the hemicellulose having been removed by alkalization.

The peak at 1440 cm^{-1} is due to plane deformation of the CH_2 group in lignin.⁴⁵

Table 3. Recapitulating sources of different recorded frequencies

Frequency (cm^{-1})	3400	2925	2350	1735	1440	1028
Possible allocations	(O-H) stretching	C-H vibration	Nitrile elongation $C\equiv N$	$C=O$ stretching vibration of	Deformation in the CH_2 group plane	C-O compound stretching
Components	cellulose, hemicellulose	Cellulose and hemicellulose	Probable atmospheric CO_2	Hemicellulose	Lignin	Cellulose and hemicellulose

IV.4 Image processing using X-ray tomography

The tomographic observations reveal the area slightly distant from the fractured zone, as illustrated in figure 4. By processing these bi-dimensional images with Fiji software,⁴⁶ we reconstructed and visualized the distribution and ratio of porosity in the scanned zone, which varies with the duration of jute fabric treatment.

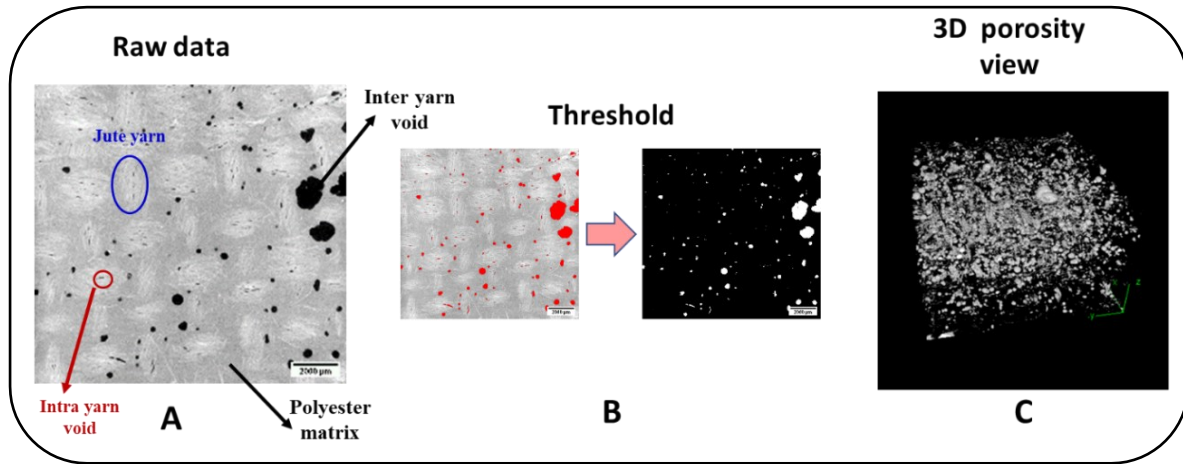


Figure 9. 3D Reconstruction Schematic Steps

We adjust the contrast of the bi-dimensional color-coded slices, where red represents porosity. After that, we use white instead of red and black for the background, giving us representation B.

The 3D viewer after 2D image reconstruction for each case of prepared composites is as follows.

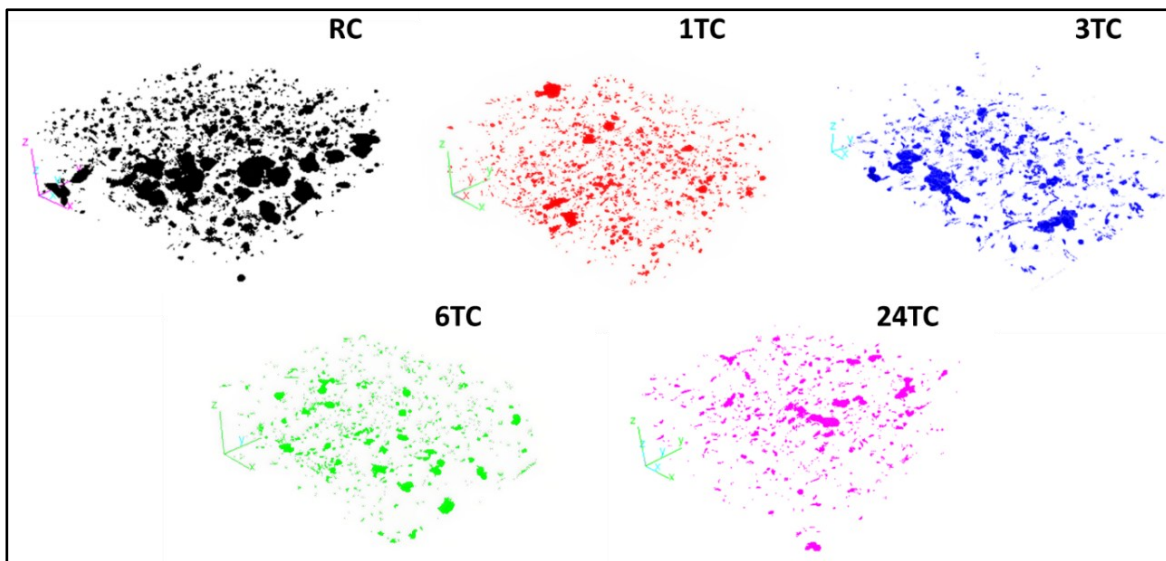


Figure 10. 3D porosity presentation for different composites

X-ray tomography image processing allows us to identify not only the three-dimensional nature of pores/voids in composites, but also their shape, location, and even the average size, as shown in figure 10.

Reliable quantification and localization of porosity in biocomposites is vital for determining its effect on mechanical and structural performance, which is the aim of this non-destructive method.

To calculate porosity fraction, we measure the area fraction of all 2D images making up the scanned volume (X=15 Y=13 Z=6) mm; this observation is conducted in a zone for two specimens for each type of elaborated composite. We changed the color of the void to make a distinction between each case of treatment.

Figure 11 shows the effect of alkali treatment time on the porosity rate in the jute/resin composite. The beginning of the curve presents the maximum ratio of porosity due to the presence of gum and impurities, which will generate air bubbles during the polymerization stage. A notable decrease occurs after 1h of treatment due to the degumming process caused by alkalization.⁴⁷ Then, the curve tends to stabilize despite the increasing treatment time.

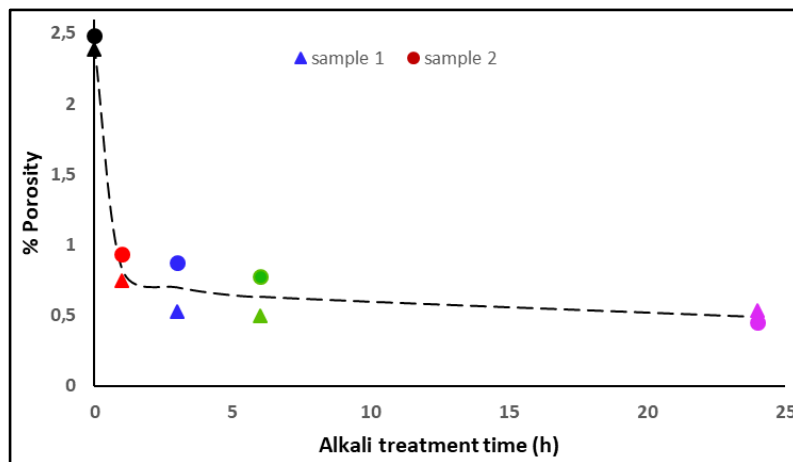


Figure 11. Alkali treatment effect on porosity ratio of jute/polyester composite

To optimize the mechanical properties (tensile test) of jute fabric/polyester resin composites for each case (raw jute, treated 1 hour, 3 hours, 6 hours, and 24 hours), we need to analyze the diagrams of figure 12 showing the impact of crystallinity index, average crystallite size, and porosity rate on tensile strength and Young's modulus.

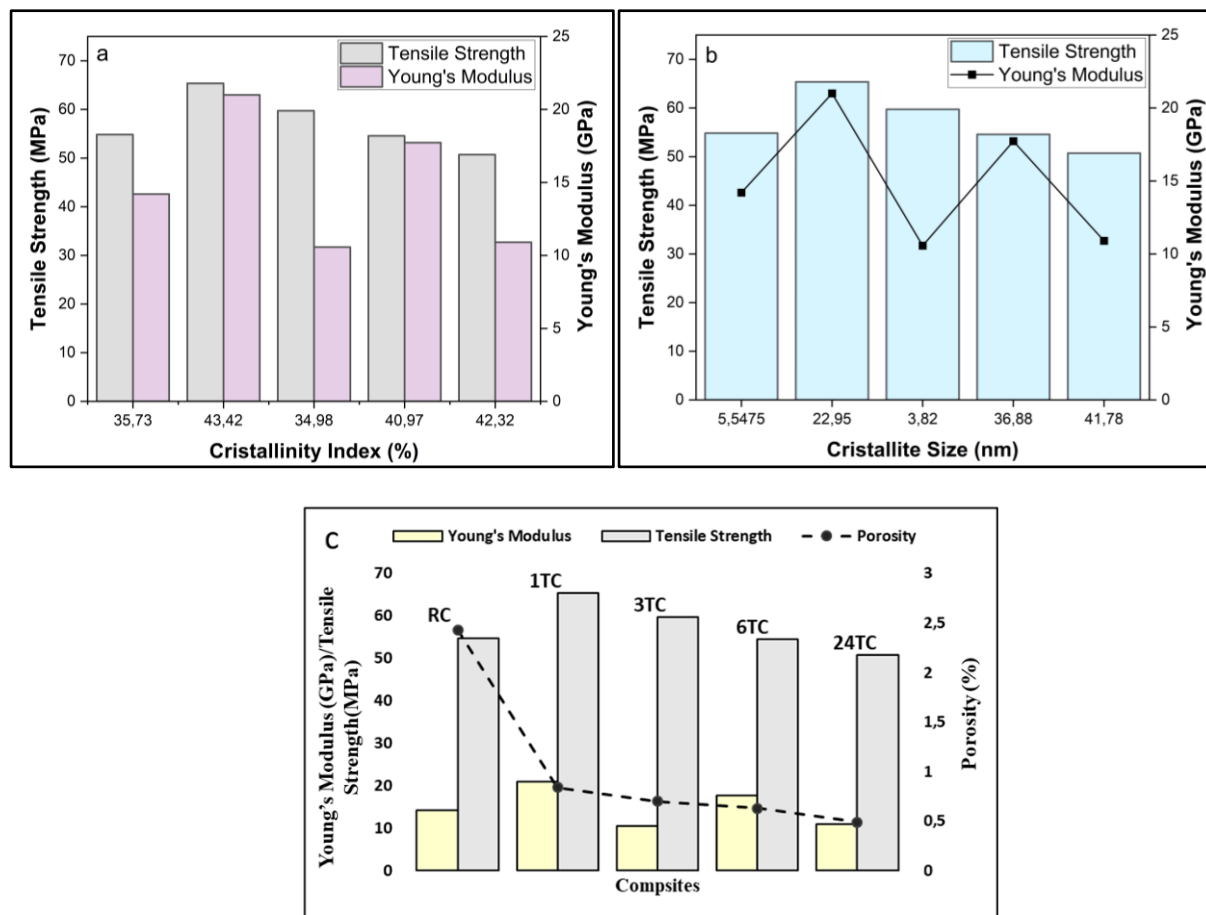


Figure 12. Impact of crystallinity index, crystallite size, and porosity rate on tensile test properties

Crystallinity index

A higher crystallinity index indicates a better structural organization within the fiber matrix, which enhances stress transfer and mechanical performance. This correlation is clearly reflected in the 1TC composite, which exhibits the highest crystallinity index (43.42%) among the tested samples and correspondingly shows the best tensile strength (65.5 ± 1.3 MPa) and Young's modulus (21 ± 1.22 GPa). These findings are consistent with the reported study of Vinod A. et al that establish a positive relationship between crystallinity and mechanical strength.⁴⁸ This improvement was attributed to the removal of amorphous components like hemicellulose and lignin, promoting a more ordered microstructure and stronger interfacial bonding.⁴⁹

Crystallite size

In addition to crystallinity, crystallite size also plays a pivotal role in determining the composite's tensile performances. As shown in Figure 11(b), tensile strength and Young's modulus peak at an intermediate crystallite size of 22.95 nm. Both smaller (3.82 nm) and larger crystallite sizes (41.78 nm) are associated with diminished mechanical properties, suggesting the existence of an optimal size range that favors structural cohesion. Smaller crystallites may represent underdeveloped crystalline domains with limited load-bearing capacity, whereas larger crystallites can induce anisotropic regions that become stress concentration points under mechanical loading.⁵⁰

Porosity rate

Figure 11(c) underscores the impact of porosity on composite mechanical performance. A clear inverse relationship is observed: as porosity decreases, both tensile strength and Young's modulus increase. 1TC composite, with the 0.84 % of porosity ratio, exhibits superior mechanical strength and the highest stiffness. Despite 6TC and 24TC composites exhibit the lowest porosity (0.6 and 0.5 respectively), their mechanical performance is compromised due to the combined effects of reduced crystallinity, non-optimal crystallite size, and possible fiber damage. The optimal balance of all these parameters is achieved in 1TC, where a relatively low porosity (0.84%) aligns with the highest crystallinity index and an optimal crystallite size, resulting in superior mechanical properties.

IV.5 SEM observations of fractured surfaces

Figure 13 shows SEM observations of the fractured surfaces of raw and alkali-treated jute fabrics reinforced composites.

The micrographs of the observed specimens indicate the presence of a multitude of fiber/matrix de-cohesion, representing the predominant mechanism for this composite. These de-cohesions are observed either between the fiber bundles and the matrix (label 2) or between the elementary fibers within a fiber strand (label 1).⁵¹

In the 24h treated composite, the matrix crack appears perpendicular to the loading direction (label 3). For the 1-hour treated jute reinforced composite (b), the SEM observations of the fracture surfaces indicate that the broken jute yarns show significant raveling with sharp edges, which perfectly explains the good wettability between the fabric and the matrix of this treatment case (label 4).

Label 5, related to 6h treated composites, refers to the longitudinal separation of the elementary jute fibrils.

The granular aspect of the matrix surface (label 6) indicates the brittle failure mode.⁵²

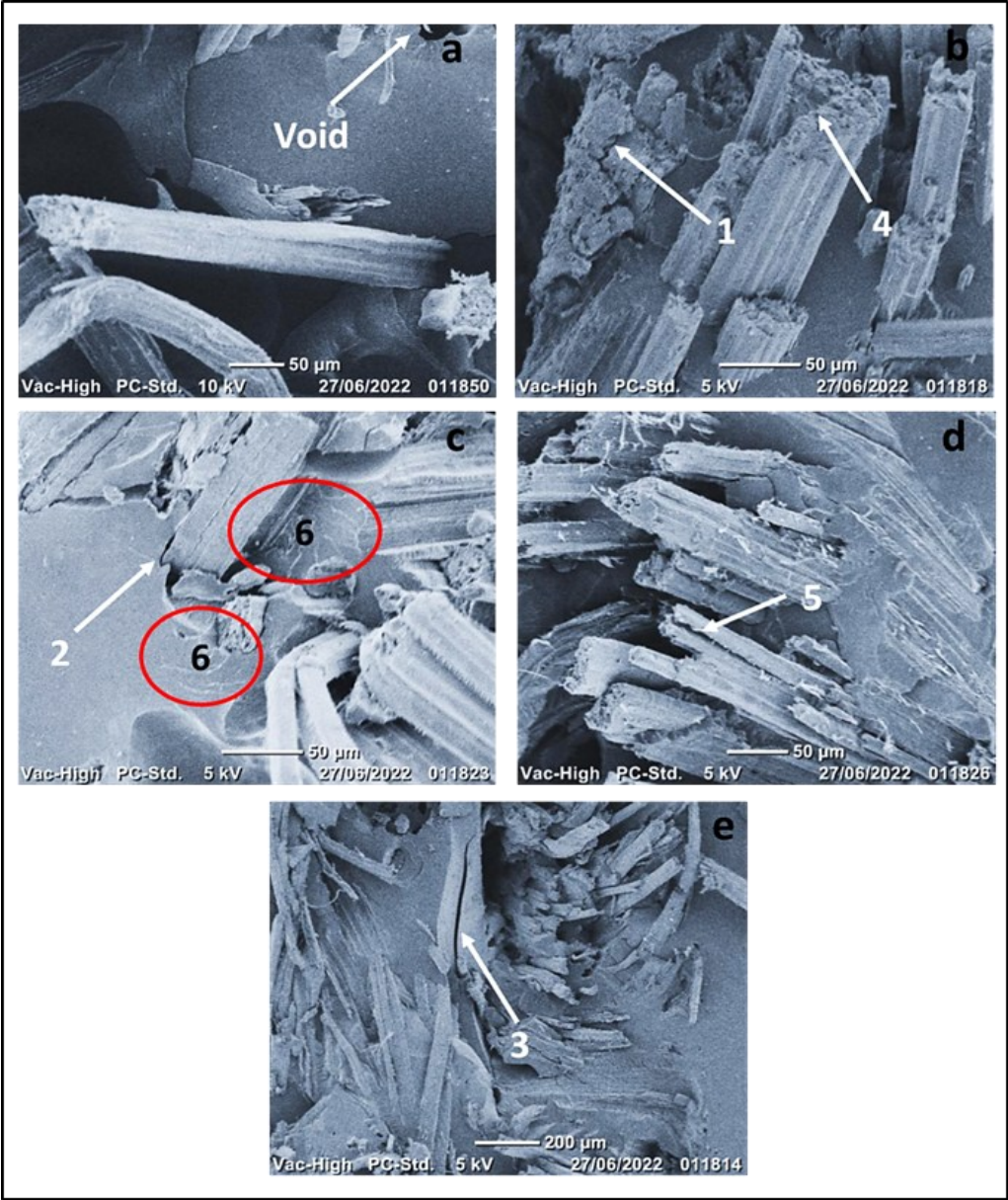


Figure 13. SEM observations of fracture surfaces (a) RC composite (b) 1TC (c) 3TC (d) 6TC (e) 24TC

However, figure 14 (f) shows a SEM micrograph of untreated jute yarn in cross-section, illustrating the internal structure of individual fibers. The image clearly reveals the circular to oval cross-sectional shape of jute fibers, with visible central lumens surrounding by cell walls.

The presence of distinct lumen cavities is characteristic of natural plant fibers, contributing to their lightweight nature and specific surface area.⁵³ The smooth surface and intact cellular arrangement indicate that the fiber has not undergone chemical modification. The observed structure is consistent with the typical morphology of raw jute, where the lumen plays a role in moisture absorption and can influence the fiber-matrix interaction in composites.⁵⁴

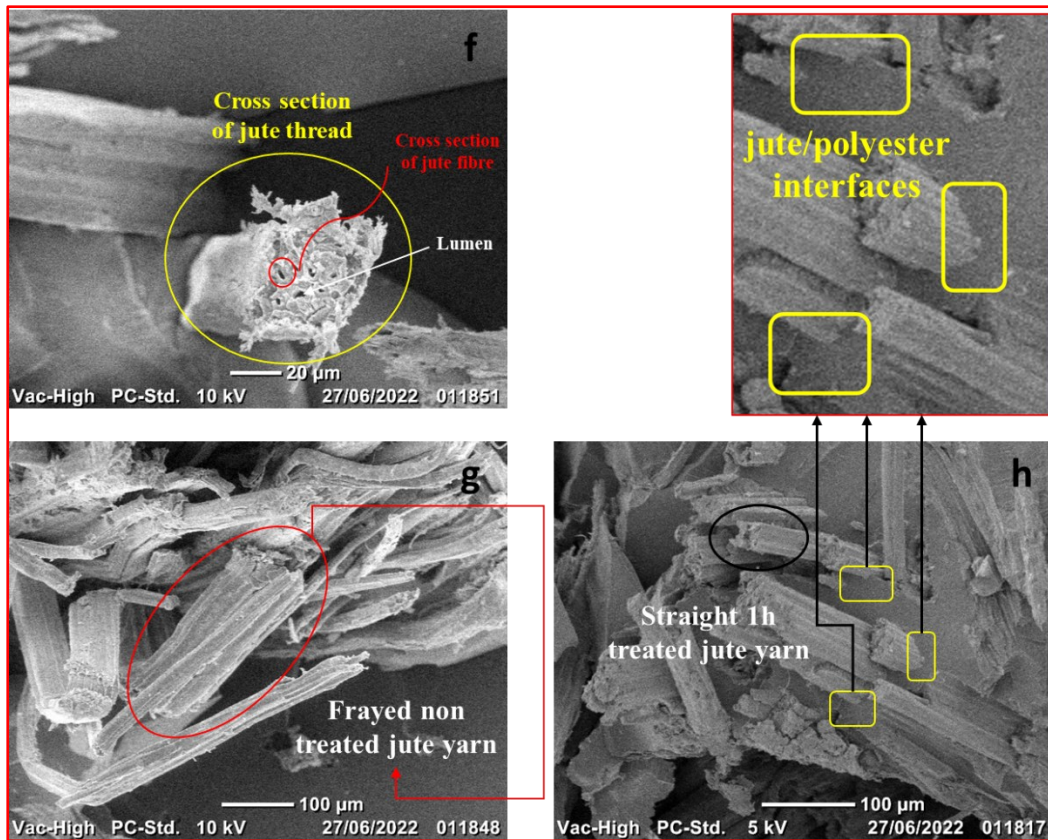


Figure 14. SEM micrographs of jute/polyester composite (f) Cross section of untreated jute yarn (g) RC yarns after tensile test (h) 1TC yarns after tensile test

SEM image of untreated jute yarns reveals that they are often frayed, loosely bundled, and poorly aligned, indicating a lack of structural integrity and cohesion figure14 (g). This fraying observed in untreated jute yarns indicates inadequate structural integrity and limited cohesion between the elementary fibers within the yarn, which compromises the overall strength and stiffness of the composite material.⁵⁵

In the other side, 1 hour alkali treatment leads to straightened and more consolidated jute yarns, as observed in figure14 (h), indicating improved fiber alignment and cohesion within the yarn structure. This transformation in fiber morphology is a direct result of the chemical changes

induced by the alkaline solution, which removes surface impurities and modifies the fiber surface. The straightened and consolidated yarns exhibit a more organized and uniform structure, which is essential for achieving optimal mechanical performance in composite materials.⁵⁶

V. Conclusion

In this study, we have investigated the effects of alkali treatment on the crystallinity index, porosity rate, crystallite size, and overall tensile test properties of jute fabric-reinforced polyester composites. Various alkali treatment durations were addressed to determine the optimal conditions for enhancing composite performance.

Overview of this study

Mechanical properties

RC composite presents lower strength and stiffness due to a less optimal crystallinity index, crystallite size, and relatively high porosity.

1TC presents the best global performance regarding mechanical strength (improvement of 20%) and Young's modulus (from 14 ± 1.22 GPa in RC composite to 21 ± 1.22 GPa in 1TC composite). This treatment time optimizes the crystallinity index and crystallite size and minimizes porosity.

24TC: Despite the high crystallinity index (42.32%), the large crystallite size (41.78 nm) does not fully compensate, resulting in lower mechanical performance (50.72 ± 1 MPa strength and 10.9 ± 0.2 GPa rigidity).

Crystallinity and Structural Integrity

X-ray Diffraction (XRD) analysis revealed that the crystallinity index of the composites improved markedly after alkali treatment, peaking at 43.42% for the 1-hour alkali treatment. Hence, a more crystalline fiber structure contributed to enhancing mechanical properties.

Fourier Transform Infrared Spectroscopy (FTIR) confirmed the effective removal of hemicellulose and other impurities, indicating successful chemical modification of the jute fabrics.

Porosity and Density

The porosity of the composites was significantly reduced with alkali treatment. The optimum porosity rate (0.84%) was achieved after 1 hour of alkali treatment. This reduction in porosity is closely associated with improved mechanical performance and better fibre-matrix adhesion.

An increase in composite density was observed with optimal treatment, further supporting the enhancement of structural integrity.

Microstructural Characteristics

Scanning Electron Microscopy (SEM) revealed improved interfacial bonding between jute fibers and the polyester matrix in the treated composites, particularly those treated for 1 hour. This improvement is indicative of better resin impregnation and stronger fibre-matrix interaction.

The fracture surface analysis showed distinct changes in fracture mechanisms with different treatment durations, with the 1TC composite showing fewer defects and a more cohesive structure.

The results highlighted the effectiveness of sodium hydroxide (NaOH) treatment in improving the interfacial adhesion between jute fibers and the polyester matrix, leading to enhanced tensile strength and reduced porosity in the composites. Specifically, composites reinforced with fabrics treated for 1 hour exhibited superior mechanical performances (breaking stress and Young's modulus).

Structural characterization using XRD and FTIR confirmed changes in crystallinity index and chemical groups after treatment, indicating improved compatibility between fibers and matrix. X-ray tomography image processing provided valuable insights into the distribution of porosity, with treated composites showing decreased porosity values, further validating the benefits of alkali treatment. SEM observations elucidated various fracture mechanisms, highlighting the importance of interfacial bonding in determining composite properties. Overall, these findings underscore the potential of alkali treatment as a cost-effective and efficient method to enhance the mechanical and structural properties of jute fabric-reinforced polyester composites, contributing to their widespread application in various industrial sectors. Future research could optimise treatment parameters and explore additional characterization techniques to understand the underlying mechanisms further and refine composite properties.

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