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Comparative Study of Chemical Modification of Natural Fibers for Improved Mechanical Properties in Composite Materials

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Abstract

Natural fibers are increasingly investigated as low-density and renewable reinforcements for polymer composites, but their hydrophilic surfaces, waxes, lignin and hemicellulose reduce compatibility with hydrophobic matrices and restrict mechanical performance. This methodology paper proposes a controlled comparative study of chemical surface modification routes for improving the tensile, flexural and impact properties of natural-fiber composites. Jute fiber and epoxy resin are selected as a representative industrial composite system. Five fiber conditions are compared: untreated fiber, sodium-hydroxide (alkali) treatment, silane treatment, sodium-bicarbonate treatment and sequential alkali-silane treatment. The experimental design maintains constant fiber loading, fiber length, resin system, curing schedule and fabrication method so that differences can be attributed primarily to surface treatment. Modified fibers are characterized by Fourier-transform infrared spectroscopy, scanning electron microscopy, water absorption and single-fiber tensile testing, while composites are evaluated for tensile, flexural, impact and interfacial performance. Literature through 2021 indicates that alkali treatment removes non-cellulosic constituents and increases roughness, whereas silanes can form chemical bridges between cellulose and polymer matrices; combined treatment can therefore provide stronger interfacial load transfer. An illustrative result set is included only to demonstrate the proposed analysis and ranking procedure. The methodology is intended to support material selection for automotive panels, construction components, furniture, packaging and other lightweight industrial products.

Introduction

Natural-fiber-reinforced polymer composites offer low density, renewability, attractive specific properties and lower abrasive wear on processing equipment than many mineral or glass reinforcements. However, natural fibers contain cellulose, hemicellulose, lignin, pectin, waxes and extractives, and the hydroxyl-rich surface readily absorbs moisture. The resulting polarity mismatch with many polymer matrices

can produce poor wetting, weak interfaces and premature debonding under load. Chemical modification is therefore widely used to alter surface chemistry, remove weak boundary layers and improve fiber-matrix adhesion [1-3].

Alkalization or mercerization is among the most common treatments because it can remove waxes and portions of lignin and hemicellulose, increase surface roughness and promote fibrillation [1,3,11]. Silane coupling agents are

used to create a molecular bridge between hydroxyl groups on lignocellulosic fibers and reactive groups in the polymer matrix [2]. Studies on kenaf, sisal, hemp and jute systems show that treatment effectiveness depends on fiber type, chemical concentration, treatment time and matrix chemistry; excessive treatment may damage cellulose or reduce intrinsic fiber strength [4,7,8]. This study therefore emphasizes a comparative, controlled procedure rather than assuming that more severe treatment necessarily gives better composite properties.

Literature Review

Li et al. [1] reviewed chemical treatments used for natural-fiber composites and described alkali, silane, acetylation, benzylation and related routes for reducing hydrophilicity and improving interfacial bonding. Xie et al. [2] specifically reviewed silane coupling agents and explained their hydrolysis, condensation and coupling mechanisms at natural-fiber/polymer interfaces. Herrera-Franco and Valadez-González [4] demonstrated that surface modification of short henequen fibers improved stress transfer in polymer composites. Asumani et al. [5] reported that sequential alkali-silane treatment of kenaf significantly improved tensile and flexural properties compared with individual treatments. Goriparthi et al. [6] showed that jute-fiber surface treatment affected both mechanical and abrasive-wear behavior in PLA composites. Orue et al. [7] found that alkali-treated sisal/PLA composites displayed improved interfacial

adhesion, although treatment could reduce the strength of individual fiber bundles.

Fiore et al. [8] investigated sodium bicarbonate as a comparatively mild and eco-friendlier chemical treatment for sisal fibers and epoxy composites, providing a useful benchmark against conventional NaOH treatment. Pickering et al. [9] reviewed natural-fiber composite mechanical performance and highlighted the importance of fiber quality, interface strength, orientation and processing. Sepe et al. [10] compared chemical treatments of hemp/epoxy composites and confirmed that interfacial changes can improve tensile and flexural behavior when treatment conditions are suitably selected.

Hamidon et al. [11] reviewed kenaf-fiber treatments and emphasized that silane can improve fiber-matrix bonding, while alkali treatment is especially effective for cleaning and roughening the fiber surface. Syduzzaman et al. [12] summarized fabrication routes and applications of plant-based natural-fiber composites. In 2021, Verma and Goh [13] reviewed mercerization effects on fiber morphology and mechanical response; Khalid et al. [14] surveyed chemical treatments and manufacturing strategies for biodegradable composites; and Alao et al. [15] experimentally evaluated alkali and silane pretreatments in hemp/PLA systems. Collectively, these studies support a methodology that compares multiple treatment chemistries under a single fabrication and testing framework.

Table 1. Comparative mechanisms and limitations of the selected chemical modifications.

Treatment	Primary surface action	Expected interface effect	Main risk/limitation	Representative sources
Alkali (NaOH)	Removes waxes, pectin and part of lignin/hemicellulose; fibrillation	Higher roughness and mechanical interlocking	Over-treatment may damage cellulose	[1,3,11,13]
Silane	Forms siloxane network and organofunctional coupling layer	Chemical bridge with polymer matrix	Sensitive to pH, hydrolysis and dosage	[2,5,15]
NaHCO ₃	Milder cleaning and compositional modification	Improved wetting with lower chemical severity	Longer treatment may be needed	[8]
Alkali + silane	Surface cleaning followed by coupling-agent grafting	Potentially strongest load transfer	Additional process steps and chemicals	[5,15]

Methodology

1. Materials and Experimental Design

Woven or aligned jute fiber is selected as the reinforcement and a room-temperature-curing epoxy resin/hardener system as the matrix. The

target reinforcement content is fixed at 30 wt% for all laminates. Fibers are cut to equal dimensions, washed in distilled water, and dried at 60 °C to constant mass before treatment. Five treatment groups are prepared: U (untreated

control), A (5 wt% NaOH), S (silane), B (5 wt% NaHCO₃), and AS (5 wt% NaOH followed by silane). At least five mechanical specimens per test and treatment group are recommended so that mean, standard deviation and statistical significance can be reported.

2. Step-by-Step Fiber Modification

Step 1 - Cleaning and conditioning: Wash jute fibers to remove dust and soluble impurities, dry to constant mass, and store in a desiccator before treatment.

Step 2 - Alkali treatment (A): Immerse fibers in 5 wt% NaOH at room temperature for 2 h. Rinse repeatedly with distilled water until the wash water is approximately neutral, then dry at 60 °C.

Step 3 - Silane treatment (S): Prepare a 2 wt% 3-aminopropyltriethoxysilane solution in

ethanol/water (95:5 v/v), adjust to mildly acidic pH to promote hydrolysis, immerse fibers for 1 h, drain, dry and condition.

Step 4 - Sodium-bicarbonate treatment (B): Immerse fibers in 5 wt% NaHCO₃ solution for 24 h at room temperature, rinse with distilled water and dry. This route is included as a milder benchmark based on the eco-friendlier approach reported by Fiore et al. [8].

Step 5 - Sequential treatment (AS): Perform the NaOH treatment first, neutralize and dry the fibers, and then apply the silane treatment. This tests whether surface cleaning and coupling-agent grafting act synergistically [5,15].

Step 6 - Conditioning: Equilibrate all treated and untreated fibers under the same laboratory conditions before composite fabrication to minimize moisture-related variability.



Figure 1. Proposed comparative workflow for chemical modification, composite fabrication and testing.

3. Composite Fabrication

Each fiber group is incorporated into the same epoxy system using hand lay-up followed by compression molding. Resin and hardener are mixed according to the supplier ratio, degassed when possible, and applied uniformly to the fibers. Laminates are consolidated under the same pressure and cured for 24 h at room temperature followed by a controlled post-cure if required by the resin system. The fiber mass fraction, laminate thickness and curing schedule are held constant. Specimens are then cut from comparable laminate regions to minimize effects of thickness gradients or edge defects.

4. Characterization and Mechanical Testing

Chemical and morphological characterization is used to verify that the treatments altered the fiber surface before interpreting composite strength. FTIR spectra are recorded to monitor changes in bands associated with hemicellulose, lignin and silane-related groups. SEM is used to compare surface roughness and fracture-surface

evidence of fiber pull-out, gaps and resin adhesion. Fiber water uptake is determined gravimetrically after controlled immersion, while selected single fibers are tensile tested to check whether the treatment has excessively weakened the reinforcement. These observations are interpreted together because stronger composite behavior can result from improved interface strength even when the intrinsic strength of individual treated fibers decreases [7,9].

Composite tensile testing is performed using specimens prepared to the geometry of ASTM D3039 where applicable; flexural performance is assessed by three-point bending using ASTM D790-type geometry, and notched impact behavior by an ASTM D256-type method. For each property, at least five valid specimens are recommended. Tensile strength, tensile modulus, flexural strength, flexural modulus and impact energy are calculated from the corresponding force-displacement data. Fractured surfaces are retained for SEM examination.

Table 2. Controlled factors, characterization techniques and response variables.

Factor response / Level method or	Controlled variable	Purpose	Output	
Fiber treatment	U, A, S, B, AS	Same jute batch	Primary comparison	Treatment effect
Fiber loading	30 wt%	Constant	Eliminate reinforcement-content bias	Comparable laminates

FTIR / SEM	Before composite fabrication	Same acquisition settings	Confirm chemical/morphological change	Surface evidence
Tensile test	ASTM D3039-type	Same geometry and crosshead rate	Load-bearing performance	Strength/modulus
Flexural test	ASTM D790-type	Same span/thickness ratio	Bending performance	Strength/modulus
Impact test	ASTM D256-type	Same notch and thickness	Damage tolerance	Impact energy
Statistics	One-way ANOVA + Tukey test	$\alpha = 0.05$	Compare treatment groups	Significant differences

5. Data Analysis and Treatment Ranking

Mean and standard deviation are calculated for every measured response. One-way analysis of variance (ANOVA) tests whether treatment has a statistically significant effect, followed by Tukey's post-hoc comparison when appropriate. Mechanical-property improvement is reported relative to the untreated control. A simple normalized performance index can be used for overall ranking: 40% tensile strength, 30% flexural strength, 15% impact energy, 10% reduction in water uptake and 5% process simplicity. A sensitivity check should also be performed because industrial priorities vary: automotive panels may emphasize impact and moisture resistance, whereas structural boards may place greater weight on flexural stiffness.

6. Quality Control

To maintain reproducibility, all solutions are prepared from the same reagent lots; treatment temperature and time are recorded; fiber drying is continued to constant mass; composite fiber fraction is verified gravimetrically; and void-rich laminates are rejected before mechanical testing. Untreated controls are processed through the same washing, drying and fabrication history. These controls allow mechanical differences to be assigned to surface modification rather than handling or curing variations.

Results

Methodological note: The numerical values below are an illustrative benchmark dataset for showing how the proposed analysis should be reported. They are not presented as measurements from a completed laboratory experiment and must be replaced with actual experimental data.

1. Illustrative Mechanical-Property Comparison

The illustrative dataset shows a consistent improvement in composite strength after treatment, with the largest gains assigned to the sequential alkali-silane condition. In this example, the untreated laminate has tensile and flexural strengths of 48 and 78 MPa, respectively. Alkali treatment increases these values to 62 and 96 MPa, while silane treatment gives 66 and 103 MPa. Sodium bicarbonate produces a smaller but still useful increase, whereas the combined alkali-silane route reaches 72 MPa tensile strength and 112 MPa flexural strength. The pattern is consistent with the mechanistic expectation that alkali treatment cleans and roughens the surface and subsequent silane coupling improves chemical interaction with the matrix [2,5,11,15].

Table 3. Illustrative benchmark results for demonstrating the comparative analysis.

Treatment	Tensile strength (MPa)	Flexural strength (MPa)	Impact energy (kJ/m ²)	Water uptake (%)	Relative tensile gain	Illustrative rank
Untreated	48 ± 3	78 ± 5	10.2 ± 0.7	8.1	0%	5
5% NaOH	62 ± 4	96 ± 6	12.8 ± 0.8	6.0	+29%	3
Silane	66 ± 3	103 ± 5	13.6 ± 0.7	5.2	+38%	2
5% NaHCO ₃	58 ± 3	90 ± 5	11.8 ± 0.6	6.5	+21%	4
NaOH + Silane	72 ± 4	112 ± 6	14.5 ± 0.9	4.6	+50%	1

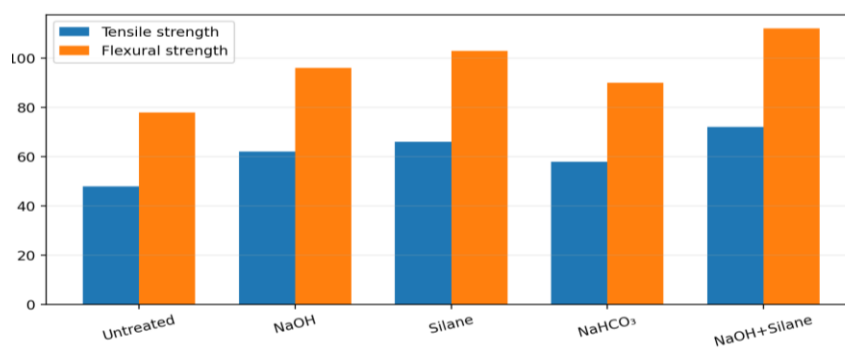


Figure 2. Illustrative tensile and flexural strength comparison for untreated and chemically modified jute/epoxy composites.

2. Expected Morphological and Chemical Trends

SEM would be expected to show a smoother, impurity-covered surface on untreated jute and progressively greater surface definition after alkali or bicarbonate treatment. Fracture surfaces of poorly bonded composites typically show long fiber pull-out and interfacial gaps, whereas successful treatment should produce more resin attachment and shorter pull-out lengths. FTIR changes associated with reduced hemicellulose/lignin contributions after alkali treatment and additional silane-related features after coupling-agent treatment provide supporting evidence that the interface has been chemically modified. These observations should be correlated with mechanical data rather than used alone to claim improved performance [1-3,7].

Conclusion and Discussion

This methodology provides a controlled framework for comparing chemical modification routes for natural fibers used in polymer composites. Jute/epoxy is used as the model system because it is representative of lightweight bio-composites and allows treatment effects to be isolated through constant fiber content and processing. The comparison includes conventional NaOH mercerization, silane coupling, a milder sodium-bicarbonate route and sequential alkali-silane treatment. Literature through 2021 supports the expectation that treatment can improve composite mechanical properties mainly by cleaning the fiber surface, increasing roughness, lowering hydrophilicity and strengthening interfacial adhesion [1-3,11-15]. The proposed interpretation deliberately distinguishes fiber strength from composite strength. Some chemical treatments can reduce the tensile strength of individual fiber bundles while still improving the final composite because stress transfer at the fiber-matrix interface becomes more efficient [7]. Therefore, treatment selection should be based on composite-level

tensile, flexural, impact and moisture performance together with FTIR and SEM evidence. The illustrative benchmark predicts the combined alkali-silane route to give the best overall performance, followed by silane and alkali treatment. Sodium bicarbonate may offer a useful compromise where reduced chemical severity and simpler waste handling are important [8]. For industrial translation, the optimum treatment must also consider cost, treatment time, chemical recovery, worker safety, effluent generation and compatibility with high-throughput manufacturing. Future studies should repeat the comparison with other fibers such as hemp, sisal, kenaf and flax, and with thermoplastic matrices such as polypropylene or PLA. Long-term moisture aging, fatigue, thermal cycling and recyclability should also be incorporated before recommending a treatment for automotive, building, furniture or packaging applications. The principal methodological conclusion is that a standardized side-by-side experimental design is more informative than comparing treatment results obtained from different fiber batches, matrices and test conditions.

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