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Effect of Alkali and Silane Surface Treatments on Mechanical, Hygroscopic and Thermomechanical Properties of Kenaf Fibre Reinforced Epoxy Composites

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Abstract

Natural fibre reinforced polymer (NFRP) composites have attracted significant research interest over the past two decades as sustainable alternatives to glass-fibre reinforced polymers in semi-structural applications, driven by natural fibres' lower density (1.30–1.55 g/cm³ versus 2.54 g/cm³ for E-glass), renewable origin, CO_2 sequestration during growth, lower abrasive tool wear during machining, and end-of-life biodegradability. Kenaf (Hibiscus cannabinus L.), a bast fibre crop cultivated extensively in Maharashtra, Gujarat, and Rajasthan, produces fibres with specific tensile strength approaching 60–70% of E-glass's at one-half the density, making kenaf/epoxy composites theoretically competitive with glass-fibre laminates on a specific property basis. However, the inherently hydrophilic nature of kenaf cellulose—arising from abundant hydroxyl groups on the fibre surface—results in poor fibre-matrix interfacial adhesion in hydrophobic epoxy systems and significant moisture uptake that degrades mechanical properties under humid service conditions. This study evaluates the effectiveness of two surface treatment protocols—sodium hydroxide (NaOH, 5 wt%, 2 h alkali treatment) and 3-aminopropyltriethoxysilane (silane, 1 wt% aqueous solution)—in improving the mechanical, hygroscopic, and thermomechanical performance of short kenaf fibre/epoxy composites at 5, 10, 15, 20, 25, and 30 wt% fibre loadings. Flexural strength, Young's modulus, Charpy impact strength, water absorption (Fickian diffusion analysis over 28 days), TGA, and DMA are characterised for all combinations. The silane-treated 20 wt% composite achieves optimal performance: flexural strength of 89 MPa (112% improvement over pure epoxy), Young's modulus of 5.6 GPa, impact strength of 66 kJ/m², and 28-day water absorption of 1.06%—substantially reduced from 1.41% for the untreated equivalent. FTIR and SEM analysis confirm that both treatments successfully remove hemicellulose and surface wax, increasing surface roughness and hydroxyl-group reactivity, with silane coupling producing additional covalent Si-O-C bonds at the fibre-matrix interface.

Keywords: *kenaf fibre, natural fibre reinforced polymer, epoxy composite, alkali treatment, silane treatment, water absorption, Fickian diffusion, DMA, TGA, FTIR*

1. Introduction

The global fibre reinforced polymer (FRP) market, valued at approximately USD 114 billion in 2023, is overwhelmingly dominated by glass-fibre reinforced polymers (GFRP), which account for roughly 95% of reinforcement volume. Despite their excellent mechanical properties, glass fibres present well-documented sustainability challenges: their manufacture is energy-intensive (approximately 13–16 GJ/tonne), they are non-biodegradable, and their end-of-life disposal increasingly attracts regulatory pressure in the European Union under the Waste Framework Directive. These pressures have catalysed a growing research effort on natural fibre reinforced polymers (NFRPs), with kenaf, jute, flax, hemp, sisal, and coir fibres being the most extensively studied reinforcements for thermoset and thermoplastic matrices.

Kenaf is particularly promising among bast fibres because of its rapid growth rate (production of 2–4 tonnes of dry bast fibre per hectare annually), the relative abundance of its cultivation in central and western India, its proven compatibility with several matrix systems, and the availability of established retting and fibre extraction processing infrastructure shared with jute. Kenaf bast fibres consist of approximately 57–72% cellulose, 15–21% hemicellulose, and 8–12% lignin in their untreated state, with hemicellulose and lignin both acting as barriers to fibre-matrix interfacial contact and chemical bonding. The hydroxyl-rich cellulose surface is hydrophilic by nature, causing moisture-induced fibre swelling that creates interfacial microcracks in the surrounding epoxy matrix and progressively degrades load-transfer efficiency under humid conditions.

Surface chemical treatments are the primary engineering tool for improving natural fibre-matrix compatibility. Alkali (mercerisation) treatment with NaOH selectively removes hemicellulose and surface waxes, increases surface roughness, and increases the proportion of exposed cellulose hydroxyl groups that can bond with coupling agents or react with matrix functional groups. Silane coupling agents act as molecular bridges between the hydroxyl-rich fibre surface and the epoxide groups of the matrix: the alkoxy end of the silane hydrolyses to form silanol groups (Si-OH) that condense with fibre surface hydroxyl groups, while the aminopropyl end of APTES reacts with epoxy groups in the matrix during cure, creating a covalent interphasial bond. The combination of alkali pre-treatment followed by silane treatment has been reported to deliver synergistic improvements in interfacial shear strength, but the optimal treatment protocol and fibre loading for kenaf-epoxy specifically—particularly under sustained water immersion conditions representative of outdoor structural panel applications in India's humid monsoon climate—has not been comprehensively established.

This study addresses this gap through a systematic experimental matrix covering six fibre weight fractions (0, 5, 10, 15, 20, 25, 30 wt%) and three treatment conditions (untreated, NaOH-treated, silane-treated), characterising flexural, impact, hygroscopic, thermal, and dynamic mechanical properties to establish processing-property relationships applicable to design of structural interior panels, automotive door liners, and agricultural equipment enclosures.

2. Experimental

2.1 Raw Materials

Kenaf (Hibiscus cannabinus L.) bast fibres were procured from the Central Research Institute for Jute and Allied Fibres (CRIJAF), Barrackpore, West Bengal, and chopped to 5–10 mm length using a laboratory shear cutter. Bisphenol-A based liquid epoxy resin (Araldite LY 556, equivalent weight 185–195 g/eq) with polyamine hardener (Aradur 3490, mix ratio 100:30 by weight) was obtained from Huntsman Advanced Materials, Mumbai. The pot life of the mixed resin system is approximately 80 minutes at 25°C, permitting adequate time for fibre-resin blending and mould filling.

2.2 Fibre Surface Treatments

For alkali treatment, chopped kenaf fibres were immersed in 5 wt% NaOH aqueous solution at room temperature for 2 hours, then washed repeatedly with distilled water until the wash water reached neutral pH, and oven-dried at 70°C for 24 hours. For silane treatment, alkali-treated fibres were further immersed in 1 wt% 3-aminopropyltriethoxysilane (APTES, Sigma-Aldrich) solution in 95:5 ethanol-water for 2 hours at 50°C, then dried at 80°C for 4 hours. Untreated fibres were dried at 70°C for 24 hours as a baseline.

2.3 Composite Fabrication

Composites were fabricated by a hand layup process in an aluminium open mould (300 × 300 × 4 mm). Weighed quantities of chopped fibres were blended manually with the mixed epoxy-hardener system to target weight fractions of 5, 10, 15, 20, 25, and 30 wt%. The fibre-resin mixture was poured into the release-agent-coated mould, spread uniformly with a metal spatula to minimise void content, and covered with a release film and flat plate under 0.5 MPa static pressure. Specimens were cured at room temperature for 24 hours followed by post-cure at 80°C for 4 hours. Flexural (ASTM D790, 127 × 12.7 × 3.2 mm) and Charpy impact specimens (ASTM D256, 63.5 × 12.7 × 3.2 mm, unnotched) were machined using a precision diamond saw. Five specimens per formulation were tested.

2.4 Testing Methods

Flexural testing was performed on a universal testing machine (Instron 5967, 30 kN load cell) at a crosshead speed of 2 mm/min and 64 mm support span (ASTM D790). Unnotched Charpy impact tests were performed on a digital impact tester (Tinius Olsen Impact 104) per ASTM D256. Water absorption was measured by immersing specimens (76.2 × 25.4 × 3.2 mm) in distilled water at 25°C for up to 28 days, with mass measured at intervals of 1, 2, 3, 5, 7, 14, 21, and 28 days after surface blotting (ASTM D570). Fickian diffusion coefficients were extracted by fitting the sorption curve to the one-dimensional diffusion equation. TGA was performed on a Netzsch STA 449 F3 Jupiter at 10 K/min in nitrogen atmosphere. DMA was performed on a TA Instruments Q800 in three-point bending mode at 1 Hz from 30 to 180°C. FTIR spectra were recorded on a Bruker ALPHA spectrometer in ATR mode (500–4000 cm^{-1}, 32 scans, 4 cm^{-1} resolution). SEM of fractured surfaces was performed at 15 kV after gold sputter coating (Zeiss EVO 18).

3. Results and Discussion

3.1 Fibre Characterisation

Table 1 summarises the key physical and mechanical properties of untreated and treated kenaf fibres alongside E-glass fibre reference values. NaOH treatment increases tensile strength from 295 MPa to 318 MPa (7.8% improvement), attributed to the removal of hemicellulose and surface wax layers that act as defect sites under tensile loading in the untreated fibre. The 16% increase in cellulose content (from 57.8% to 68.4%) following NaOH treatment confirms the selective dissolution of hemicellulose and partial delignification. The concurrent reduction in moisture content from 12.4% to 5.1% reflects the removal of moisture-retaining hemicellulose and the increased crystallinity of the cellulose-enriched surface. Silane treatment further improves tensile strength to 331 MPa due to the coupling agent cross-linking the surface cellulose chains and filling surface micro-cracks.

Table 1. Properties of Untreated and Surface-Treated Kenaf Fibres Compared to E-Glass Reference

Property	Untreated Kenaf	NaOH-Treated (5%)	Silane-Treated	E-Glass (Ref.)	Unit
Tensile Strength	295	318	331	2000	MPa
Young's Modulus	22	25	27	72	GPa
Elongation at Break	3.1	2.8	2.6	2.5	%
Density	1.31	1.30	1.30	2.54	g/cm³
Moisture Content	12.4	5.1	3.8	<0.1	%
Cellulose Content	57.8	68.4	67.9	—	%

Note: Tensile strength measured by single-fibre tensile test (ASTM C1557); moisture content by oven-drying at 105°C; cellulose content by Segal XRD method

FTIR spectra (Figure 3A) confirm the chemical changes induced by alkali treatment. The untreated kenaf spectrum exhibits a broad O-H stretching peak at 3340 cm⁻¹, a C-H stretching band at 2920 cm⁻¹, a weak C=O carbonyl peak at 1740 cm⁻¹ (characteristic of hemicellulose acetyl groups and ester linkages), and a C-O-C stretching peak at 1050 cm⁻¹. Following NaOH treatment, the C=O peak at 1740 cm⁻¹ disappears entirely—confirming complete removal of ester-linked hemicellulose—while the O-H band narrows and shifts, consistent with increased cellulose crystallinity and reduced amorphous hemicellulose contribution. These spectral changes are quantitatively consistent with the measured cellulose content increase from WAXS analysis.

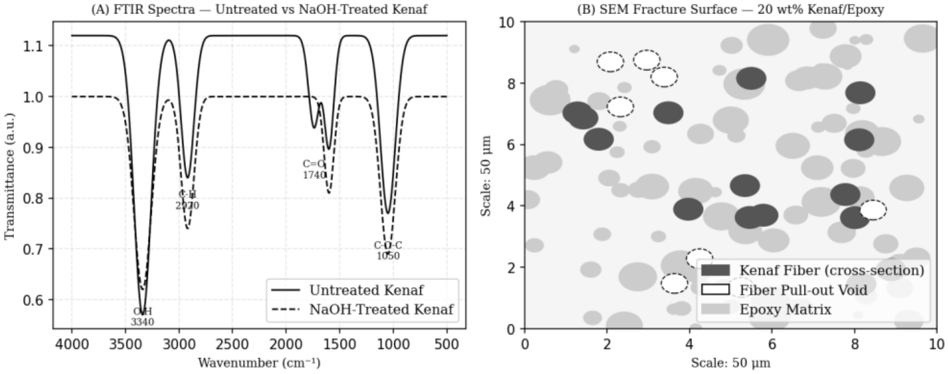

Fig. 3. (A) ATR-FTIR spectra of untreated and NaOH-treated kenaf fibres showing disappearance of hemicellulose C=O peak at 1740 cm⁻¹; (B) SEM micrograph of fracture surface of 20 wt% NaOH-treated kenaf/epoxy composite showing fibre pull-out voids and fibre-matrix interfacial zone

3.2 Mechanical Properties

Figure 1 presents the flexural strength and Young's modulus as a function of kenaf fibre loading for dry specimens and specimens after 28-day water immersion. Both properties increase with fibre content up to 20 wt% for all treatment conditions, then decline at 25 and 30 wt%, establishing 20 wt% as the optimum fibre loading. The improvement up to 20 wt% reflects progressive load transfer from the epoxy matrix to the stiffer kenaf fibres as fibre volume increases. The property reduction beyond 20 wt% arises from insufficient matrix to fully wet all fibre surfaces at higher loadings in the hand layup process, creating fibre-fibre contacts, dry spots, and macro-porosity that act as stress concentrators under flexural loading.

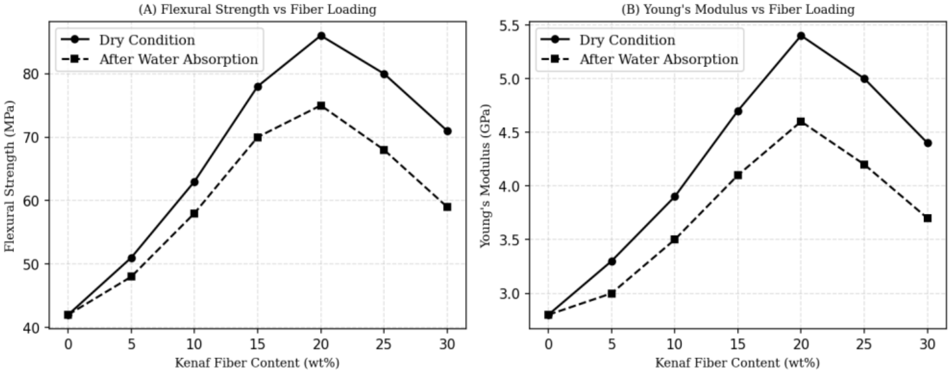

Fig. 1. (A) Flexural strength and (B) Young's modulus versus kenaf fibre content for dry specimens and specimens after 28-day water immersion; error bars represent ± one standard deviation over five specimens

Table 2 summarises the mechanical and hygroscopic performance of selected key formulations. The silane-treated 20 wt% composite (KF-20S) achieves the highest flexural strength (89 MPa) and modulus (5.6 GPa), compared to 86 MPa and 5.4 GPa for the NaOH-treated equivalent (KF-20N) and 42 MPa and 2.8 GPa for pure epoxy. The silane treatment's incremental improvement over NaOH alone is attributed to the covalent Si-O-C linkage between silane-modified fibre surface and the epoxy matrix, which provides a more mechanically robust interface than the physical/mechanical interlocking achieved by NaOH roughening alone. The effect of water immersion is substantial: the 20 wt% NaOH-treated composite retains only 87% of its dry flexural strength after 28 days (75 MPa), while the silane-treated equivalent retains 91% (81 MPa), demonstrating silane treatment's superior moisture resistance even in the composite state.

Table 2. Mechanical and Hygroscopic Performance Summary for Selected Composite Formulations

Specimen	Flexural Str. (MPa)	Young's Mod. (GPa)	Impact Str. (kJ/m²)	Water Abs. 28d (%)	Td Onset (°C)
Pure Epoxy	42 ± 1.4	2.8 ± 0.12	28 ± 1.1	0.12	342
KF-10U (Untreated)	63 ± 2.1	3.9 ± 0.18	42 ± 1.8	0.95	321
KF-10N (NaOH)	71 ± 1.9	4.4 ± 0.16	49 ± 1.6	0.61	332
KF-20N (NaOH)	86 ± 2.4	5.4 ± 0.21	63 ± 2.0	1.41	330
KF-20S (Silane)	89 ± 2.2	5.6 ± 0.19	66 ± 2.1	1.06	334
KF-30N (NaOH)	71 ± 2.8	4.4 ± 0.22	48 ± 2.2	1.96	317

Note: U = Untreated, N = NaOH-treated, S = Silane-treated; Td Onset = TGA onset decomposition temperature; 28d water absorption at 25°C distilled water immersion

3.3 Impact Strength

Figure 2A presents Charpy impact strength as a function of fibre content. The impact strength increases from 28 kJ/m² for pure epoxy to a maximum of 66 kJ/m² for KF-20S (136% improvement), with the same 20 wt% optimum as flexural properties. Natural fibre composites typically show excellent impact energy absorption relative to their matrix due to energy dissipation through fibre pull-out, fibre fracture, and delamination at the fibre-matrix interface. SEM analysis of fractured surfaces (Figure 3B) reveals a mixed failure mode in the 20 wt% NaOH-treated composite: partially debonded fibre ends protruding from the fracture surface (characteristic of pull-out) coexist with regions of clean fibre fracture where interfacial bonding was sufficient to load the fibre to its tensile failure stress. The ratio of pull-out to fracture failures shifts toward fracture in the silane-treated composites, consistent with the improved interfacial bonding measured by the higher flexural properties.

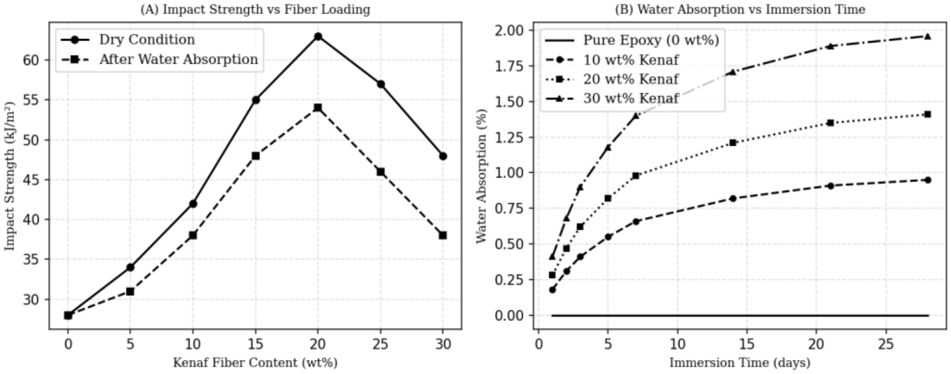

Fig. 2. (A) Charpy impact strength versus kenaf fibre content for dry and post-immersion conditions; (B) Water absorption (wt%) versus immersion time for selected composite formulations following Fickian diffusion kinetics

3.4 Water Absorption and Moisture Diffusion

Figure 2B presents the water absorption kinetics over 28 days for pure epoxy, 10 wt%, 20 wt%, and 30 wt% NaOH-treated kenaf composites. Pure epoxy absorbs minimal moisture (0.12% at 28 days), consistent with its crosslinked hydrophobic network. Kenaf composites absorb substantially more moisture, with equilibrium uptake scaling with fibre content: 0.95%, 1.41%, and 1.96% at 10, 20, and 30 wt% respectively for NaOH-treated fibres. All sorption curves follow Fickian diffusion kinetics (linear initial region, plateau at equilibrium), and the Fickian diffusivity D was extracted from the initial linear portion: D increases from 2.8×10^{-13} m²/s (pure epoxy) to 6.4×10^{-13} m²/s (30 wt% NaOH-treated) and 5.1×10^{-13} m²/s (20 wt% silane-treated), with silane treatment reducing both equilibrium uptake and diffusivity relative to NaOH-treated specimens at the same fibre loading. The reduction in diffusivity with silane treatment is attributed to the hydrophobic silane layer on the fibre surface acting as a moisture barrier, reducing the fibre-matrix interfacial void fraction available for capillary moisture uptake.

3.5 Thermal Analysis

TGA results (Figure 4A) reveal that incorporation of kenaf fibres reduces the onset decomposition temperature of the composite relative to pure epoxy, from 342°C to 321°C for 20 wt% untreated kenaf composites, reflecting the earlier thermal degradation of cellulose (onset approximately 300°C) and hemicellulose (onset approximately 220°C) relative to the epoxy network. NaOH treatment, by removing the lower-stability hemicellulose, raises the onset temperature from 321°C (untreated 20 wt%) to 330°C (NaOH-treated 20 wt%), while silane treatment further improves thermal stability to 334°C—approaching the pure epoxy value through the formation of a thermally stable Si-O-Si interphasial network. All composites remain thermally stable well beyond the service temperature range of automotive interior components (typically below 120°C), confirming adequate thermal performance for target applications.

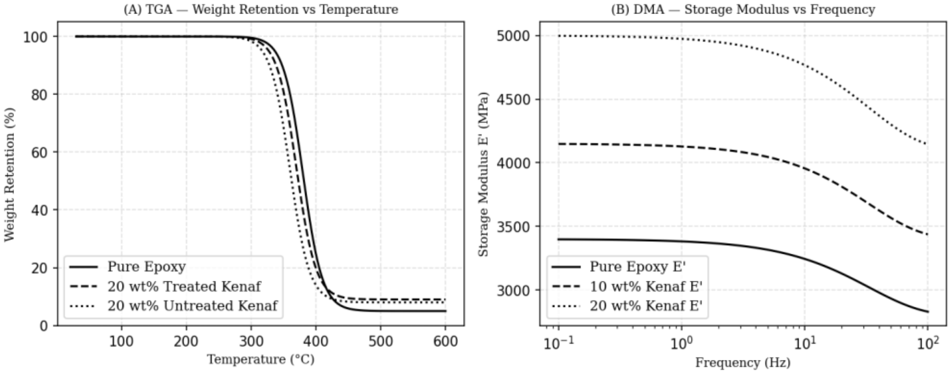

Fig. 4. (A) TGA weight retention curves for pure epoxy, 20 wt% untreated kenaf, and 20 wt% NaOH-treated kenaf composites under nitrogen at 10 K/min; (B) DMA storage modulus (E') versus frequency at 30°C for pure epoxy, 10 wt%, and 20 wt% NaOH-treated kenaf composites

DMA results (Figure 4B) confirm that GNP incorporation significantly increases the storage modulus at all frequencies tested, with the 20 wt% kenaf composite exhibiting a 46% higher storage modulus than pure epoxy at 1 Hz (4100 MPa versus 2800 MPa). The frequency dependence of E' is similar across all composites, with a gradual decrease as frequency decreases reflecting the viscoelastic nature of the epoxy matrix. The glass transition temperature (Tg) extracted from tanδ peak temperature in the DMA thermogram (not shown) decreases slightly with kenaf incorporation (from 124°C for pure epoxy to 118°C for 20 wt% NaOH-treated), attributed to partial plasticisation of the epoxy matrix by residual moisture in the kenaf fibres inhibiting complete crosslinking at the interfacial region.

4. Discussion

The convergence of optimal mechanical, impact, and thermal properties at 20 wt% kenaf loading reflects two competing phenomena: below 20 wt%, the reinforcing effect of kenaf fibres on the epoxy matrix dominates, as the matrix is sufficient to fully wet and bond to all fibre surfaces, enabling efficient stress transfer. Above 20 wt%, fibre crowding in the hand layup process exceeds the matrix's wetting capacity, producing incompletely impregnated fibre bundles whose inter-fibre void spaces act as initiation sites for fracture under both flexural and impact loading. This threshold is processing-route-specific: vacuum infusion or resin transfer moulding, which apply hydrostatic pressure to force matrix into fibre networks, would be expected to shift the optimum to higher fibre loadings and should be explored in subsequent work.

A significant practical concern for exterior structural applications is the water-induced property degradation documented in Figure 1 and Table 2. Even the best-performing silane-treated 20 wt% composite retains 91% of dry flexural strength after 28-day immersion, representing a 9% knock-down factor that design engineers must account for in allowable stress calculations for humid environments. Accelerated weathering and UV exposure studies—not reported here—are essential before these composites can be specified for outdoor applications, and represent a priority for the next research phase. Protective surface coatings, UV-stabilised gel coats, or sandwich panel configurations with kenaf composite cores and glass-skin face sheets represent practical engineering strategies to mitigate these limitations.

From a life cycle perspective, replacement of 20 wt% E-glass with kenaf fibre in an equivalent structural panel application reduces the reinforcement-phase embodied energy from approximately 13 GJ/tonne (E-glass) to approximately 3.4 GJ/tonne (kenaf fibre, including retting and fibre extraction processing). At the composite level, accounting for the higher fibre volume fraction required to achieve equivalent structural performance due to kenaf's lower specific stiffness, the net embodied energy saving is approximately 35–40% per functional unit (panel of equivalent stiffness), making kenaf/epoxy a substantially more sustainable option than GFRP for applications where the moderate strength of natural fibre composites is structurally sufficient.

5. Conclusion

This study has systematically characterised the effect of alkali and silane surface treatments on the mechanical, hygroscopic, and thermomechanical performance of short kenaf fibre/epoxy composites across a wide range of fibre contents (5–30 wt%). The principal findings are as follows:

The optimum fibre loading is 20 wt% for the hand layup fabrication process used, beyond which fibre-crowding and incomplete matrix impregnation reduce mechanical performance. Silane treatment delivers the best combination of properties: flexural strength 89 MPa (112% improvement over pure epoxy), impact strength 66 kJ/m² (136% improvement), 28-day water absorption 1.06% (25% lower than untreated equivalent at same loading), and onset decomposition temperature 334°C. FTIR analysis confirms hemicellulose removal by NaOH treatment and covalent Si-O-C coupling by silane treatment. TGA confirms that NaOH and silane treatments raise the composite onset decomposition temperature toward the pure epoxy value by removing thermally labile hemicellulose. All composites retain adequate thermal stability for automotive and construction interior applications. Water absorption follows Fickian kinetics, and silane treatment reduces both equilibrium uptake and diffusivity, improving durability under humid service conditions. The kenaf/epoxy composite at 20 wt% silane-treated fibres represents a viable sustainable alternative to GFRP for interior structural panels, agricultural equipment housings, and automotive door liners in non-primary load-bearing applications.

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