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Impact of fibre surface treatment on the mechanical properties of waste mulberry silk/epoxy composites

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Natural fibre-reinforced composites are increasingly investigated as sustainable alternatives to synthetic materials; however, their hydrophilic nature and weak fibre–matrix adhesion often limit structural performance. This study explores the reinforcement potential of waste mulberry silk fabric, sourced from discarded textiles, in epoxy composites, with emphasis on the role of silane surface treatment. Raw and γ-aminopropyltriethoxysilane (APTES)-treated silk fabrics were incorporated into epoxy resin through a hand lay-up process at varying fibre weight fractions (11.13–33.38 wt%). Mechanical tests, including tensile, flexural, interlaminar shear strength (ILSS), impact strength, and hardness, were conducted in accordance with ASTM standards. Silane-treated composites consistently outperformed untreated counterparts, exhibiting nearly twofold improvements in tensile strength and modulus, ~20% higher flexural strength, ~35–45% higher ILSS, and substantially enhanced impact resistance. Hardness values and dimensional stability under moisture exposure were also superior in treated composites. SEM analysis confirmed that silane modification introduced surface roughness and improved interfacial bonding, leading to reduced fibre pull-out and enhanced stress transfer. The findings demonstrate that silane-treated waste mulberry silk fabrics are effective reinforcements for epoxy, enabling lightweight, high-strength, and moisture-resistant composites suitable for engineering and structural applications.

Keywords: Epoxy composites, Fibre–matrix adhesion, Silane treatment, Waste mulberry silk fibres

1 Introduction

The increasing global emphasis on sustainability and the depletion of petroleum-based resources have accelerated the development of natural fibre-reinforced polymer composites (NFRPCs). Compared to synthetic fibres such as glass, carbon, and aramid, natural fibres offer several advantages including low cost, light weight, biodegradability, renewability, and reduced environmental impact during production and disposal. As a result, they have gained significant attention in automotive, construction, aerospace, and consumer product applications[1-4].

Silk is one of the most remarkable natural fibres, known for its unique molecular configuration and outstanding mechanical performance. Mulberry silk, obtained predominantly from the *Bombyx mori* silkworm, is prized in the textile industry for its lustre, softness, and strength[5-10]. Nevertheless, large volumes of silk fabric are discarded after 5–10 years of use, leading to significant waste and environmental concerns. Repurposing this post-consumer mulberry silk as a reinforcement in polymer composites presents an innovative pathway for sustainable material development. This strategy not only reduces resource inefficiency but also enhances the value of textile waste while aligning with the circular economy framework[11]. Despite the inherent advantages of natural fibres, certain drawbacks limit their effective utilization in composite applications[12]. Natural fibres are hydrophilic in nature due to the presence of hydroxyl groups in cellulose, sericin, and other protein-based structures. This hydrophilicity causes poor compatibility with hydrophobic polymer matrices such as epoxy resin, resulting in weak interfacial adhesion, inefficient stress transfer, and reduced mechanical strength[5,10]. Additionally, moisture absorption by untreated fibres can cause dimensional instability and long-term degradation of composites[13,14].

To overcome these limitations, chemical surface modification techniques have been extensively studied[15,16]. Among them, silane coupling agents are particularly effective in improving fibre–matrix

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adhesion. Silane molecules form covalent bonds with hydroxyl groups on the fibre surface while simultaneously interacting with the polymer matrix, thereby creating a stable interfacial bridge. This dual functionality reduces fibre pull-out, enhances load transfer efficiency, and ultimately improves the overall mechanical performance of the composite. Previous studies have reported significant improvements in tensile, flexural, and impact properties of natural fibre composites following silane treatment, highlighting its importance in composite engineering[13, 17, 18].

Yang *et al.*[19] studied the toughening mechanisms of natural silkworm silks and their composites, reporting outstanding strength and fracture resistance due to silk's unique hierarchical structure. These findings highlight the suitability of silk fibres for structural applications, which aligns with the present research on developing waste mulberry silk–based composites for lightweight engineering components.

Chen *et al.*[20] fabricated poly(butylene succinate) bio-composites reinforced with waste silkworm silk fabric and observed notable improvements in mechanical and thermal properties, along with eco-friendly performance. Their findings further establish waste silk fabric as a valuable reinforcement material, which resonates with the present research aimed at utilizing waste mulberry silk fabrics in polymer composites for engineering applications.

Hamidi *et al.*[21] studied the processing and performance of silk/epoxy composite laminates. Their findings revealed that silk fibres substantially enhanced the tensile, flexural, and impact properties of epoxy composites, while also improving their fracture resistance. This research confirmed the effectiveness of silk as a natural reinforcement in polymer matrices.

Epoxy resin is widely used as a thermosetting polymer matrix in high-performance composites due to its excellent mechanical strength, chemical resistance, dimensional stability, and strong adhesion to reinforcements. When reinforced with suitably modified natural fibres, epoxy composites can combine performance with sustainability, offering a viable replacement for conventional synthetic fibre composites in several applications[22-24].

In this context, the present study investigates the role of fibre surface treatment on the mechanical properties of epoxy composites reinforced with waste mulberry silk fabrics. Raw and silane-treated silk fabrics were incorporated into epoxy resin, and the resulting composites were evaluated for tensile, flexural, and impact performance. The study aims to establish the effectiveness of silane surface treatment in enhancing interfacial bonding and mechanical properties, thereby demonstrating the potential of waste mulberry silk as a sustainable reinforcement for high-performance composite materials.

2 Methods and Materials

2.1 Materials

Poly (bisphenol A-co-epichlorohydrin) epoxy resin was procured from Sanghvi Chemicals, India, and used as the primary matrix material. The curing agent employed was diethylenetriamine (DETA; Sigma Aldrich, USA), mixed with the resin in a weight ratio of 10:6. The mixture was thoroughly stirred until slight exothermic warming was observed, ensuring homogeneity. Waste mulberry silk fabric, sourced from an old silk saree, was used as the reinforcement. To enhance interfacial adhesion between fibre and matrix, γ-aminopropyltriethoxysilane (APTES; Sigma Aldrich, USA) was used as a surface coupling agent. Ethanol of absolute grade (Merck, India) served as the solvent during silane treatment and fabric preparation.

2.2 Silane Treatment

Waste mulberry silk fabric was subjected to surface modification using γ-aminopropyltriethoxysilane (APTES; Silquest A-1100) to enhance interfacial adhesion with the epoxy matrix. Prior to treatment, the fabric was cleaned with a mild non-ionic detergent (0.5 wt%) in deionized water, rinsed thoroughly, and oven-dried at 60 °C to remove impurities. The silane solution was prepared by mixing ethanol and deionized water in a 95:5 (v/v) ratio, adjusting the pH to 4.5–5.5 with glacial acetic acid as shown in Fig. 1, and adding APTES under stirring to achieve a final

Fig. 1 — pH Meter Used for Adjusting the pH of Silane Solution

concentration of 2 vol%. The silk fabric layers were immersed in the hydrolysed silane bath for 5 min with gentle agitation to promote uniform adsorption, withdrawn, and briefly rinsed in ethanol to remove loosely bound species. Curing of the deposited silane layer was carried out either at 60 °C for 20 min or at ambient temperature (≈60% relative humidity) for 24 h, enabling condensation of silanol groups into siloxane networks and covalent bonding with hydroxyl functionalities on the silk surface. This treatment rendered the silk fabric more chemically compatible with epoxy by introducing reactive amino groups, thereby reducing moisture uptake, improving fibre–matrix interfacial bonding, and mitigating the risk of delamination in the resulting composites.

2.3 Fabrication of Composite

The composite laminates were fabricated using the conventional hand lay-up technique. Epoxy resin and diethylenetriamine (DETA) hardener, mixed in a weight ratio of 10:6, were thoroughly stirred until a homogeneous blend was obtained. Silane-treated waste mulberry silk fabrics were used as reinforcement to enhance fibre–matrix adhesion. The fabrics were sequentially placed in the mould, and the resin mixture was carefully applied to ensure uniform wetting and impregnation of fibres.

Laminates were prepared with varying reinforcement levels by stacking 4, 6, 8, 10, and 12 fabric layers[25], corresponding to silk weight fractions of 11.13%, 16.69%, 22.26%, 27.82%, and 33.38%, respectively. The lay-ups were consolidated under moderate pressure to expel entrapped air and left to cure under ambient conditions for 24 h. post-curing was carried out at 60 °C for 3 h to achieve complete crosslinking of the matrix and final laminate as shown in Fig. 2 (a) silane treated waste mulberry silk laminate and Fig. 2 (b) raw waste mulberry silk laminate.

The cured laminates were cut into test specimens using a CNC water-jet cutting machine to maintain dimensional accuracy and avoid thermal degradation. Samples were prepared according to standard ASTM specifications: ASTM D638 for tensile testing, ASTM D790 for flexural testing, ASTM D2344 for interlaminar shear strength (ILSS), and ASTM D256 for impact testing.

2.4 Preparation and Evaluation of Composite Test Samples

2.4.1 Tensile Test

The tensile specimens were prepared according to the dimensions specified in ASTM D638. Each specimen

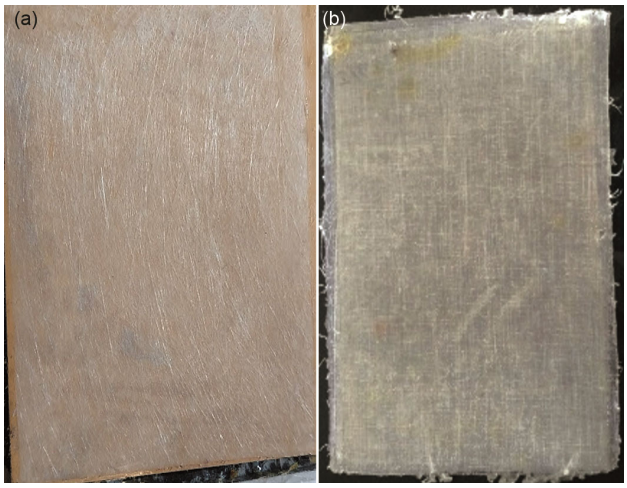

Fig. 2 — (a) Composite Sheet Using Silane Treated Waste Mulberry Silk Fabric, and (b) Composite Sheet Using Raw Waste Mulberry Silk Fabric

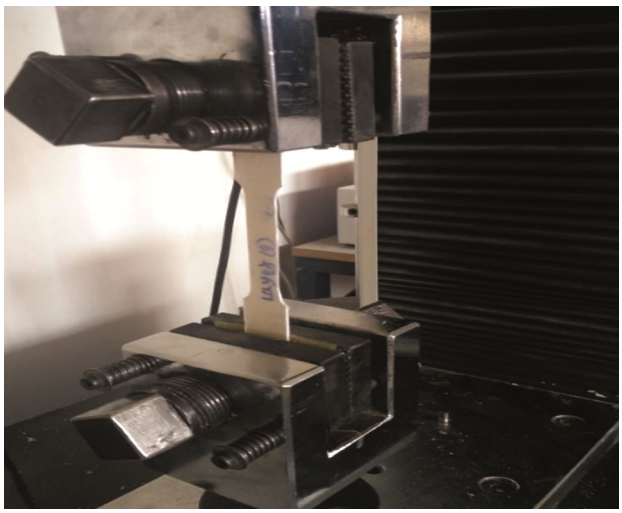

Fig. 3 — Schematic Representation of Tensile Test Specimen and Loading Arrangement (ASTM D638)

was clamped vertically in the grips of the Universal Testing Machine (UTM) as illustrated in Fig. 3. The grips were tightened uniformly to prevent slippage during testing. The crosshead speed was maintained at 0.2 in/min**.** As the specimen elongated, the applied load was continuously monitored through a load cell, and elongation was recorded until specimen failure. Both the maximum load at break and elongation at rupture were documented for further analysis.

2.4.2 Flexural Characteristics

Flexural strength evaluation was carried out using the same UTM setup in accordance with ASTM D618. Test specimens were prepared as rectangular bars with dimensions 1/8 × ½ × 4 in. The load was

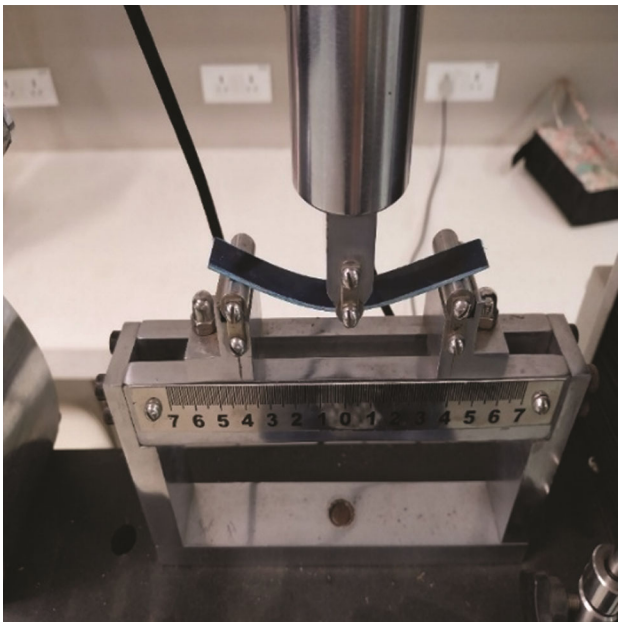

Fig. 4 — Three-Point Bending Setup for Flexural Testing (ASTM D618)

applied centrally at a constant crosshead speed as mentioned in figure 4 and deflection was measured either by a displacement gauge placed beneath the specimen or by tracking the movement of the loading nose relative to the supports. The flexural stress (σ_f) was calculated using:

$$\sigma_f = \frac{3PL}{2bd^2} \quad \ldots (1)$$

where P is the applied load, L is the support span, b is the specimen width, and d is the specimen thickness. The maximum stress in the outer fibres at fracture was reported as the flexural strength.

2.4.3 Interlaminar Shear Strength (ILSS) Test

The interlaminar shear strength of the composites was determined using the short-beam shear test as per ASTM D2344 as shown in Fig. 5. Rectangular specimens were loaded under three-point bending with a small span-to-thickness ratio to promote interlaminar shear failure.

The ILSS was calculated using:

$$\tau = \frac{0.75P}{bd} \quad \ldots (2)$$

where P is the maximum load applied, b is the specimen width, and d is the specimen thickness. This test provided insights into the fibre–matrix adhesion and interlaminar bonding strength of the hybrid laminates.

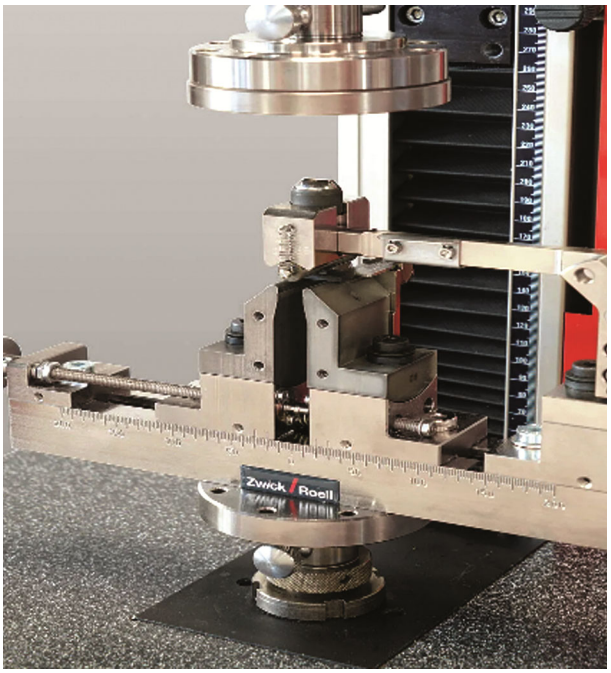

Fig. 5 — Short-Beam Shear Test Setup for ILSS Evaluation (ASTM D2344)

Fig. 6 — Izod Impact Test Configuration (ASTM D256)

2.4.4 Impact Test

The izod impact strength was measured following ASTM D256. Rectangular specimens of size 2.5 × 0.5 × 0.125 in were either directly moulded or cut from composite laminates. Each specimen was mounted horizontally on supports with the notch facing towards the striking pendulum as illustrated in figure 6. The pendulum was released from its locked position, striking the specimen until fracture. The

absorbed impact energy was displayed by the equipment and normalized with respect to specimen thickness to obtain impact strength (kJ/m²). Only specimens exhibiting complete fracture were considered valid[26, 27].

2.4.5 Scanning Electron Microscopy

SEM characterization was performed on cleaned single fabric specimens and fractured composite surfaces. Samples were ultrasonically cleaned in ethanol: water (1:1) for 10 min, rinsed in DI water and dried at 60 °C. Fracture surfaces were produced by mechanical testing. Specimens were mounted on aluminium stubs with conductive carbon tape and sputter coated with Au ~3 nm. Imaging was carried out on a quanta 200 model using the secondary electron detector at 15 kV and a working distance of ~12 mm. Images were acquired at multiple magnifications ×300, ×500, ×1,500 and ≥5 random fields per sample.

3 Results and Discussion

From an economic and industrial standpoint, the present study indicates promising potential for future large-scale implementation. The reinforcement material is obtained from post-consumer mulberry silk waste, sourced from discarded silk textiles and garments, which can significantly reduce raw material costs compared to virgin natural or synthetic fibres. Although the market for waste silk fabric is currently unorganized, the establishment of a structured collection and supply chain in the future could substantially facilitate large-scale utilization of this resource. The silane surface modification process employed in this work is straightforward, requires a low concentration of coupling agent, and is performed under mild processing conditions, leading to low energy demand and minimal additional processing cost. Moreover, both the surface treatment methodology and composite fabrication routes are compatible with conventional textile finishing and polymer composite manufacturing techniques, such as hand lay-up and compression moulding. Collectively, these factors demonstrate that silane-treated waste mulberry silk/epoxy composites are not only technically effective but also economically feasible for potential commercialization.

3.1 Tensile Characteristics

The tensile characteristics of raw and silane-treated waste mulberry silk/epoxy composites are presented in Fig. 7. A clear influence of fibre surface modification and fibre loading on the mechanical behaviour of the composites is observed. The untreated composites exhibited a gradual increase in elongation illustrated in figure 7(a) with increasing fibre content, indicating limited load transfer and higher fibre slippage at the fibre–matrix interface. In contrast, the silane-treated composites displayed a slightly lower elongation at lower fibre loadings, which progressively decreased

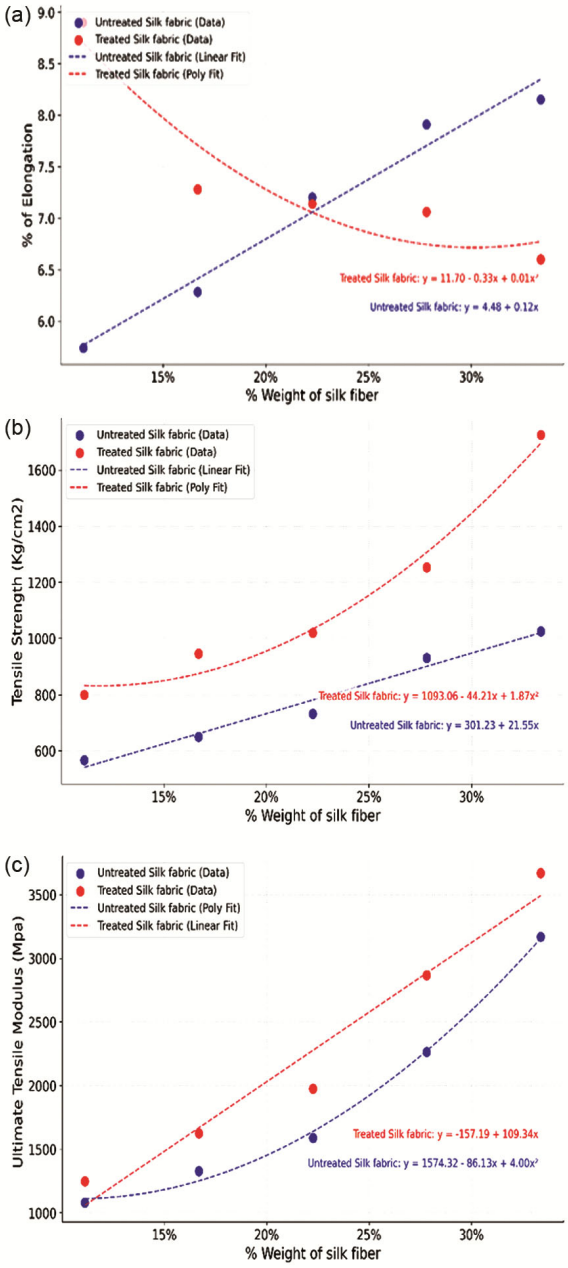

Fig. 7 — Effect of Silk Fibre Weight Fraction on (a) Elongation, (b) Tensile Strength, and (c) Tensile Modulus of Treated and Raw Silk Fabric–Epoxy Composites

with higher reinforcement levels. This behaviour suggests that improved interfacial adhesion due to silane treatment restricted fibre pull-out and deformation, resulting in stiffer composites with lower ductility. A significant enhancement in tensile strength, Fig. 7 (b) was observed for silane-treated composites across all fibre loadings. At 30 wt% fibre content, the tensile strength of treated composites reached values nearly double that of untreated composites, highlighting the effectiveness of silane coupling in enhancing stress transfer between the silk fibres and epoxy matrix. The untreated composites, although showing a marginal increase with fibre loading, suffered from weak bonding and interfacial defects, which limited their strength improvement.

The tensile modulus, Fig. 7 (c) exhibited a linear rise with increasing fibre content for both treated and untreated systems. However, the treated composites consistently displayed higher modulus values, with the gap widening at higher fibre loadings. At 30 wt% fibre content, the modulus of silane-treated composites was more than twice that of untreated composites, indicating that surface modification enabled the fibres to carry and distribute applied loads more efficiently.

The results confirm that chemical surface modification is crucial for optimizing the mechanical performance of natural fibre-reinforced epoxy composites.

3.2 Flexural Characteristics

The flexural modulus illustrated in Fig. 8 (a) of both untreated and silane-treated waste mulberry silk/epoxy composites increased linearly with fibre content, indicating that higher fibre reinforcement enhances stiffness. However, silane-treated composites consistently exhibited higher modulus values across all fibre loadings. The difference between the two systems widened at higher reinforcement levels, with treated composites showing an improvement of approximately 10–15% compared to untreated ones. This enhancement can be attributed to the stronger fibre–matrix interfacial adhesion imparted by silane treatment, which enabled more efficient stress transfer and reduced fibre pull-out under flexural loading. A similar trend was observed in Fig. 8 (b) for flexural strength. Both untreated and treated composites showed progressive improvements with increasing fibre weight percentage, but the silane-treated composites consistently outperformed the untreated group. At 33 wt% fibre loading, the flexural strength of treated composites was nearly 18–20% higher than

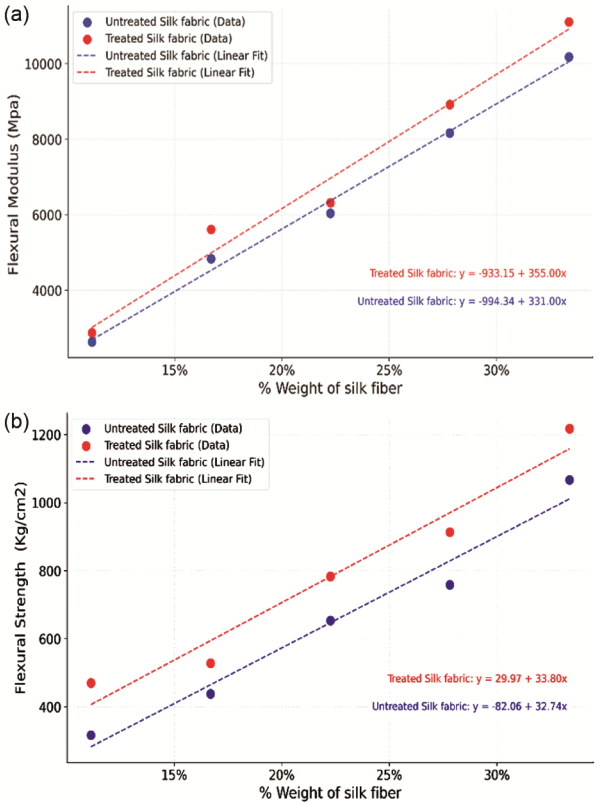

Fig. 8 — Influence of fibre treatment on (a) flexural modulus, and (b) flexural strength

untreated composites, underscoring the effectiveness of surface modification in improving load-carrying capacity. The improved interfacial bonding not only delayed crack initiation but also enhanced resistance to crack propagation during bending.

The results confirm that silane-modified waste mulberry silk fibres are effective in reinforcing epoxy matrices, producing composites with higher stiffness and strength under flexural loading.

3.4 Interlaminar Shear Strength

The interlaminar shear strength (ILSS) of both untreated and silane-treated silk–epoxy composites exhibited distinct trends with varying fibre weight fractions shows in figure 9. A steady increase in ILSS was observed with fibre loading up to approximately 22–25 wt%, beyond which the untreated silk composites showed a decline. This behaviour may be attributed to poor stress transfer efficiency and fibre–matrix debonding at higher fibre concentrations in the untreated system. In contrast, the treated silk composites demonstrated a continuous enhancement in ILSS, reaching a peak value of ~1600 kg/cm² at 33.38 wt% fibre loading.

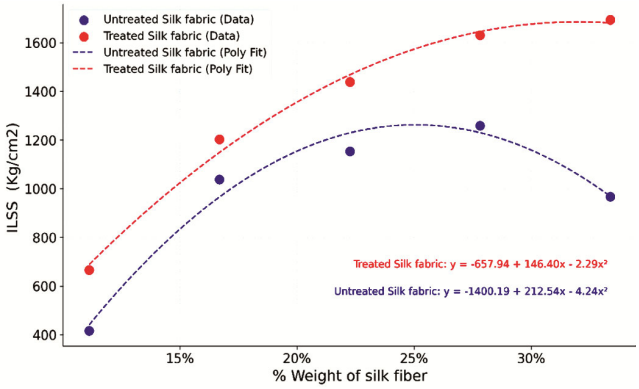

Fig. 9 — Effect of fibre loading on ILSS of treated and untreated silk fabric–epoxy composites

The polynomial regression fits confirm these observations, with the untreated composites following a quadratic trend (ILSS = 1400.19 + 212.54x – 4.24x², $R^2 > 0.95$), where the negative quadratic term explains the downturn at higher fibre content. Conversely, the treated composites fit a quadratic relationship with a positive reinforcement effect (ILSS = 657.94 + 146.40x – 2.29x², $R^2 > 0.97$), showing sustained improvement with increasing fibre weight.

The superior performance of treated silk composites can be ascribed to improved interfacial adhesion due to silane treatment, which enhances fibre surface roughness, increases wettability, and promotes mechanical interlocking with the epoxy matrix. This modification reduces interfacial voids and suppresses crack initiation along the fibre–matrix boundary, thereby delaying shear failure.

Kirmasha *et al.*[28] examined unstitched silk fibre–stitched woven kenaf/epoxy composites and found that silk stitching considerably improved interlaminar shear strength and fracture resistance.

3.4 Impact Strength

The Izod impact strength of both untreated and silane-treated silk–epoxy composites showed a clear dependence on fibre weight fraction in Fig. 10. For untreated composites, impact strength exhibited a gradual rise with increasing silk fibre loading, following a polynomial trend (Impact strength = 74.91 – 8.39x + 0.46x², $R^2 > 0.95$). This non-linear increase suggests that while additional fibre content enhances energy absorption capacity through crack deflection and fibre pull-out, excessive loading may cause localized stress concentrations and incomplete wetting at lower fibre–matrix compatibility levels.

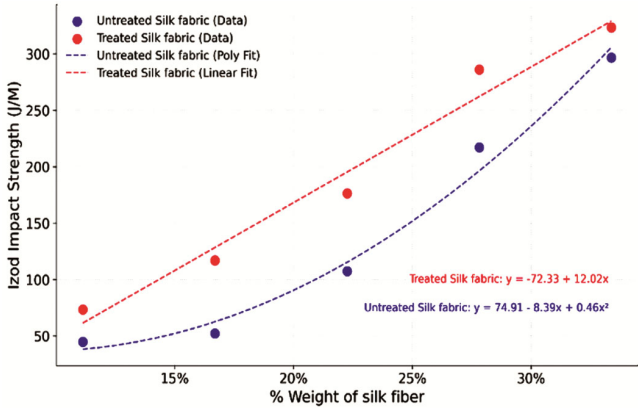

Fig. 10 — Impact strength of treated and untreated silk fabric composites as a function of fibre content

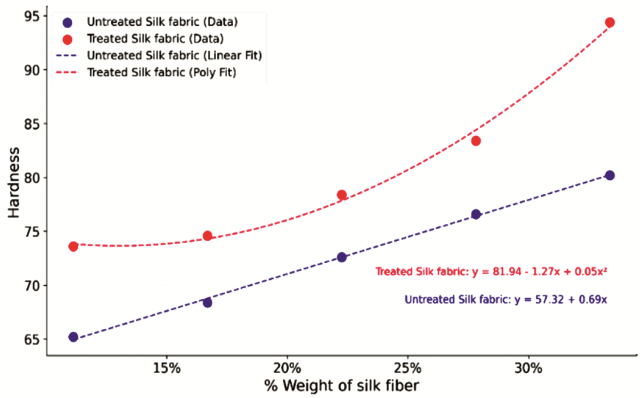

Fig. 11— Hardness behaviour of treated and untreated silk fabric composites at different fibre loadings

In contrast, treated silk composites displayed a nearly linear increase in impact resistance with fibre loading (Impact strength = –72.33 + 12.02x, $R^2 > 0.97$), achieving a maximum of ~320 J/m at 33.38 wt% fibre. The enhanced performance of treated fibres can be attributed to improved interfacial adhesion, which facilitates efficient stress transfer and dissipates impact energy through fibre breakage and matrix yielding, rather than premature debonding. The treatment-induced surface roughness and increased wettability also minimize interfacial voids, thereby enhancing the composite's ability to resist sudden fracture.

3.5 Hardness

The hardness behaviour of untreated and silane-treated silk–epoxy composites as a function of fibre loading is shown in Figure 11. For untreated silk composites, hardness increased linearly with fibre content (Hardness = 57.32 + 0.69x, $R^2 > 0.96$), rising from ~66 at 13.89 wt% to ~82 at 33.38 wt% fibre.

This gradual improvement reflects the inherent contribution of silk fibres to restricting localized plastic deformation within the epoxy matrix, despite limited fibre–matrix bonding.

In comparison, treated silk composites exhibited a more pronounced increase in hardness, best described by a quadratic relationship (Hardness = 81.94 – 1.27x + 0.05x², $R^2 > 0.97$). Unlike untreated composites, treated samples maintained a consistently higher hardness across all fibre loadings, reaching a maximum of ~94 at 33.38 wt%. The enhanced performance is primarily attributed to improved interfacial adhesion induced by silane treatment, which enhances stress transfer efficiency and reduces matrix-rich regions susceptible to indentation. Furthermore, the roughened fibre surface and increased wettability due to treatment promote better epoxy infiltration, effectively constraining polymer chain mobility under applied load.

3.6 Water Absorption

The variation in water absorption of untreated and silane-treated silk–epoxy composites with increasing fibre loading is presented in Figure 12. For untreated silk composites, water uptake followed a polynomial trend (WA = –0.80 + 0.20x – 0.00x², $R^2 > 0.97$), increasing sharply up to ~2.0% at 27.77 wt% fibre before exhibiting a slight decline at higher loadings. This behaviour can be attributed to the hygroscopic nature of silk fibres, where higher fibre contents provide more hydroxyl groups capable of hydrogen bonding with water molecules. However, at very high loadings, fibre–fibre contact zones limit the diffusion pathways, marginally reducing the water uptake.

In contrast, treated silk composites exhibited significantly lower water absorption across all fibre loadings, with a nearly linear increase (WA = 0.53 + 0.03x, $R^2 > 0.95$). Maximum water absorption was only ~1.5% at 33.38 wt% fibre, which is considerably lower than the untreated counterpart. The reduced uptake is primarily attributed to silane treatment, which removes hemicellulose, waxes, and amorphous components from silk fibres, thereby decreasing the number of hydrophilic sites available for moisture interaction. Furthermore, improved interfacial bonding between treated fibres and epoxy restricts the formation of interfacial voids and capillaries, effectively lowering moisture ingress.

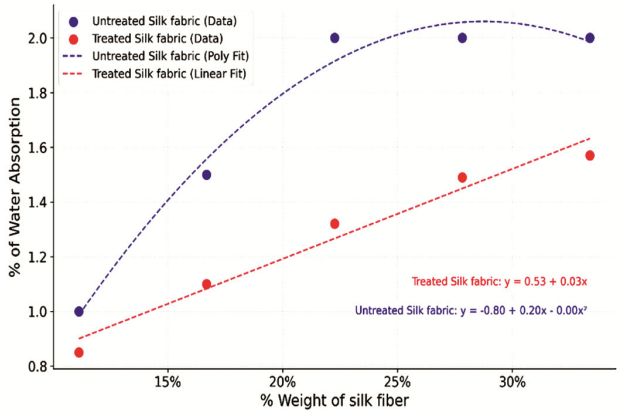

Fig. 12 — Variation of water absorption with silk fibre weight fraction for treated and untreated silk fabric–epoxy composites

The results clearly establish that silane treatment enhances the dimensional stability of silk–epoxy composites by mitigating water absorption. This improvement is crucial for structural and aerospace applications, where moisture-induced degradation can adversely affect mechanical reliability and long-term performance[29, 30].

3.7 Scanning Electron Microscopy

3.7.1 SEM for Waste Mulberry Silk Fabric

The surface morphologies of untreated and silane-treated waste mulberry silk fibres were examined using SEM at 1500× magnification and 15.0 kV accelerating voltage, as shown in Figure 13 (a) (untreated) and Figure 13 (b) (silane-treated).

The SEM micrograph of untreated waste silk fabric, figure (a) exhibits a relatively smooth and featureless surface with minimal irregularities. The absence of surface roughness or functional groups indicates limited surface reactivity and poor wettability when incorporated into a polymer matrix. Such smooth surfaces reduce the available interfacial area for bonding, thereby restricting mechanical interlocking and fibre–matrix adhesion. As a result, composites prepared with untreated fabrics are prone to fibre pull-out, weak interfacial shear strength, and reduced load transfer efficiency.

In contrast, the silane-treated fabric surface, figure (b) displays increased surface roughness and deposition of irregular patches, consistent with the formation of a silane coating layer. The morphological changes are attributed to the hydrolysis and condensation of silane coupling agents, which graft reactive silanol groups onto the silk fibroin surface. These groups subsequently form covalent bonds with the epoxy resin during curing, thereby enhancing chemical compatibility between the hydrophilic fibre and hydrophobic matrix. The

presence of a roughened topography also contributes to improved mechanical interlocking at the fibre–matrix interface. Such features are expected to reduce fibre pull-out and debonding, thereby increasing the tensile, flexural, and interlaminar shear strengths of the composites.

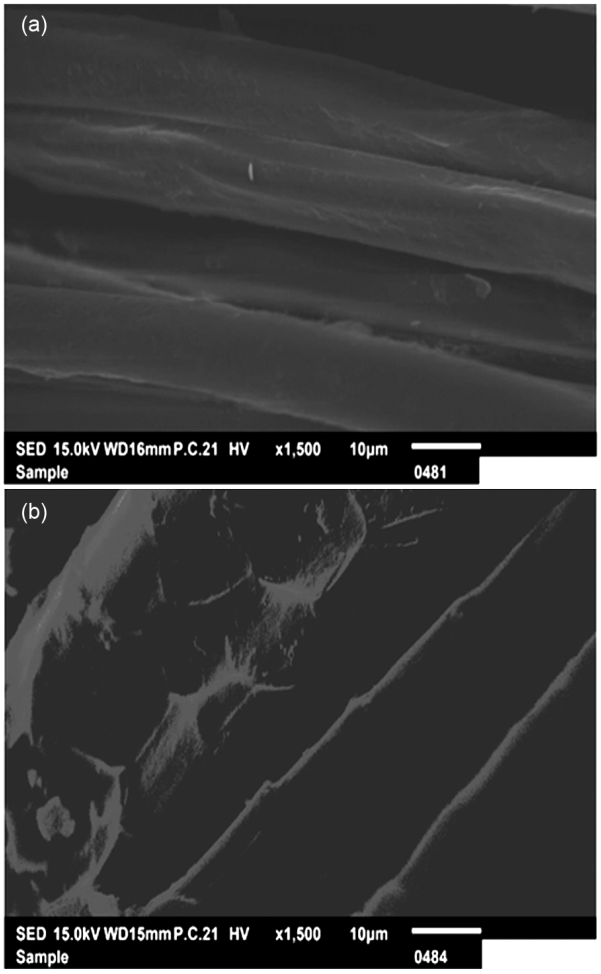

Fig. 13 — Sem Micrograph of Waste Mulberry Silk Fabric (a) Untreated Silk Fabric, and (b) Silane Treated Silk Fabric

3.7.2 SEM for Silk/Epoxy Composite (Untreated and Treated)

The fractured morphologies of silane-treated and untreated waste mulberry silk/epoxy composites were investigated by scanning electron microscopy (SEM), as presented in Fig. 14 (a–c) (untreated) and Fig.14 (d–f) (silane-treated). Distinct differences in fibre–matrix interfacial characteristics were observed, demonstrating the crucial role of silane surface modification in tailoring adhesion and fracture behaviour.

By contrast, untreated composites demonstrate weak fibre–matrix bonding. In Figure a, extensive fibre pull-out is observed, leaving behind clean fibre surfaces devoid of matrix residues. This indicates poor interfacial adhesion, causing premature debonding under applied load.

Figure b highlights longitudinal fibre regions with resin-rich zones and voids, which act as stress concentrators and facilitate crack initiation. The smooth fracture surface suggests that the fibres slipped out of the matrix with minimal energy absorption. In Figure c, large interfacial gaps and delaminated regions are visible, confirming the absence of chemical anchoring between untreated fibres and epoxy. The dominance of fibre pull-out as the primary failure mechanism limits the efficiency of stress transfer, resulting in reduced tensile, flexural, and ILSS values for untreated composites.

The SEM micrographs of silane-treated composites reveal enhanced fibre–matrix adhesion. In Figure (d), the fibres are seen embedded firmly within the epoxy matrix, with resin layers adhering to the fibre surfaces. This indicates strong interfacial bonding, which restricts fibre pull-out and improves stress transfer efficiency. Figure (e) shows fibre surfaces covered with epoxy residues, suggesting chemical coupling between the silane-treated silk fibroin and the epoxy resin. The fractured surfaces exhibit

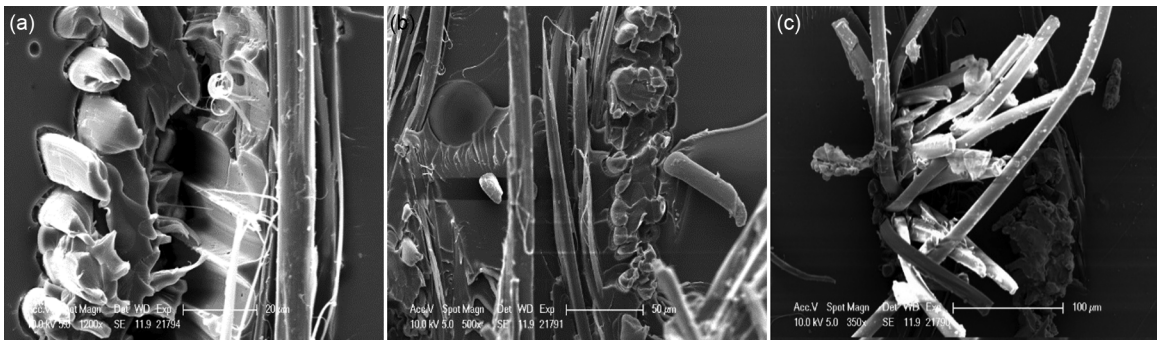

Fig. 14 — Factrography of Silane-Treated Waste Mulberry Silk/Epoxy Composites (d) Fibre Reduced Interfacial Gap, (e) Strong Matrix Adhesion to Fibre Surfaces, and (f) Rough Fracture and Fibre Breakage

irregular resin topographies, further supporting the improved compatibility between the fibre and the matrix. In Figure (f), broken fibre ends surrounded by resin fragments are evident, pointing to a mixed mode of failure involving both fibre fracture and cohesive resin cracking. These morphological features reflect efficient load sharing and higher energy absorption during fracture, which directly explain the improved tensile, flexural, and interlaminar shear strengths of silane-treated composites.

The microstructural differences between treated and untreated composites are consistent with their mechanical performance. Silane treatment enhances fibre wettability and introduces covalent linkages at the fibre–matrix interface, leading to improved load transfer and reduced interfacial sliding. The treated composites exhibit rough, irregular fracture morphologies with fibre breakage and resin adherence, indicating ductile-like failure with higher toughness. Conversely, untreated composites show smooth fracture surfaces, interfacial voids, and fibre pull-out, indicative of brittle failure and poor stress dissipation. These observations confirm that surface treatment is essential to exploit the reinforcing potential of waste mulberry silk in polymer composites, thereby enabling their application in structural and engineering fields[4, 31].

4 Conclusion

This study established the feasibility of repurposing waste mulberry silk fabric as a sustainable reinforcement for epoxy composites and highlighted the importance of fibre surface modification in enhancing performance. Key conclusions are:

1. Effect of silane treatment: Surface modification with APTES significantly improved fibre–matrix adhesion, as confirmed by SEM analysis, resulting in superior load transfer and reduced interfacial defects.
2. Mechanical enhancements: Treated composites demonstrated marked improvements in tensile, flexural, interlaminar shear, impact, and hardness properties compared to untreated composites. The effect was more pronounced at higher fibre loadings, where interfacial adhesion becomes critical.
3. Moisture resistance: Silane treatment effectively reduced water absorption, thereby enhancing the dimensional stability and long-term durability of the composites.
4. Sustainability perspective: The use of discarded silk textiles aligns with circular economy principles by valorizing post-consumer waste into high-performance composite materials.

This study demonstrates the feasibility of using waste mulberry silk fabric as a cost-effective and sustainable reinforcement for epoxy composites and highlights the effectiveness of silane surface treatment in improving fibre–matrix interfacial adhesion. Silane-treated composites showed substantial enhancements in mechanical performance and moisture resistance compared to untreated systems. The use of post-consumer silk waste reduces material costs, while the low-energy, scalable surface treatment is compatible with conventional composite manufacturing methods. Although the work is limited to laboratory-scale investigation, the processing simplicity and material availability indicate strong potential for future industrial-scale applications.

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