

Moisture Ageing Behavior of Flax-Epoxy Composites Modified with Silane-Functionalized Graphene Oxide

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1. Abstract

Natural fiber-reinforced composites are attractive sustainable alternatives to synthetic composites but remain susceptible to moisture-induced degradation. This study investigates the influence of silane-functionalized graphene oxide (sGO) treatment and fiber architecture on the moisture ageing behaviour of flax–epoxy composites. Flax fibers were coated with sGO and fabricated into unidirectional (UD) and woven composites. Water absorption, yarn behaviour, and tensile performance under dry and wet conditions were evaluated. Results show that woven composites exhibited greater resistance to moisture-induced strength loss, while sGO-treated UD composites demonstrated improved mechanical property retention after ageing. These findings suggest that sGO enhances moisture durability primarily through improved interfacial stability rather than reduced water uptake.

2. Introduction

The increasing demand for sustainable engineering materials has accelerated interest in replacing conventional synthetic reinforcements, such as glass fibers, with renewable natural fibers. Among these, flax fiber has attracted considerable attention due to its low density, renewability, biodegradability, and relatively high mechanical properties, making it a promising reinforcement for lightweight and environmentally friendly composites. However, the hydrophilic nature of lignocellulosic fibers remains a major limitation restricting their use in structural applications.

Flax fibers contain cellulose, hemicellulose, and pectin, which introduce abundant hydroxyl groups capable of forming hydrogen bonds with water molecules. Consequently, flax fiber composites readily absorb moisture during humid exposure or water immersion, resulting in dimensional instability, matrix plasticization, and degradation of the fiber–matrix interface [1], [2]. Swelling mismatch between hydrophilic fibers and relatively hydrophobic polymer matrices can further generate internal stresses that promote interfacial debonding and microcrack formation.

Numerous studies have demonstrated the detrimental effects of moisture on flax–epoxy composites. Habibi et al. [3] reported significant reductions in tensile properties of nonwoven flax/epoxy composites after water immersion, particularly at elevated temperatures, while Huner [4] observed progressive decreases in tensile and flexural performance with increasing moisture exposure. However, moderate water uptake may also temporarily improve fiber–matrix contact through fiber swelling. Muñoz et al. [5] reported increased tensile strength but reduced tensile modulus in water-aged flax bio-epoxy composites, suggesting competing mechanisms involving temporary interfacial stabilization and long-term degradation. Because the fiber–matrix interface governs stress transfer, various surface modification strategies have been explored to improve moisture durability. Chemical treatments such as alkalization, acetylation, and silane coupling can reduce fiber hydrophilicity and improve fiber–matrix compatibility [6]. Among these approaches, silane coupling agents are particularly attractive because they react with hydroxyl groups on the fiber surface while simultaneously forming covalent linkages with polymer matrices, thereby improving interfacial bonding and resistance to moisture-induced degradation [7], [8].

Graphene-based nanomaterials have recently emerged as an alternative strategy for enhancing both mechanical performance and environmental durability. Graphene oxide (GO) possesses high surface area and reactive oxygen-containing functional groups, but poor dispersion and excessive hydrophilicity often limit its reinforcing efficiency. To address these limitations, silane-functionalized graphene oxide (sGO) has been developed by grafting organosilane molecules onto GO surfaces, thereby improving compatibility with polymer systems and enhancing interfacial bonding [9]. Graphene-based materials have also shown promise in improving moisture resistance in flax composites through reduced water uptake and improved mechanical retention after ageing [10], [11]. However, most studies have focused on graphene as a matrix additive rather than a fiber surface modifier. Consequently, the role of sGO coatings applied directly onto flax fibers, particularly in improving the hydrolytic stability of the fiber–matrix interface under moisture exposure, remains largely unexplored.

In previous work, the authors demonstrated that sGO surface treatment significantly improved the interfacial and mechanical performance of flax–epoxy composites under dry conditions through enhanced fiber–matrix bonding and load transfer [12]. However, it remains unclear whether the improved interphase stability provided by sGO can be maintained under moisture exposure. Therefore, this study investigates the moisture ageing behaviour of sGO-modified flax–epoxy composites by evaluating water absorption characteristics and mechanical property retention after immersion. It is hypothesized that sGO improves the hydrolytic stability of the fiber–matrix interface, thereby mitigating moisture-induced degradation and enhancing mechanical performance retention under wet conditions.

3. Materials and methods

3.1. Composite Fabrication and Experimental Design

Based on previously optimized sGO treatment conditions, flax fiber sheets were surface-treated and fabricated into flax–epoxy composites with UD and woven architectures. Three fiber treatment conditions were investigated: water treated control, 0.2 wt.% sGO for 2 h, and 1 wt.% sGO for 30

min. To isolate the effect of sGO treatment from moisture exposure during dip coating, control fibers were immersed in DI water for 2 h and dried under identical conditions. Six composite groups were fabricated: control woven, 0.2 wt.% woven, 1 wt.% woven, control UD, 0.2 wt.% UD, and 1 wt.% UD. Four laminate plates were manufactured for each group. From each plate, eight tensile coupons and six interlaminar shear strength (ILSS) specimens were prepared, yielding 32 tensile and 24 ILSS specimens per group. Specimens were assigned to dry, 24 h, 7 days, and saturated conditioning states. Moisture saturation duration was determined from water absorption analysis.

3.2. Moisture Conditioning and Water Absorption Analysis

Water absorption behaviour was evaluated according to ASTM D5229. Dry specimen mass (W_0) was recorded before immersion in deionized water at 23 °C. Specimens were removed every 24 h, surface-wiped, and weighed to determine moisture uptake:

$$M_t(\%) = \frac{W_t - W_0}{W_0} \times 100 \quad (1)$$

where W_t is the specimen mass at time t , and W_0 is the initial dry mass.

Immersion continued until equilibrium moisture content (M_E) was reached and subsequently used to define saturated conditioning. Moisture uptake was analyzed as a function of time and $\sqrt{t}$, and diffusion coefficients (D) were calculated using a Fickian plate diffusion model:

$$D = \frac{\pi}{16} \left(\frac{kh}{M_E} \right)^2 \quad (2)$$

where k is the slope of the initial linear region, h is specimen thickness, and M_E is the equilibrium moisture content.

3.3. Yarn Tensile Testing

Flax yarn tensile properties were measured according to ASTM D2256 using an Instron 5969 universal testing machine (50 N load cell, 250 mm gauge length, 25 mm/min). Yarns were extracted from UD flax mats corresponding to the three treatment conditions. For dry testing, control yarns underwent identical water immersion and drying procedures as treated fibers. Wet yarns were immersed in water for 24 h and tested immediately after removal. Twenty specimens were tested per condition. Tensile performance was reported as tenacity (cN/tex), and modulus was determined from the tenacity–strain response.

3.4. Mechanical Testing of Water-Aged Composites

Tensile properties were measured according to ASTM D3039 for dry, 24 h, 7 days, and saturated specimens. Following conditioning, specimens were lightly surface-dried and immediately tested in the wet state. Tensile strength, modulus, and strain at failure were evaluated and compared with

dry-state controls. ILSS was measured using ASTM D2344 under the same conditioning states. For UD composites, additional transverse-direction testing was conducted to improve sensitivity to matrix- and interface-dominated behaviour. Wet specimens were tested immediately after ageing to minimize moisture loss.

4. Results and Discussions

4.1. Moisture Absorption Behaviour of sGO-Modified Flax-Epoxy Composites

The moisture absorption behaviour of flax-epoxy composites with different sGO treatments is shown in Fig. 1, and the corresponding diffusion coefficients are summarized in Table 1. All groups exhibited similar moisture uptake profiles, characterized by rapid absorption during the first 7–10 days followed by a gradual approach to equilibrium after approximately 21–28 days, consistent with diffusion-controlled transport in natural fiber composites.

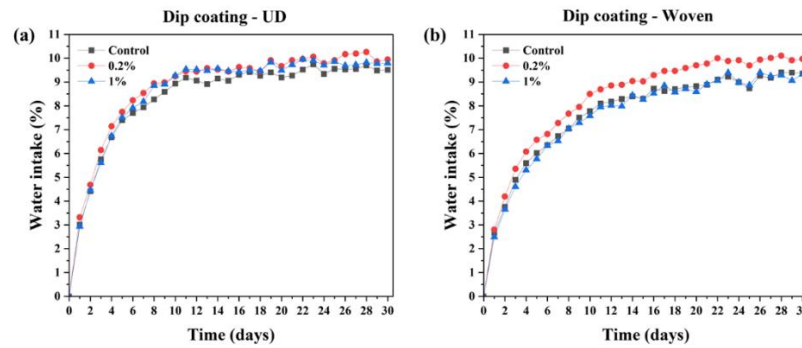


Figure 1. Moisture absorption behaviour of flax-epoxy composites with different sGO treatments during water immersion at room temperature: (a) UD composites and (b) woven composites.

For UD composites (Fig. 1a), equilibrium moisture contents (M_E) ranged from 9.55–10.02 wt.%, with the 0.2 wt.% sGO-treated group exhibiting slightly higher uptake than the control and 1 wt.% sGO groups. A similar trend was observed for woven composites (Fig. 1b), where the 0.2 wt.% treatment again showed the highest moisture uptake (9.94 wt.%), while the control and 1 wt.% groups exhibited the same equilibrium moisture contents of 9.18 wt.%. Diffusion coefficients indicated minimal influence of sGO treatment on moisture transport kinetics. UD composites exhibited similar diffusion coefficients across all conditions (9.75×10^{-7} – 9.93×10^{-7} mm²/s), whereas woven composites showed slightly higher values (1.09×10^{-6} – 1.20×10^{-6} mm²/s), suggesting faster moisture transport. This behaviour may be attributed to woven architecture, such as inter-yarn gaps and resin-rich regions, which facilitate moisture penetration.

Table 1. Equilibrium moisture content (M_E) and diffusion coefficient (D) of flax-epoxy composites with different sGO treatments and fiber architectures.

Treatment	UD		Woven	
	M_E (wt.%)	D (mm ² /s)	M_E (wt.%)	D (mm ² /s)
Control	9.55	9.77×10^{-7}	9.18	1.2×10^{-6}
0.2% treatment	10.02	9.75×10^{-7}	9.94	1.09×10^{-6}
1% treatment	9.80	9.93×10^{-7}	9.18	1.15×10^{-6}

Interestingly, sGO treatment did not substantially reduce moisture uptake or diffusion behaviour in either architecture, indicating that sGO does not primarily act as a moisture barrier. Instead, its contribution may lie in stabilizing the fiber–matrix interphase through improved chemical compatibility and interfacial bonding. This distinction is important for interpreting subsequent mechanical results, where improved wet property retention may arise from enhanced resistance to moisture-induced interfacial degradation rather than reduced water absorption. Comparable moisture uptake among treatment groups further suggests that water remained capable of penetrating the composite structure regardless of surface treatment.

4.2. Effect of Moisture on Flax Yarn Mechanical Behaviour

The influence of moisture exposure on flax yarn mechanical behaviour is shown in Fig. 2. Tensile performance was evaluated through tenacity, representative tenacity–strain curves, and modulus measured at different strain regions to assess fiber-level mechanical response.

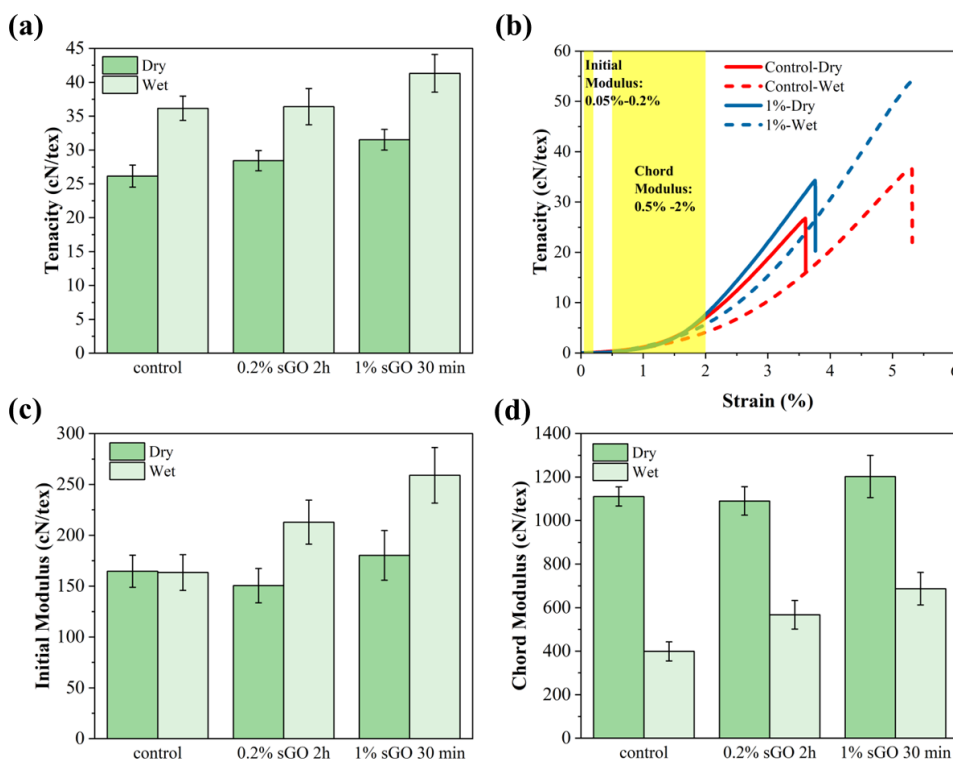


Figure 2. Mechanical behaviour of flax yarns under dry and water-conditioned states: (a) apparent tenacity, (b) representative tenacity–strain curves, (c) initial modulus (0.05–0.2% strain), and (d) chord modulus (0.5–2% strain).

As shown in Fig. 2a, dry yarn tenacity increased following sGO treatment, from 26.1 cN/tex for the control to 28.8 cN/tex and 31.8 cN/tex for the 0.2 wt.% and 1 wt.% sGO-treated yarns, respectively, which was reported in authors' previous work [12]. After 24 h water immersion, apparent tenacity increased across all groups, reaching 36.2 cN/tex for the control and 41.3 cN/tex for the 1 wt.% sGO-treated yarn. Similar behaviour has been reported in moisture-aged flax composites, where moderate water absorption temporarily enhanced load transfer through fiber swelling and improved interfacial contact by Muñoz et al.[5]. However, wet tenacity was calculated using the original dry linear density (tex); therefore, the observed increase should be

interpreted cautiously, as moisture uptake increased yarn mass and diameter after immersion. Nevertheless, the higher maximum load observed in the tenacity–strain curves (Fig. 2b) suggest enhanced fiber rearrangement and load redistribution rather than intrinsic strengthening of flax fibers. Representative tenacity–strain curves further illustrate the effect of moisture on yarn deformation behaviour (Fig. 2b). While sGO-treated yarns maintained superior tensile performance under both dry and wet conditions, water immersion altered deformation behaviour, resulting in lower stiffness at intermediate strains but higher load-bearing capacity at larger strains.

To further examine this behaviour, modulus was evaluated in two strain regions: an initial modulus calculated from 0.05–0.2% strain and a chord modulus determined between 0.5–2% strain. As shown in Fig. 2c, the initial modulus remained stable or increased slightly after water conditioning, particularly for treated yarns. For example, the initial modulus of the 1 wt.% sGO-treated yarn increased from 180.2 to 258.9 cN/tex, while the control remained nearly unchanged (164.6 to 163.4 cN/tex). This behaviour may result from temporary stiffening caused by moisture-assisted fiber swelling and enhanced compaction between elementary fibers within technical flax bundles, which may improve resistance to initial deformation [13].

In contrast, the chord modulus decreased substantially after moisture exposure for all groups (Fig. 2d), dropping from 1110.6 to 399.1 cN/tex for the control and from 1202.6 to 686.9 cN/tex for the 1 wt.% sGO-treated yarn. This reduction likely reflects moisture-induced plasticization and increased mobility between fiber bundles, reducing stiffness during continued deformation. The simultaneous increase in initial modulus and reduction in chord modulus suggests a strain-dependent response, where early-stage stiffness is maintained through swelling-induced consolidation, while larger-strain behaviour becomes governed by plasticization and fiber slippage. These yarn-level observations provide important insight into subsequent composite behaviour under wet conditions. Although moisture altered yarn deformation characteristics, sGO-treated yarns maintained improved tensile performance, suggesting that interfacial stabilization mechanisms may continue to contribute to mechanical property retention after moisture exposure.

4.3. Mechanical Performance Retention of Water-Aged Flax–Epoxy Composites

The influence of moisture exposure on the tensile performance of flax–epoxy composites is shown in Fig. 3. Tensile strength and modulus were evaluated for woven and UD composites after 1 day and 7 days of water immersion. At the time of manuscript submission, longer-term ageing experiments remain ongoing and will be completed prior to the conference presentation.

Woven composites exhibited relatively minor changes in tensile strength following moisture exposure (Fig. 3a). Tensile strength remained generally comparable to dry-state values for all groups, indicating good tolerance to short-term water exposure. For example, the 0.2 wt.% sGO-treated woven composite maintained the highest tensile strength, changing only slightly from 112.4 MPa (dry) to 113.0 MPa (1 day) and 108.8 MPa (7 days). Similarly, the 1 wt.% woven composite increased slightly from 104.0 MPa to 106.0 MPa after 7 days. In contrast, tensile modulus decreased substantially after immersion (Fig. 3b). The control woven composite decreased from 11.7 GPa (dry) to 8.1 GPa after 7 days, while the 1 wt.% woven composite decreased from 11.0 to 6.8 GPa. These reductions likely resulted from matrix plasticization and moisture-induced softening of the flax network. Despite modulus loss, tensile strength remained

relatively stable, suggesting that woven architecture mitigated moisture-induced stress concentrations through yarn interlacing and load redistribution.

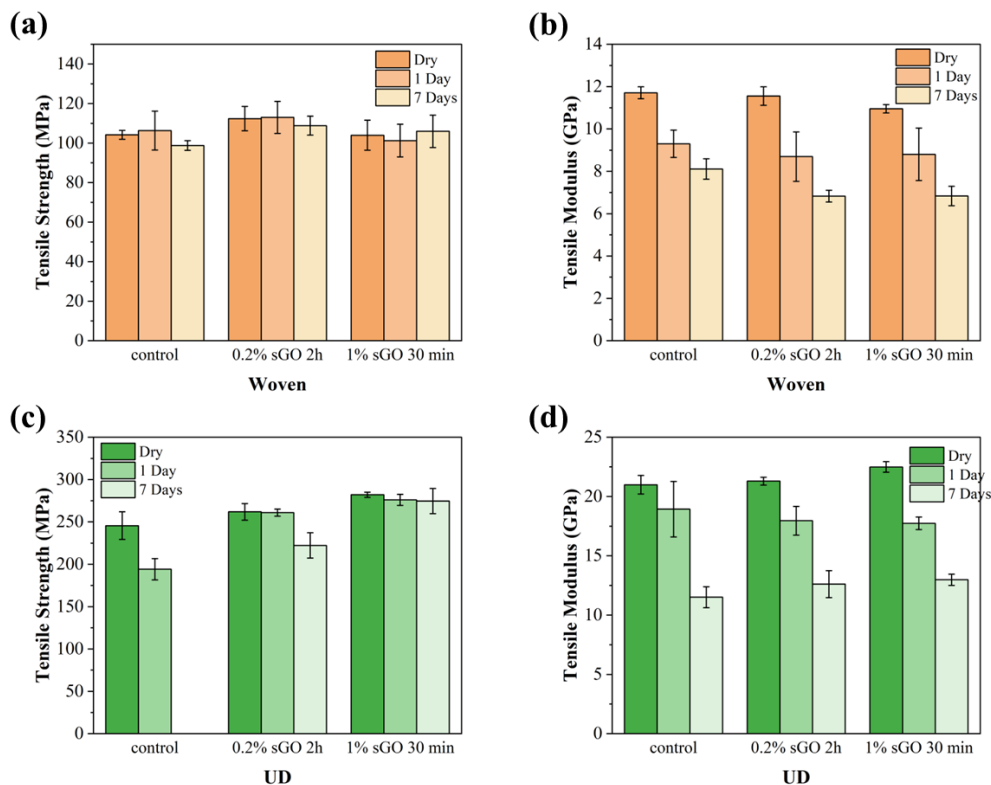


Figure 3. Tensile properties of flax-epoxy composites after water ageing: (a) tensile strength of woven composites, (b) tensile modulus of woven composites, (c) tensile strength of UD composites, and (d) tensile modulus of UD composites under dry, 1-day, and 7-day immersion conditions. Error bars represent the 95% confidence intervals. Each column is based on eight replicates.

In contrast, UD laminates exhibited greater sensitivity to moisture exposure (Fig. 3c–d). The control showed a substantial reduction in tensile strength, decreasing from 245.6 MPa (dry) to 194.2 MPa after 24 h. This behaviour suggests pronounced moisture-induced degradation associated with fiber swelling, matrix plasticization, and weakening of the fiber–matrix interface. Interestingly, sGO-treated UD composites demonstrated substantially improved strength retention following water immersion. The 0.2 wt.% sGO-treated UD composite retained 222.2 MPa tensile strength after 7 days, compared with 261.9 MPa in the dry state, while the 1 wt.% group exhibited minimal strength reduction (282.1 MPa dry vs. 274.6 MPa after 7 days). Although tensile modulus decreased for all UD groups after immersion, treated composites maintained higher post-ageing stiffness than control. After 7 days, the 1 wt.% UD composite retained 13.0 GPa modulus, compared with 11.5 GPa for the control.

These preliminary results suggest that sGO treatment improves moisture durability primarily through stabilization of the fiber–matrix interphase rather than reduced moisture ingress, consistent with the moisture absorption behaviour discussed in Section 4.1. Although water remained capable of penetrating the composite structure, sGO-treated composites exhibited improved resistance to moisture-induced mechanical degradation. Ongoing long-term ageing

experiments will further clarify whether these trends persist after extended exposure and equilibrium saturation.

4.4. Interfacial Stability During Moisture Ageing

The influence of moisture exposure on interfacial behaviour was evaluated using short-beam shear testing, and the corresponding results are shown in Fig. 4. Under dry conditions, the measured ILSS values (21.1-39.8 MPa) remained within the range commonly reported for flax–epoxy composites using ASTM D2344 short-beam testing. For example, Javanshour et al. [14] reported an apparent interlaminar shear strength of ~ 27.4 MPa for flax/epoxy laminates, which is comparable to the dry values measured in the present study. However, after moisture conditioning, calculated values increased substantially beyond the expected range for natural fiber composites, particularly for woven and UD longitudinal specimens. Because moisture-conditioned specimens exhibited large deformation and altered failure behaviour during testing, the wet-condition results are referred to herein as apparent short-beam strength rather than true ILSS.

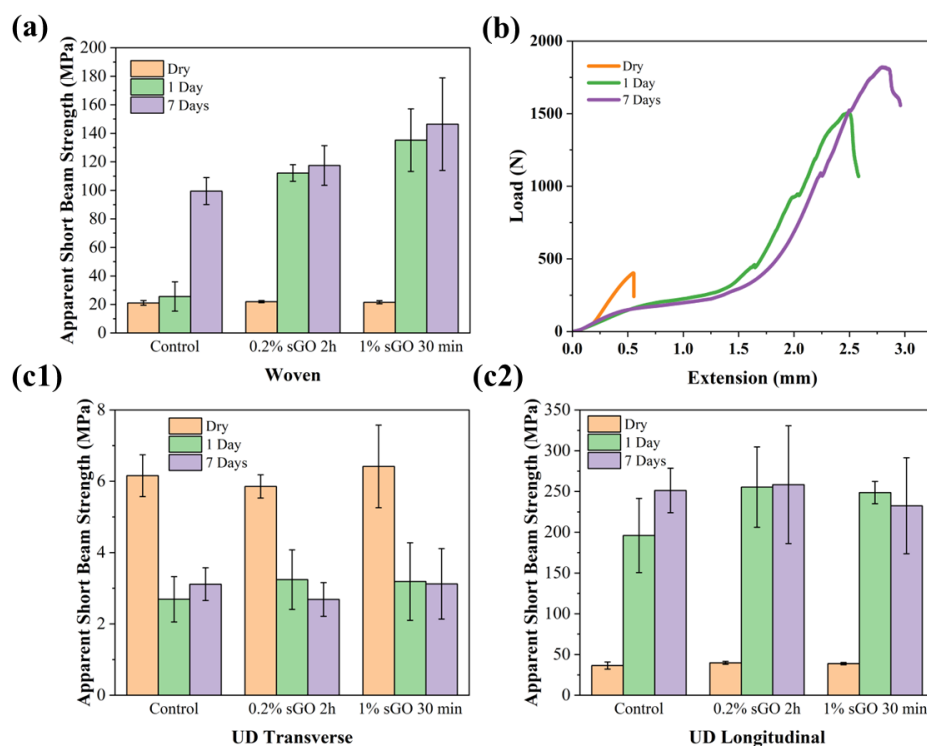


Figure 4. Apparent interlaminar shear behaviour of flax–epoxy composites under moisture ageing: (a) ILSS of woven composites, (b) representative load–extension curves for 0.2 wt.% sGO woven composites, (c1) Apparent ILSS of UD longitudinal specimens, (c2) Apparent ILSS of UD transverse specimens.

For woven composites (Fig. 4a), apparent short-beam strength increased markedly after water immersion. The control increased from 21.1 MPa (dry) to 25.6 MPa after 1 day and 99.5 MPa after 7 days. Similarly, the 0.2 wt.% sGO-treated woven composite increased from 22.0 to 117.4 MPa, while the 1 wt.% group reached 146.4 MPa after 7 days. Comparable trends were observed for UD longitudinal specimens (Fig. 4c1), where the control increased from 36.5 MPa (dry) to 251.2 MPa after 7 days, while the 0.2 wt.% and 1 wt.% sGO groups reached 258.3 and 232.5 MPa,

respectively. In contrast, UD transverse specimens (Fig. 4c2), which are more sensitive to matrix- and interface-dominated deformation, exhibited reductions after conditioning, with the control decreasing from 6.16 MPa to 3.12 MPa after 7 days.

The contrasting behaviour between longitudinal and transverse loading directions suggests that short-term moisture exposure altered the deformation mechanism of the composites rather than simply improving interfacial strength. Representative load–extension curves of woven composites (Fig. 4b) illustrate this transition clearly. Dry specimens failed abruptly at low displacement, exhibiting brittle fracture and steep load drops. In contrast, water-conditioned specimens sustained substantially greater deformation before failure, with extended displacement and more gradual load transfer, indicating increased flexibility and compliance after immersion.

Fracture morphology further supports this observation (Fig. 5). Dry specimens exhibited relatively flat fracture surfaces and limited deformation, whereas water-conditioned specimens developed pronounced curvature corresponding to the loading nose of the short-beam fixture, suggesting substantial elastic deformation prior to failure. The retained curvature after fracture indicates that moisture conditioning increased flexibility and delayed catastrophic fracture.

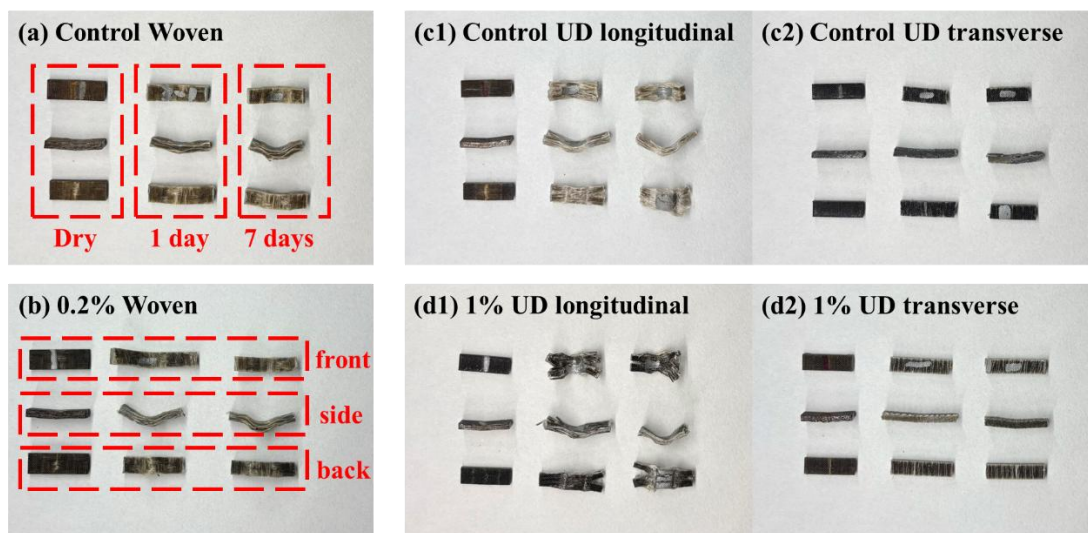


Figure 5. Representative fracture morphology of flax–epoxy composites after short-beam shear testing: (a) control woven composite after dry, 1 day, and 7 day immersion; (b) 0.2 wt.% sGO-treated woven composite (front, side, and back views); (c1–c2) control UD longitudinal and transverse specimens; and (d1–d2) 1 wt.% sGO-treated UD longitudinal and transverse specimens. Water-conditioned specimens exhibited increased deformation and curvature compared with dry specimens.

The elevated apparent short-beam strength observed after short-term ageing may therefore arise from moisture-induced plasticization and fiber swelling rather than improved intrinsic interfacial shear resistance. Matrix plasticization likely reduced brittleness and promoted stress redistribution during short-beam loading, while flax fiber swelling may have temporarily improved interfacial contact and mechanical interlocking, consistent with yarn-level observations (Section 4.2) and previous reports of moisture-induced plasticization in flax–epoxy systems [5]. Notably, sGO-treated specimens maintained higher apparent short-beam strength than controls after conditioning, suggesting improved stability of the fiber–matrix interphase despite continued moisture uptake (Section 4.1). Ongoing long-term ageing experiments will determine whether this

temporary enhancement persists or transitions to interfacial degradation at equilibrium moisture saturation.

5. Conclusions

This study investigated the influence of sGO treatment and fiber architecture on the moisture ageing behaviour of flax–epoxy composites. Moisture absorption results showed that all composites exhibited comparable equilibrium moisture contents and diffusion behaviour, indicating that sGO treatment did not substantially reduce water ingress. However, mechanical testing demonstrated that sGO-treated composites, particularly UD laminates, exhibited improved retention of tensile properties after moisture exposure compared with controls. Woven composites showed greater tolerance to short-term water ageing, maintaining tensile strength despite reductions in modulus.

Notably, short-term moisture conditioning increased the apparent interlaminar shear strength of woven and UD longitudinal composites, accompanied by increased deformation and flexibility during short-beam shear testing. These findings suggest that moisture-induced plasticization and fiber swelling temporarily improved stress redistribution and interfacial contact under localized loading. Overall, the results indicate that sGO enhances moisture durability primarily through improved interfacial stability rather than reduced moisture uptake. Ongoing long-term ageing experiments will further evaluate whether these benefits persist under equilibrium moisture saturation conditions. In addition, post-ageing microstructural and chemical characterization using scanning electron microscopy (SEM) and Fourier transform infrared spectroscopy (FTIR) will be conducted to further elucidate the mechanisms governing moisture-induced degradation and assess the stability of the sGO-modified fiber–matrix interface.

6. References

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