



Ageing of out-of-autoclaved hybrid flax/carbon fibre composites: effects on water absorption and flexural behaviour

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ABSTRACT

The growing demand for high-performance yet sustainable composites highlights the need to replace synthetic composites and energy-intensive processing with more efficient alternatives. However, limited research has been conducted on how out-of-autoclave (OOA) fabrication affects the durability of the natural/synthetic hybrid composites under hygrothermal ageing. This study examines the ageing behaviour of flax/carbon fibre hybrids fabricated using a vacuum-assisted oven-curing process, focusing on moisture diffusion, swelling, and flexural response. Hybridisation reduced diffusion coefficients under hot-wet conditions by 76–96 % and swelling by up to 80 % compared with flax composites. It also enhanced flexural strength and stiffness up to 200 %. The hybrids retained 60–80 % of their flexural strength and 70–90 % of their stiffness after 23, 45, 70 °C water exposure, confirming the effectiveness of the carbon layers in limiting water-induced deformation and preserving interfacial integrity.

1. Introduction

Growing interest in sustainable engineering materials has challenged research into reducing environmental impact without sacrificing performance. Natural fibre composites are a biodegradable, renewable alternative to synthetic composites with lower energy input for manufacture. Their use in high-performance applications is yet limited by low mechanical properties, high water absorption, and poor dimensional stability under hygrothermal conditions [1].

Hybridisation—combining natural and synthetic fibres—has been shown to counter these shortcomings. The combination of synthetic fibres such as carbon or glass with natural fibre systems (i.e., flax, jute, coir, kenaf, or pineapple leaf) can enhance tensile and flexural strength, toughness, and water resistance, particularly if the stacking sequence and fibre volume fraction are optimum [2–5]. These properties position hybrid laminates as promising alternatives for durable structural applications [6]. Nevertheless, the majority of hybrid composites are presently still produced by compression moulding, autoclave moulding, or resin infusion. These methods are time-consuming and energy-hungry, and prone to void and inhomogeneous consolidation [7,8].

This study explores flax/carbon fibre hybrid composites fabricated via a vacuum-assisted out-of-autoclave (OOA) process, focusing on their

water absorption and flexural performance. Unlike conventional methods, the vacuum-assisted OOA route offers a scalable, cost-effective, and energy-efficient means to produce high-quality composites. The novelty of this work lies in examining the unique interaction of hygrothermal ageing, flexural loading, and stacking sequence in OOA-fabricated flax/carbon prepreg hybrids—an aspect not previously studied for this material system. This approach aims to enhance fabrication quality, mechanical performance, and hygrothermal resistance, advancing the accessibility of high-performance hybrid composites. By analysing flexural behaviour, moisture uptake, and swelling under varied environmental conditions, this study provides new insights into the potential of OOA-produced flax/carbon hybrids for sustainable, lightweight, and high-performance applications, contributing to the progress of eco-efficient composite technologies.

2. Materials and methods

2.1. Materials and design

Unidirectional prepreg flax fibre (FLAXPREG 110 g) and unidirectional prepreg carbon fibre (XC130 300 g) were sourced from Easy Composites. Symmetrical composite panels were designed and

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fabricated in a $\pm 45^\circ$ orientation using both flax and carbon fibres, as shown in Fig. 1.

2.2. Preparation and vacuum assisted OOA curing

Each composite panel was prepared by hand lay-up of six prepreg layers onto a mould, then vacuum-bagged as shown in Fig. 1. The moulds were oven-cured under -1 bar vacuum to ensure consolidation. Curing was carried out by heating to 80°C at $2^\circ\text{C}/\text{min}$ with 40 mins dwell, followed by a ramp to 120°C held for 1 h, and then cooled to room temperature at $2^\circ\text{C}/\text{min}$. Samples ($80 \times 20 \times 1.8 \pm 0.3$ mm) were cut by waterjet. Density measurements showed flax had the lowest density (0.97 g/cm^3), Hybrid-1 (HY1) the highest (1.28 g/cm^3) from its three carbon layers, and Hybrid-2 (HY2) intermediate (1.21 g/cm^3) due to its denser skins and lighter core.

2.3. Environmental conditioning

The specimens were dried in a desiccator to constant weight, then conditioned by immersion in water at 23 , 45 , 70°C , and in a controlled -20°C frozen environment. Weights were recorded at regular intervals using a 0.01 mg precision balance after removing surface water. Moisture uptake was monitored gravimetrically until reaching effective saturation, with three specimens of each material tested for averaging. The percentage weight gain was calculated following ASTM D570–98 (Eq. 1) and plotted against $\sqrt{\text{time}}$, exhibiting the expected Fickian linear trend [9]. As the focus of this work is on the initial Fickian diffusion stage, long-term non-Fickian behaviour was not addressed [10]. The swelling rate was determined using Eq. 2.

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

$$\text{Swelling Ratio\%} = \frac{\text{wet area} - \text{dry area}}{\text{dry area}} \times 100 \quad (2)$$

The diffusion coefficient (D) was obtained from Eq. 3, where G is the slope of the initial linear portion, h the sample thickness, and M_∞ the equilibrium moisture content. An edge correction factor was applied, accounting for sample dimensions (h , l , w). The $\pm 45^\circ$ composite geometry was assumed to have isotropic diffusion, consistent with the resin phase [10].

$$D = \frac{\pi G^2 h^2}{16 M_\infty \left(1 + 0.54 \frac{h}{l} + 0.54 \frac{h}{w} + 0.33 \frac{h^2}{lw} \right)} \quad (3)$$

2.4. Three-point bending test

Flexural testing was performed using a Hounsfield testing machine following the ASTM D790–17 standard. A three-point bending test with a 64 mm support span and a crosshead rate of 2 mm/min was performed. Five specimens were tested for each condition.

3. Results and discussion

3.1. Moisture diffusion properties

The water absorption behaviour of flax and hybrid composites was strongly influenced by fibre hybridisation, laminate lay-up, and conditioning temperature (Fig. 2 (a–c)). Flax composites showed the highest moisture uptake (Fig. 2 (e)) due to the hydrophilic cellulose and hemicellulose that readily form hydrogen bonds with water [8]. Water diffusion through amorphous regions disrupts these bonds causing fibre swelling [11], while crystalline cellulose restricts diffusion. Overall, combined sorption and multilayer absorption mechanisms lead to high moisture retention. Hybridisation significantly reduced moisture uptake by up to 80% and 65% for HY1 and HY2, respectively. Hybrid-1 exhibited the lowest water content, demonstrating the moisture-barrier function of the carbon layers [12]. Hybrid-2, with a thicker flax core, absorbed about 6.5% more water than HY1 due to its greater flax volume and diffusion pathways. For all composites, water uptake increased with temperature as epoxy chain mobility and free volume expanded, facilitating deeper penetration. Elevated temperature may also have promoted microcracking or interfacial degradation [12–14]. Diffusion coefficients (Fig. 2 (d)) reflected the same trend: flax composites had the highest diffusion from their high polarity and porosity. Hybrid-2 matched Hybrid-1 at 23°C and 45°C but exceeded it at 70°C , attributed to its thicker flax core and enhanced temperature-driven diffusion. The 96% diffusion reduction in HY1 confirms the efficient consolidation from vacuum-assisted oven curing.

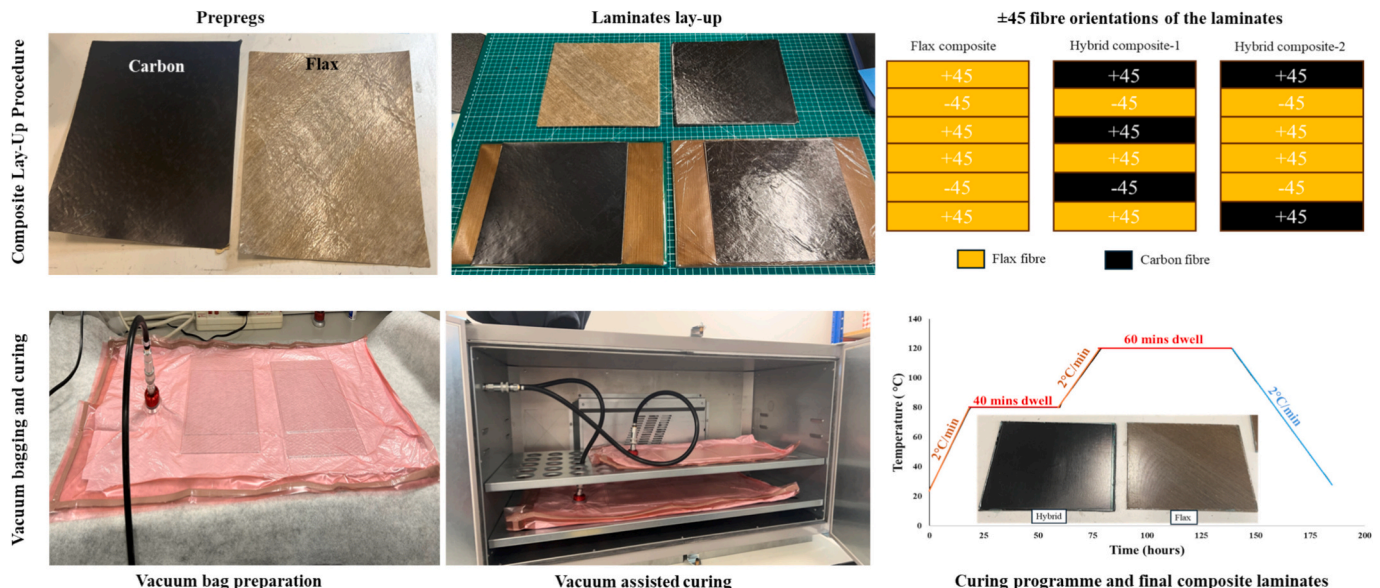


Fig. 1. Composites fabrication process

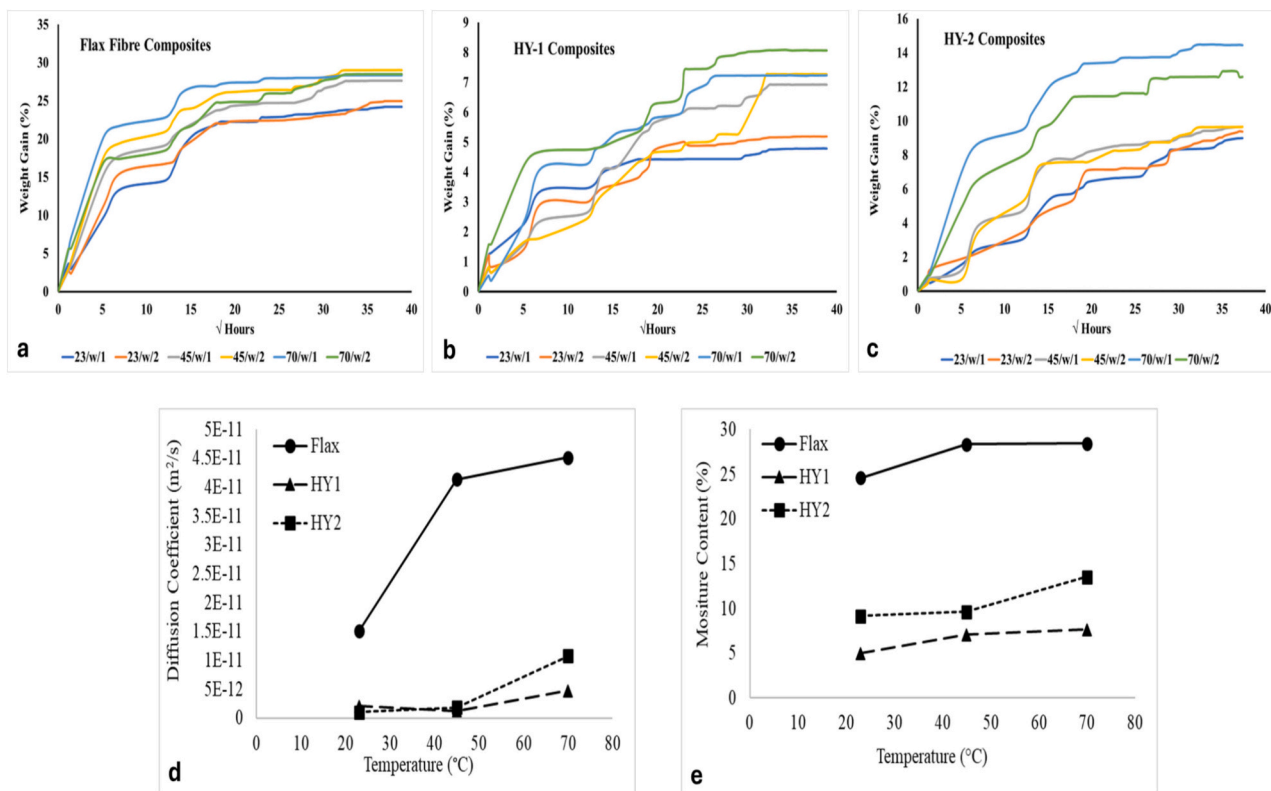


Fig. 2. Moisture uptake at different temperatures (a,b,c), diffusion rates (d) and moisture content (e) of the composites.

3.2. Swelling

Swelling behaviour (Fig. 3) differed clearly among flax, Hybrid-1, and Hybrid-2 composites. Flax composites exhibited the highest swelling at all temperatures due to their hydrophilic cellulose and hemicellulose, whose hydroxyl groups form hydrogen bonds with water, leading to greater moisture uptake and fibre expansion [12,15]. Swelling decreased from $\sim 14\%$ at 23°C to $\sim 9\%$ at higher temperatures due to partial fibre network stabilisation through plasticisation or internal stress relaxation [14]. Despite this reduction, flax composites remained most prone to moisture expansion. Hybrids showed 50–85 % lower swelling rates because of the reduced flax content and the restraining effect of carbon fibres acting as dimensional stabilisers [12,16]. Both hybrids displayed similar dimensional stability despite differences in composition revealing the carbon skins effectiveness in

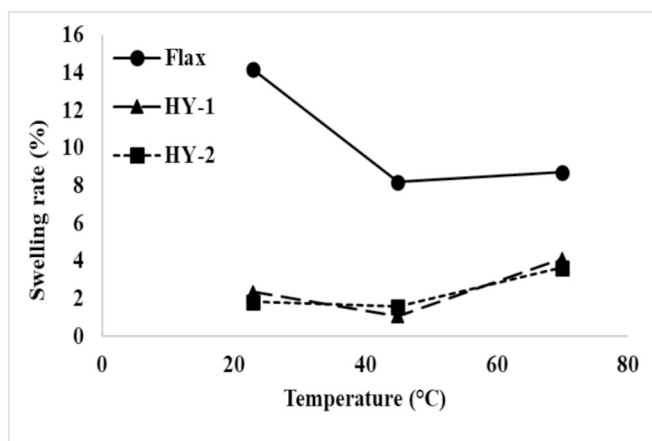


Fig. 3. The swelling rate. vs. temperature.

limiting flax expansion. The swelling rate dropped slightly at 45°C due to polymer relaxation relieving internal stress. The slight swelling rise at 70°C is caused by the increased polymer mobility and partial fibre–matrix debonding, reflecting competing effects of plasticisation and interfacial weakening [16].

3.3. Flexural analysis

The hybrid composites outperformed flax-only laminates, indicating the effectiveness of hybridisation. Moisture exposure caused a decline in flexural performance across all samples (Fig. 4). Flax composites showed a sharp strength drop from 85.9 MPa at room temperature to 21.1 MPa after 70°C water ageing ($\sim 75\%$ loss), caused by swelling-induced fibre–matrix debonding and matrix softening [16]. At -20°C , strength (56.4 MPa) remained higher, indicating that sub-zero embrittlement was less damaging than moisture, though thermal contraction likely caused microcracking [17]. Hybrid-1 retained 156 MPa ($\sim 84\%$) at -20°C and 110 MPa ($\sim 60\%$) at 70°C , whereas Hybrid-2 retained 138 MPa ($\sim 90\%$) and 96 MPa ($\sim 63\%$) under the same conditions. These results confirm the protective effect of carbon skins against moisture and temperature damage [14], while the lower strength of Hybrid-2 reflects its higher flax content and greater moisture sensitivity. Flexural modulus decreased with temperature for all materials. Flax composites dropped from 3.5 GPa at RT to 0.7 GPa after 70°C exposure, indicating severe softening and plasticisation, and declined to 1.8 GPa at -20°C due to microcracking and limited load transfer [1,17]. Hybrid-1 remained stable across temperatures, decreasing from 7.0 GPa at RT to $\sim 6\text{ GPa}$ at $(-20)\text{--}45^{\circ}\text{C}$ and 4.7 GPa at 70°C . Hybrid-2 showed higher stiffness, retaining $9.5\text{--}10\text{ GPa}$ of its 11.5 GPa RT modulus after 23°C and 70°C exposure. The minor modulus reduction at -20°C and 45°C is likely caused by thermal contraction microcracking and slight polymer softening. The carbon skins effectively constrained strain, ensuring stable load transfer consistent with sandwich theory [18].

The present flax/carbon hybrids fabricated via vacuum-assisted OOA

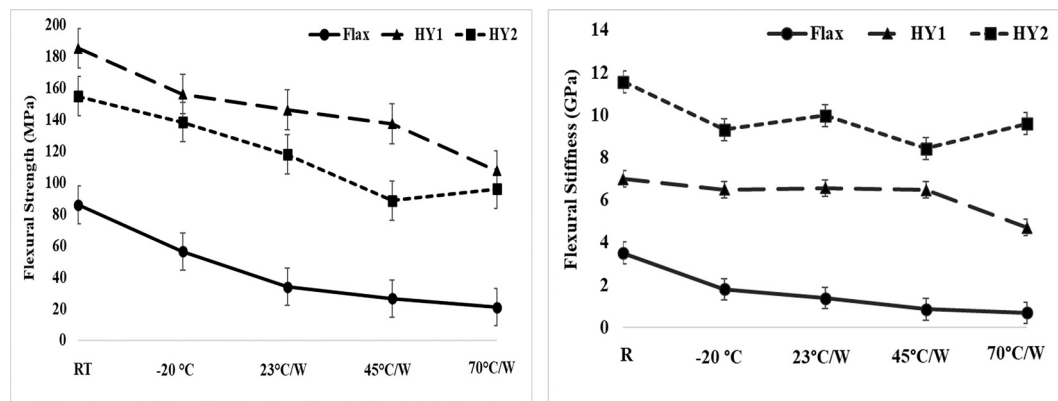


Fig. 4. Flexural properties under different conditions.

outperform previously reported systems [14–16]. While flax/epoxy and glass/flax (compression-moulded or autoclave) retained only 40–55 % strength, and carbon/flax via resin infusion 60 %, the vacuum-assisted, oven-cured hybrids retained on average up to ~70 % strength and ~80 % stiffness under hot-wet and frozen conditions. High stiffness and strength retention confirm the strong bonding from vacuum-assisted oven curing. These results highlight the novelty of combining hybridisation with energy-efficient OOA processing to achieve superior dimensional stability and mechanical durability.

4. Conclusion

$\pm 45^\circ$ flax/carbon fibre hybrid composites were successfully fabricated using a vacuum-assisted curing oven to evaluate the effects of ageing, laminate design, and hygrothermal conditioning on water absorption and flexural performance. Flax-dominant composites suffered from severe moisture uptake, swelling, and major strength loss under hot-wet and freezing conditions due to fibre-matrix debonding and microcracking from flax's hydrophilic nature. In contrast, both hybrid systems exhibited lower water absorption properties and swelling while maintaining strength and stiffness after ageing. Hybrid-1 showed lower moisture uptake and diffusion with higher strength but lower stiffness than Hybrid-2, which exhibited comparable dimensional stability and better rigidity. The strategic integration of hydrophilic flax with hydrophobic carbon fibres provided an effective balance between sustainability, durability, and mechanical performance. Vacuum-assisted curing ensured strong bonding, confirming a cost-effective alternative to other curing processes.

CRediT authorship contribution statement

Feras Korkees: Writing – original draft, Supervision, Resources,

Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Alison Sharratt:** Writing – original draft, Methodology, Formal analysis, Data curation. **Mazen Alqathami:** Writing – review & editing.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

The data that has been used is confidential.

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