INFLUENCE OF WET/DRY AGEING ON MECHANICAL PROPERTIES AND DAMAGE MECHANISMS OF HEMP-REINFOCED ECOCOMPOSITES

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ABSTRACT

This study aims to assess the effect of wet/dry cycling ageing on the mechanical properties and the damage mechanisms of hemp and glass reinforced composites. Samples were successively immerged in water and dried in an oven for 30 wet/dry cycles. Three different matrices were used to manufacture four fibre/matrix combinations: hemp/Epolam, hemp/Greenpoxy, hemp/Elium and glass/Epolam. Tensile tests, instrumented with an axial extensometer and an acoustic emission system, were performed for 0 (ambient storage), 1, 5, 10, 20 and 30 wet/dry cycles. Micro-computed tomography acquisitions were carried out before and after each mechanical test to follow the occurrence of damage during the ageing phases and the consequences on their development during mechanical loadings. Results enable a thorough understanding of the wet/dry cycling ageing consequences on the behaviour of biocomposites.

1 INTRODUCTION

Despite the increasing interest of manufacturers in the use of ecocomposites in the transport, energy or furniture industry to replace glass-reinforced composites [1]–[3], outdoor conditions are still a limiting factor. Indeed, the hydrophilic behaviour of natural fibre reinforced composites and the related consequences on interfacial adhesion have to be taken into account for structural applications [4], [5]. Some studies simulated the service conditions to which flax-reinforced composites could be subjected [6], [7]. This work aims to study the effect of wet/dry cycling ageing on the mechanical properties and damage mechanisms of three hemp-reinforced biocomposites. To reproduce the outdoor conditions, wet/dry cycles are applied so that absorption and desorption are accelerated. Repeated progressive tensile loading tests, instrumented with acoustic emission (AE), are performed on standard rectangular samples. For each selected number of wet/dry cycles, micro-CT acquisitions are carried out before and after tensile testing. Results are compared with glass-reinforced composites.

2 MATERIALS AND METHODS

2.1 Tested materials

In this study, hemp and glass-reinforced composites are made of seven plain-woven plies of fabrics with an areal weight of 290 ± 10 g/m² and 280 ± 10 g/m², respectively. Three matrices were used to manufacture plates using the vacuum infusion process. Two matrices are thermoset resins (Epolam2020 and Greenpoxy56) and one is a thermoplastic polymer (Elium180). Glass reinforcement is only used with the Epolam resin. Some properties of raw materials are provided in Table 1. Samples are then cut to have two reinforcement orientations (±45° and 0°/90°). All the samples were manufactured to obtain an equivalent fibre volume fraction (v_f) to facilitate comparisons. The measured values of v_f and the overall dimensions for all the studied materials are given in Table 2.

	density ($g.cm^{-3}$)	Young's modulus (GPa)	strength (MPa)	References
hemp yarn	1.48	23	601	[8], [9]
Epolam 2020	1.16	3.1	69	[4], [9]
Greenpoxy 56	1.18	2.8	67	
Elium 180	1.19	3.3	76	manufacturer's data sheet

Table 1: Properties of used materials.

Composite	v_f (%)	thickness (mm)	width (mm)	length (mm)
hemp/Epolam	40.3 ± 2.4	3.5	20	140
hemp/Greenpoxy	41.8 ± 1.7			
hemp/Elium	39.7 ± 1.6			
glass/Epolam	47.0 ± 2.5	1.5		

Table 2: Fibre volume fractions and overall dimensions of the studied composites.

2.2 Wet/dry ageing

Each wet/dry cycle consists in immersing samples in water at 60°C for 12 days (enough to reach the maximum water absorption) and then drying them in an oven at 40°C for 2 days. Up to 30 ageing cycles were performed, representing a duration of 420 days. The water uptake of the samples has been measured with a precision balance during the immersion phases. Weight gain, noted M_t, follows the Eq. (1):

$$M_t = 100 * \frac{m(t)-m_0^i}{m_0^i} \quad (1)$$

where m_0^i is the initial weight of the sample for the wet/dry cycle number i and $m(t)$ is the sample weight at time t. An approximation of the one-dimensional Fick's model has been used [10] (Eq. (2)) and the diffusion coefficient for each wet/dry cycle is expressed by Eq. (3):

$$\frac{M_t}{M_\infty} = 1 - \exp\left(-7.3\left(\frac{Dt}{h^2}\right)^{3/4}\right) \quad (2)$$

$$D = \pi\left(\frac{hk}{4M_\infty}\right)^2 \quad (3)$$

where M_∞ is the maximum of weight gain, D is the Fick diffusion coefficient (mm²/s), h is the thickness of the sample (mm) and k is the initial slope of the M_t-$\sqrt{t}$ curve.

2.3 Instrumented tensile tests

Tensile tests were performed by using an *Instron 5982* tensile testing machine with a crosshead speed of 0.5 mm/min. The axial strain was measured by a 12.5 mm gauge length extensometer. Samples were also instrumented with an acoustic emission (AE) system *PCI-2* from the company *Mistras Group* composed of two *Micro-80* sensors.

2.4 Micro-CT

Image acquisition was carried out using an *UltraTom CT* scanner manufactured by the company *RX Solutions*. In this work, a 15-µm resolution, an intensity of 300 µA and a voltage of 50 kV were used for an acquisition time of about 4 hours per sample.

3 RESULTS AND DISCUSSION

3.1 Water diffusion

The curves of water absorption measured during fifteen wet/dry cycles are shown in Fig. 1 for the four studied composites. For the sake of clarity, only the first fifteen wet/dry cycles are shown. It has been observed that the reinforcement orientation (±45° or 0°/90°) has no influence on the water uptake. Therefore, only one example for each material is plotted in Fig. 1. Figure 1 shows that the wet/dry cycle parameters chosen allow to reach the water saturation in less than twelve days and samples are almost dry after two days in an oven at 40°C. The maximum water uptake of the hemp-reinforced composites are located between 7 and 8% during all the cyclic hydrothermal ageing. Glass/Epolam samples logically absorb far less water than biocomposites. Whatever the composite and the number of wet/dry cycles, it has been checked that the water diffusion behaviour of each composite follows the one-dimensional Fickian law.

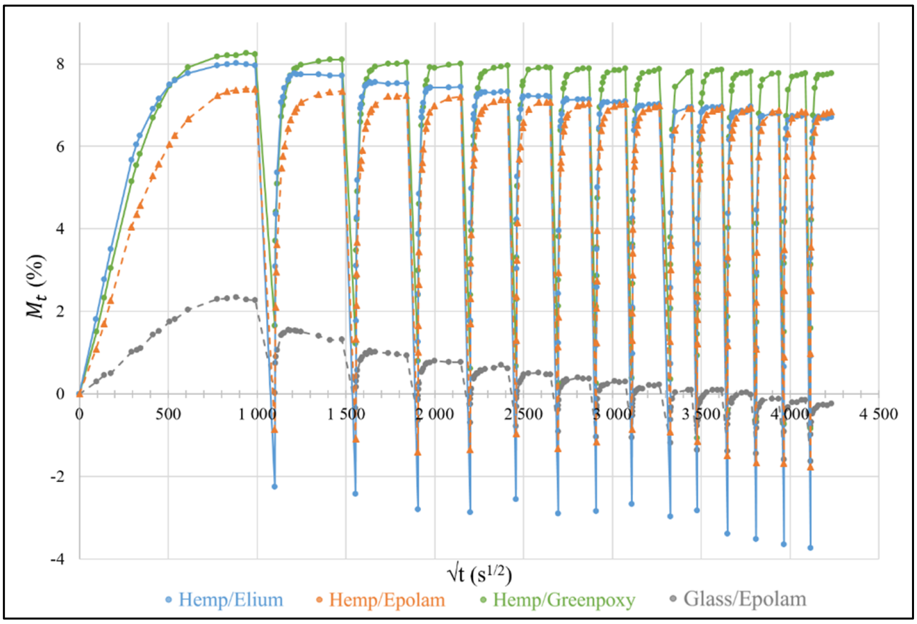

Figure 1: Weight variations during fifteen wet/dry cycles for all the studied materials.

Figure 2 shows for example the evolution of the Fick diffusion coefficient D for the ±45° hemp/Greenpoxy samples after 1, 5, 10, 20 and 30 wet/dry cycles. In addition, micro-CT images of these samples before mechanical testing for three different ageing states are shown. The evolution of D shows a high increase between the first and the second wet/dry cycles, and then the slope of the curve decreases. Micro-CT images in Fig. 2 show that fibre/matrix debondings occurred as early as the first wet/dry cycle. The debondings are more developed after 10 cycles and more again after 30 wet/dry cycles.

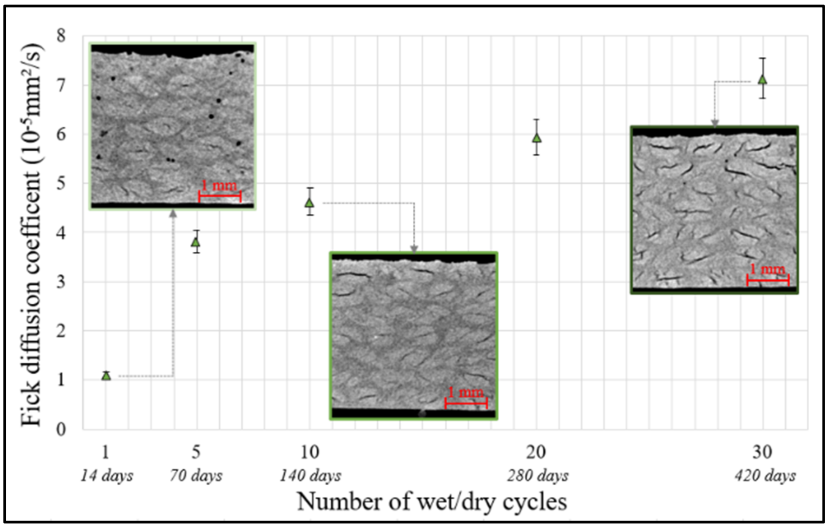

Figure 2: Fick diffusion coefficient evolution for hemp/Greenpoxy [±45°]$_7$ samples and related micro-CT images after 1, 10 and 30 wet/dry cycles.

3.2 Tensile properties

After ageing, all the materials were subjected to tensile tests. Figure 3 shows, for the three different biocomposites, for ±45° orientation, the evolution of the maximal stress value up to 30 wet/dry cycles. It can be seen that, whatever the matrix, there is a decrease in the maximum stress values when the number of wet/dry cycles is increasing. The decrease is significant after only one wet/dry cycle, and then a plateau can be observed up to 30 cycles.

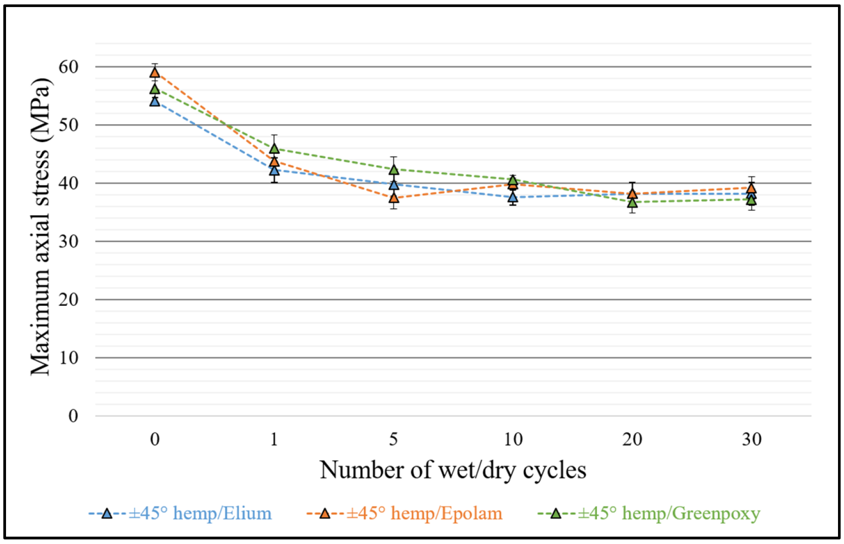

Figure 3: Maximum axial stress of ±45° hemp-reinforced composites versus the number of wet/dry cycles (0 means “Ambient storage”).

3.3 Damage quantification

Each tensile test was instrumented with the AE system. It allows to determine the evolution of the AE cumulated energy during mechanical testing. Figure 4 shows, for all the biocomposites, the applied stress values to reach 5% of the total AE cumulated energy during the tensile test. It can be seen that these stress values, considered as damage thresholds, decrease as the number of wet/dry cycles increases. This means the more aged the samples are, the earlier they become damaged. The tendency observed in the analysis of the AE energy is the same as for the mechanical properties; there is a decrease when the number of wet/dry cycles is increasing and it is more significant after the first wet/dry cycle.

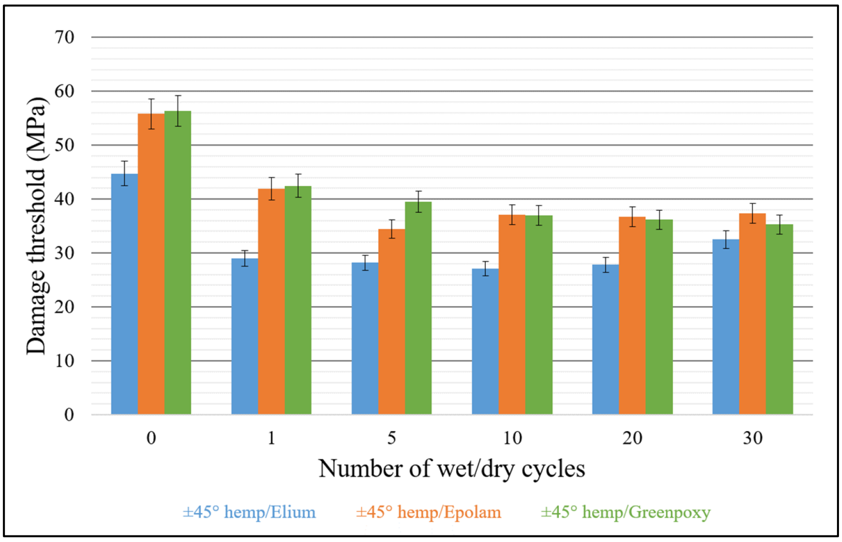

Figure 4: Applied stress values to reach 5% of the total AE cumulated energy during tensile testing of ±45° hemp-reinforced composites versus the number of wet/dry cycles (0 means "Ambient storage").

X-ray microtomography analyses have also been carried out after tensile failure to better understand the damage evolution. Figure 5 presents examples of pictures obtained after tensile tests for the three $[0°/90°]_7$ biocomposites of the study for ambient storage (AS) and ten wet/dry cycles. The images shown are from the core of each sample. The horizontal x-axis represents the direction of the tensile loading and the z-axis corresponds to the sample thickness.

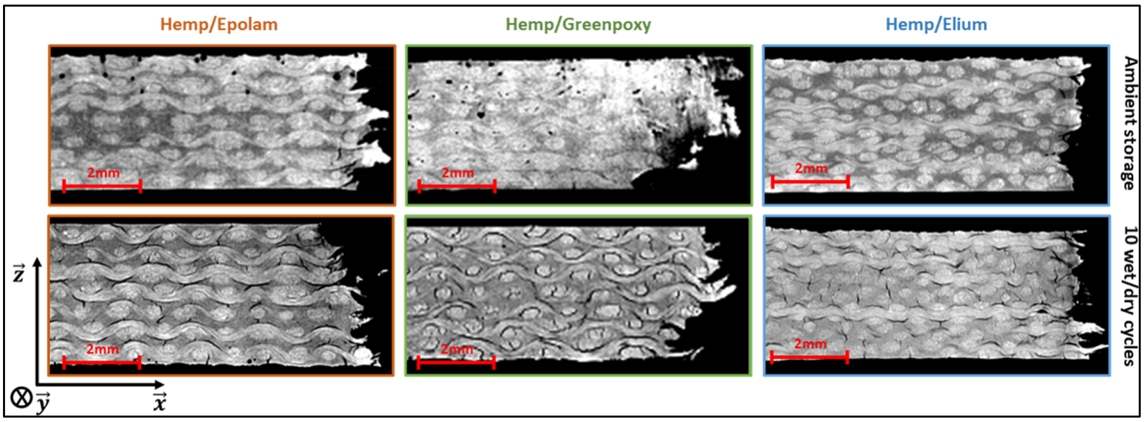

Figure 6: Micro-CT images after tensile testing for the three different biocomposites (orientation 0°/90°) after ambient storage and ten wet/dry cycles.

Micro-CT observations show that some specimens contain porosities due to the manufacturing process. For example, it is the case for the Hemp/Epolam and Hemp/Greenpoxy samples with AS condition presented in Figure 6. However, there is no specific damage due to the presence of these porosities. Figure 6 shows that, for all biocomposites, the damage is localised in the failure zone for AS conditionning. On the opposite, after 10 wet/dry cycles, the damage is present all along the observed zone of the samples. Debonding at fibre/matrix interfaces and matrix cracks are observed. Damage is far more developed than for ambient storage condition, demonstrating the role of wet/dry cycles on damage mechanisms. The water absorption fatigue leads to the degradation of the fibre/matrix interfaces, facilitating the development of damage during the tensile tests. This explains the decrease in the mechanical properties of the materials.

4 CONCLUSION

Up to 30 wet/dry cycles were applied to hemp-reinforced composites with Epolam, Greenpoxy or Elium matrix and to glass/Epolam composites. Micro-CT images of hemp composites reveal that interfacial debonding increases as the wet/dry cycles progress. This damage leads to the acceleration of the water absorption kinetics by increasing the Fick diffusion coefficient. At the same time, the mechanical properties are highly affected and the evolution of maximal stress values can be correlated to the acoustic emission activity.

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