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Development of a sustainable flax fiber-reinforced composite for low energy impact

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Abstract. In this study, a unidirectional layer of flax fibers is backed to a thin mat binder made of short flax fibers, the combination being used as reinforcement in an epoxy matrix. Using this reinforcement, two types of composite laminates were examined: a unidirectional $[0]_8$ and a cross-ply $[0/90]_{2s}$. These laminates were tested under uniaxial tension before and after impact at low energy levels. For comparison, synthetic glass fiber laminates made of the same stacking sequences and fiber volume fraction (40%) were tested under the same loading conditions. The findings indicate that the specific stiffness of the flax fiber composite is approximately 7% higher than that of the glass fiber composite, regardless of the stacking sequence used. When it comes to low-energy impact resistance, the cross-ply laminate demonstrates superior performance with greater impact resistance and less permanent deformation compared to the unidirectional laminate. The study also explores the hybridization of flax and glass fibers, suggesting a promising approach that leverages the synergistic effects of employing two different types of fibers in the laminate. The comparison of energy absorption during impact shows that the hybrid flax/glass composite has a greater energy absorption capacity than the glass fiber composite. Additionally, hybridization helps mitigate the degradation of tensile properties caused by impact, representing an effective strategy to enhance the post-impact mechanical properties of the flax fiber composite.

Introduction

Flax fiber is the most used vegetable fiber in composites, thanks to its exceptional specific mechanical characteristics [1], good vibration acoustic properties and its biodegradability [2, 3]. During the manufacturing process and during their use, composite materials are subjected to impacts, especially low energy impacts. The induced damages caused by low energy impacts can be barely visible, but they can affect the residual properties of parts and devices [4]. Habibi et al. [5] investigated the post-impact tensile properties of a unidirectional-mat flax/epoxy composite. They showed that the tensile strength was strongly affected by the impact, with a decrease of 41% after an impact of 5 J. In addition, Yuanjian and Isaac [6] showed that, for glass/polyester $[0/90]_{2s}$ composites, the residual tensile strength decreased when the impact energy threshold was reached between 5 J and 10 J. The same trend was observed for stiffness. Therefore, it is necessary to characterize the impact and post-impact behavior of biocomposites for better part designs in engineering applications.



Materials and processes

Reinforcements and composites fabrication

Two kinds of reinforcements were used: flax fibers and glass fibers. The flax reinforcement is a combination of a continuous unidirectional (UD) flax layer and a thin mat layer of short fibers which is used as a binder for the UD layer. This reinforcement is commonly named a UD-mat reinforcement. In the fabrication process, continuous flax yarns are first aligned, using a flat winder system, to make the UD layer. Next, short flax fibers, chopped into 6 ± 1 mm of length, are projected on the surface of the UD layer (to form the UD-mat reinforcement) using a dynamic former machine commonly used in the paper industry, as described in detail in a previous study [7]. After fabrication, the short fibers act as a binder to the UD yarns to allow reinforcement manipulation and maintain the UD yarns side-by-side during composites fabrication. The continuous flax yarns of the UD layer consist in Tex 400 strands supplied by Safilin Inc. (France), and a Tex 5000 flax ribbon, also supplied by Safilin, were chopped and used for the short mat binder of the reinforcement. After fabrication, the average surface density of the UD-mat reinforcement is 299 ± 11 g/m², including a 50 g/m² mat binder. Before molding, the stack of reinforcements is dried at 105°C for 15 minutes to remove humidity. The UD glass reinforcement, supplied by Texonic Inc. (Québec, Canada), has been chosen with a surface density of 295 ± 2 g/m², close to that of UD-mat flax reinforcement.

Composites were manufactured using the Resin Transfer Molding (RTM) process in which the SikaBiresin resin CR72, supplied by Sika Industry (USA), was used. For both types of laminate (flax and glass), two stacking sequences have been studied: a unidirectional [0]₈ and a cross-ply [0/90]_{2s} (Table 1). All laminates were produced with the same fiber volume fraction of around 40%. Moreover, a hybrid composite (combining layers of flax and glass reinforcements) was manufactured with the cross-ply stacking sequence [(0/90)^G/(0/90)^F]_s keeping the same 40% fiber volume fraction. The characteristics of the laminates are presented in Table 2.

Table 1- Configurations of studied composites.

	configurations	composite plates
FUM0 (flax UD-mat)	[0] ₈	
FUM090 (flax UD-mat cross-ply)	[0/90] _{2s}	
GUD0 (glass UD)	[0] ₈	
GUD090 (glass cross-ply)	[0/90] _{2s}	
FGUD090 (hybrid cross-ply)	[(0/90) ^G /(0/90) ^F] _s	

The flax fiber volume fraction (V_f) was calculated using the grammage of flax reinforcement and the composite plate thickness according to Equation 1:

$$V_f = \frac{n \times m_r}{e \times \rho_f} \quad (1)$$

With:

V_f : fiber volume fraction ;

n : number of plies in the composite ;

m_r : flax layer grammage (surface density) (g/m^2);

e : thickness of the composite (m) ;

ρ_f : flax fiber density (g/m^3).

For the glass laminates, the fiber volume fractions were measured by calcination of samples in an oven at 650°C for 30 minutes to determine the residual glass fiber weight. The density of composites was measured using the Archimedes' principle [REF.]. For the measurements and for each laminate in Table 1, five samples of 1 cm^2 were cut from the composite plates. Each sample was weighed using a Sartorius electronic scale (with an accuracy of 10^{-3} mg) and the density was next calculated using Equation (2):

$$\rho_c = \frac{m_{c,air}}{m_{c,air} - m_{c,water}} \times \rho_{water} \quad (2)$$

Here, ρ_c is the composite density (g/cm^3), ρ_{eau} the water density at room temperature (g/cm^3), $m_{c,air}$ the composite weight measured in air (g) and $m_{c,water}$ the weight of composite submerged in water (g).

Table 2- Physical properties of the composites

	Thickness t [mm]	Fiber volume fraction, V_f [%]	Density ρ [g/cm^3]
FUM0 (flax UD-mat)	4.25 ± 0.07	39.1 ± 0.8	1.28 ± 0.01
FUM090 (flax UD-mat cross-ply)	4.36 ± 0.02	38.1 ± 0.2	1.27 ± 0.01
GUD0 (glass UD)	2.34 ± 0.04	39.7 ± 0.9	1.69 ± 0.01
GUD090 (glass cross-ply)	2.32 ± 0.03	40.1 ± 0.6	1.72 ± 0.02
FGUD090 (hybrid cross-ply)	3.28 ± 0.08	39.5 ± 1.0	1.42 ± 0.01

Tensile and impact tests

Tensile tests before and after impact were performed with an Instron 8801 servo-hydraulic machine (Fig. 1a). The tests were conducted at a crosshead speed of $2 \text{ mm}/\text{min}$ and the longitudinal strain was measured by an INSTRON 2620-601 extensometer with a gauge length of 12.5 mm . Tensile samples of 150 mm in length x 25 mm in width were cut for the non-impacted specimens base on the

recommendations of ASTM D3039/D3039M. The width of post-impact tensile specimens was chosen at 30 mm, large enough to completely encompass the impact damage area. It should be noted that glass/epoxy tabs were bonded in the GUD090 (see Table 1 for the nomenclature) composite to ensure a failure inside the gauge length.

Low velocity impact tests were performed using an Imatek IM 10 ITS drop tower (Fig.1b). A load cell of 30kN was used to measure the force applied by the impactor during testing and the displacement was recorded by a laser sensor. To prevent multiple strikes, the drop tower machine was also equipped with an anti-rebound system. The specimens were fixed between an upper movable support and a lower fixed support, at a clamping pressure of 7 bar. The internal and external diameter of supports are 40 mm and 60 mm, respectively. ASTM D7136/D7136M standard was followed for the sample dimensions, which were as follows: 150 mm \times 90 mm \times t for the thickness of the composite. A 20 mm diameter hemispherical impactor was used to perform the tests and the impact energy was fixed at 5J for all the studied composites. After impact, the impacted samples were cut into the rectangular tensile samples in the center of impact zone with the dimensions 150 mm length \times 30 mm width for post-impact tensile tests.

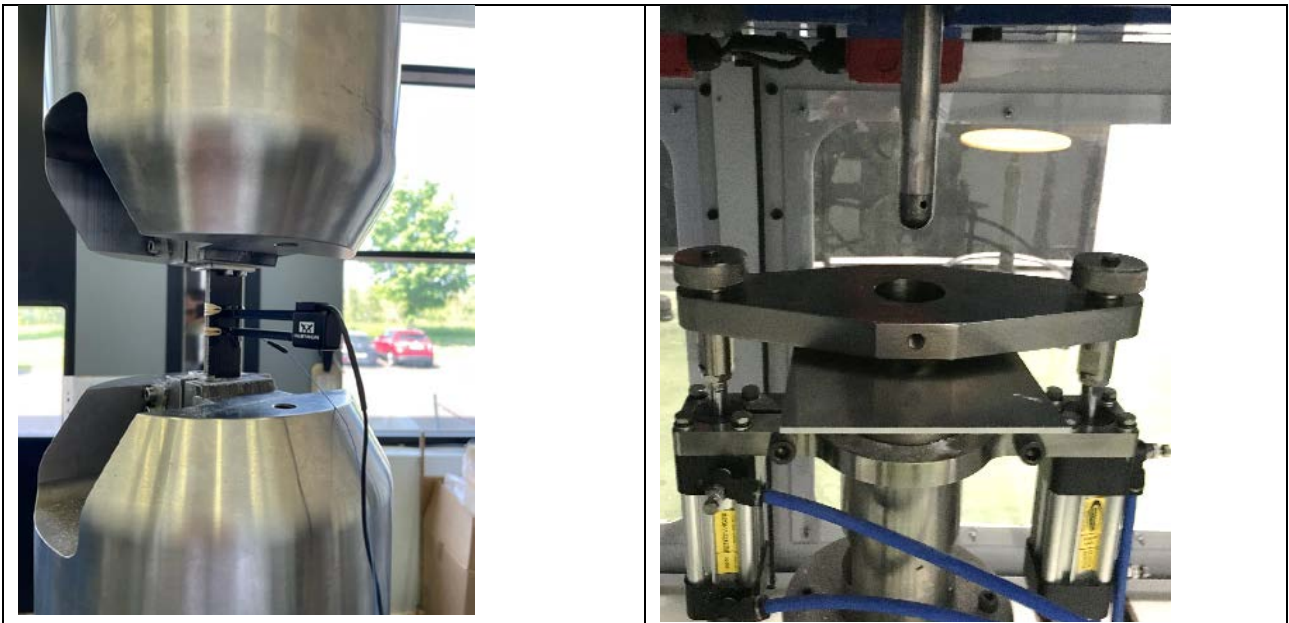


Figure 1- (a) tensile test, (b) impact drop tower with supports.

Micro-CT analysis

An UltraTom CT scanner manufactured by RX Solutions (France) was used to perform image acquisition. A resolution of 30 μ m was applied, along with a beam current of 141 mA and an accelerating voltage of 70 kV. In this study, the X-ray flat panel detector used had 1920 \times 1536 pixels with a pixel size of 127 μ m. For each specimen, around 2h were taken for the image acquisition. The reconstruction in the three different planes was performed using an algorithm based on the filtered back-projection procedure for Feldkamp cone-beam geometry.

Results and discussion

Mechanical properties of non-impacted composites

Fig. 2 displays representative stress-strain curves for the non-impacted composites for both stacking sequences. The unidirectional glass fiber composite (GUD0) exhibits a quasi-linear behavior up to failure. However, the unidirectional flax FUM0 shows a well-known bilinear behavior with a transition spread from 0.1% to 0.3% of strain commonly named the knee of the curve. The curves of the cross-ply composites (Fig. 2b) show lower elastic moduli and tensile strength than those of unidirectional laminates. As expected, the hybrid composite (green curve) has an intermediate

behavior between the glass fiber and flax fiber laminates. Its properties increase 34% for tensile strength and 13% for failure strain compared to the 100% flax fiber cross-ply laminate. However, no significant change was observed for the Young's modulus, which was expected to increase given the rule of mixtures. Table 3 groups the values of the experimental tensile properties along with the theoretical tensile Young's modulus (third column) determined by the rule of mixtures.

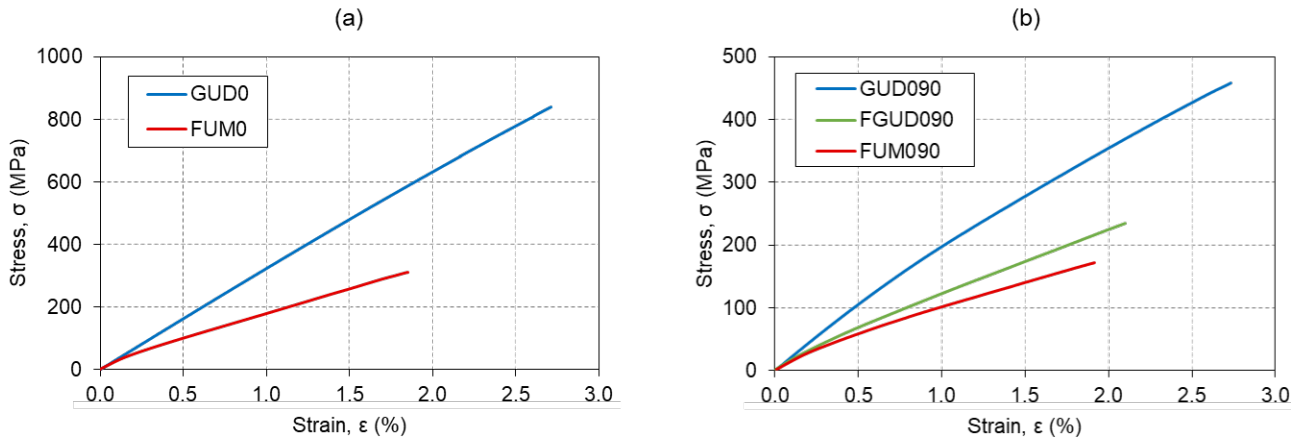


Figure 2-Stress and strain curves of non-impacted composites: (a) unidirectional flax UD-mat and glass fibers composites, (b) flax UD-mat cross-ply, glass fibers and hybrid cross-ply composites.

It is observed that the highest elastic modulus is obtained with the unidirectional glass laminate and the lowest one for the flax fibers cross-ply laminate. The theoretical values of the Young's modulus are very close to the experimental ones with a derivation of less than 14%. This means that the rule of mixtures can reasonably predict the modulus of elasticity even for a composite including the mat phase of the UD-mat reinforcement. The mat phase has a relatively small influence on the measured mechanical properties.

Table 3- Tensile properties of non-impacted composites

	E_1 [GPa]	E_{theory} [GPa]	σ_R [MPa]	ϵ_R [%]
FUM0	26.1 ± 0.4	24.2	311 ± 13	1.90 ± 0.13
GUD0	32.0 ± 0.4	29.7	838 ± 45	2.85 ± 0.19
FUM090	15.1 ± 0.4	15.5	172 ± 3	1.85 ± 0.09
GUD090	21.7 ± 1.2	19.9	438 ± 39	2.71 ± 0.03
FGUD090	15.5 ± 0.3	17.7	230 ± 5	2.09 ± 0.08

By dividing the properties by the composite density, to obtain the specific properties, the difference between the two types of composites is reduced (Fig.3). The flax/epoxy composites compare favorably with glass/epoxy composites in terms of specific modulus, but the specific strength in cross-ply remains significantly higher for the glass/epoxy laminates and for the hybrid composites (88% and 20% higher for the specific strength in GUD090 and FGUD090, respectively).

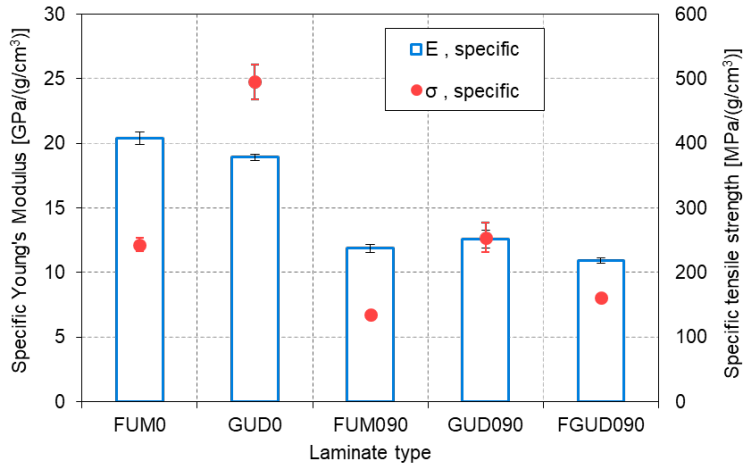


Figure 3- Specific Young's modulus and specific strength of the composites

Impact test results

The load-displacement curves at an impact of 5J are shown in Figure 3 for the UD laminates (Fig. 4a) and the cross-ply laminates (Fig. 4b) which also include the hybrid laminate. Each curve shows the load and unload parts of the test, forming a closed loop with the final residual displacement at the end of the test. It should be noted that all curves have a closed loop shape, meaning that no tested sample was completely perforated. The loop shapes of the flax fiber composites are always wider than those of glass fiber composites, regardless of the stacking sequences. The loop shape of the cross-ply hybrid composite lies between that of the flax and glass cross ply composites. Flax fiber composites exhibit a plateau where the load stabilizes almost at a constant value for about 2 mm. The curves of the glass fiber composites do not show this stabilization. According to Panciroli and Giannini [8], this plateau indicates that fiber breakage is taking place in the material. The average peak load in the unidirectional flax fiber composite is lower than in its cross-ply configuration, showing the benefit of bidirectional layup (see Table 4). The lowest impact load (2443 ± 41 N) is observed in the hybrid composite compared to other configurations. The impact time remains almost unchanged, and the permanent displacement is highest in the flax fiber composites. The energy absorption ratio, calculated by dividing the absorbed energy (the area inside the loop of the load-displacement curve) by the impact energy, are also shown in Table 4.

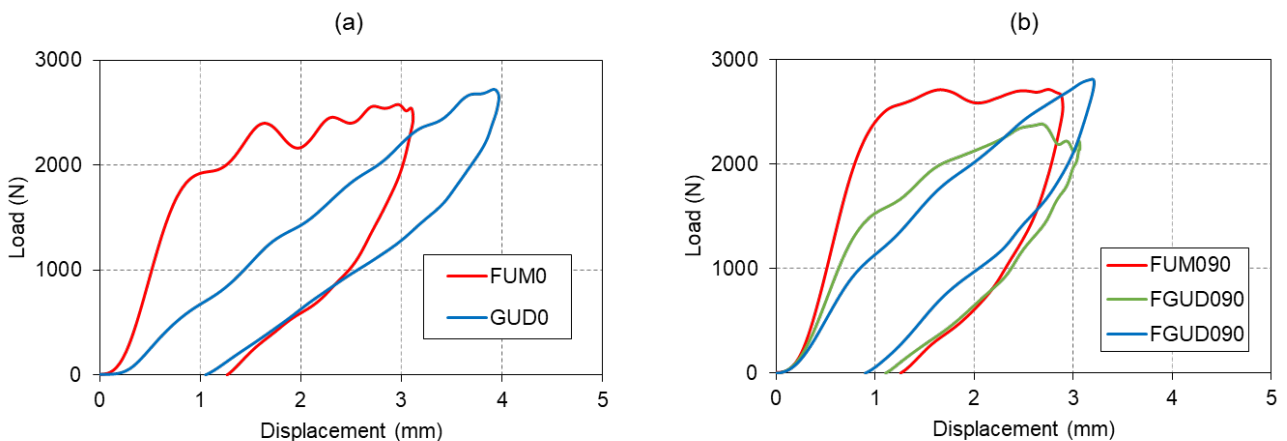


Figure 4-Load-displacement curves of the composites impacted at 5J: (a) UD laminates and (b) cross-ply laminates

Flax fiber/epoxy composites absorb more energy than glass fiber composites, for both stacking sequences. The energy absorption of the hybrid composite lies between that of glass-only and flax-only composites. For a given type of composite, the energy absorption ratio of the UD is lower than that of the cross-ply configuration. A higher energy absorption is related to the damage mechanisms

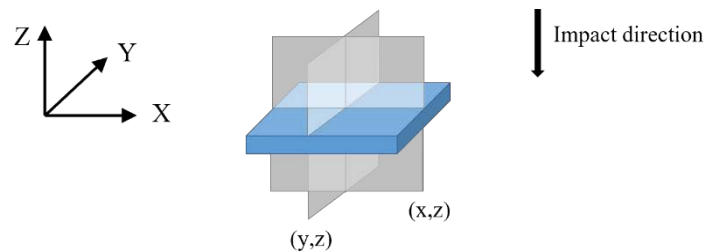
occurring in the specimens such as matrix cracks, delamination and fiber breakage which can be observed by micro-CT scans.

Table 4- Impact properties of the composites

Impact energy [J]	Peak load [N]	Impact time [ms]	Permanent Displacement [mm]	Energy absorption ratio [%]
FUM0	2566 ± 11	8.89 ± 0.29	1.21 ± 0.06	73.0 ± 0.3
GUD0	2777 ± 40	10.19 ± 0.19	1.07 ± 0.11	49.2 ± 2.5
FUM090	2738 ± 124	7.92 ± 0.26	1.29 ± 0.05	76.6 ± 0.6
GUD090	2842 ± 57	9.09 ± 0.16	0.90 ± 0.03	52.0 ± 1.0
FGUD090	2443 ± 41	9.48 ± 0.04	1.10 ± 0.01	67.7 ± 0.2

Impact damage observation by micro-CT and visual inspection

Figure 5 shows 3D reconstructed micro-CT images and the corresponding photographs of UD and cross-ply flax laminates impacted at 5J. Figure 5a reveals delamination, a bending crack on the rear face oriented parallel to the fiber direction and fiber breakages. The latter case was predictable since the tensile stress is highest at this location. In Figure 5b for the cross-ply composite, delamination is more pronounced than in the UD case (Fig. 5a). Damage in cross-ply laminates are also generally conical in shape due to delamination and transverse cracks. The different damage mechanisms contribute to dissipating energy in these flax/epoxy composites [8], consistent with the observations of Lepaupin et al. [9]. Micro-CT scans highlight significant damage inside the flax fiber composites impacted at 5J, while a simple visual inspection of the back side only reveals few visible damage.



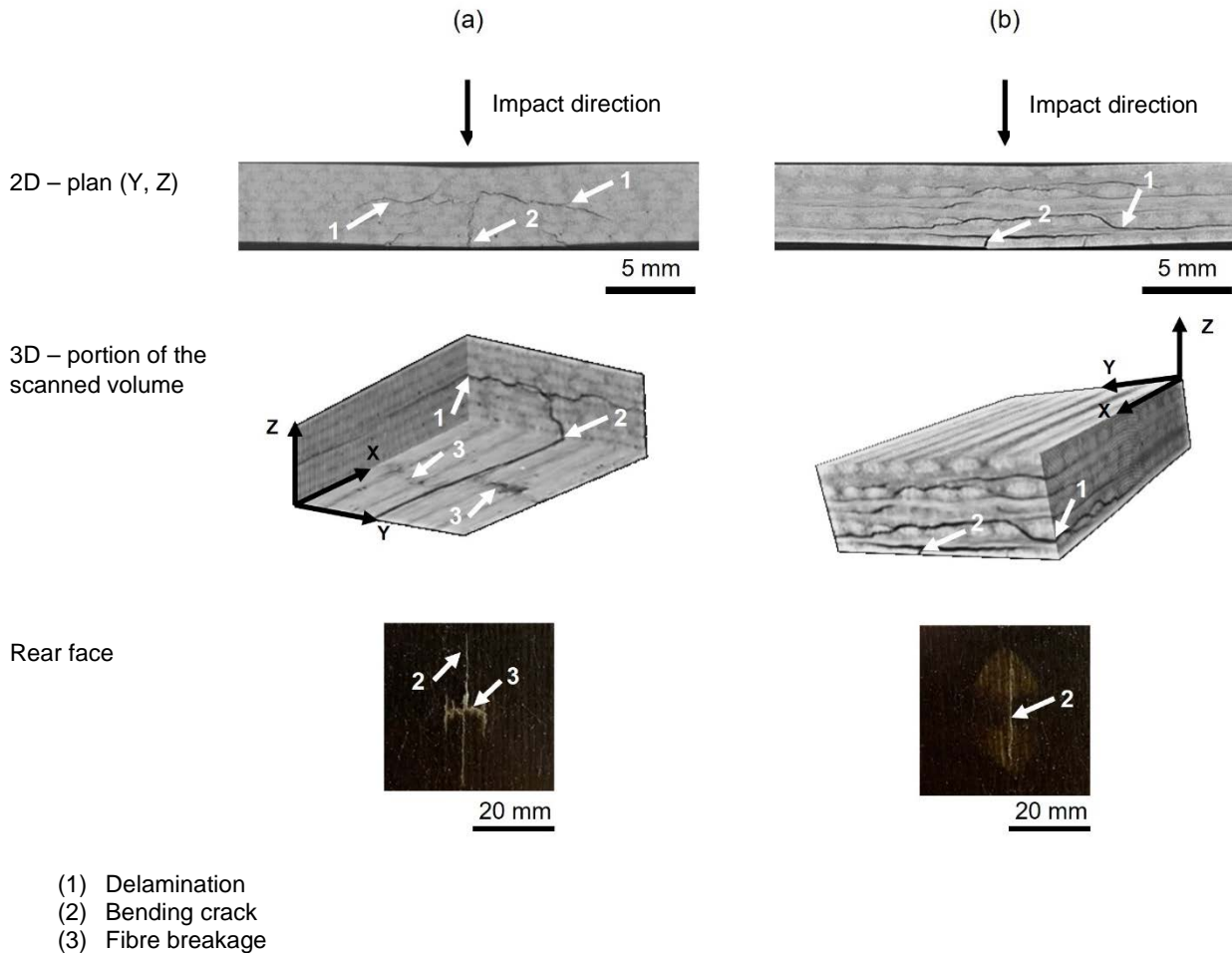


Figure 5 - 2D et 3D views of damage detected by micro-CT and visual inspection after an impact at 5J (a) unidirectional Flax UD-mat FUM0, (b) Flax UD-mat cross-ply FUM090

Figure 6 shows micro-CT scans and photographs of the damage observed in the impacted cross-ply hybrid composite in the front and rear faces. In Figure 6a on the impacted face, little damage is visible. On the rear face, however, the damage has a general butterfly shape delimiting the delamination between the two lower plies as well as a bending crack and fiber fractures. The micro-CT scans (Fig. 6b) show several delamination and bending cracks in the two bottom plies. Matrix and fiber cracks adjacent to these plies are observed in the X-Z plane, as well as a delamination between flax fiber and glass fiber plies.

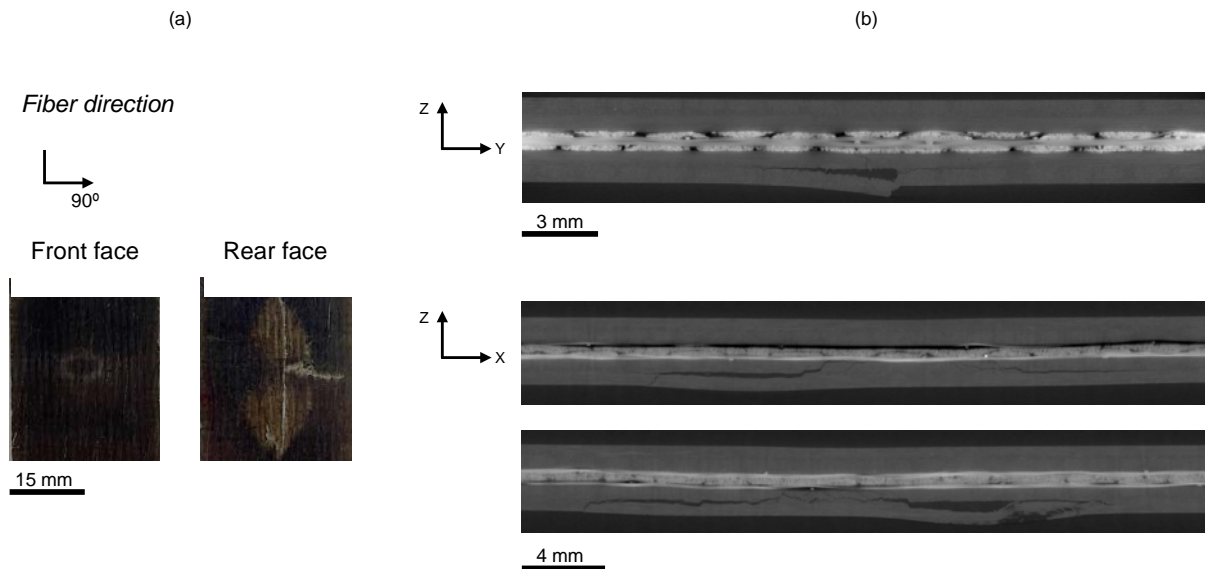


Figure 6. Images of damage of a hybrid composite FVUD90 impacted at 5J: (a) visual inspection and (b) micro-CT images in the Y-Z et X-Z planes.

It should be noted that due to the transparency of the glass fibers/epoxy composites, there are no differences between the damages observed on the front and rear faces of the samples (Fig. 7). The main visible damage are matrix cracks and delamination in accordance with the literature [10,11]. The horizontal lines are the weft threads in the unidirectional glass fabric.

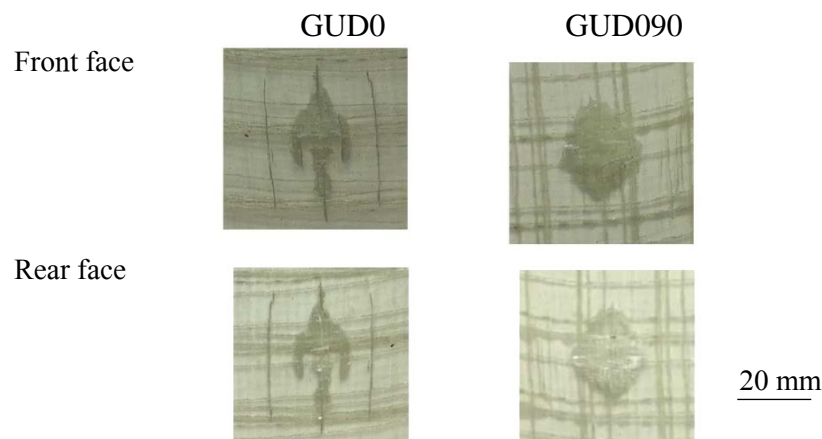


Figure 7- Visible transparency damage in the glass fibers /epoxy composites impacted at 5J.

Residual tensile properties of impacted composites

The stress-strain curves of the impacted composites (solid lines) are compared to those of the non-impacted specimens (dashed lines) in Figure 8. It is shown that the tensile properties of flax fiber composites are strongly influenced by the impact, unlike the glass fiber composites which are almost unaffected regardless of the fiber stacking sequences. The degradation of tensile properties is also detected in the hybrid composite, in particular for the tensile strength and fracture strain due to the induced damage after impact. The residual properties are summarized in Table 5 for the different laminates.

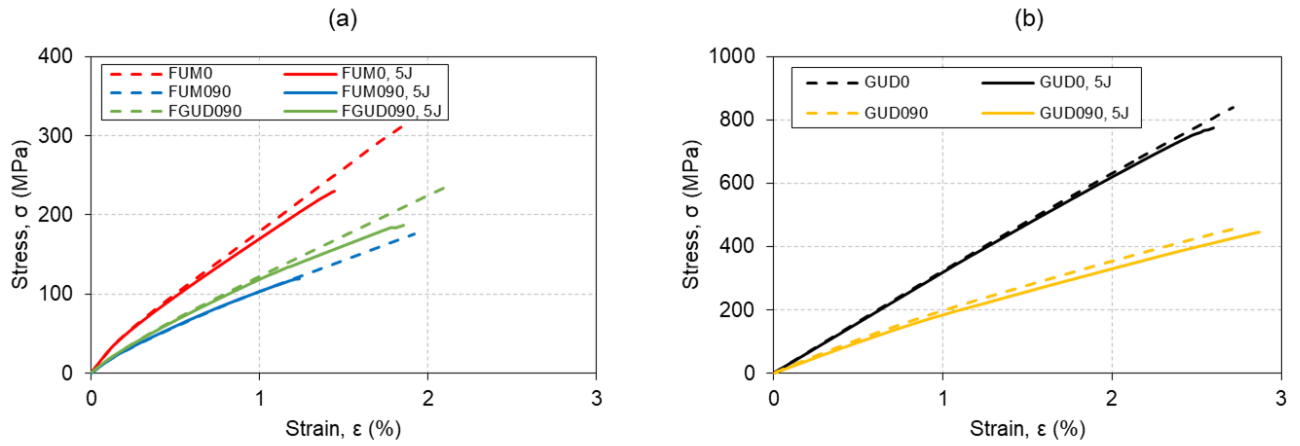


Figure 8 - Stress and strain curves in tensile tests of the composites before and after an impact of 5J (a) Flax fibers /epoxy composites, (b) Glass fibers/epoxy composites.

The elastic moduli of flax fiber composites increase slightly after impact: +3% for FUM0 and +5% for FUM090. It is almost unaffected in the unidirectional glass fiber GUD0 and hybrid composites. On the other hand, the cross-ply glass fiber composite shows a significant reduction of 11% compared to non-impacted specimen. The degradation can be explained by the strong delamination created by the impact in the GUD090 (Fig. 7). The impact of 5J produces notable decreases of tensile strength and failure strain in the flax fiber and hybrid fiber FGUD090 composites, but these decreases in hybrid laminates are lower than in 100% flax fiber laminates, in particular regarding the failure strain. According to Selver et al. [12], placing flax fiber plies on the faces of the laminate (as external plies) reduces the damage in the glass fiber plies of a hybrid laminate. After impact, the impacted tensile properties of the hybrid composite (Table 5) are similar to those of non-impacted flax/epoxy composite. Otherwise, these properties are very little influenced by the impact in glass fibers/epoxy composites.

Table 5 - Residual tensile properties of the impacted composites

	FUM0	FUM090	GUD0	GUD090	FGUD090
E_{5J} [GPa]	27.0 ± 0.6	15.8 ± 0.3	31.7 ± 0.3	19.2 ± 0.8	15.2 ± 0.7
ΔE [%]	+3%	+5%	-1%	-11%	-2%
$\sigma_{R,5J}$ [MPa]	232 ± 20	123 ± 6	797 ± 14	432 ± 27	181 ± 8
$\Delta \sigma$ [%]	-25%	-28%	-8%	-1%	-21%
$\epsilon_{R,5J}$ [%]	1.43 ± 0.12	1.25 ± 0.10	2.61 ± 0.02	2.93 ± 0.21	1.86
$\Delta \epsilon$ [%]	-25%	-32%	-8%	+8%	-11%

Conclusion

Flax fibers have demonstrated a strong potential as reinforcement in composites for industrial applications, thanks to their specific properties that are competitive to those of glass fibers. However, to combine safety, performance and environmental virtues, it is necessary to understand the mechanical performances of flax fiber composites under real conditions such as dynamic loadings. In this work, three types of composite laminates were investigated under uniaxial tension before and

after impact at a low energy of 5J, flax fibers, glass fibers and a hybrid composite. Impact results show that the cross-ply laminates exhibit a higher impact resistance and a lower permanent displacement than the unidirectional laminates. The internal damage of flax fiber composites, detected by CT-scan analysis, highlight damages such as delamination, bending cracks and fiber fracture forming a conical distribution in the cross-ply laminate. This resulted in flax fibers/epoxy composite having a higher energy absorption capacity than glass fibers/epoxy composites, which are mainly affected by matrix cracks and delamination. The post impact tensile tests show tensile strength decreases of 25% and 28% for unidirectional $[0]_8$ and cross-ply $[0/90]_{2s}$ flax/epoxy composites respectively, while the strength remains relatively stable in glass/epoxy composites. Finally, hybridization could provide an interesting solution for engineering applications requiring intermediate energy absorption with a low degradation of the tensile properties.

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