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IMPACT AND POST-IMPACT DAMAGE CHARACTERISATION OF HYBRID COMPOSITE LAMINATES BASED ON BASALT FIBRES IN COMBINATION WITH FLAX, HEMP AND GLASS FIBRES MANUFACTURED BY VACUUM INFUSION

R. Petrucci¹, C. Santulli², D. Puglia¹, E. Nisini³, F. Sarasini⁴, J. Tirillò⁴, L. Torre¹, G. Minak³, J.M. Kenny¹

¹ Università di Perugia, Civil and Environmental Engineering Dept., Materials Science and Technology, strada di Pentima 4, 05100 Terni, Italy

² Università degli Studi di Camerino, School of Architecture and Design, viale della Rimembranza, 63100 Ascoli Piceno, Italy

 ³ DIEM Alma Mater Studiorum, Università di Bologna, viale Risorgimento 2, 40136 Bologna, Italy[;]
⁴ Sapienza Università di Roma, Dept. of Chemical Engineering Materials Environment, via Eudossiana 18, 00184 Roma, Italy

ABSTRACT

The impact and flexural post-impact behaviour of ternary hybrid composites based on epoxy resin reinforced with different types of fibres, basalt (B), flax (F), hemp (H) and glass (G) in textile form, has been investigated, namely FHB, GHB and GFB. The reinforcement volume employed was in the order of 21-23% throughout. Laminates based exclusively on basalt, hemp and flax fibres were also fabricated for comparison. Hybrid laminates showed an intermediate performance between basalt fibre reinforced laminates on the high side, and flax and hemp fibre reinforced laminates on the low side. As for impact performance, GHB appears to be the worst performing hybrid laminate and FHB slightly overperforms GFB. In general, an increased rigidity can be attributed to all hybrids with respect to flax and hemp fibre composites. The morphological study of fracture by SEM indicated the variability of mode of fracture of flax and hemp fibre laminates and of the hybrid configuration (FHB) containing both of them. Acoustic emission monitoring during post-impact flexural tests confirmed the proneness to delamination of FHB hybrids, whilst they were

able to better absorb impact damage than the other hybrids.

KEYWORDS: A. Hybrid; A. Polymer matrix composites (PMCs); B. Impact behaviour; D. Acoustic emission; Basalt fibres

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Corresponding author, e-mail: carlo.santulli@unicam.it

INTRODUCTION

In recent years, there has been substantial interest on the application of basalt fibres as the reinforcement of polymer matrices. These can be proposed as a replacement for glass fibres in view of their advantages in terms of environmental cost relative to their chemical and physical properties. Basalt fibres are continuously extruded from a high temperature melt (around 1500°C) of selected basalt stones (volcanic, over-ground, effusive rocks saturated with 45–52% silica) [1]. In particular, their similar chemical structure to glass, even though their density is slightly higher (2.8 g/cm³ compared to 2.54 g/cm³ of glass), eases such a replacement. Also, the chemical stability of the basalt fibres is higher than that of glass fibres, especially in an acidic environment [2]. This characteristic allows basalt fibres to more effectively binding sizing agents, such as organosilanes, resulting in the need for a reduced amount of these chemicals with respect to glass fibres [3]. Even more than glass, basalt fibres can be used in a wide range of temperatures, from -200°C to +600°C [4].

The above mentioned similarity led to attempting the hybridisation of glass and basalt fibre laminates, aiming at tailoring to needs their mechanical properties. In particular, continuous basalt fibre has a not very different content in silica and alumina from glass fibres and also a comparable, if not superior, tensile strength [5]. It was also noted that basalt fibre reinforced laminates and intercalated basalt-glass hybrid laminates exhibited better impact energy absorption capability when compared to glass fibre reinforced laminates and glass/basalt sandwich hybrid laminates with glass fibres serving as reinforcement of the core and basalt fibre as reinforcement of the skin [6].

In an attempt to produce more sustainable materials, reducing carbon footprint during production and at their end-of-life, retaining nevertheless sufficient mechanical resistance and particularly an acceptable impact performance, glass/plant fibre hybrid laminates have often been considered [7-8]. The interest of this procedure has been recently confirmed by a study on hybrid composites with different respective amounts of glass and flax fibres, though always summing to a 67 vol.%, which indicated the presence of a remarkable bridging between flax fibres, flax yarn and glass fibres with benefits for interlaminar shear strength and interlaminar fracture toughness [9].

As regards impact properties, different works have been carried out, which suggest that the most effective performance is achieved whenever glass fibre laminates are used as skins and plant fibre laminates as core [10]. Other configurations can be possibly produced, such as those including intercalated layers of plant and glass fibre laminates in the core, retaining glass fibre laminates as skins: the adoption of this type of stacking sequence results in a more gradual degradation of the laminate, when subjected to impact loading [11].

An option that has been neglected so far is the possibility to produce hybrids including plant fibre and basalt fibre laminates. A recent study, which the present follows in view of its completion, concentrated on the manufacturing of hybrids made using basalt fibre composites as the core laminates, in particular introducing, as skins, glass/flax, glass/hemp and hemp/flax laminates [12]. In general terms, the results indicated that hybrid configurations including hemp fibres had a lower quality interface.

In this study, the same hybrids examined in [12] are now evaluated for their impact and post-impact properties, assisted this time by acoustic emission monitoring in the characterization of their post-impact flexural behaviour, a possibility that was explored already with promising results in a number of studies on glass/plant fibre composites [10-11].

MATERIALS AND METHODS

1. Composite manufacturing

The resin system used was an EC360 (resin)/W160 (hardener) epoxy, provided by Elantas Camattini s.p.a. The reinforcements based on flax, hemp, and the basalt grid were all provided by Fidia Srl, while the glass mat has been provided by G. Angeloni Srl.

All the composite panels have been produced by means of a vacuum infusion process, which has been described already in [12]. For this purpose, for each configuration, a stack of dry reinforcement plies has been laminated over a glass mould. After the lamination stage, the layout has been completed with the flow media and the infusion network, and finally the mould has been sealed with the vacuum bag. The curing stage of the resin, after the infusion is concluded, has been carried out at room temperature, while the post curing took place in an oven in two sub-stages: at 60°C for three hours and subsequently at 80°C for four hours. The in-mould pressure measured during the infusion stages was equal to 20 mbar. Vacmobiles model 20/2 vacuum pump, provided by Vacmobiles Europe by Filter Technics, has been used.

Rectangular 260 mm x 450 mm sheets have been obtained. Subsequent cutting operations have been carried out with the aim to produce specimens suitable for mechanical characterization, in accordance to the related standards.

Three laminates were based on hemp, flax and basalt fibres respectively, which were used for comparison with the three hybrids, similar to those produced in [12] and lettered with the same notation, which are summarised below.

The measured surface densities of the flax textile, of the hemp textile and of the basalt textile were equal to 292, 234 and 242 g/m², respectively. All these reinforcements have a balanced configuration on warp and weft yarns. Finally the glass mat surface density was equal to 100 g/m^2 .

The following stacking sequences have been employed:

• Hemp, flax and basalt laminates have all been produced by stacking together 4 layers of biaxial textiles, according to a $[0/90]_{2S}$ lay-out. Fibre volume fraction was 20.16 (± 1.37)% for hemp; 24.82 (±0.83)% for flax; 28.23 (±0.85)% for basalt.

• GFB laminate (stacking sequence GFBBFG): total fibre volume fraction equal to 21.18 (± 0.32) %, of which flax 11.72 (± 0.18) %, basalt 7.16 (± 0.11) % and glass 2.30 (± 0.04) %;

• GHB laminate (stacking sequence GHBBHG): total fibre volume fraction 22.53 (± 0.48)%, of which hemp 8.56 (± 0.16)%, basalt 11.38 (± 0.21)% and glass 2.59 (± 0.05)%;

• FHB laminate (stacking sequence FHBBHF): total fibre volume fraction 22.63 (± 0.36)%, of which flax 9.11 (± 0.20)%, hemp 7.95 (± 0.12)% and basalt 5.57 (± 0.05)%.

For all hybrids, the fibre direction into each textile network is 0/90.

2. Impact testing

Impact tests were carried out using an in-house built falling weight impact tower on square samples with planar dimensions equal to 100x100 mm. The impactor diameter was 12.7 mm and its mass was 1.25 kg. Sampling frequency of the signals was 100 kHz with no external filtering.

The variables measured on the falling weight tower were:

- Contact force, measured using a piezoelectric load-cell for dynamical loading;
- Velocity, measured through elaboration of the signal supplied by a Laser sensor, placed about 50 mm above the quote of the sample;
- Impactor speed and position as a function of time obtained by double numerical integration of force signal (as suggested by ASTM D7136-12 standard).

Samples are supported between two steel plates with a central circular opening (diameter 76 mm), realised in accordance with ASTM D3763-10 standard, at the centre of which the impact event takes place. The bolts constraining the two plates are serrated by hand till they allowed no movement of the laminates.

Keeping the mass constant at 1.25 kg, this was dropped from a height of 3 meter, resulting in an impact energy of 36.75 Joules, which was deemed sufficient to allow for full penetration of the laminates. The energy absorbed from these samples was obtained from impact hysteresis cycles (Force vs. displacement), measuring the total area under the curve and was considered equal to the penetration energy for the laminates. To compare different materials, the energy value obtained was then normalised dividing it by the section of the sample which is left free to deform, equal to d*t, where d is the diameter of the central circular opening in the fixture and t is the laminate thickness. This is a common procedure adopted in IFW testing of composite materials [13-14]. Once established the penetration energy, all laminates were impacted from a height of 1 meter, therefore with an energy of 12.25 Joules, which was considered sufficient to produce some significant damage to the laminate without resulting in its penetration. In practice, this energy resulted in excessively significant damage for some laminates, as detailed and explained below, in which case impact loading from a reduced height of 0.5 meter was performed, resulting in impact energy of 6.12 Joules.

3. Post-impact flexural testing monitored with acoustic emission

Specimens for mechanical characterization were obtained both from impacted and from nonimpacted plates: three-point flexural tests, in accordance with ASTM D 790, were performed. The length of the specimens was selected in order to enable the placement of two acoustic emission sensors at their extremities, whilst their width needed to be sufficient to fully contain the damaged area due to the low-velocity impact. These tests were performed in a Zwick/Roell Z010 universal testing machine equipped with a 10 kN load cell using a fixed support span length of 40 mm, a cross-head speed of 1.7 mm/min and a pre-load of 3 N. Five impacted and five non-impacted specimens were tested, the latter serving as reference materials. The impacted surface was consistently in compression during bending loading of all laminates. It is noteworthy that the support span was in [12] equal to 16 times the laminate thickness, while in this study a fixed span length of 40 mm was applied, as the consequence of the presence of impact damage, which suggested that a sufficient span was needed for all laminates to be much greater than the delamination area surrounding the impact point. In this way, the whole of the damaged area was included inside the region delimited by the support span and therefore subjected to flexural loading. As a consequence, flexural tests results reported here for non-impacted laminates are different from those reported in [12].

The monotonic flexural tests were monitored by acoustic emission until final fracture occurred, using an AMSY-5 AE system by Vallen Systeme GmbH. The AE acquisition settings used throughout this experimental work were as follows: threshold = 35 dB, RT (Rearm Time) = 0.4 ms, DDT (Duration Discrimination Time) = 0.2 ms, and total gain = 34 dB. Two broadband (100–1500 kHz, Fujicera 1045S) PZT AE sensors were used. The sensors were placed on the surface of the specimens at both ends to allow linear localization, using silicone grease as coupling agent.

4. SEM characterisation

The fracture surfaces of the specimens were investigated using a scanning electron microscope (Philips XL40) and a field emission (FE) SEM (Zeiss, Auriga). All specimens were sputter coated with gold prior to examination.

RESULTS AND DISCUSSION

Flexural tests carried out on the different materials gave the results that are reported in Table 1, from which it is apparent a clear hierarchy in terms of flexural performance, that is GFB>FHB>GHB. In Figure 1 typical flexural stress-strain curves are reported for all the materials configurations, which show that the two hybrid configurations including glass fibres (GFB and GHB) present a more gradual failure process, not breaking at the reaching of the maximum load, which is the case instead for FHB, as well as for basalt fibre laminates (B). As a different span was used, the comparison with the flexural results described in [12] may appear questionable. However, some general observations are worthy pointing out: in particular, the superior performance of GFB among the three hybrid configurations was already revealed in [12]. Also, it appears rather surprising here that the two hemp-based hybrids, GHB and FHB, show a performance in the order of that of the hemp fibre reinforced laminates (H), or even inferior, which wasn't the case in [12]. It needs to be observed that a shorter span concentrates flexural loading in a smaller region, which may be detrimental whenever interlaminar strength of the composite is limited: this might be the case for GHB and FHB, as shown by the ILSS results in [12], where in particular GHB was clearly inferior to H laminates.

To investigate whether impact damage does markedly change this picture, especially as regards the hierarchy of mechanical performance, falling weight impact tests have been carried out. Impact at 36.75 J, which enabled full penetration of all the laminates, allowed determining the level of energy absorbed, normalised, as explained above, to take into account the different thickness. Normalised penetration energy data for all configurations are reported in Figure 2. In the case of impact, while GHB continues to be the worst performing hybrid laminate, here FHB slightly overperforms GFB. Note also that the hybrids show a quite larger standard deviation with respect to pure laminates.

Some indications are supplied by the mode of failure of penetrated laminates, which is shown in Figure 3: it appears that in all configurations where hemp and/or flax fibres are present, the penetration shows an obvious directionality: in other words, only in the basalt fibre reinforced laminates the hole produced by penetration is quasi-circular. In other cases, they have a pronounced cross-like feature (hemp, flax and FHB) or an elongated shape (GHB and GFB), the latter being likely to be connected to the effect of glass fibres introduction on impact damage. It is suggested that the regularity of glass fibre laminates structure does result in an enhanced directional propagation of impact damage in the rest of the hybrid material: similar observations have been performed on abaca/jute/E-glass hybrids [15]. In contrast, cross-like features of impact damage on woven hemp fibre laminates have already been observed in [16].

After the aforementioned measurement of impact penetration energy, impact testing at lower energies was also carried out, aimed at the evaluation of flexural post-impact properties. This is a procedure widely followed to allow on one side obtaining information on the residual performance of laminates, while on the other side it enables using acoustic emission as a real-time monitoring technique to assist damage characterisation: it has been used already in a number of instances, in particular, considering works whose aim is closer to the present one, in [5.6]. The idea in this case was applying energy sufficient to produce substantial damage without coming too close to penetration. Based on data presented in Table 2, and considering the different thickness of the laminates, it was suggested that impact at 12 Joules did not result in complete penetration for basalt, GFB and FHB laminates, and it was close to penetration for flax fibre reinforced laminates. Hemp fibre laminates and GHB were impacted at a lower energy, equal to 6 Joules, to allow easier comparison of data among these two configurations, since impact at 12 Joules on hemp resulted in penetration. The severity of damage produced by impact et the different energies was evaluated from the patterns observed on the rear surface of the laminates, which are depicted in Figure 4.

Impact hysteresis cycles at relevant energies (6 or 12 Joules, depending on the laminates), reported in Figure 5a, indicate that the laminates reinforced with plant fibres, namely F and H, are clearly more prone to deformation than all the hybrids and the B laminates. Impact penetration curves, from impact at 36.75 J and measurement of absorbed energy from the area under the load vs. deflection curves, show that the hybrids are clearly superior, the best one being FHB, where both plant fibres are coupled, so that possibly their mutual locking during impact loading can provide some additional energy absorption. This is confirmed by the linear stiffness values (Figure 6), which are calculated in N/mm from the slope of the quasi-linear part of the impact curve i.e., until the peak load is reached, then normalised by the thickness of the laminate, expressed in mm, so that finally a value in (N/mm)/mm is obtained: this procedure was followed already, e.g., in [16-17] on hemp fibre laminates. In Figure 6, the values of normalised linear stiffness for hybrids are quite obviously intermediate between those for B laminate (on the higher side) and F and H laminate (on the lower side). Among the three hybrids, the worst performance is offered once again by the GHB laminate, whilst in this case the GFB is superior to the FHB one. This suggests that glass fibre laminates confer to the GFB, when compared with FHB, an improved rigidity during nonpenetrating impact, though leading to collapse for a lower value of impact energy, as from data in Table 2. This indicates that the presence of glass fibre laminates results generally in a higher damage tolerance, although once a given threshold for unrecoverable damage is exceeded, final failure is much more abrupt than it is the case for hybrids not containing glass fibre laminates. This may be related to the pull-out and tear-off of vegetable fibres in flax and hemp fibre laminates, which the basalt fibre reinforced laminates in the core of the hybrids are not fully able to hinder, while they contribute as a whole to the improved impact performance of the hybrid laminate. In other words, the controlling factor for resistance is offered by the non uniform structure of flax and hemp fibre laminates. On the other side, the FHB laminates show higher penetration energy: this can be explained by the fact that the irregular structure of flax and hemp laminates, once locally compressed during impact loading, is beneficial for the possible mechanical locking between the 2-D fabric and the matrix of the adjacent layers, which in turn increases the resistance to delamination. It can be noticed that, among vegetable fibres, flax and hemp present a significant effect of mechanical locking, due to their limited porosity, which hinders penetration of the matrix under their surface [18].

The increased rigidity offered by the presence of glass fibres is revealed also by residual flexural properties, reported in Table 3, which on the other side appear to form a sounder interface with flax fibre laminates than with hemp fibre ones. An action of impact damage dissipation on glass/flax

fibre hybrids has been observed already elsewhere [19]. In this regard, excluding the F and H laminates that basically did not offer any residual performance after impact, the superiority of the GFB laminate over other hybrid laminates can be noted.

Acoustic emission monitoring of post-impact flexural tests, which is a well known procedure to assess the global amount of damage produced by the impact event on the laminates, is particularly aimed at comparing different types of configurations: this proved effective on laminates with various types of fibre reinforcement [20]. In particular, it can be noticed that high amplitude events are mostly present in the impacted configurations including flax, namely FHB and GFB (Figure 7). This might suggest that more fibres fail as a consequence of the flexural loading subsequent to impact in these laminates, whilst possibly hemp fibres tend to be more subjected to fibrillation than being fractured in a fragile way. Fibrillation, which has been widely observed, as from SEM micrographs presented below, has controversial effects: on one side, it facilitates wetting of the fibres by the resin, thereby increasing the contact area [21], whilst on the other side the single filaments are weaker than the whole fibre, so that they are more easily displaced and fractured by increasing stress [22]. Considering AE localisation plots during tensile loading (Figure 8) it can be noticed that acoustic emission has a much earlier appearance, therefore at low loads, in the case of FHB laminates, and events appear much more concentrated than in the case of the other hybrids around the geometrical centre of the flexural beam (abscissa x = 5 cm approximately). This is likely to indicate that the effect of flexure of the laminate is much more pronounced, in other words the absence of glass fibres eases bending of FHB laminates, although because of the larger deflection achievable with the combination of flax and hemp fibres, this does still result in a performance superior to GHB laminates. In general, the improvement of rigidity was clearly demonstrated by the addition of outer layers reinforced with basalt fibres to glass fibre reinforced laminates [23], while it had been suggested elsewhere that the production of intercalated hybrids including layers in a asymmetrical configuration, such as BGBG, though in general terms less performing, would result in more controllable crack propagation after impact [10-11]. It may be suggested that this effect, alternative to bare intercalation of plant fibre composites with glass (or basalt) fibre composites, may be possibly obtained also by a suitable combination of flax, hemp and glass fibres with basalt fibres, which will obviously result in a higher technological complexity in laminate production.

SEM micrographs of flexural fracture surfaces in hemp fibre layers of GHB laminates (Figure 9) confirm the diffuse presence of fibrillation (particularly to the centre-right region of Figure 9a) and widespread debonding (Figure 9b) after flexural loading with limited fibre-matrix adhesion (hemp

fibres appear quite clean after debonding). This raises some concern on the interfacial strength of these laminates. It is important to note that a common issue with hemp fibres, as represented in Figure 10, is that fibrillation is an indication of a considerable variety in terms of mode of fracture for the fibres themselves. Problems are quite different in the case of GFB laminates, where fibrillation does appear to be much rarer, but on the other side flax fibre layers appear to be considerably bent and displaced in the laminate during loading, leading to diffuse damage (Figure 11). As a matter of fact, obvious fractures were observed in particular between glass fibre and flax fibre layers in GFB laminates: this can be explained as an effect of the higher flexural stresses this hybrid configuration is able to withstand (Figure 12).

In general, the manufacturing of ternary hybrid reinforced laminates including basalt fibre laminates in the core and two out of three of glass, flax and hemp fibres on the more external layers, therefore producing three configurations, namely GHB, GFB and FHB, provided sufficient resistance to impact and an acceptable post-impact performance with some weight gain with respect to basalt fibre laminates. Some reason for concern remains nonetheless, related particularly to the unpredictable mode of fracture of flax and hemp fibres and to the interfacial strength of the layers including these fibres.

CONCLUSIONS

The impact and flexural post-impact behaviour of different configurations of ternary hybrid composites based on epoxy resin with a core including a basalt fibre laminate and different types of fibres, basalt (B), flax (F), hemp (H) and glass (G) in textile form, have been investigated.

A clear hierarchy in terms of flexural performance is apparent, that is GFB>FHB>GHB: this is preserved also in the case of post-impact flexural loading, suggesting that the addition of glass fibre reinforced laminates offers a much better result in presence of flax fibres than in presence of hemp fibres. This has been explained by the greater tendency to fibrillation and the inherent irregularity of textile structure in the case of hemp fibres. Passing to impact properties, it is worth noting that among the hybrids the best performance in terms of penetration energy is offered by the FHB laminates, possibly as the result of an effective mechanical locking between the layers. However, this higher resistance to penetration is offered at the expense of a larger deformation of the laminate, as demonstrated by the linear stiffness data, obtained from impact hysteresis cycles, where the hierarchy obtained is in contrast GHB>GFB>FHB: in general terms, the absence of glass fibres allows the laminate being more easily bent.

This study indicated the suitability of using basalt fibre laminates as the core of more complex laminates, including different fibres, in which impact performance is reduced less in comparison with the weight gain obtained. However, it has indicated a number of critical conditions, in particular the limited quality of flax and particularly hemp plain weave textile structures, which appear to provide an interesting resistance to penetration, but on the other side are quite unpredictable in terms of fracture behaviour. In principle, various routes for improvements could be proposed. The availability of more punch-resistant textile structures, such as e.g., flax and hemp satin, would improve considerably that picture. Another cause for concern is the improvement of interfacial properties of layers including flax and hemp laminates, which could be obtained e.g., by treatments of the fibres tailored for their use in laminates. Future developments would also possibly include the adoption of matrices with higher degree of compatibility with plant fibres, such as for example phenolics or starch-based or oil-based biopolymers.

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Table 1 Flexural data (avg. and standard deviation) and dimensions of non impacted laminates
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Laminates	Flexural strength	Flexural modulus	Dimensions (mm)
	(MPa)	(GPa)	(l×w×t)*
Basalt (B)	339.01 ± 30.15	12.55 ± 0.86	$100 \times 32 \times 2$
Hemp (H)	188.26 ± 15.27	7.45 ± 0.74	$100 \times 32 \times 3.7$
Flax (F)	142.87 ± 12.73	4.77 ± 0.26	$100 \times 35 \times 3.2$
GHB	149.92 ± 12.34	5.30 ± 0.35	$100 \times 33 \times 3.2$
GFB	271.71 ± 24.31	10.12 ± 0.81	$100 \times 35 \times 3.9$
FHB	215.65 ± 26.27	5.98 ± 0.25	$100 \times 36 \times 3.4$

 $*length \times width \times thickness$



Figure 1 Flexural stress vs. strain curves for all configurations as-received





FHB

GHB

GFB

Figure 3 Back surfaces of penetrated laminates

Laminate	Thickness	Impact penetration energy
	(mm)	(J)
В	2	16.7 ± 1.1
Н	3.7	8.2 ± 0.9
F	3.2	12.4 ± 1
GHB	3.2	14.3 ± 1.7
GFB	3.9	19.8 ± 2
FHB	3.4	25.1 ± 2.4

Table 2 Predicted average penetration energies for the laminates



FLAX (12 J)

HEMP (6 J)

GHB (6 J)





Figure 5a Impact hysteresis cycles for all laminates



Figure 5b Impact penetration curves for all the laminates



Laminate	Impact energy	Flexural strength	Flexural modulus
	(J)	(MPa)	(GPa)
В	12	246.20 ± 21.89	11.00 ± 0.53
FHB	12	124.41 ± 10.73	3.40 ± 0.29
GFB	12	218.55 ± 22.55	8.23 ± 0.74
GHB	6	109.71 ± 9.87	4.43 ± 0.24
F	12	6.08 ± 0.94	0.45 ± 0.13
Н	6	18.68 ± 3.61	10.21 ± 0.49

Table 3 Post-impact flexural performance of the laminates



Figure 7 AE hits localisation vs. amplitude for the different hybrid laminates, non-impacted and impacted (the positions of the sensors are marked with S)



Figure 8 AE hits localisation vs. tome for the different hybrid laminates, non-impacted and impacted (the positions of the sensors are marked with S)



Figure 9 Fracture surface of GHB laminates (hemp fibre layers are shown)



Figure 10 Fractured hemp fibres in FHB laminates



Figure 11 Fracture surface of flax fibre reinforced laminates



Figure 12 Fracture surface of GFB laminates at the interface between glass (top) and flax fibre layers (bottom)