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Mechanical and impact characterisation of flax and basalt fibre vinylester composites and their hybrids

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Abstract

The present experimental investigation is aimed at performing an analysis of mechanical and impact properties of flax and basalt fibres and their hybrids using a vinylester resin to produce reinforced thermosetting composites. Laminates were fabricated by hand lay-up and resin infusion. Cure processes were accelerated and controlled by applying heat and pressure in autoclave. Tensile, flexural and falling weight impact tests were carried out, the latter with energies of up to 40 J. The results indicated that hybrid laminates did not mostly offer properties to the level predicted by an application of the rule-of-mixtures, especially as regards flexural performance. On the other side, advantages provided concerned in particular reducing the brittleness of basalt offering some evidence of plastic behaviour, especially related to the fact of flax fibre reinforced laminated providing a quite long period at quasi constant load during impact tests, therefore resulting in delayed failure, while extensive damage is produced. The results tend to challenge the idea that basalt/flax fibre hybrid laminates would offer a good performance only with the presence of basalt fibres in the outer layers and would suggest the possible adoption in future of more complex stacking sequences, involving intercalation of flax and basalt layers.

1. Introduction

Currently, a large interest and considerable research activity is dedicated to elicit solutions to minimize the environmental impact in the production and use of composite materials, leading therefore to their improved sustainability [1]. New environmental regulations and evolving governmental attitudes are a powerful key-driver, stimulating the research of more environmentally friendly products and processes [2]. As a reinforcement, natural fibres (as flax, hemp, kenaf, wood, bamboo, etc.) are largely investigated as an alternative, involving total or partial substitution, to synthetic fibres (mainly to glass, since carbon and Kevlar offer more specific properties, in terms of mechanical performance) [3-4]. The aforementioned "partial substitution" of glass fibres is normally obtained by hybridization, normally achieved by stacking layers reinforced with glass fibres with other layers reinforced with vegetable fibres, such as hemp, jute, etc. [5-8].

In recent years, basalt fibres have often been proposed as an alternative to glass, in view of some significant advantages: these include the fact that basalt is directly spun from the molten rock, and then finished with the application of sizers not dissimilar from those applied on glass fibres. In addition, the surface of basalt fibre fibres contains groups taking part in ionic exchange, such as hydrogen-bound silanol, which form active adsorption sites and can interact with components of the sizing agent [9]. In some cases, the improved resistance of basalt to acid environments has also been revealed, much more than what had been reported in the case of their exposure to basic environments [10]. In other studies, the reverse was reported, hence that the exposure to alkali

would be less damaging for basalt fibres [11]. In general, resistance to acids would be particularly desirable e.g., in the automotive sector [12-13]. On the other side, when employing vegetable fibres, such as flax and hemp, these are normally alkali treated: hybrid composites using vegetable fibres in combination with basalt fibres were realised in [14-15].

Analysis of falling weight impact properties of basalt/flax/hemp hybrids suggested that despite their outstanding impact resistance, improved even with respect to glass/flax/basalt layers, they showed some proneness to delamination under post-impact flexural tests [16]. A recent study on basalt/flax hybrids, where basalt fibre reinforced layers were placed externally ensured a significant improvement of resistance under salt and fog conditions, which only partially reflected on Charpy impact properties, where basalt fibre external layers showed a limited resilience under prolonged ageing [17]. Hybrid composites with vegetable fibre reinforcement coupled with basalt are far from being optimised though, especially with respect to the possible effect of different stacking sequences on their properties [18]. Considering hybrid laminates obtained by combining basalt with fibres of vegetable origin, it needs to be considered that the latter are compounds of cellulose, hemicellulose and lignin in variable amounts and with different microstructures, hence with non negligible inherent variation in properties. A number of more specific reasons can also be accounted for this scattering in experimental data on vegetable fibres: these include the presence of internal voids, or lumens, of variable geometry and extent, which are therefore limiting homogeneity of vegetable fibres, and leads to possible problems in terms e.g., of significant water absorption and the irregular diameter of these fibres [19]. On the other side, vegetable fibres are biodegradable and "carbon positive" since they absorb more carbon dioxide than they produce. They are non-irritating and tend to be non-abrasive, with reduced wear on tooling and manufacturing. In a composite, vegetable fibres have lower density values: considering flax with respect to glass (or basalt), fibres density is reduced by about 50%. Flax is particularly suitable since easy to process and recycle, can be customized to meet a variety of specifications and different manufacturing systems for its great flexibility in terms of production of textile products with different architecture and areal weights. Flax properties are among the highest for vegetable fibres (a concise compared evaluation of flax and basalt fibres from relevant literature is offered in Table 1). However, composites obtained with flax need still to be optimised from a structural point of view. A number of investigations concerning impact properties of flax reinforced composites, mainly based on Charpy and Izod impact tests, have been carried out, and the results obtained were very far from what measured on fibreglass [20-21]. Even if not outstanding resistance to impact does limit service use of flax fibres, hybridisation with stronger fibres, such as basalt, would result in properties more tailored to requirements [7]. Of course, in this case, an impact performance-centred investigation would need to involve falling weight impact tests, especially because these offer more information on the gradual progression of damage with growing impact energy and of the prevalent mode of absorption together with the particular damage morphology with respect to the originating materials (in this case basalt and flax fibre reinforced laminates) [4, 22].

In this investigation, flax/basalt hybrids have been produced along the lines of work performed in [23]. Here, a specific attention was reserved to the factual evaluation on the influence of basalt as hybrid reinforcement in combination with flax fibre, focusing especially on the modes of damage observed as the consequence of impact energy absorption.

2. Material and Methods

2.1. Fibres

Flax fibres were used in the form of 0/90 balanced fabrics, with areal weight of 300 g/m² (*LINEO*[®] *FLAXPLY BL*). Basalt fibres were used also in the form of 0/90 balanced fabric, with areal weight 350 g/m² (*BASALTEX*® *BAS 350.1500.A*). The idea was to select fibres with areal weight as close as possible from each other, of course since basalt fibres have a density close to twice the one of

flax fibres, the thickness of each basalt fabric layer was of 0.24 ± 0.01 mm, while that of the single flax fabric layer was of 0.53 ± 0.02 mm, considering the higher amount of voids in the fabric.

2.2. Matrix

A *DISTITRON*[®] *VEef 220 STZ* vinylester resin, pre-accelerated and thyxotropic was used in this work, a resin with low (35%) volatile content (VOC), low styrene and low hazardous air pollutant (HAP), obtained via a mix between vinylester and bisphenolic epoxy. This resin is able to polymerize at ambient temperature and pressure, its viscosity being not superior to 2700 MPa*s at 25°C. However, a cure in autoclave does offer a higher performance in materials and a better control in processing.

2.3. Manufacturing

Composite laminates were produced by a hand layup procedure, stacking dry fabric layers by hand onto a planar support to form a laminate stack. Resin was applied to the dry plies after layup and then the amount of resin needed for full impregnation was added by means of resin infusion under pressure, as described below. The fibre volume content was evaluated by separate weighing of the fabric layers and of the resin used for production: the production was carried out with the idea to use the least possible amount of resin needed for full impregnation. In practice, by weighing three different laminates of each type of composite, an evaluation of the fibre volume fraction from the respective thickness of the layers compared to that of the whole composite was performed. This is not intended to be very precise, but to give a rough idea of the possible range for these values. Values obtained were equal to around 23.5 (\pm 2)% for flax, 27 (\pm 1.5)% for basalt and 25.5 (\pm 2.5)% for flax/basalt composites.

Cure was realized by applying controlled heat and pressure in autoclave, according to instruction provided by the resins' manufacturers. In practice, laminates underwent an initial 24 hours curing at environmental conditions, then a post-cure, which consisted of a 3 hours treatment in autoclave at 100°C under a pressure of 6 bars.

All the laminates throughout this study were fabricated using eight sheets of fabric reinforcement. In the case of hybrid laminates, obtained using both basalt and flax fibres, it was considered more suitable, as per common practice in impact-resistant hybrids [15-16], to use the softer fibre in the core layers and the harder one in the skin ones. Therefore laminates were manufactured using flax as the reinforcement of the four internal plies, whilst on both sides the two external layers were reinforced by basalt fibres.

Void measurements were carried out on small squares of material of 25 mm side.

2.4. Samples

A minimum of five samples were tested in the case of both tensile tests and flexural tests with planar dimensions 250x25 mm for tensile tests, 120x15 mm for flexural tests and 100x100 mm for falling weight impact tests. In the case of falling weight impact tests, three samples were impacted at the maximum energy of 40 Joules in the case of flax/basalt and basalt fibre laminates: in both cases the energy was proved not sufficient to produce the penetration. After this, two lower heights were selected in order to offer information about impact damage progression and three specimens were impacted for each of these heights. In the case of flax fibres, three samples were also tested at 40 Joules, which resulted in penetration and in the absorption of a much lower amount of energy, in particular equal to around 32.5 ± 2 J, as measured from the impact hysteresis cycles. Geometrical differences in the samples were found to be much lower than \pm 10%, as prescribed by the standards. In particular, the thickness of the laminates was in the region of 7.1 (\pm 0.3) mm for flax fibre laminates.

3. Experimental Tests

3.1. Tensile tests

Tensile tests were performed according to ASTM D3039-14 standard, using an Instron universal tester, model 8033, equipped with a 200 kN load-cell. Loading was performed in displacement control mode using a 2 mm/minute velocity. The ultimate tensile strength of each sample was determined from the maximum load supported before failure, whereas the yield tensile strength of each sample was evaluated by the 0.2% yield offset method: the 0.2% deviation from linearity is automatically computed by the system, hence setting also two points for the region of the stress-strain curve in which measurement of Young's modulus is carried out. In this way, ultimate and yield stresses were obtained; similarly, from displacements, strains were calculated. By using four strain gauges, two per each side of the sample, additional measurements of strain were carried out for a direct estimation of Young's, Poisson's and shear moduli. When the gage axes of a two-gage 90-deg rosette are aligned with the principal axes, the output of the half bridge is numerically equal to the maximum shear strain. A full shear-bridge (with twice the output signal) is then composed of four gages. It is suggested that ensuring that the laminate is really loaded through its centroid axis, therefore being perfectly aligned, would offer reasonably accurate values of shear strain, from which shear modulus and Poisson's ratio have been calculated. This method is as suggested in [24-25].

3.2. Flexural tests

Three-point flexural tests were performed according to ASTM D790-10 standard, using again an Instron universal tester, model 8033. The flexural rig included an upper cylindrical support of 12.7 mm diameter, whilst the lower supports had a 6.35 mm diameter each. The planar dimensions of the samples are 120x15 mm: span to thickness ratios are equal to approximately 15 ± 1 . The ultimate flexural strength of each specimen was determined from the maximum load carried before failure. Loading was performed in displacement control mode using a 2.5 mm/min velocity. The yield flexural strength of each sample was also evaluated by the 0.2% yield offset method.

3.3 Falling weight impact tests

Falling weight impact tests were performed according to ASTM D7136/D7136M-15 standard: damage was imparted through out-of-plane concentrated impact, perpendicular to the plane of the laminated plate, using a falling weight of mass 1.25 kg with a hemispherical striker tip of 6.35 mm diameter. The use of a small mass with a relatively high velocity (around 7-8 m/s) of impact is aimed at reproducing the conditions of most severe strikes during maintenance (an example could be the dropping of a tool on an automotive interior panel). The 100 mm side samples were mechanically clamped between two plates, the upper ones having a circular opening of 60 mm diameter. To perform the impact test, the mass was raised to a known height and released for a free fall: theoretical impact energies obtained are given in Table 2. This height is carefully chosen and modified (during the sequence of tests) with the aim of permitting to explore the different mechanisms of failure. The damage resistance was quantified in terms of the resulting size and type of damage in the specimen. A specific sensor also permitted to measure the impact force, providing useful information for retracing the dynamic of impact. The time-dependent response of materials to impact was presented in diagrams in consideration of the height of release and consequent impact energy.

4. Results and discussion

Composites reinforced by flax or basalt fibres and hybrid (flax + basalt) fibres were tested and compared. Tensile and flexural results are reported in Table 3a and 3b, respectively. As quite obvious, the hybrid laminates offered intermediate properties between the basalt and the flax fibre laminates, although closer to the former in the case of tensile performance, whereas closer to the latter in the case of flexural ones. In other words, flexural performance appears rather deceiving with respect to tensile one.

Typical tensile and flexural curves are reported in Figure 1 and 2, respectively. In general terms, flax fibre composites present during tensile loading a typical curve in which stress-strain curve is hardly linear, which appears to be the case for basalt instead, whereas hybrids present a quasi-linear behaviour with some evident load drops, possibly the effect of onset of internal damage. In reality, it is well known that flax fibres present a linear tensile behaviour [26], therefore the departure from linearity needs to be attributed to the interaction between the fibre and the matrix. In flexural curves, hybrids laminates show, a clear change of slope, in other words a proportionality limit. This is different from what observed on both basalt and flax fibre composites, where no change of slope is obvious from the curve.

In terms of the mechanical results obtained from tensile and flexural tests (all tensile and flexural results are summarised in Table 3 and b, respectively), quite obviously the results obtained from hybrid laminates do come at an intermediate value between flax fibre composites and basalt fibre composites. It may be suggested that in principle a rule-of-mixtures approach could be applied to static tests of hybrid laminates, as it is the case for flexural and tensile loading. This was applied from early studies on hybrid laminates including vegetable fibres [27]. In other words, the hypothetical static properties of the hybrid composites (hence tensile strength and stiffness) can be considered an average, weighed over respective volume, of the two original composites that form it, hence a flax fibre reinforced and a basalt fibre reinforced one. The calculations leading to this hypothesis are exposed as follows:

(1)

where:
vc= Value predicted on the composite
Vb= Volume fraction of basalt fibre composite
vb = Average value obtained on basalt fibre composite
Vf = Volume fraction of flax fibre composite
vf = Average value obtained on flax fibre composite

As for values, we intend tensile strength and stiffness and flexural strength and stiffness. The volume fraction can be calculated by considering that hybrid fibre laminates includes an equal number of layers of flax and basalt fibre laminates, considering the average thickness of the laminates reported in Section 2.4, it may be suggested that in the hybrids the thickness of basalt fibre composite layers is not reduced, due to their rigidity, therefore being equal to 3.8/2=1.9 mm, while the remainder is flax fibre composite, hence (5.1-1.9) = 3.2 mm. In this way, it can be suggested that the volume fraction of basalt fibre composite in the hybrid is equal to 1.9/5.1=0.37, while the volume fraction of flax fibre composite is equal to 3.2/5.1 = 0.63.

Therefore equation (1) becomes: vc=0.37* vb +0.63* vf

Results predicted for the hybrid laminates from results obtained on flax and basalt ones are given in Table 4, where they are compared with the experimental results actually obtained on hybrids and the percentage variation is also given, to assist the evaluation. It can be noticed in this regard that as for tensile and flexural stress, the results obtained with hybrids were slightly inferior to what could be predicted by the rule-of-mixtures, as can be observed in Figure 1 and Figure 2, as far as tensile and flexural stress are concerned, respectively. It needs to be considered that the amount of voids is not accounted for in the rule-of-mixtures, which may be ultimately the reason for the differences observed. More generally, the defects in composites, including void content, fibre–matrix debonding, and fibre defects should be considered: therefore some degree of inaccuracy is expected, although the entity of variation would in any case give some information on the quality of hybrid laminate processing. The only positive value is offered by tensile stiffness, in which case it might be suggested that the effect of the voids is possibly overwhelmed by the rigidity during tension offered by basalt layers. As a matter of fact, it is suggested that flax fibre composites produced with similar techniques than the one adopted may present a quite considerably large amount of voids, due to variability of crimp characteristics, which may result in resin starvation [28-29].

A selection of the samples and their mode of failure are reported in Figure 3 and 4 for tensile and flexural laminates, respectively. It can be observed that in basalt/flax hybrids the samples are clearly delaminated before failure, while flexural loading produces early failure on flax with a limited bending angle. It has been observed elsewhere that the tendency to delamination in laminates including flax fibres may strongly depend on the stacking sequence selected [17], therefore the results presented are not conclusive, but would be specific for the configuration presented here.

SEM observations following flexural fracture (Figure 5a-c), suggested as a whole that the limited bending angle needed for fracture of flax fibre reinforced laminates was prevalently due to the presence of fibrillation, hence fibres splitting into filaments, a well known phenomena on these composites, which is evident also in Figure 5a [30]. As a matter of fact, the opening of much larger holes as an effect of bending are needed for basalt fibre composites to lead them to collapse, as represented in Figure 5b. It is also suggested that the combination of both phenomena into flax/basalt hybrid composites, such as in Figure 5c, would result in these offering a quite deceiving flexural performance.

In the case of impact testing, these were performed by gradually increasing the height of the falling weight. In the case of flax fibre composites, the initial height was 1 meter, whereas in the case of basalt and flax/basalt fibre composites, the initial height was set at 2.5 meters. Comparing force vs. time curves obtained in impact tests that yielded the maximum impact force on the laminate, it can be noticed, as from Figure 6a-c, that in the case of flax fibre laminates a larger part of the energy is absorbed through non elastic mode, hence after reaching the maximum load on the laminate. In particular, a large plateau at around maximum load is observed. The extension of this plateau is in contrast more limited in the case of basalt fibre laminates, whereas the behaviour of flax/basalt hybrid laminates is limited between the two. This is not unexpected, since it has been also emphasised in other works on falling weight impact of plant fibre composites [7, 31], as an effect of the capability to deflect the progression of the impacting head during the impact event. In Table 5a and 5b more information about impact tests is offered at two different impact energies, namely 30.62 and 36.75 J, which indicates also that the difference in terms of maximum load between the flax and basalt fibre laminates is not very high. The real difference is in the penetration energy, in fact impact with fall from the maximum height of 3.26 meters results in penetration of flax fibre laminates, while this does not occur for basalt fibre laminates and flax/basalt fibre laminates not

even in the case of impact from the maximum height allowed by the falling weight tower employed in this study. Hysteresis cycles at energy of 30.62 J have also been reported in Figure 7: in this case, it is noticeable that flax/basalt hybrid laminates do present a particularly high residual deformation compared to the other laminates. In other words, the more complex structure presented by the hybrid, including two materials with different strength, is likely to reduce the extent of the striker rebound.

In Figures 8-10 damage occurring as the effect of impact on the different laminates is represented. It can be noticed as in flax fibre composites extensive tearing of the fabric is obtained during impact even at low energies. In contrast, in the case of basalt fibre laminates the fabric does not tend to be fractured away from the impact point, most damage is instead concentrated as an indentation or protrusion (depending on the side) around the impact region with some additional damage in the form of resin abrasion around the impact point can be noticed. Some marked effect of orientation is observable in hybrid laminates, where impact damaged areas considerably depart from a circular geometry. This is to be attributed to the anisotropy of reinforcement: this is particularly evident when using vegetable fibres, such as flax, where usually weave structure shows some waviness, which influences the path of damage propagating from the impact point [32]. In terms of impact performance, this is likely to occur also due to the influence of micro-defects and weave mismatches in the fabric [33-34].

Conclusions

The production of hybrids including flax and basalt fibres in vinylester matrix using flax fibre reinforced layers as the core between basalt fibre skins demonstrated some possibility of synergistic behaviour. This appeared mainly limited to tensile behaviour though, where the mutual behaviour of the two reinforcement fibres led to an improvement of performance with respect to the weighed average of the two component materials. In particular, this combination reduces the stiffness and brittleness of basalt, demonstrated by a plastic behaviour after yielding, confirmed also from the results of falling weight impact testing, which provides greater flexibility to the material. Conversely, hybridisation considerably increases impact performance of flax fibre composites with a non excessive increase in weight. On the other side, however, the introduction of flax fibres increases the dependence of the laminates from weave configuration and its defects, hence leading to orientation of impact damage according to areas of lower resistance in the laminate and generally to proneness to delamination of hybrids. In addition, the evaluation of more complex stacking sequences with intercalation of flax and basalt layers inside the laminate would be needed, although the concept of using the latter as skins may show some merit during impact loading.

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Figures



Figure 1 Typical tensile stress vs. strain curves for the different laminates



Figure 2 Typical flexural stress vs. strain curves for the different laminates



Figure 3 Tensile samples removed from different laminates and their mode of failure: flax (left), basalt (centre), flax/basalt hybrids (right)



Figure 4 Flexural samples removed from different laminates and their mode of failure flax (left), basalt (centre), flax/basalt hybrids (right)



Figure 5a SEM observation following flexural fracture on flax fibre reinforced laminates



Figure 5b SEM following flexural fracture on basalt fibre reinforced laminates



Figure 5c SEM observation following flexural fracture on flax-basalt hybrid laminates



Figure 6a Typical force vs. time diagrams obtained from flax fibre composites impacted at different energies



Figure 6b Typical force vs. time diagrams obtained from basalt fibre composites impacted at different energies



Figure 6c Typical force vs. time diagrams obtained from flax/basalt hybrid fibre composites impacted at different energies



Figure 7 Typical force vs. displacement diagrams obtained from impact tests on the different laminates with nominal energy of 30.62 J (impact height = 2.5 metres)



Figure 8 Images of flax fibre composites after impact at different energies







Figure 10 Images of flax/basalt fibre hybrid composites after impact at different energies

Tables

FIBRES	Density	σ_{u}	E	٤ _u
	g/cm ³	MPa	GPa	%
FLAX	1.3-1.5	345-1500	27-80	2.7-3.2
BASALT	2.65-2.8	4000-4700	84-87	3.1-3.6

Table 1 Main properties of flax and basalt fibres [9, 22-25]

Impact height (m)	Impact energy (J)	Applied on
1	12.25	Flax
1.5	18.37	Flax
2	24.5	Flax
2.5	30.62	All laminates
3	36.75	All laminates
3.26	40	Basalt and flax/basalt

Table 2 Impact energies applied

LAMINATES	σ _y [MPa]	σ _{max} [MPa]	ε _y %	E [MPa]	G [MPa]	v -
FLAX	27.7 ± 10.1	47.5 ± 3.9	0.93 ± 0.07	4854 ± 166	2001 ± 69	0.21 ± 0.02
BASALT	108 ± 17.5	165 ± 13.3	1.47 ± 0.11	11153 ± 456	4960 ± 183	0.12 ± 0.02
FLAX/BASALT	67.3 ± 9.8	86.5 ± 5.5	1.12 ± 0.1	8151 ± 386	3879 ± 127	0.13 ± 0.01

Table 3a Tensile properties of the three laminates

LAMINATES	σ _y [MPa]	σ _{max} [MPa]	ε _y %	E [MPa]
FLAX	85.1 ± 4.8	118.3 ± 6.5	1.66 ± 0.21	6930 ± 190
BASALT	212.3 ± 33.2	265 ± 17.8	1.84 ± 0.25	14481 ± 515
FLAX/BASALT	106.7 ± 11	144.8 ± 7.3	1.69 ± 0.2	8275 ± 333

Table 3b Flexural properties of the three laminates

Property	Predicted value	Experimental value	Difference (%)
Tensile strength (MPa)	90.3	86.5	- 4.2
Tensile modulus (GPa)	7.14	8.15	+ 14.1
Flexural strength (MPa)	172.6	144.8	- 16.1
Flexural modulus (GPa)	9.72	8.27	- 14.9

Table 4 Predicted and experimental values for hybrid flax/basalt fibre laminates

Laminates	Peak force (kN)	Max. displacement (mm)	Absorbed energy (J)
FLAX	3.87 ± 0.43	7.9 ± 0.4	22.1 ± 1.3
BASALT	4.63 ± 0.40	9.1 ± 0.5	21.2 ± 2
FLAX/BASALT	4.17 ± 0.54	7.3 ± 0.8	21.9 ± 1.9
		L	

Table 5a Avg. maximum force (kN) and non-elastic energy during impact at 30.62 J

Laminates	Peak force (kN)	Max. displacement (mm)	Absorbed energy (J)
FLAX	3.90±0.24	11.50±0.96	33.69±2.17
BASALT	5.61±0.09	10.55±0.36	30.23±0.56
FLAX/BASALT	5.45±0.15	9.32±0.45	32.33±2.74

Table 5b Avg. maximum force (kN) and non-elastic energy during impact at 36.75 J