

# XIV DBMC 14th International Conference On Durability of Building Materials and components











Edited by

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## XIV DBMC 14<sup>th</sup> International Conference on Durability of Building Materials and Components

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- optimise and harmonise test methods for measuring properties and performance of building and civil engineering materials and structures under laboratory and service environments,
- prepare technical recommendations for testing methods,
- prepare state-of-the-art reports to identify further research needs,
- collaborate with national or international associations in realising these objectives.

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# XIV DBMC – 14th International Conference on Durability of Building Materials and Components

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## **Preface**

The DBMC Conferences have had a long history that started in 1978 and travelled to different cities: Ottawa 1978, Gaithersburg 1981, Espoo 1984, Singapore 1987, Brighton 1990, Omiya 1993, Stockholm 1996, Vancouver 1999, Brisbane 2002, Lyon 2005, Istanbul 2008, Porto 2011, Sao Paulo 2014. The aim of DBMC is to bring together those concerned about durability and sustainability of all building materials and components and thus provide a forum for discussion of research and practice for all classes of building materials.

It is our privilege and honour to organize the 14<sup>th</sup> event in Ghent, May 2017. The relevance of the DBMC conference series is illustrated by the large number of active contributions. About 300 abstracts have been submitted, resulting in nearly 200 peer reviewed papers. We are very grateful to the members of the international technical committee who contributed in a significant way to the review of the submitted papers.

We also acknowledge the international organisations in the international steering committee, ASTM, CIB, NIST, NRC-CNRC and RILEM, for their important role in the DBMC series, and for their support in spreading the information to their members.

The support of our sponsors is thankfully mentioned: Carmeuse, PermeaTORR AC, Wienerberger, and Sanacon. We also acknowledge the substantial support by the Belgian Building Research Institute, BBRI.

Finally, our gratitude goes to the many co-workers of Ghent University in general, and the Magnel Laboratory for Concrete Research more specifically, for the endless list of organizational and practical issues that have been taken care of in a professional and efficient way. We specially mention Mrs. Marijke Reunes, for taking the lead of the conference secretariat.

We hope that the scientific contents of the conference will largely meet your expectations. And at the same time we hope that networking with your colleagues and friends during XIV DBMC in Ghent will open new perspectives and offer new opportunities for further ground-breaking research activities.

Geert De Schutter, Nele De Belie, Arnold Janssens Conference chairs.

## The assessment over time of the performance of jute-basalt hybrid composites for cladding panels

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Keywords: Durability, Hybrid composites, Basalt fiber, Jute fiber, Cladding panels.

#### **Abstract**

The technology of dry assembly to produce multi-layer panels is an efficient alternative to traditional systems. Eco-sustainable systems are increasingly studied, particularly fiber reinforced composites for external cladding, based on natural fibers. Nevertheless, durability data on these composites are still lacking, being this is a really actual theme, especially for their potential use in outdoor applications. In this way, hybridization of natural fibers with mineral fibers as basalt ones appears promising. In the present paper, the performance of jute-reinforced laminate was compared with those of two jute/basalt reinforced hybrid laminates. The laminates were manufactured by means of vacuum infusion process and then cured, varying the number of layers in order to achieve both thickness and fiber content almost constant. In particular, not hybridized jute reinforced composite is made of eight layers of jute plain weave fabric (290 g/m² areal density). The hybridization of jute was performed by using unidirectional basalt fabrics (300 g/m²), to manufacture two hybrid laminates: in the first one, layers were stacked as a sandwich sequence with six jute fiber reinforced layers as core and two basalt fiber layers as skins, for each side of the laminate; in the second laminate, fabrics of basalt and jute fibers were alternatively stacked with basalt fabrics as outer layers (Fig. 1).

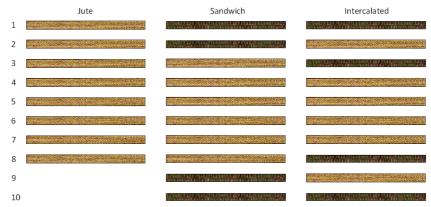


Fig. 26. Stacking sequences of the three types of laminate composites

The number of the layers (i.e. 8 and 10 for Jute and Hybrid laminates, respectively) was varied in order to obtain the same volume fraction, approximately equal to  $0.294 \pm 0.03$ , so that the thickness of all the produced laminates was almost constant ( $5.50 \pm 0.12$  mm). All laminates were exposed to 4 steps of accelerated aging into climatic chamber, for a whole period of 84 days, by developing aging cycles of hygrothermal stress (Phase A: 300 min made of Rain: 75 min, Cold: 40 min at 2°C, Hothumid: 115 min at 35°C and RH=87%, Hot-dry: 70 min at 50°C and HR=56%; phase B made of 120 min of UV irradiation at 35°C and HR=87%), reproducing the climatic conditions of the Mediterranean context of South Italy. During the aging exposition, the samples were removed from

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the climatic chamber and mechanically tested (i.e. by means of quasi-static three point bending, dynamic-mechanical and impact tests) after 1, 2, 4 and 8 steps (i.e. after 14, 28, 56 and 84 days of artificial aging), respectively. To complete a single step of 14 days, Phase A and Phase B were sequentially repeated 24 times and this alternation was further repeated twice. From literature survey, it is well known that water exposition can induce a plasticization of the laminates' matrix thus leading to an increase of the mobility of the polymer chains. This phenomenon is responsible for the increments of both the deformation at break and the impact energy absorption. Moreover, the temperatures reached in "Hot-humid" and "Hot-dry" stages as well as the UV exposition lead to a post-curing effect of the polymer matrix, mainly in the first part of the exposition. This results in an increase of the matrix stiffness and an enhancement of the fibre-matrix interface that is reflected by the increase of the dynamical mechanical properties (i.e. the glass transition temperature increases and the  $\tan\delta$  peak decreases) of the laminates. On the other hand, the improvement of matrix stiffness and fibre-matrix adhesion, can contribute to the decrease of the impact energy absorption capability of the laminate, as stated in previous studies. It is worth nothing that the residual heat reaction measured by DSC decreases with the increase of the aging exposition time, thus evidencing that postcuring reactions occur during the aging exposition. As expected, the maximum temperature of the exothermic peak does not change by varying the aging time exposition, remaining almost constant to  $76 \pm 0.5$  °C. Another phenomenon to take into account when a polymer composite is subjected to alternate cycles of hygrothermal stress and UV radiation consists in a progressive damage of the resin surface due to the formation of micro-cracks. In particular, UV radiation creates a vicious cycle of hydrogen abstraction from the polymer molecules initiating at the surface of the material. This leads to the formation of free radicals which initiates other reactions causing embrittlement of the materials thus leading to an overall deterioration in material properties. The molecular chain scission occurring on the polymer surface generates the radicals that may bind themselves to the main chain of a neighbouring molecule to give a branched molecule with a higher molecular weight. The excessive embrittlement is mainly responsible for the formation of micro-cracks and their broadening thus accelerating the photo-degradation process. Moisture further accelerates the photo-degradation process through the enhanced mobility of free radicals and ions. Moreover, the degradation phenomena act on the fibre-matrix interface and it worsens the stress transmission efficiency. Furthermore, jute layers tend to absorb a greater amount of water than basalt ones during the aging exposition, due to the lignocellulosic nature of jute fibre. Consequently, the hygroscopic expansion may induce residual stresses within the stacking sequences of hybrid laminates leading to a premature failure of the samples, as confirmed by optical and SEM micrographs. From these experimental results, it is possible to assert that the interplay hybridization represents a promising solution to overcome those drawbacks that characterize the use of jute fibres as reinforcement in composite materials for outdoor applications. Sandwich laminates showed better aging resistance to the external environment than Intercalated laminates, due to the barrier effect of the external basalt layers that protect the jute internal ones from the degradation phenomena. This is reflected by the flexural quasistatic properties that highlighted a slighter influence of the aging environment on the Sandwich laminates if compared to the others. Overall, Jute laminates showed lower flexural properties than hybrid ones, regardless the aging exposition time. Moreover, it was shown that the use of thicker external basalt layer, made of two basalt layers, in the Sandwich configuration allows to postpone the degradative effect of the aging exposition on the dynamic mechanical properties. Similarly, the worsening of the impact properties of Sandwich laminates is attenuated in comparison to Intercalated laminates. In terms of durability, the hybrid laminate composites showed higher initial mechanical performance compared to Jute laminates, suffering more intense decrease in flexural strength due to artificial aging. Comparing the two hybrid laminates, the Sandwich configuration is more suitable than the Intercalated one for the realization of technical elements more lasting over time, giving higher static and dynamic performance.

Edited by Geert De Schutter, Nele De Belie, Arnold Janssens, Nathan Van Den Bossche

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Influence of hydrophobic treatments applications on the concrete carbonation delay

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### The assessment over time of the performance of jute-basalt hybrid composites for cladding panels

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#### **Abstract**

The technology of dry assembly to produce multi-layer panels is an efficient alternative to traditional systems. Eco-sustainable systems are increasingly studied, particularly fiber reinforced composites for external cladding, based on natural fibers. Nevertheless, durability data on these composites are still lacking, being this is a really actual theme, especially for their potential use in outdoor applications. In this way, hybridization of natural fibers with mineral fibers as basalt ones appears promising. In the present paper, the performance of jute-reinforced laminate was compared with those of two jute/basalt reinforced hybrid laminates. The laminates were manufactured by means of vacuum infusion process and then cured, varying the number of layers in order to achieve both thickness and fiber content almost constant. In particular, not hybridized jute reinforced composite is made of eight layers of jute plain weave fabric (290 g/m<sup>2</sup> areal density). The hybridization of jute was performed by using unidirectional basalt fabrics (300 g/m<sup>2</sup>), to manufacture two hybrid laminates: in the first one, layers were stacked as a sandwich sequence with six jute fiber reinforced layers as core and two basalt fiber layers as skins, for each side of the laminate; in the second laminate, fabrics of basalt and jute fibers were alternatively stacked with basalt fabrics as outer layers. All laminates were exposed to 4 steps of accelerated aging into climatic chamber, for a whole period of 84 days, by developing aging cycles of hygrothermal stress and UV radiation, reproducing the climatic conditions of the Mediterranean context of South Italy. After each step of accelerating aging, several mechanical tests (i.e., quasi-static three point bending tests, Charpy impact tests and dynamic thermal tests) were performed according to International standards. Initial results show the effectiveness over time of produced composite laminates and initial increase of measured performances after the 1st step of accelerated aging.

#### Introduction

The technology of dry assembly for the production of multi-layer panels has evolved over the past 30 years, becoming an efficient alternative to traditional systems.

Eco-sustainable systems have been increasingly tested and used, particularly those based on laminated composites for cladding.

This technology focuses on the assembly of multi-layer composite panels, introducing new materials in the construction process, optimizing the main performance of the various layers [1].

Natural fibres reinforced composites have received growing attention in the last two decades both from the academic world and industrial manufacturers thanks to their specific mechanical properties, price and advantages for health and recyclability. A great variety of different natural fibres is widely used as reinforcement in composite materials for their properties and availability as flax [2], hemp [3], jute [4, 5] and sisal [6].

Jute is an abundant natural fibre used as a reinforcement in bio-composite [7]: i.e., it is the second most important fibre in terms of world production levels of cellulosic fibres, next to cotton. White and dark jute fibre is obtained from the bast layer of the plants. It is an annual plant that flourishes in monsoon climates and grows up to 4.5 m, primarily grown in Africa and Asia. The tensile strength, Young's modulus and elongation at break of jute fibre have been reported to be in the ranges 393-773 MPa, 13-26.5 GPa and 7-8%, respectively whereas its density is between 1.3 g/cm<sup>3</sup> and 1.45 g/cm<sup>3</sup> [8].

Nevertheless, the hydrophilic nature, highly variable mechanical properties, poor adhesion with several different polymeric matrices and low aging resistance in critical environments (e.g. humid and/or hot ambient) represent the main drawbacks that limit the use of natural fibres to non-structural or semi-structural components not exposed to aggressive condition. To overcome this problem, a hybridization of natural fibres with basalt fibres having superior aging resistance and thermal stability is proposed in this work.

Basalt fibres have recently gained an increasing attention as reinforcement of composite materials, thanks to their advantages in terms of environmental cost in addition to high mechanical, chemical and thermal properties [9-15]. The production technology of basalt fibres can be considered environmentally friendly and non-hazardous since through melting of basalt rock with no other additives, thus needing a lower amount of energy if compared to glass fibres [9].

In literature, there are several works related to the hybridization of natural fibres with synthetic ones such as glass [16] and carbon [17], some of these focused on the evaluation of their aging resistance [21, 25]. Basalt fibre has been recently used in the hybridization of natural reinforced composites [21, 22] even if very limited papers concerning their durability in critical environment of such kind of hybrid composites exist [23].

In this work, the assessment over time of the performance of three types of fibre/epoxy composite - one jute fibres based and two jute/basalt fibres based, with different stacking sequences - were compared. This comparison aims to investigate the suitability of extending the use of jute fibre as reinforcement in composite structures useful for outdoor applications such as external cladding, through basalt hybridization.

#### 1. Material and methods

#### 1.1 Materials

All the laminates were manufactured through vacuum assisted resin infusion and they were cured for 12 h at room temperature and for 4 h at 50 °C. A bio-based epoxy resin Super Sap 100 and its own hardener Super Sap 1000 (mix ratio in weight =100/48) supplied by Entropy Bio-Resins, USA, were used as matrix for the laminates.

Fig. 1 shows all the manufactured laminates, the one named "Jute" was made stacking 8 layers of jute plain weave fabric with areal density of 290 g/m<sup>2</sup>, supplied by Composites Evolution (UK).

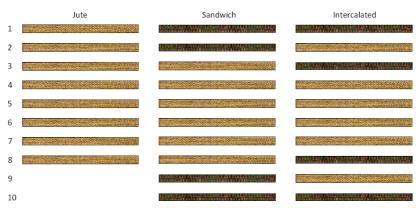


Fig. 1. Stacking sequences of the three types of laminate composites

The interplay hybridization of jute was performed using unidirectional basalt fabric with areal density of 300 g/m², supplied by HG Europe (Italy). In the "Sandwich" hybrid configuration, layers were stacked in a symmetrical sequence with six jute fabrics across the laminate mid plane and two basalt fabrics as outer layers (Fig. 1b). In the "Intercalated" hybrid configuration, four jute fabrics were stacked across the laminate mid plane whereas basalt and jute fibres fabrics were alternatively stacked as external layers of the laminate (Fig. 1c). The number of the layers (i.e. 8 and 10 for Jute and Hybrid laminates, respectively) was varied in order to obtain the same volume fraction, approximately equal to  $0.294 \pm 0.03$ , so that the thickness of all the produced laminates was almost constant  $(5.50 \pm 0.12 \text{ mm})$ .

#### 1.2 Artificial aging

The laminates were exposed to accelerated aging in a climatic chamber model ACS 1200 from Angelantoni (Italy), by developing aging cycles of hygrothermal stress (i.e. Phase A) and UV radiation (i.e. Phase B), by means of 125 W UV lamp, producing irradiance of 5 W/m<sup>2</sup>, as shown in Table 1.

Phase ID	Aging condition	Time [min]	T [°C]	RH [%]
	Rain	75	20	95
A	Cold	40	2	-
	Hot-humid	115	35	87
	Hot-dry	70	50	56
	<b>Total Phase A</b>	300		
В	Hot-humid/UV radiation	120	35	87
	Total Phase A+B	420		

Table 1. Aging parameters for single cycle

During the aging exposition, the samples were removed from the climatic chamber (Fig. 2) and mechanically tested (i.e. by means of quasi-static three point bending, dynamic-mechanical and impact tests) after 1, 2, 4 and 8 steps (i.e. after 14, 28, 56 and 84 days of artificial aging), respectively. To complete a single step of 14 days, Phase A and Phase B were sequentially repeated 24 times and this alternation was further repeated twice.



Fig. 2. Samples inside the climatic chamber

This accelerating aging cycle was derived from the study of the last 20 years weather forecast data of the city of Palermo [24]. Unaged samples were tested for comparison purpose.

### 1.3 Experimental Characterization

Five prismatic samples (5.5 mm x 20 mm x 110 mm) of all laminates were tested for each aging condition by means of three point bending according to ASTM D790 by using a 5 kN Universal

Testing Machine, model Z005 (Zwick/Roell, Germany) and setting the span length equal to 96 mm and the crosshead speed to 2.35 mm/min.

Dynamic mechanical tests were performed in tensile mode according to ASTM D 4065 standard, using a dynamic mechanical analyser model DMA+150 (Metravib, France). Five prismatic samples (5.5 mm x 3 mm x 46 mm) for each aging condition were tested from room temperature to 150  $^{\circ}$ C with heating rate of 2  $^{\circ}$ C/min, in nitrogen atmosphere.

Impact tests were carried out according to EN ISO 179 standard, using a Charpy pendulum model 9050 (Ceast, Italy), equipped with a pendulum of potential energy equal to 25 J and impact speed of 3.8 m/s. Five un-notched prismatic samples (5.5 mm x 10 mm x 80 mm) of both laminates were tested for each aging condition.

Thermal properties of the laminates were studied by using a differential scanning calorimeter model DSC-60 (Shimadzu, Japan). In particular, the differential scanning calorimetry (DSC) was used to determine the residual heat of the curing process as a function of aging exposition time. To this aim, samples of approximately 10-20 mg were heated under nitrogen atmosphere from room temperature up to 150 °C, with temperature rate of 5 °C/min.

Morphological analysis was performed on the laminates by using a scanning electron microscopy (SEM) model Phenom Pro X (Phenom World, Netherlands). Before analysis, each sample was sputter coated with a thin layer of gold to avoid electrostatic charging under the electron beam.

#### 2. Results and discussion

#### 2.1 Aging phenomena

To better understand the phenomena acting on each laminate, a brief literature survey is reported here and it will shortly recalled in the subsequent sections.

The water exposition can induce a plasticization of the matrix thus leading to an increase of the mobility of the polymer chains. This phenomenon is responsible for the increments of both the deformation at break and the impact energy absorption. Moreover, the temperatures reached in "Hot-humid" and "Hot-dry" stages (i.e. 35°C and 50°C, respectively) [25] as well as the UV exposition [26] during Phase B lead to a post-curing effect of the polymer matrix, mainly in the first part of the exposition. As widely discussed, this results in an increase of the matrix stiffness and an enhancement of the fibre-matrix interface that is reflected by the increase of the dynamical mechanical properties (i.e. the glass transition temperature increases and the tanδ peak decreases) of the laminates. On the other hand, the improvement of matrix stiffness and fibre-matrix adhesion, can contribute to the decrease of the impact energy absorption capability of the laminate, as stated in previous studies [23, 27].

The occurrence of post-curing reactions during the aging exposition is here evidenced via differential scanning calorimetry (DSC).

For sake of conciseness, considering that post-curing not affect the reinforcement, the DSC curves of the hybrid laminates was not shown here. Fig 3 reports the residual curing heat curves for all the investigated laminates, as a function of the aging exposition time.

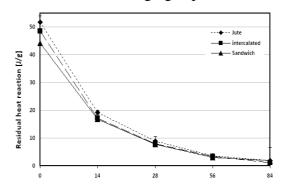


Fig. 3 Residual curing heat curves at varying the artificial aging for laminates

It is worth nothing that the residual heat reaction decreases with the increase of the aging exposition time, thus evidencing that post-curing reactions occur during the aging exposition. As expected, the maximum temperature of the exothermic peak does not change by varying the aging time exposition, remaining almost constant to  $76 \pm 0.5$  °C.

Another phenomenon to take into account when a polymer composite is subjected to alternate cycles of hygrothermal stress and UV radiation consists in a progressive damage of the resin surface due to the formation of micro-cracks. In particular, UV radiation creates a vicious cycle of hydrogen abstraction from the polymer molecules initiating at the surface of the material [28]. This leads to the formation of free radicals which initiates other reactions causing embrittlement of the materials thus leading to an overall deterioration in material properties [29]. The molecular chain scission occurring on the polymer surface generates the radicals that may bind themselves to the main chain of a neighbouring molecule to give a branched molecule with a higher molecular weight. The excessive embrittlement is mainly responsible for the formation of micro-cracks and their broadening thus accelerating the photo-degradation process. Moreover, moisture further accelerates the photo-degradation process through the enhanced mobility of free radicals and ions [28]. Moreover, the degradation phenomena, after a certain exposition time, acts on the fibre-matrix interface and it worsens the stress transmission efficiency.

Furthermore, Jute layers tend to absorb a greater amount of water than basalt ones during the aging exposition, due to the lignocellulosic nature of jute fibre. Consequently, the hygroscopic expansion may induce residual stresses within the stacking sequences of hybrid laminates leading to a premature failure of the samples.

#### 2.2 Flexural properties

By observing the fractured samples, it is possible to relate the stress-strain trends slopes to the failure modes experienced by the laminates. Fig. 4 shows selected optical micrographs of the tested samples.

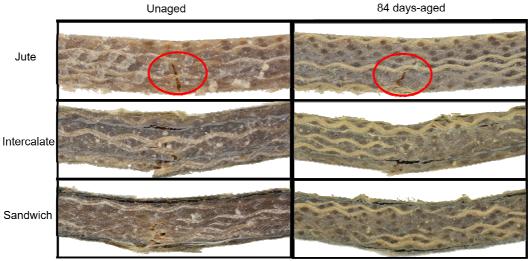
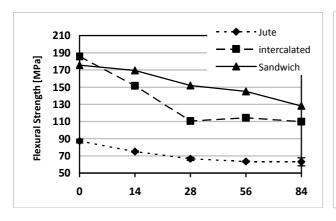


Fig. 4. Optical micrographs of the thickness side of the three unaged and 84 days-aged laminates

A brittle collapse of Jute laminate samples is observed, the failure occurs when the critical tensile stress is reached in the external lower jute layers, regardless the aging exposition.

For hybrid composites, a combination of buckling, delamination and tensile failure is evidenced for all the tested samples and the area interested by buckling phenomena becomes larger after the aging exposition.

Laminates' flexural strengths and moduli at varying aging exposition time are showed in Fig. 5.



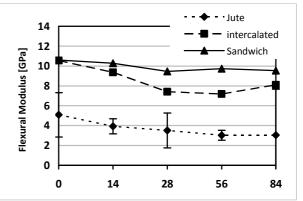


Fig. 5. Quasi-static mechanical properties at varying aging exposition time: (a) Flexural strength and (b) Modulus

Overall, hybrid laminates perform better than jute laminates in terms of quasi-static mechanical properties, regardless the aging condition. Moreover, the hybrid laminate configuration does not affect the flexural properties at the beginning of the aging campaign, whereas Sandwich laminates show higher flexural strength and modulus than Intercalated ones, during the aging exposition time. In all the cases, a decrement of the absolute values of the flexural modulus and strength was registered and it is explained by the occurrence of the degrading phenomena presented before (i.e. free radicals action, photo-degradation, formation of micro-cracks, degradation of the fibre-matrix interface).

As already discussed, for short exposition time, two competitive phenomena mainly influence the quasi-static mechanical properties of the laminates: i.e. post-curing reactions limit the degradative effects due to hygrothermal stress [23, 24] and UV radiation [25, 26].

As for flexural strength, even for modulus, during the artificial aging, Sandwich configuration shows higher values whereas Jute laminates the lower ones, showing a monotonic decrease until a stabilization for long time exposure. Jute laminates evidence the highest degradation rate among the laminates due to the presence of lignocellulosic fibres that, without any protection, tend to mostly suffer the degradative effect due to alternate aging cycles, thus leading to noticeable decrements of flexural stiffness.

Taking into account the presence of basalt external layers in Sandwich configuration, the degradation process acts in a slighter way, thanks to the barrier effect of the outer layers. Between 14 and 28 days, a noticeable drop in stiffness can be observed. It is believed that, in this period, degradation phenomena start to reduce the interlaminar properties and the water absorption resulting in hygroscopic deformation inducing additional stresses that influence the global behaviour of the samples. Moreover, in the first phase of the aging exposition, the presence of higher number of basalt-jute interfaces leads to a more remarkable drop of the flexural modulus for Intercalated laminates. For long exposure time, the flexural stiffness of all the laminates tent to an asymptotic value.

As concerns the flexural strength (Fig. 5a), Jute laminates show a monotonic decrease of this property in the first 28 aging days, followed by a stabilization for long exposure time. For hybridized laminates, a drop in strength is found between 14 days and 28 days of aging that can be explained taking into account both the arising of hygroscopic stresses and the onset of cracks and defects, due to the degradation phenomena. This behaviour is amplified for Intercalated laminates due to higher number of jute-basalt interfaces representing the weakest zones, easily damageable by the aging exposition. To confirm this, SEM micrographs of samples before and after the exposition to artificial aging are reported in Fig. 6.

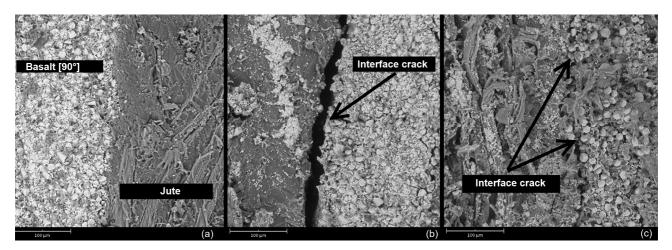


Fig. 6. SEM micrographs of unaged Intercalated (a) and 84 days-aged Intercalated (b) and Sandwich (c) samples

The morphology of Intercalated and Sandwich laminates are shown for unaged and 84 days-aged samples, respectively. For Intercalate laminates, it is possible to notice, comparing Fig. 7a and Fig. 7b, the arising of wide basalt-jute interface cracks due to the aging exposition. On the other hand, smaller and less evident cracks were observed in Sandwich laminates. In addition, different morphology of the jute layers is evidenced by more jagged surfaces probably due to bundle swelling and twisting phenomena as a consequence of the water absorption during the aging exposition.

Overall, it is evident that Sandwich laminates show better aging resistance to the critical environment due to the barrier effect of the external basalt layers that protect the jute internal ones from the degradation phenomena.

#### 2.3 Dynamic mechanical tests

The damping behaviour and the Tg value of a polymeric material depends exclusively on the capability of movement of the macromolecules that constitute the polymer. On the other hand, the incorporation of fibres in a polymeric matrix affects the  $\tan\delta$  (i.e. the damping factor) vs. temperature curve of the composites, due both to the energy dissipation within the matrix and to shear stress concentrations at the fibre-matrix interfaces. Consequently, Tg value and height of the  $\tan\delta$  peak of composites depend on the fibre-matrix adhesion: i.e. a weak filler-matrix adhesion leads to higher values of  $\tan\delta$  peak and lower values of glass transition temperature while a good fibre-matrix adhesion limits the mobility of the polymer chains thus reducing the damping and increasing the Tg.

The following tables show the effect of the aging exposition on the glass transition temperature and  $tan\delta$  peak of Jute and hybrid composites.

As concerns the unaged samples, no noticeable differences in the Tg of the laminates are found. As expected, the Tg of the unaged laminates is equal to about  $60\,^{\circ}\text{C}$  (i.e.  $61.5\pm0.7\,^{\circ}\text{C}$ ,  $62.6\pm3.4\,^{\circ}\text{C}$  and  $62.0\pm2.1\,^{\circ}\text{C}$  for Jute, Intercalate and Sandwich hybrid laminates, respectively). On the other hand, the tan $\delta$  peak of unaged Jute laminates (i.e. 0.32) is higher than those of hybrid ones (i.e. 0.17 for both Intercalated and Sandwich configuration). This difference can be explained taking into account that the fibre-matrix adhesion is weaker for the Jute laminate than for both hybrid laminates, due to the hydrophilic nature of jute fibre and the hydrophobic nature of the epoxy resin used as matrix.

By observing Tables 2 and 3, it is evident a step change in the dynamical mechanical properties of the investigated laminates, as function of the aging time exposition. In the first phase of the aging campaign, noticeable improvements were found in the dynamical mechanical properties (i.e. the glass transition temperature increases and the  $\tan \delta$  peak decreases for each laminate).

Table 2. Glass transition temperature [°C] at varying aging exposition

Aging days	0	14	28	56	84
Jute laminate	$61.5 \pm 0.7$	$67.0 \pm 0.2$	$68.3 \pm 2.0$	$64.7 \pm 0.6$	$64.5 \pm 0.4$
Intercalated laminate	$62.6 \pm 3.4$	$80.2 \pm 0.6$	$93.4 \pm 1.0$	$92.2 \pm 1.5$	92.9 ± 1.4
Sandwich laminate	$62.0 \pm 2.1$	$73.7 \pm 1.8$	$88.9 \pm 3.6$	$95.5 \pm 3.1$	91.9 ± 1.3

Table 3. Tan  $\delta$  peak at varying aging exposition

Aging days	0	14	28	56	84
Jute laminate	$0.317 \pm 0.029$	$0.117 \pm 0.01$	$0.235 \pm 0.001$	$0.234 \pm 0.004$	$0.228 \pm 0.01$
Intercalated laminate	$0.175 \pm 0.018$	$0.152 \pm 0.01$	$0.152 \pm 0.012$	$0.193 \pm 0.007$	$0.195 \pm 0.01$
Sandwich laminate	$0.174 \pm 0.008$	$0.160 \pm 0.002$	$0.150 \pm 0.011$	$0.192 \pm 0.006$	$0.195 \pm 0.01$

This initial behavior is clearly due to the post-curing reactions leading to increase the matrix stiffness. Moreover, the predominant post-curing in the early phase of the aging exposition improves the fibre-matrix adhesion thus limiting the polymer chain mobility.

After this first phase, the dynamic mechanical properties begin to worsen (i.e. the Tg values decrease whereas the tanô peak increases for all the investigated laminates). These results can be explained taking into account that, after a certain period of exposition, the degradative effect of the hygrothermal stress and UV radiation plays a predominant role on the mechanical behaviour of the laminates. As deeper discussed, UV radiation leads to the embrittlement of the materials and formation of micro-cracks thus leading, with the aid of moisture that further accelerates the photo-degradation process, to an overall deterioration in material properties. This means that in the second phase of the exposition, the degradation effect due to the aging environment becomes predominant for the dynamic properties of the laminates.

The above trend inversion has been found for all the investigated laminates even if it occurs at different exposition time as dependence of the lay-up sequence. The Tg of Jute laminates increases until 28 days of aging exposition changing from 61.5 °C to 68.3 °C whereas the tanδ peak decreases until 14 days of aging from 0.32 to 0.12. Afterwards, the Tg and the tanδ peak of the jute reinforced composite reach 64.5 °C and 0.23 after 84 days of artificial aging, respectively. As concerns the hybrid jute-basalt composites, the Tg of Intercalated laminates increases from 62.6 °C to 93.4 °C and the tanδ peak decreases from 0.17 to 0.15, in the first 28 days. Otherwise, in the aging time interval between 28 days and 84 days, the Tg remains almost constant (i.e. equal to 92.9 °C at the end of the aging exposition) whereas the tanδ peak slightly increases up to 0.19. On the other hand, the Sandwich laminate configuration shows an increment of the glass transition temperature in the first 56 days of aging (i.e. from 62.0 °C to 95.5 °C) and decrease of the peak height from 0.17 to 0.14 in the first 28 days of aging. After this first phase of aging, the Tg and the tanδ peak of jute-basalt sandwich structures invert their trends reaching 91.9 °C and 0.20 after 84 aging days, respectively.

These results mean that, the degradative phenomena due to the alternate cycles of hygrothermal stress and UV radiation become predominant in comparison to the post-curing reactions between 14 and 28 days of aging for the Jute laminate. This change happens after about 28 days and in the interval time between 28 and 56 aging days for Intercalated and Sandwich hybrid laminates, respectively. Consequently, it is possible to state that the presence of the external basalt layers in hybrid structures screens the internal jute layers from the degradation phenomena thus delaying their worsening effect on the dynamic mechanical properties of the composite laminates. By comparing the hybrid structures, it is evident that thicker external basalt layer in the stacking sequence of the

Sandwich configuration allows to postpone the degradative effect of the aging exposition, in comparison to the intercalated hybrid configuration.

### 2.4 Impact tests

The evaluation of the impact strength gives important information about the fibre-matrix interfacial adhesion and the properties of the matrix and the fibre. In particular, when a fibre reinforced material undergoes a sudden load, the impact energy is dissipated by the combination of fibre pull-outs, fibre fractures and matrix deformations.

As shown in Fig. 7, it is evident that the capability to absorb impact energy is higher for both hybrid laminates than Jute laminates, regardless the aging time exposition. The impact strength of unaged Jute samples (i.e.  $11.2 \text{ kJ/m}^2$ ) is about 1/7 than those of unaged hybrid laminates (73.6 kJ/m² and 74.3 kJ/m² for Intercalate and Sandwich configurations, respectively). This means that the use of basalt fibres as reinforcement of the outer layers allows to remarkably decrease the impact energy of Jute laminates. Moreover, it is evident that the stacking sequence of the hybrid laminates does not influence the impact strength of these composites when not jet exposed to artificial aging: i.e. the impact energy capability does not change if the jute and basalt layers are Intercalated or they are stacked as Sandwich configuration.

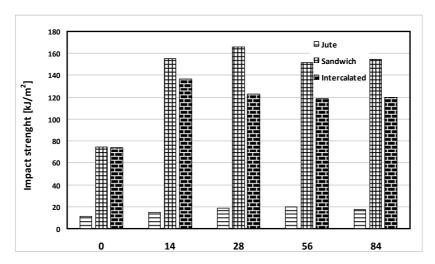


Fig.7. Impact strength [kJ/m<sup>2</sup>] at varying aging exposition

Even at the end of the aging exposure, after 84 days, Jute laminates still show the lowest impact energy capability in comparison to hybrid laminates (i.e. 88.6% and 91% lower than those of Intercalated and Sandwich laminates, respectively).

As concerns the effect of the aging exposition on the impact energy, it is worth noting that all the investigated laminates increase their impact energy capability in the first phase of the aging exposition. Afterwards, the impact strength of the laminates begins to decrease.

The impact energy of Jute laminate increases until 56 aging days from 11.2 kJ/m² to 19.7 kJ/m², then decreasing to 13.5 kJ/m² after 84 days. On the other hand, Intercalated hybrid laminates experience an improvement of the impact strength just in the first 14 days of aging exposition (from 73.6 kJ/m² to 136.6 kJ/m²) and subsequently it reaches a plateau (~120 kJ/m²) during the remaining part of the aging exposition (i.e. in the interval time 28-84 days). As concerns Sandwich laminates, the impact strength increases until 28 aging days (from 74.3 kJ/m² to 156.5 kJ/m²) then it decreases to about 150 kJ/m² in the final phase of the aging exposition.

The improvement of the impact energy experienced by the laminates in the initial phase of the aging campaign can be explained taking into account the plasticization effect due to the water exposition that leads to an increase of the mobility of the polymer chains. This phenomenon is clearly evident also observing the deformation at break improvements showed by the laminates during the aging exposition. Consequently, it is possible to state that the resin plasticization can be

considered as the predominant phenomenon that mainly influences the impact properties of the laminates in the early phase of the aging exposition.

Likewise the dynamic mechanical properties, the decrement of the impact strength experienced by the laminates in the final phase of the aging campaign can be attributed to the degradative effect of the hygrothermal stress and UV radiation that becomes predominant leading to the material embrittlement and, consequently, to the micro-cracks formation.

Overall, it is possible to state that hybrid laminates perform better than Jute laminates also in terms of impact properties (both at the beginning of the aging campaign and during the entire aging exposition). Moreover, the hybrid laminate configuration affects the aging resistance of the structures, with Sandwich laminates that outperform Intercalated ones. Indeed, although the hybrid laminates show similar impact energies in the "unaged" state (i.e. 73.6 kJ/m² and 74.3 kJ/m²), the Sandwich stacking sequence allows to reach higher impact energy (i.e. 150.3 kJ/m²) than the Intercalated one (i.e. 118.4 kJ/m²), after 84 aging days. These results mean that the degradative effect due to hygrothermal stress and UV radiation is attenuated by thicker external basalt layer of Sandwich configuration thus emphasizing the positive effect of the resin plasticization on the impact energy of the laminates.

#### 3. Conclusions

From the experimental results, it is possible to assert that the interplay hybridization represents a promising solution to overcome those drawbacks that characterize the use of jute fibres as reinforcement in composite materials for outdoor applications.

In particular, Sandwich laminates showed better aging resistance to the external environment than Intercalated laminates, due to the barrier effect of the external basalt layers that protect the jute internal ones from the degradation phenomena. This is reflected by the flexural quasi-static properties that highlighted a slighter influence of the aging environment on the Sandwich laminates if compared to the other studied hybrid configuration. Overall, Jute laminates showed lower flexural properties than hybrid ones, regardless the aging exposition time.

Moreover, it was shown that the use of thicker external basalt layer, made of two basalt layers, in the Sandwich configuration allows to postpone the degradative effect of the aging exposition on the dynamic mechanical properties. Similarly, the worsening of the impact properties of Sandwich laminates is attenuated in comparison to Intercalated laminates.

In terms of durability, the hybrid laminate composites showed higher initial mechanical performance compared to Jute laminates, suffering more intense decrease in flexural strength due to artificial aging.

Comparing the two hybrid laminates, the Sandwich configuration is more suitable than the Intercalated one for the realization of technical elements more lasting over time, giving higher static and dynamic performance.

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