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# Hybrid biocomposites: properties and performance for exoskeleton applications

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**Abstract:** Bio composites are biocompatible composites, having organic or inorganic components in their composition. A 100% ecological bio composite material consists of a matrix (resin) and reinforcement represented by natural fibers. The matrix can be from two resources, i.e. renewable or non-renewable polymers. The matrix has a major importance in protecting the reinforcement against exposure to the environment through mechanical and/or chemical damage and for supporting the loads. The paper analysis the hybrid (basalt/flax) bio composites for determination of mechanical properties on DMA tests, in order to evaluate possibility to use the composite bio basalt/flax made of 8-ply flat woven laminates for realizing exoskeleton for transport and weights handling and to compare the performance for the stacking configurations considered.

Keywords: bio composites, natural fibers, mechanical, DMA

# **1. INTRODUCTION**

The composites industry is in a permanent modeling, led by the market, by the component materials and by their technology. The components of a composite, the matrix and the fibers, play a significant role in the structure and contribute greatly to the remaining on the market. Biocomposites are considered as new materials from the fourth generation, having organic or inorganic components [1]. Biocomposites have natural fibers as reinforcement in their structure, being an alternative to composites reinforced with synthetic fibers (glass, carbon)[2]. Depending on their origin, natural fibers can be obtained from plants, animals or minerals. Used alone or in hybrid combinations with already tested materials (glass fibers or carbon fibers), the organic materials can improve quality by being lighter and visually attractive. Thus, flax can compete with glass fibers due to the breaking resistance given by the long cellulose fibers inside the stems of the plant. Natural fibers have good sustainability [3] but they have disadvantages (sensitivity to moisture, their structure depends on the growing conditions and the harvesting period), leading to an uncertainty of the final properties as low thermal stability. Adherence to matrix and the tendency to form agglomerates with resins implies the solutioning of

manufacturing technological problems and a control of the behavior in the intended applications [4]. Basalt fiber with mineral origin from volcanic rock is obtained by extruding the melted material into fine fibers, used for reinforcing composite with durability and good heat resistance.

The transport industry requires the movement and handling of heavy materials. Adding a wearable could assist the transport activity or workflow in some areas. Thus, the optimal design of the bio-composites used in exoskeleton construction (for transport and weights handling), require to know the mechanical properties of the component materials. For this, the influence of the orientation and stacking sequences of the basalt and flax layers over mechanical properties were evaluated, the results were compared with those of carbon fiber reinforces plastics with same characteristics.

#### 2. SPECIMEN PREPARATION AND METHODS

The influence of stacking sequences on the mechanical properties of composites is recognized [5], the elastic properties of the composite are dependent on the significant differences in the packaging of the laminae as well as the laminae themself [6]. The fibers provide strength and rigidity and act as reinforcement in composites and determine their properties. The unidirectional fibers in the fabric structure obtained in two layers sewn together give it improved mechanical properties. The rigidity of the laminate is given by the twill 2/2 fabric and the twist-free threads. In the case of basalt, the multiaxial fabric leads to a significant increase in the modulus of elasticity and resistance, promoting them to construction of ships, cars, high-speed trains, wind blades.

In the case of the studied biocomposites, for the evaluation of their mechanical performances, plates having 295x205 mm<sup>2</sup> with a thickness of 5.38 mm for flax and 3.45 mm for basalt were made by the vacuum infusion process with eight layers of fabric for both types of fibers. The stacking order for flax was  $[0^{\circ}]_{8}$  and  $[45^{\circ}]_{8}$  and for basalt it was  $[+45^{\circ}/-45^{\circ}]_{4}$  respectively  $[0^{\circ}/90^{\circ}]_{4}$ . For both materials, bioepoxy with 56% of plant source GreenPoxy 56 and multipurpose hardener series SD7561 with a high carbon content of vegetable origin were used. In order to compare the performance of bio composites with those of the carbon fiber reinforced polymer (CFRP), plates with dimensions of 295x205x3.20mm<sup>3</sup> were made by the same process. The multilayer stacking sequence of the T240 type pre-impregnated carbon fibers for FRP plates is face and opposite surface  $[0^{\circ}/90^{\circ}]$  with intermediate layers  $\{[0^{\circ}]_{3}$  and  $[+45^{\circ}/-$ 45°]<sub>2</sub>}. Table 1 shows mechanical properties of different bio fibers/CF.

Tables1. Mechanical properties of different bio fibers/CF					
Materials/ width (mm)	V <sub>f</sub> (%)	Areal weight (g/m2)	Tensile Strength of filament (MPa)	Young's modulus (GPa)	Elongation at break (%)
Flax- 5.38	30-35	200-350	500-900	50-70	1.5-4.0
Basalt – 3.45	50	620	3000-4840	79.3-110	3.1-3.2
CF – 3.43	45	193	3600-4100	235	2.8

Changing the stacking sequence generally modifies the parameters of the composite and joining with adhesives generates a behavior that is influenced

both by the total stacking sequence, the sequence and the local distribution of the layers near the interfaces. Dynamic mechanical analysis (DMA) specimens were prepared in accordance with ASTM D5023 [14] for testing with DMA 242C – Netzsch Germany, three-point bending test using Protheus software v.4.8.5 under sinusoidal cyclic loading. The maximum allowed dimensions of the specimens are  $50 \times 10 \times 5 \text{ mm}^3$ . The viscoelastic properties of the specimens as complex modulus of elasticity E', the loss modulus E'', the damping or loss factor, tan  $\delta$ , depending on the temperature were determined in the range of  $25^{\circ}\text{C}-250^{\circ}\text{C}$ . The parameters obtained provide information on vitrification, referred to T<sub>g</sub> (glass transition), resulting from the cross-linking reaction. The apparent activation energy for the glass transition process, at a preload of 6N was determined for 1 and 5Hz in a temperature-controlled room.

The absence/presence of porosity in the samples were analyzed using the US method. For this, the PHASOR XS equipment coupled with a phased array with 32 US sensors working at a frequency of 5MHz and a delay line was used.

#### **3. RESULTS AND DISCUSSION**

Through their microscopic structure, bio composites are made of fibers embedded in a matrix, arranged with different orientations to the longitudinal axis, forming a layered composite. Figure 1 shows the non-crimp biax fabric used as reinforcements in bio-composites.





**Figure 1:** Reinforcements used a) bidirectional flax 0/90; b) basalt woven fibers 0/90/±45

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Composite interaction with the ultrasounds (US) depends on the US energy but also on its structure as the dimensions and cohesion of the fibers, the quality and composition of the bio-resin, the moisture content, the temperature of the environment and, in particular, the orientation of the wrapping sequences in relation to US source. The time-of-flight method is the common technique in US testing. From the time interval between transmission and reception, either the speed of sound or the distance traveled is obtained. The effect of the percentage of fibers ( $V_f$ ) in the composite mass and the orientation of the fibers correlates with the speed of the longitudinal US waves as well as with the attenuation of the US wave [7]. For the fiber content for flax alkali treated,  $c_f=2764$  m/s was obtained and for basalt  $c_b=2556$  m/s. The porosity content of the material is directly related to the pressure applied during the hardening of the composite. The pressure regulation can control the volume of void content in the first sequence. Here, the porosity was assessed by image analysis, knowing that the degree of porosity is in linear correlation with the US attenuation in composites (Figure 2). The displacement of the transducer and establishing the tested location was achieved using an encoder.



Figure 2: Combined A-scan and sectorial scan views for: a) flax; b) basalt; c) CF

The US tests were performed in approximately 20-25 areas of the bio and carbon epoxy composites. It is possible to evaluate the way of distribution of the fibers as well as the lack of delaminations/gaps. Thus, from figure 1b, it can be seen that the basalt has a different arrangement of fibers near the surface compared to those in the volume. It is found that flax and basalt fibers have a higher attenuation compared to FRP, the US absorption coefficient is defined as the energy absorbed reported to the total energy used [8].

The DMA tests performed for the samples show that the changes in the dynamic mode of the materials under the vibration load with temperature differ for the type of bio-composites compared to the carbon-based composites. The mechanical properties as well as the effect of the frequency on the viscoelastic properties of the composites are presented in figures 3-5.



The loss modulus E' (figure 3) measures the energy absorbed by the composite during the oscillation cycle. For  $V_f=50\%$  fibers E'=6GPa for flax and 15.8GPa for basalts. It can be observed that, at the same stacking sequence, the modulus of elasticity for basalt decreases much faster in the range of 80-100°C compared to flax, while the loss modulus also for basalt fluctuated violently for the same temperature range. This indicates that temperature has a significant role on the interface's properties and adhesion activity of the resin. The stress is distributed equally in both directions. It is observed that the variation of E' of the biocomposites is greater in the glassy region, so that later at a temperature of 80°C it decreases due to the reinforced fiber that loses its rigidity at elevated temperatures. The difference between the properties of the composites with flax and basalt fibers after the temperature of 100°C is no longer significant. The transition temperature to the vitreous state for both types of fibers in this packaging does not differ significantly with frequency, thus at 1 and 5 Hz for basalt  $T_q = \{85.2; 86.6\}^{\circ}C$  and for flax  $T_q = \{80.4;$ 82.2}°C. The analysis shows that T<sub>q</sub> represents the critical limit of good functioning of the composite material. The advanced characterization provides data for knowing the performance/limits of the use of bio-epoxy composites.



In relation to the packing mode, it can be observed that due to the presence of fibers with [0/90]<sub>4</sub> orientation, the flax and basalt composites in the vitreous region have an increased storage modulus compared to figure 3. The same behavior is noted for the E' and E" values at the 80-100°C range for the glass transition region, E" provides information on the amount of energy dissipated in the form of heat per oscillation cycle. The increased values may be due to the 0° orientation of the fibers, knowing that the unidirectional shows an increased stiffness. A slight increase in  $T_q = \{91.4; 92.8\}^{\circ}C$  for basalt and  $T_q = \{83.6; 86.5\}^{\circ}C$  for flax at 1 and 5 Hz. As the material passes from the vitreous state to the "highly elastic" state, the properties of the biopolymer become predominant. Tan $\delta$  (damping) measures the impact properties of the biocomposite. Tan $\delta$  for  $[0/90]_4$  has a lower value, because the bioepoxy resin has a high molecular mobility that is stopped by the incorporation of fibers in the given structure. Thus, the strong adhesion of the fibers to the matrix is established, resulting in a uniformly distributed tension. The lower damping curve for flax compared to basalt indicates that the material can absorb more energy, becoming stiffer at a slightly higher temperature. The curves have a high peak as the frequency increases. The DMA analysis of FRP in the presented stacking mode, shows that as the temperature increases and at the two vibration frequencies, the loss modulus E' respects the three transition zones of the phases.



The vitrous state remains approximately constant up to  $80^{\circ}$ C so that then in the vitreous state a slight increase can be noticed at a temperature of  $125^{\circ}$ C, after which it decreases in the range [130-150°C]. For both directions of FRP testing (given by the layout of the layers) the transition temperature to the vitreous state is T<sub>g</sub>={115.8; 119.3°C}. The orientation of the fibers seems to reduce the variation range of T<sub>g</sub> by a few degrees and therefore increases T<sub>g</sub> as the critical upper limit for safe design. The same behavior of rapid decrease of E' is noted due to the reaction of the resin groups in the matrix. The loss modulus curve fluctuates violently with temperature between [110-150°C], the area demonstrating a high energy dissipation, the synthetic epoxy sample has the peak at a higher temperature than the bio epoxy samples but having close transition temperatures. FRP has a narrower width of the E" curve compared to bio-composites. Tan $\delta$  which provides information on the fiber and matrix interaction, measures the impact properties of FRP. The peak value is close to that of the flax biocomposites with the sequence [0]<sub>8</sub>, demonstrating a high

molecular mobility that is stopped by the incorporation of fibers in the given structure. The strong adhesion of the fibers with the matrix, in the created structure, results in a uniformly distributed tension.

# 4. CONCLUSIONS

The research demonstrated that the use of biocomposites with natural fibers ie flax or basalts with the volume of fibers Vf=50% could replace with good results the composites reinforced with carbon fibers in the face and opposite surface structure  $[0^{\circ}/90^{\circ}]$  with intermediate layers  $\{[0^{\circ}]_{3} \text{ and } [+45^{\circ}/-45^{\circ}]_{2}\}$  in making the exoskeleton for transport and baggage handling. The prospects of using basalt and flax fibers for the production of bio composite materials with a complex of improved mechanical properties were highlighted. The volume fraction of flax fiber/basalts is very important for establishing the quality of the biocomposite designed for exoskeleton application. It directly affects both mechanical performance and vibration resistance. The width of the curve could suggest the degree of homogeneity of the composite. The informative characterization parameters were analyzed the complex modulus of elasticity E, the loss modulus E", which represents the viscous component of the material and the damping or loss factor, tan  $\delta$ , in a temperature range following the dynamic behavior of the biocomposite after passing over transition temperature to the vitreous state. The integration of monitoring improves the understanding of the mechanical behavior of biocomposites, the correlation of dynamic parameters with the dominant stresses in the realization of the exoskeleton for transport and baggage handling could offer possibilities for stress/damage prediction in these applications.

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# References

- [1] Faruk O., Bledzki A.K., Fink H.P. and Sain M., 2012. Biocomposites reinforced with natural fibers: 2000–2010. Progress in polymer science, 37(11), pp. 1552-1596.
- [2] Akampumuza O., et al., 2017. Review of the applications of biocomposites in the automotive industry. Polymer composites, 38(11), pp.2553-2569.
- [3] Bogard F., et al., 2022. A comparative review of Nettle and Ramie fiber and their use in biocomposites, particularly with a PLA matrix. Journal of Natural Fibers, 19(14), pp.8205-8229.
- [4] Xu X., Hu D. and Ma W., 2021. Synergistic improvement of mechanical and thermal properties in epoxy composites via polyimide microspheres. Journal of Applied Polymer Science, 138(35), p.50869.
- [5] Mohanavel V., et al. 2022. Influence of stacking sequence and fiber content on the mechanical properties of natural and synthetic fibers reinforced penta-layered hybrid composites. Journal of Natural Fibers, 19(13), 5258-5270.
- [6] NagarajaGanesh B. and Rekha B., 2020. Intrinsic cellulosic fiber architecture and their effect on the mechanical properties of hybrid composites. Archives of Civil and Mechanical Engineering, 20, pp.1-12.
- [7] Faktorová, D., et al. (2024). Analysis of the Anisotropy of Sound Propagation Velocity in Thin Wooden Plates Using Lamb Waves. Polymers, 16(6), 753.
- [8] Markiewicz E, PauksztaDand Borysiak S 2012 Acoustic and dielectric properties of polypropylene-lignocellulosic materials composites, Polypropylene InTech. 30:193-217.