ELABORATION OF HYBRID BIO-COMPOSITES WITH THERMOPLASTIC MATRIX: MATERIAL FORMULATION AND MODELLING OF THE QUASI-STATIC BEHAVIOUR

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ABSTRACT

Environmental impact is becoming increasingly important in the automotive industry, with car manufacturers looking to reduce CO2 emissions through cleaner engines and structural weight reduction. Composite materials offer an excellent alternative to standard steels with significant weight reduction and the ability to produce functional parts [1],[2],[3]. The main objective of this study is to investigate the potential of a new and unique hybrid bio-composite material combining flax and basalt fibers and PA11 polymer. This material design is studied with the idea of reducing the moisture sensitivity, variabilities and uncertainties of vegetal fibers by the presence of basalt fibers. The first step consists of developing a new hybrid composite material and studying its quasi-static mechanical behaviour when subjected to different humidity levels. Then, a multi-scale non-linear homogenization approach is proposed to support the interpretation of the characterization test results. The experimental results show the benefits of the hybridization. In fact, the hybrid composite is 20% stiffer than the flax/PA11 composite, 8% lighter than the basalt/PA11 composite and has a reduced dispersion of its mechanical properties, 52 % lower than the flax/PA11 composite.

1. INTRODUCTION

The application of composite materials remains limited in the automotive industry due to various technical, economic, and environmental constraints. Knowledge of the technical aspects of manufacturing (processes and production rate), the mechanical behaviour of these heterogeneous materials (behaviour law and simulation tools) and their lifespan are still insufficient for mass production. On the economic aspect, the high price of fibers, such as carbon fibers, and the high manufacturing cost related to cycle time are the main obstacles to developing these materials. The main objective of this research is to define a new concept for semi-structural automotive parts (hood, door, dashboard…) that can meet the various industrial requirements while respecting environmental regulations. Therefore, the initial work has focused on selecting materials from a wide range of vegetal fibers and bio-sourced polymers with suitable properties. Then, a focus was made on the characterization of hybrid composite materials through various quasi-static mechanical tests at different humidity levels. Hybridization is a frequently employed process to create intermediate properties between the two original materials [4].

Hybridization can result in a trade-off between mechanical properties and economic cost in order to meet the application requirements. Several recent studies show that the mechanical properties can be adapted by using hybridizations based on basalt fiber laminates with other natural fibers [5], [6], [7], intending to provide a more durable material. These studies mainly focused on the impact and mechanical characterization properties of a basalt/flax hybrid composite. All the composites involved were thermoset composites (epoxy, vinylester) with laminate hybridization. This study analyzed an original combination of flax and basalt fibers and a thermoplastic matrix (PA11) with a fabric scale hybridization by co-weaving. The hybridization concept uses two or more fibers reinforcing the same matrix. It provides sufficient flexibility to design the optimal material [10], [11],[12]. The main idea is to mitigate the weaknesses of one of the fibers while keeping the advantages of the other(s) [13],[14],[15]. This composite material offers many advantages but requires in-depth study to define the best combination of compatible fibers [14].

A first selection of natural fibres was made based on studies of bio-composite materials and their application fields [8],[9]. The industrial aspect of fibre production (quantity and quality of the products) represented an essential factor in this selection. It was decided to select flax and basalt as fibre reinforcement and a PA11 matrix from Arkema [9]. These different constituents are compatible and have complementary mechanical properties. Flax fiber has good mechanical properties [8] and low density (1.4 g/cm^3) [8], while basalt fiber provides high stiffness (Young's modulus of 93 GPa) [8], high moisture resistance (water absorption rate of 0.03%) [8] and excellent temperature resistance (maximum application temperature: 850°C) [8]. The choice of a thermoplastic matrix and natural fibers improves the recyclability of this hybrid bio-composite.

This article is structured as follows. First part concerns the results of the experimental study of the developed materials, basalt/PA11, flax/PA11 and hybrid5050/PA11 for different directions (0°/45°/90°). Second part is dedicated to the numerical study, we used Multiscale Designer (Altair) to predict the mechanical response of different RVEs beyond elastic regimes.

2. MECHANICAL TESTS

The main objectives of the experimental phase were to identify the mechanical characteristics (Young's modulus, Shear modulus, Poisson's ratio, strength and strain at failure…) of the composites flax/PA11, basalt/PA11 and hybrid50/50. Then to understand the evolution of the hybrid5050/PA11 mechanical behavior according to the different humidity ranges RH0, RH50 and RH85 the quasi-static tensile and in-plane shear tests were carried out on a universal testing machine (INSTRON 5584). The specimens were manufactured by thermocompression in accordance with the ISO527 standard as follow: flax/PA11 (twill, 4 plies, 2 mm thick, Vf 42%), basalt/PA11 (UD, 4 plies, 2 mm thick, Vf 27%) and hybrid5050 (twill, 4 plies and 2 mm thick, Vf 33 %).

Quasi-static tests have been carried out in three Relative Humudity (RH0, RH50 and RH85) at ambient temperature(+23°C) and for different directions (0°/45°/90°). The 0° orientation provides the longitudinal mechanical properties, the 45° orientation gives the shear properties, and the 90° orientation provides the transverse mechanical properties of the composite. Five samples were used for each of the tests reported in Table 1 to ensure the reproducibility of the results.

	Testing directions	Relative humidity RH T +23°C
Flax twill/PA11	0°/45°/90°	RH0/RH50/RH85
UD Basalte/PA11	0°/45°/90°	RH0/RH50/RH85
Twill Hybrid5050/PA11	0°/45°/90°	RH0/RH50/RH85

Table 1:Characterization test matrix for flax/PA11, basalt/PA11, hybrid5050/PA11

3. EXPERIMENTAL STUDY

Figure 1 compares the different mechanical behaviors of the flax/PA11, basalt/PA11 and hyb5050/PA11 composites. True stresses were calculated to take into account the potential effects of large deformations. The true stress σ and strain ε are determined from:

$$\sigma = \sigma_n(1+\varepsilon_n) \quad (2)$$

$$\varepsilon = ln\,(1+\varepsilon_n) \quad (3)$$

$$\varepsilon_n = \frac{l-l_0}{l_0} \quad (4)$$

$$\sigma_n = \frac{F}{S_0} \quad (5)$$

ε_n : nominal strainσ_n: nominal stress

F: applied forceS_0: samples initial section

l: samples lengthl_0: samples initial length

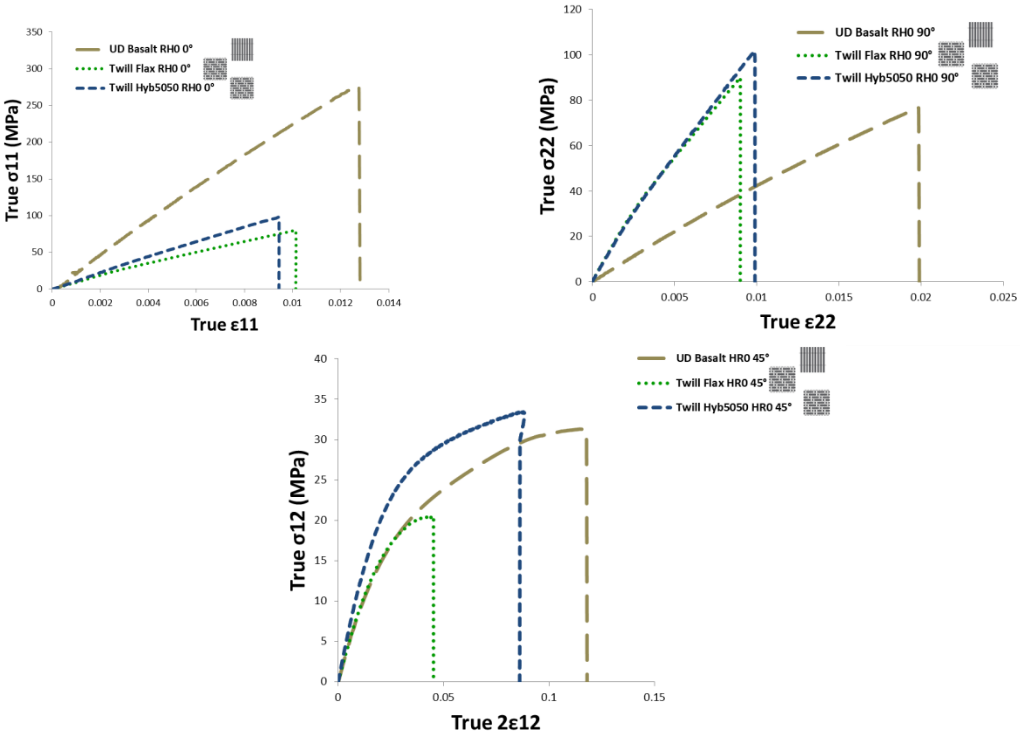


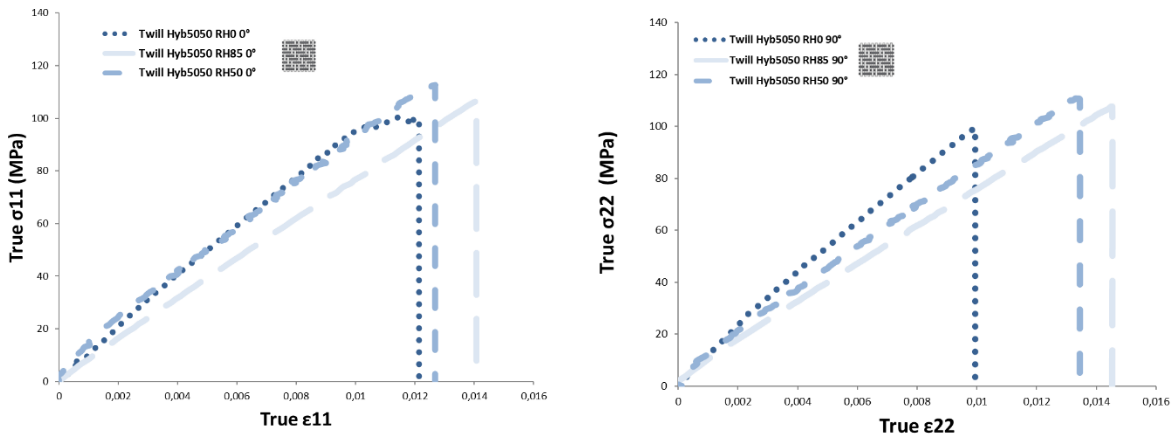
Figure 1: σ true= f (ε true) in the global coordinate system for the flax/PA11, Hyb5050/PA11 & basalt/PA11 composites for RH0 and 0°,45° and 90° at 23°C

The basalt/PA11 composite is stiffer than the hybrid 5050/PA11 and flax/PA11 composites for the 0° direction due to the relatively high mechanical properties of the basalt fiber [18] compared to the flax fiber [8]. Furthermore, the basalt/PA11 composite is unidirectional, and the hybrid5050/PA11 and flax/PA11 composites are balanced. The deformation of the basalt composites is relatively higher than that of the hybrid 5050/PA11 and flax/PA11 composites for the different directions (0° and 90°). As conclusion, the hybridization has improved the mechanical characteristics of the hybrid 5050/PA11 versus he flax/PA11 composite. Figure 1 shows that the hybrid composite is 20% stiffer than the flax composite. As expected, the Young modulus E22 of the unidirectional basalt/PA11 is significantly lower than the hybrid5050/PA11 and flax/PA11 woven composites (Figure 1). Also, the longitudinal and transverse Young's moduli of the 5050/PA11 and Flax/PA11 are very similar because they are made from balanced twill fabrics. The different mechanical properties of the studied composites are reported in Table 2. It is important to emphasise that it is possible to have a more consistent comparison if fibre content Vf for the hybrid5050/PA11 and flax/PA11 composites were the same. In fact, the fibre content for the different composites: flax/PA11 (Vf 42%), basalt/PA11 (Vf 27%) and hybrid5050/PA11 (Vf 33%) are different. This difference in fibre content is due to the process of powdering the fabric during the pre-impregnation manufacturing (50% by weight for the fibres, 50% by weight for the matrix). The hybrid5050/PA11 composite would have better mechanical properties with a higher fibre content, thus highlighting the interest in hybridization.

	Flax		**Basalt**		**Hyb 50 50**	
0°	**E11(MPa)**	**υ 12**	**E11(MPa)**	**υ 12**	**E11(MPa)**	**υ 12**
	10100 ±1340	0.04±0.02	23300±3900	0.18±0.02	10220±650	0.05±0.05
	σ 11 failure (MPa)	**ε 11 failure**	**σ 11 failure (MPa)**	**ε 11 failure**	**σ 11 failure (MPa)**	**ε 11 failure**
	60±11	0.008±0.01	255±37	0.01±0.001	70±17	0.01±0.04
90°	**E22(MPa)**	**υ 21**	**E22(MPa)**	**υ 21**	**E22(MPa)**	**υ 21**
	12807±1200	0.05±0.153	4220±545	0.032±0.07	11400±680	0.50±0.2
	σ 22 failure (MPa)	**ε 22 failure**	**σ 22 failure (MPa)**	**ε 22 failure**	**σ 22 failure (MPa)**	**ε 22 failure**
	75±4	0.007±0.002	57±4	0.015±0.01	85±3	0.008±0.0003
45°	**G12(MPa)**		**G12(MPa)**		**G12(MPa)**	
	949±143		801±156		1240±150	
	σ 12 failure (MPa)	**2 ε 12 failure**	**σ 12 failure (MPa)**	**2 ε 12 failure**	**σ 12 failure (MPa)**	**2 ε 12 failure**
	35±5	0.06±0.032	54±10	0.14±0.04	55±5	0.1±0.02

Table 2: Mechanical characteristics of Hybrid5050/PA11, basalt/PA11 and flax/PA11 composites for RH0 and for directions (0°/45°/90°) at 23°C.

The second phase of the experimental study investigates the effect of moisture on the evolution of the mechanical behaviour of our materials, flax/PA11, basalt/PA11 and Hybrid 5050/PA11 composites. In this paper a focus is placed on the impact of moisture on the mechanical behaviour of the hybrid 5050/PA11 composite. It will enable to verify the interest of hybridization in reducing the material's sensitivity to humidity. Figure 2 shows the evolution of the mechanical properties of hybrid5050/PA11 for the three moisture levels RH0, RH50 and RH85:



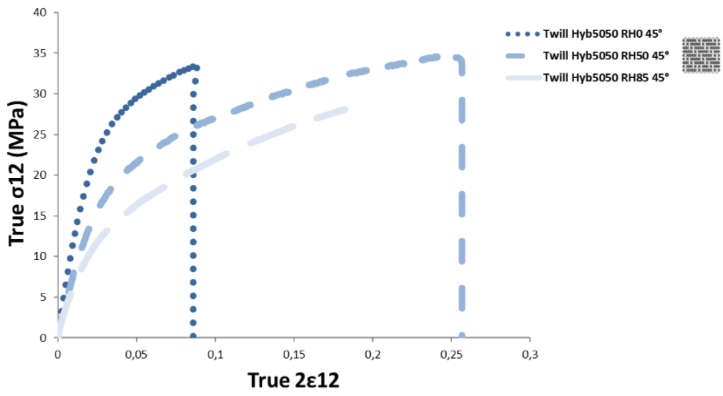


Figure 2 σ true= f (ε true) for the tensile test of hybrid5050/PA11 for RH0 RH50 & RH85 at 23°C

The different mechanical properties of the hybrid5050/PA11 composite for the different Relative humidity (RH0, RH50 & RH85) are reported in Table 3

	hybrid5050/PA11					
RHO	Orientation 0°		Orientation 45°		Orientation 90°	
	$E_{11}(MPa)$	$\nu_{12}\ (-)$	$G_{12}\ (MPa)$		$E_{22}(MPa)$	$\nu_{21}\ (-)$
	10220 ±650	0.05±0.05	1240±150		11400±680	0.05±0.2
	$\sigma_{11}^{R}\ (MPa)$	$\varepsilon_{11}^{R}\ (-)$	$\sigma_{12}^{R}\ (MPa)$	$\gamma_{12}^{R}\ (-)$	$\sigma_{22}^{R}\ (MPa)$	$\varepsilon_{22}^{R}\ (-)$
	70±17	0.01±0.04	55±5	0.1±0.02	85±3	0,008±0.0003
RH50	Orientation 0°		Orientation 45°		Orientation 90°	
	$E_{11}(MPa)$	$\nu_{12}\ (-)$	$G_{12}\ (MPa)$		$E_{22}(MPa)$	$\nu_{21}\ (-)$
	9440±390	0.09±0.03	828±90		8886±401	0.08±0.03
	$\sigma_{11}^{R}\ (MPa)$	$\varepsilon_{11}^{R}\ (-)$	$\sigma_{12}^{R}\ (MPa)$	$\gamma_{12}^{R}\ (-)$	$\sigma_{22}^{R}\ (MPa)$	$\varepsilon_{22}^{R}\ (-)$
	95±9	0.01±0.001	58±2	0.2±0.08	92±4	0.01±0.001
RH85	Orientation 0°		Orientation 45°		Orientation 90°	
	$E_{11}(MPa)$	$\nu_{12}\ (-)$	$G_{12}\ (MPa)$		$E_{22}(MPa)$	$\nu_{21}\ (-)$
	7462±360	0.1±0.009	623±62		7428±175	0.09±0.004
	$\sigma_{11}^{R}\ (MPa)$	$\varepsilon_{11}^{R}\ (-)$	$\sigma_{12}^{R}\ (MPa)$	$\gamma_{12}^{R}\ (-)$	$\sigma_{22}^{R}\ (MPa)$	$\varepsilon_{22}^{R}\ (-)$
	110±55	0.004±0.005	47±1.5	0.008±0.002	94±5	0.01±0.005

Table 3 Mechanical characteristics of hybrid5050/PA11 composites for RH0, RH50 & RH85 and for directions (0°/45°/90°) at 23°C.

From the values in Table 3, We find that the stiffness of the hybrid5050/PA11 composite decreases as the moisture level increases: the longitudinal modulus E11 decreases from 10220 MPa (RH0) to 7462 MPa (RH85), the transverse modulus E22 decreases from 11400 MPa (RH0) to 7428 MPa (RH85) and the shear modulus decreases from 1240 MPa (RH0) to 623 MPa (RH85). We note that the reduction in mechanical properties of the hybrid5050/PA11 composite is lower than the flax/PA11 composite. It can be explained by the sensitivity of both the flax fiber [16], [19], [20] and the PA11 matrix [17]. For RH85 the longitudinal modulus of the hybrid5050/PA11 is 7462 MPa whereas the longitudinal modulus of the flax/PA11 composite is 5001 MPa, a difference of 32%. The inclusion of basalt fibres has considerably reduced the effect of humidity on the mechanical behaviour of the 5050/PA11 hybrid composite.

Following the mechanical characterization tests of the different materials studied, a microscopic study was conducted using a Scanning Electron Microscope (Figure 3). There are different damage mechanisms for composite materials depending on the nature of the fibres, the matrix and the fabric used. The damage modes are fibre breakage, fibre/matrix debonding and matrix cracking. The analysis of the SEM results (Figure 4) shows that fibre breakage as the damage mode for the flax/PA11 composite is fibre breakage and fibre/matrix delamination for the basalt/PA11 composite. These damage modes were retained for the hyb5050/PA11 composites.

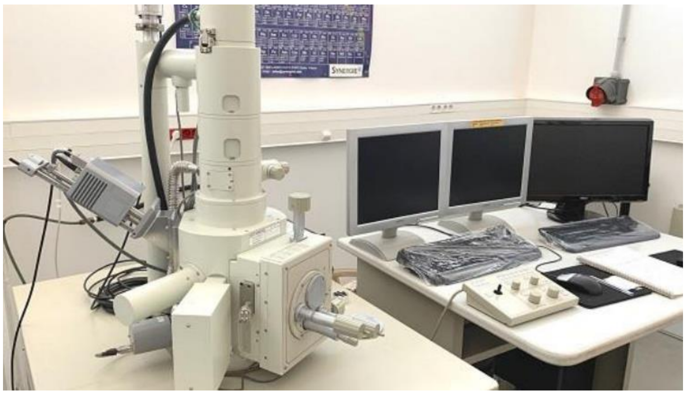
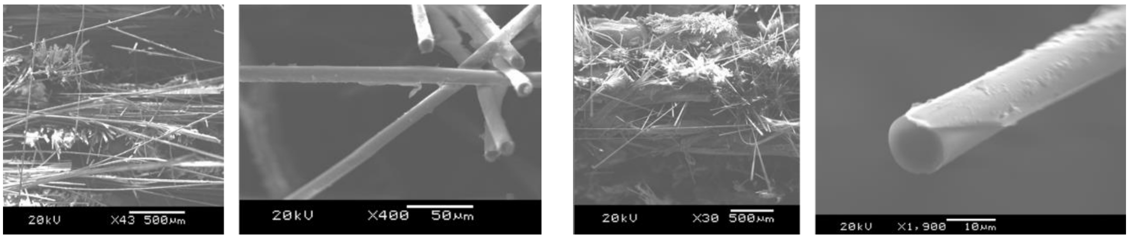


Figure 3 Scanning Electron Microscope (SEM)



Flax/PA11 specimen *Basalt/PA11 specimen*

Figure 4 SEM view of flax/PA11 and Basalt/PA11 composites after 0° quasi-static tensile tests

4. NUMERICAL STUDY

The second part of the research work is devoted to the numerical study based on homogenization methods; it allows the prediction of the mechanical behaviour of a heterogeneous material from the mechanical properties of its phases. It will be able to determine the effective properties at the macroscopic scale of the material from a Representative Elemental Volume (REV) that depends on the scale of hybridization. In this study, a numerical method based on a multi-scale homogenization (Figure 5) will be used to capture the geometrical features of the materials under study and to integrate the hygrometry that impacts the behaviour of the composite.

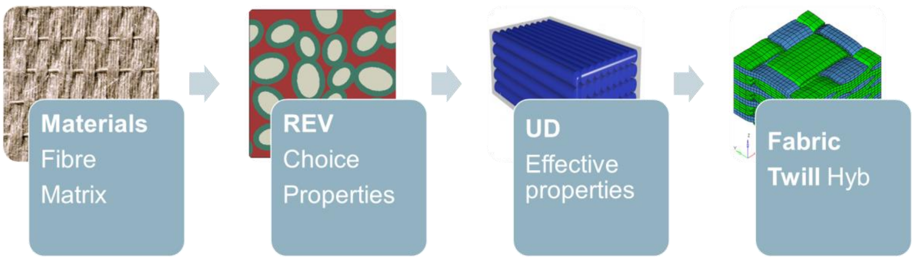
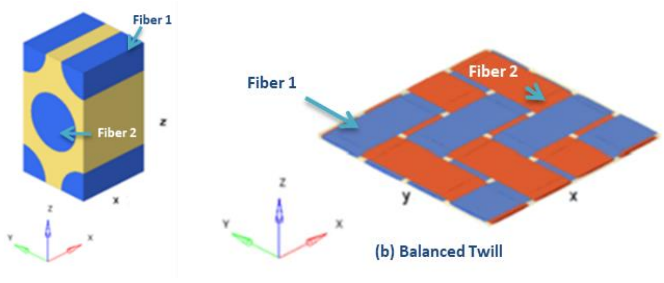


Figure 5: Multi-scale Homogenization protocol for the hybrid composite

The multi-scale numerical homogenization method is divided into two phases. The first phase concerns the homogenization of the constituents (fiber and matrix) at the microscopic scale. It provides the behaviour of the polymer reinforced by the fiber tow. The second phase represents homogenization at the macroscopic scale (fiber tow and matrix). It allows having the behaviour of the studied composite [21]. Initially, the elastic phase of the composite material's mechanical behavior is studied. Therefore, this part focuses on the determination of the effective properties for the hybrid5050/PA11 (twill) for RH0,RH50 and RH85. This tool allows different types of REV to be represented, depending on the composite studied (UD, twill, with or without the fiber/matrix interface) (Figure 6) and uses the periodic boundary conditions.



a) Fiber tow *b) Twill weave composite*

Figure 6: Representative Elemental Volume of the composites studied [43]

The REV type (Figure 6) was chosen for its ability to integrate two different fibers (flax and basalt fiber) to represent a hybrid composite. The first numerical study is carried out without adding an interface between the matrix and fibers due to the lack of information on their mechanical properties. The REV dimensions were defined from the micrographic inspection of the different composite materials (Figure 7) using the image processing tool Image J. The following dimensions were determined: Tow major radius ry, Tow minor radius rz and Tow spacing sy (Figure 8).

Figure 7: Microscopic view of basalt/Pa11 UD composite, flax/PA11 twill composite and hybrid5050/PA11 twill composite

Figure 8 presents the different REV dimensions for the flax/PA11 composite: the tow major radius (ry), the minor tow radius (rz), the tow spacing (sy) and the fiber volume fraction (Vf). This unit cell does not include the crimp of the twill weave.

Dimensions	(micrometer)
Tow major radius ry	1025
Tow minor radius rz	158
Towspacingsy	2470
Vf (%)	42

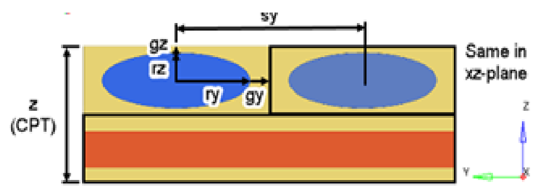


Figure 8: Dimensions of REV of the flax/PA11 twill composite

The multi-scale homogenization was carried out using the intrinsic properties of flax fibers, basalt fibers and PA11. The indices 1, 2 and 3 refer to the main axes of the fiber, with axis 1 referring to the direction of the fiber [8]. The homogenization results for the hybrid5050/PA11 composites for the three moisture levels are presented in Table 4

Relative Humidity		E11(MPa)	E22(MPa)	G12(MPa)
Hybrid5050/PA11	HR0	15000	15000	1090
	HR50	14100	14100	893
	HR85	13400	13400	782

Table 4 Numerical elastic properties of hybrid5050/PA11 composites for HR0, HR50 and HR85

A significant difference was found between the experimental and numerical properties, reflected in a high error value for the hybrid5050/PA11 composites. This can be explained by the limitation of including the fiber/matrix interface and the flax/basalt interface for the hybrid composite [17]. The type of VER has a strong influence on the numerical results. On the other hand, a difference could be observed between the intrinsic mechanical properties of the materials used and the values available in the bibliography.

Then we were interested in the mechanical behaviour of the composite for the non-linear parts. Predefined constitutive law in Multiscale Designer (Rate-independent plasticity (RI) model) was used to represent the stress-strain curve for hybrid5050/PA11. Figure 9 shows the numerical behaviour of the hybrid5050/PA11 composite for RH0, RH50 & RH85.

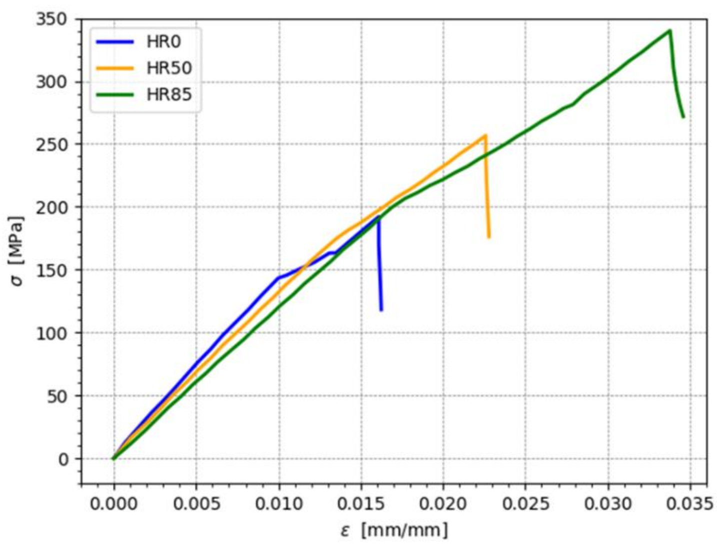


Figure 9 Numerical simulation results for hybrid5050/PA11 (0°) for RH0, RH50 & RH85

The numerical simulations show that the hybrid composite made of commingled fibres and the hybrid co-woven composite have the same mechanical properties. In fact, the hybridization is carried out in the same layer. Only the distribution of the fibres in the same layer was modified with an equal distribution in the longitudinal and transverse directions. The behaviour of the composite is balanced in both directions of the material. However, the results show a difference in the mechanical properties of the laminated composite with the mixed fibre composite and the co-woven composites. The longitudinal properties are also different from the transverse properties. This difference is related to the nature of the fabrics, in fact a combination of flax twill and basalt UD layer was used. These results show the effect of the nature of the hybridization on the mechanical properties of the hybrid composite material.

5. CONCLUSION

Firstly, natural fibers and thermoplastic matrices were studied to determine the best fibre/matrix combination. The thermo-compression parameters, i.e. temperature, dwell time and pressure were then validated to meet the industrial requirements. The second phase was devoted to the experimental characterization of the different flax, basalt and hybrid composites and the PA11 matrix. This part aimed to identify the mechanical properties of the studied materials in the dry state and at room temperature, then the influence of humidity on the evolution of the mechanical properties of the hybrid5050/PA11composite was analyzed. The results of the characterization tests highlight the interest of hybridization. Indeed, the use of basalt fibers with flax fibers improves the mechanical performance of the hybrid composite compared to the flax/PA11 composite and reduces its density by increasing the potential mass gain compared to the basalt/PA11 composite. Furthermore, the hybridization allowed a significant reduction in the scatter of the mechanical properties of the hybrid5050/PA11 compared to the flax/PA11 and the basalt/PA11 composites. The results of the experimental study are in agreement with those obtained in published research works. Indeed, hybridization improves the mechanical performance of the hybrid composite and reduces its sensitivity to moisture, especially when a plant fibre is combined with a synthetic fibre that has a low moisture absorption [24]. The research work of [22,23] approved that positive impact of using basalt fibres with flax/vinylester compositeon the interlaminar fracture toughness. Basalt fibres enhance the durability and limit the sensitivity of flax fibres.

In the second part of this paper, the multiscale numerical homogenization approach is used to determine the effective properties of the studied materials. It also allowed to understand the influence of hygrometry on the composite behaviour and to complete our empirical study by homogenizing new composite materials with identical fiber content and weave. The significant discrepancy requires the study and the integration of the fiber-matrix interface and the verification of the intrinsic properties of the flax fiber through a reverse homogenization based on the experimental curves. It helped to understand the effect of moisture on the mechanical behaviour of plant fibres. The numerical tool allows us to compare the three scales of hybridization; the laminate scale, the layer scale (by co-weaving) and the fibre scale (by blending two different fibres) and helps us to make the appropriate choice of hybridization for the intended application.

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