DEVELOPMENT OF FLAX-REINFORCED BIO-COMPOSITES FOR HIGH-LOAD BEARING AUTOMOTIVE PARTS

C. Avril[1*], P. A. Bailly[1], J. Njuguna[2], E. Nassiopoulos[2], A. De Larminat[3]

[1]*Mahytec, 210 avenue de Verdun 39100 Dole, France*
[2]*Centre for Automotive Technology, Cranfield University, College road, Bedforshire, MK43 OAL, UK*
[3]*Citi Technologies, 4 route Gisy 91570 Bièvres, France*
**christophe.avril@mahytec.com*

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Abstract

The present study was led in the frame of the ECOSHELL European-funded project which aims at developing fully renewable composite materials for the manufacturing of high-load bearing parts in automotive applications. As a first step, three different flax reinforcements (non-woven thick mat, balanced fabric and unidirectional fabric) were combined with a bio-sourced epoxy resin for the characterization of their mechanical capabilities. This paper describes the manufacturing processes of each material with an emphasis on the handling specificities inherent in the vegetal fibers. Numerous laminates were produced and specimens were machined in order to carry out tensile, compression shear and impact tests following the requirements of ASTM standards in force. This work allowed to identify the most suitable material and opened very promising industrial perspectives.

1 Introduction

Owing to their neutral carbon footprint, advantageous bio-degradability characteristics and very good specific mechanical properties, natural fibers have attracted the interest of many industries, including car manufacturers. Natural fibers-reinforced composites have mainly been used for interior components like door, dashboard and under floor trims. The objective of the ECOSHELL European-funded project is more ambitious: introducing bio-composites as a real alternative to non-renewable synthetic fibers for the manufacturing of high-load bearing parts in automotive applications.

The ECOSHELL project aims at implementing innovative bio-materials for the manufacturing of a superlight electric vehicle body, the Citi-Zen, designed by Citi Technologies (figure 1). The use of bio-composite materials shall strongly decrease the environmental footprint of the vehicle and enable to have acceptable performance at an affordable price, due to lower power of the engine and lower energy consumption.

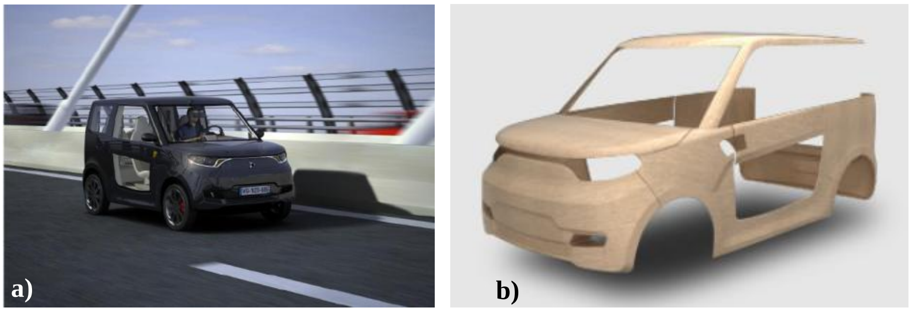

Figure 1. The superlight electric vehicle Citi-Zen (a) and its 100% bio-material body (b)

The achievement of these objectives relies on three main development axes: identification of the best materials (natural fiber, resin, glue), definition of the optimum geometry of the structural parts and definition of the optimum architecture for the whole vehicle assembly. All three aspects are addressed from the point of view of manufacturing, life cycle and end of life. The present study deals with the material section of the project: selection of the most suitable resin and natural reinforcement, identification of appropriate manufacturing protocol on lab-scale but with the perspective of an industrial transfer, laminates production, test specimens machining and experimental characterization.

2 Materials selection

2.1 Natural fibers

The choice of the natural fibers to be used for the reinforcement of the vehicle structural parts is of utmost importance since their properties will determine for most part the mechanical resistance of the bio-composite material. The selection criteria were a high longitudinal Young's modulus, a low density, an industrial-scale production within European Union and the availability on the market of technical-purpose semi-finished products. The respect of these requirements imposed flax and hemp fibers as the most relevant candidates. The table 1 presents the mechanical properties of both fibers in comparison with glass fibers:

Fiber	Young's Modulus (GPa)	Elongation at break (%)	Tensile Strength (MPa)	Density
Flax	70	1-4	600-2000	1.54
Hemp	35	1.6	389	1.56
Glass	72-73	4.6-4.8	3200-3400	2.54

Table 1. Mechanical properties of hemp and glass fibers [1]

The survey of natural fibers market revealed the difficulty to find technical hemp products owing to the nature of hemp applications: domestic insulation, decoration, etc. On the contrary, several flax-based semi-finished products were identified. Three of them were selected and purchased for manufacturing trials: flax balanced fabric, flax non-woven thick mat, flax unidirectional fabric (figure 2). Their properties are listed in the table 2:

Type	Dry fabric areal weight	Dry fabric thickness	Density
Balanced fabric	300 $g.cm^{-2}$	0.6	1.4
Non-woven	1200 $g.cm^{-2}$	5	N.C.
UD fabric	180 $g.cm^{-2}$	0.35	1.4

Table 2. Flax fiber products properties

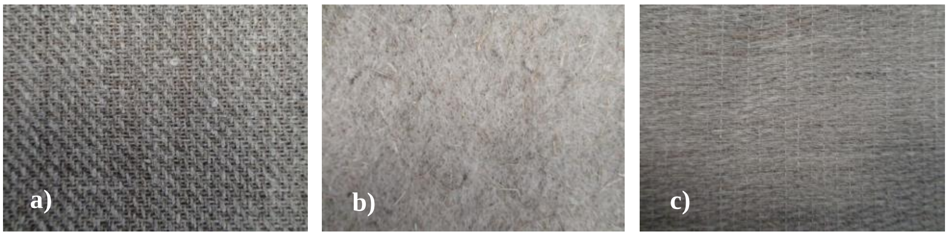

Figure 2. Dry flax balanced fabric (a), non-woven mat and (b) and unidirectional fabric (c)

2.2 Resins

In 2009, 55 millions of tons of plastics were produced within EU (EU27 + Norway + Switzerland) what represents 24% of the worldwide production. The main application markets remains packaging (40.1%), building (20.4%) and automotive industry (7%) [2]. The postconsumption waste represent 24.3 millions of tons. 54% are re-used (5.5Mt through recycling, 7.6Mt through direct energy conversion) but 46%, i.e. 11.2Mt, go to waste disposal. The environmental impact of the plastic industry is a real concern and the replacement of fossil fuel-derived plastics by renewable bio-mass-derived plastics appears as one of the most promising solution. The table 3 gives examples of various polymers sorted according to their origin and biodegradability:

	100% oil-derived	**Partially biomass-derived**	**100% biomass-derived**
Biodegradable	PBS, PCL	Starch/synthetic biodegradable polymer PLA/synthetic biodegradable polymer	Tannin-based resin Plasticized starch PLA, PHA
Non-biodegradable	PA12, PA6, PA66 PE, PP, PET, PBT Epoxy, elastomers	Starch/Polyolefin Bio-PET, Bio-PVC Bio-epoxy (plant oil)	Bio-PE PA11 (ricin oil)

Table 3. Origin and biodegradability of various polymers

In the frame of the ECOSHELL project the choice was made to investigate and compare a conventional epoxy resin, a bio-sourced epoxy resin and a self-developed fully renewable tannin-based resin. This paper focuses on the mechanical behavior of the bio-sourced epoxy/flax fibers composite materials. In order to overcome a sudden shortage in the supply of bio-epoxy resin, the unidirectional flax fabric was impregnated using a conventional epoxy system under the assumption that it won’t significantly affect the mechanical performance of the composite specimens. It should be noted that other criteria like water absorption were investigated in parallel to fully assess such biomass-derived products in comparison with non-biodegradable conventional thermosetting resin.

3 Composite laminates manufacturing

3.1 Fabrication means

The manufacturing of all bio-composites laminates was carried out by Mahytec using a hot compression molding process. The composite plates were fabricated using a 100t hydraulic press with servo-components allowing an accurate control of the consolidation pressure. The pressing blocks of the press are surrounded by an electric furnace with multi-segments controller for the programming of any polymerization cycle. In addition to the furnace temperature sensor, a small-diameter thermocouple was placed in the mold in order to measure the temperature as close as possible to the composite material. The first trials highlighted the necessity to reduce the thermal inertia of the mold in the perspective to identify the optimal curing and consolidation cycles. On that purpose, a thin-walled aluminum mold was used. Quality and repeatability requirements imposed to systematically respect a strict fabrication protocol from the mold preparation (polishing, suppression of polymeric residuals, application of cleaning solvent and release agent) to the composite curing. Each flax product was associated to a specific consolidation cycle and the achievement of homogeneous and well-impregnated composite materials required taking into account their specificities.

3.2 Flax fibers preparation

One step was mandatory and common to every material: the drying of the flax fibers. Like all natural fibers, flax fibers are very prone to water absorption when stored in normal conditions. A high humidity content of the fibers leads to a poor adhesion between fibers and matrix what means an unsatisfying stress transfer to the fibers under mechanical load and much lower mechanical properties. Such a poor interfacial strength between resin and fibers caused by insufficient drying was observed during the first manufacturing trials (figure 3) when the plates were demolded: in spite of the application of a release agent to the surfaces of the pressing blocks, fragments of resin were torn out from the composite plate and remained stuck to the mold owing to the absence of any adhesion with the flax fibers. A suitable drying protocol was finally identified and allowed the production of well-impregnated flax-reinforced plates.

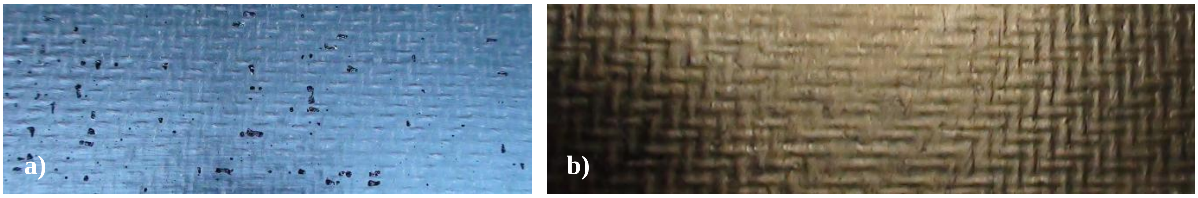

Figure 3. (a) Flax/bio-epoxy plate with resin detachments owing to poor fiber drying before consolidation
(b) Flax/bio-epoxy plate showing a hole-free surface thanks to proper fiber drying

3.3 Flax layers impregnation and consolidation protocol

Depending on the flax product, a varying number of layers were used to reach the composite plate target thickness of around 3mm. Five layers were used for the balanced fabric, seven for the unidirectional fabric while a single layer was sufficient for the non-woven mat. Each layer was cut to the dimensions of the mold (270*270mm). The cutting of the unidirectional layers demanded a special care to avoid the detachment of the weft fibers and a prejudicial distortion of the fabric that could lead to a misalignment of the warp fibers. Once the flax fibers were properly dried, the layers were manually impregnated by the epoxy resins. Owing to its larger thickness, the flax mat turned out to be more difficult to get fully impregnated. The problem was remedied by adapting the manual impregnation process and increasing the consolidation pressure. Cross-section cuts of the mat-reinforced plates after curing showed a homogeneous impregnation of the plate through the whole thickness.

For each materials combination a specific maturation time was then observed in order to let the resin viscosity increase before applying the consolidation pressure. The pressure was applied gradually and curing cycle started simultaneously. Pressing parameters were chosen to reach a weight fiber content of 50%. Depending on the type of resins and hardeners, one or two temperature plateaus were programmed. Figure 4 presents well-impregnated plates after demolding:

Figure 4. Unidirectional flax fiber- and non-woven flax fiber-reinforced composite plate after demolding

4 Testing specimens machining

The plates were used for the machining of various specimens in the perspective of an extensive mechanical characterization campaign including tensile, compression, shear, and impact testing. The table 4 reports for each test the ASTM standard which was followed, the expected outcomes, sample geometries and loading velocities:

	Static tests		
	Tensile	**Compression**	**Shear**
Standard	ASTM D3039	ASTM D3041	ASTM D7078
Outcomes	Tensile strength, strain, modulus Poisson's ratio Stress-strain response	Compressive strength, strain, modulus Poisson's ratio Stress-strain response	Shear strength, modulus Stress-strain response
Sample geometry	250*25mm	150*25mm	V-notched
Loading speed	2 mm/min	1.3 mm/min	2 mm/min

Table 4. Operating conditions for the experimental characterization campaign

Impact tests were done using a drop-weight equipment and involved energies of 1.4, 4.5 and 10J. Eight specimens were machined for each material and each test, except impact tests which involved four specimens. For tensile and compressive testing, four 50 mm long aluminum tabs were glued on each specimen by means of a bi-components epoxy adhesive. The composite plates were machined using a numerical milling machine. Figure 5 illustrates the machining operations:

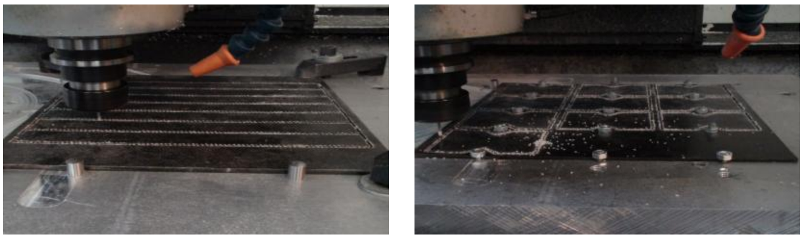

Figure 5. Fabrication of the testing specimens by means of a numerical milling machine

Figure 6 presents the set of testing specimens extracted from non-woven flax/bio-epoxy composite plates:

Figure 6. Set of flax mat/bio-epoxy testing specimens

5 Experimental results

All mechanical tests were realized by Cranfield University. Altogether, 82 specimens had to be tested. At the time of writing the experimental campaign was not fully achieved and only partial results can be presented here. However, these first results already give a clear view of the flax materials mechanical capabilities.

5.1 Tensile testing

Table 5 summarizes the tensile tests results obtained on the three flax/epoxy materials:

Reinforcement	Ultimate strength (MPa)	Young's modulus (GPa)	Ultimate strain (%)
Balanced fabric	90.11	7.69	1.86
Non-woven	76.28	8.04	1.26
UD fabric	222.94	22.30	1.24

Table 5. *Tensile testing results*

Figure 7 presents the tensile stress-strain curves (UD: blue, 0/90: red, mat: green):

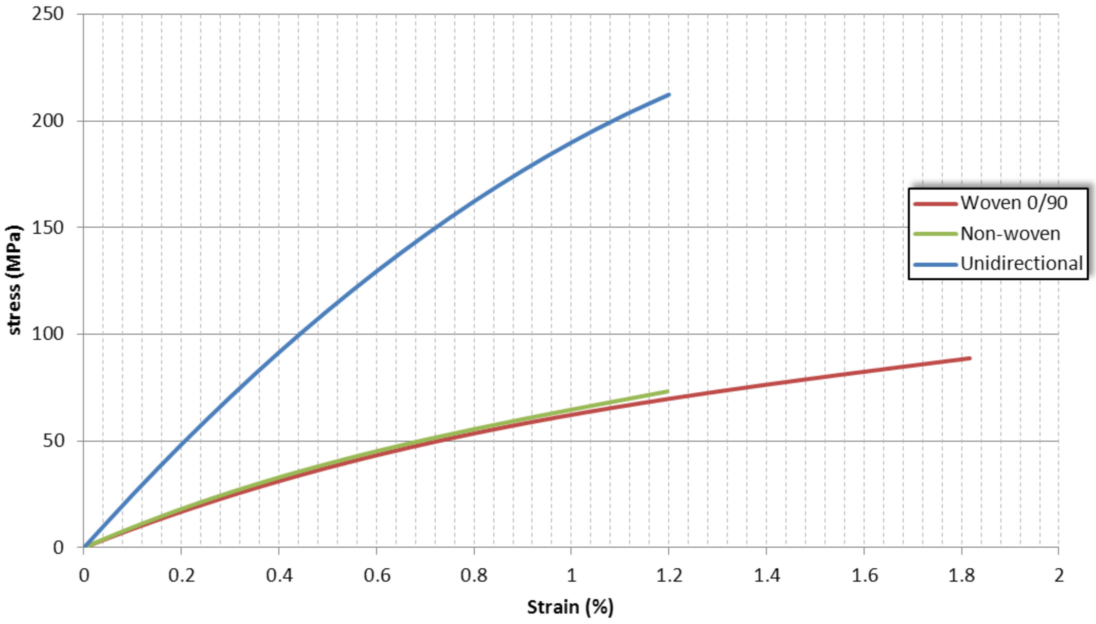

Figure 7. Stress-strain curves describing the tensile behavior of UD flax/epoxy laminates

As expected, the UD reinforcement leads to much higher tensile strength and stiffness with very interesting values meeting the mechanical requirements given by the vehicle design. More surprisingly, the balanced fabric showed performance at the same level than the thick mat, probably owing to the coarse weave pattern and undulation of warp and weft fibers.

5.2 Compression results

The results from compression tests conducted for the woven and non-woven materials are presented in table 6:

Reinforcement	Comp. strength (MPa)	Comp. modulus (GPa)	Strain at failure (%)
Balanced fabric	106	8	2-2.5
Non-woven	109.3	7.9	2-3

Table 6. *Compression test results*

As expected the compressive strength of both materials is higher than their tensile strength respectively whilst the modulus remains approximately the same. Interestingly the non-woven material has higher compressive strength than the balanced fabric, explained due to the waviness of the fibers in the last. The strain at failure cannot be accurately indicated as the samples do not fail catastrophically but with brooming in the middle of the gage length.

5.4 Shear results

Figure 8 shows the failure of the three materials under shear loading were different mechanisms can be observed. The fracture in the balanced material is almost parallel to the samples notch, connecting the two opposite edges. The failure is sudden and brittle without any visible plastic deformation and is controlled through the strength of the matrix. Although the fibers do not fail less than 40% of the initial composite shear strength is present and thus the sample is considered failed. The shear strength and modulus for the balanced material were measured 52.1 MPa and 2.2 GPa respectively. The failure paths of the non-woven material are random, following the weakest link between the fiber and matrix interface. The initiation however occurs at the notch edge in the middle of the sample, where the stress is maximum. No cracks can be observed prior to the failure, and the failure happens again in a sudden and brittle manner, with the strength being 49.5 MPa and a modulus of 2.1 GPa. Within the ASTM standard D7078 the testing of unidirectional specimens is not recommended since the measurement do not appear to be accurately estimated. In this work the results of unidirectional samples are also presented for comparative reasons. A visible crack develops in the notch root after approximately 1,5% strain and 32 MPa shear stress dropping the load around 5%. The crack then develops parallel to the fiber direction up to failure. Although the force and hence the stress after the appearance of the crack keeps increasing, the test was stopped as the samples were considered already failed.

Figure 8. Shear sample after testing, showing the different failure patterns followed by the balanced fabric, the non-woven and finally the unidirectional reinforcing material

Figure 9 depicts the curves obtained from the shear testing for all three materials.

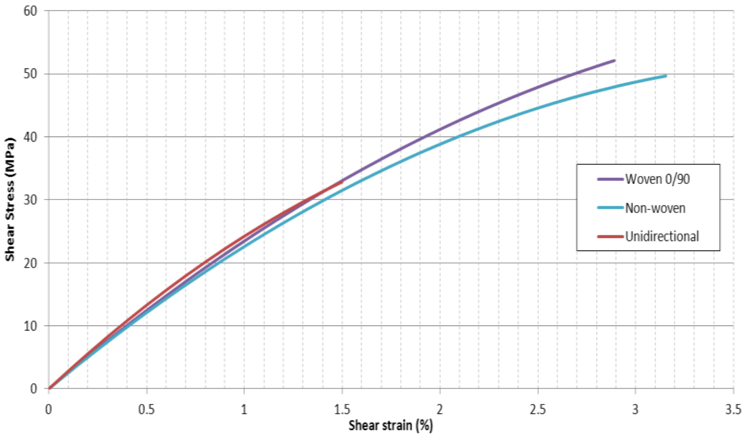

Figure 9. Shear stress-shear strain curves describing the shear behavior of all three laminates

5.5 Impact results

As can be observed in figure 11, the woven flax composite is the most resistant to impact among the materials tested. In the case of the 3 m/sec, the material resisted the load with the creation of four large cracks. The result in all other velocities followed the same principle, with the specimen stopping the impactor with the creation of four cracks perpendicular to the axis of the sample and parallel to the two fiber directions forming a characteristic cross. In the case of the non-woven material and the 3 m/sec velocity penetration was observed, as the specimen shattered in two pieces when the cracks extended up to the specimen edges. In all other velocities extended damage occurred but the impactor bounced back after few milliseconds. The unidirectional material showed poor impact resistance as well with the shuttering of the plate parallel to the fiber direction, as depicted in figure 11. For all three materials brittle failure was observed with very limited plastic deformations remaining after the impact. The shapes of the force-time histories for the woven material in the three different velocities are depicted in figure 10. An instant almost linear increase is first observed up to a maximum force. Once the maximum is reached the force remains approximately constant to this plateau level having an oscillating behavior to finally start decreasing linearly to eventually become zero. The average plateau force was 850 N and was reached in less than a millisecond for all three tests, and its duration was different for all three tests, approximately 7, 5 and 4 msec for the 3, 2 and 1 m/sec testing velocity respectively. Only in the lower speed test the linear increase showed a less steep slope and then maximum force was achieved after almost 1.5 to 2 msecs. The amount of energy absorbed by the material for each material velocity is shown in figure 10. This value includes also the energy given back as elastic rebound.

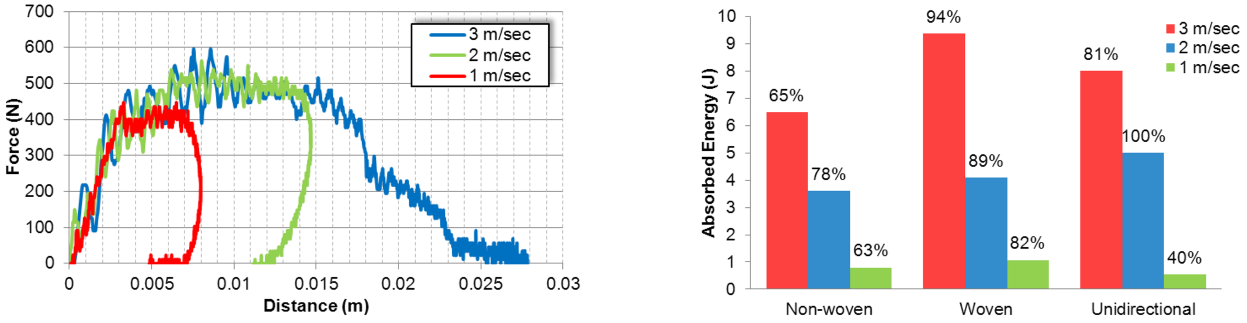

Figure 10. Force-displacement response of the 0/90°woven materials and amount of energy absorbed per material and testing velocity


0/90^0 woven **Non-woven** **Unidirectional**

Figure 11. Fractographs of the specimens after the drop weight impact test (3 m/sec velocity)

6. Conclusions and perspectives

This study, still in progress, led to a better knowledge and understanding of flax-reinforced materials from the points of view of both manufacturing protocol and mechanical behavior. The strength and stiffness values revealed by the experimental tests under different loading confirmed that such material can be used for structural applications. In the next months, hybridation of flax reinforcements within a single laminate will be investigated to take the most of all materials and implement the optimal combination for each vehicle parts.

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