Internship report

Presented by

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Master II Research

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Utilization of natural fibers as reinforcement of polymer based structure - Study of the impact strength.

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1 Introduction

Sustainable development and other concern about the save of energy incite many companies, especially in the field of transport, to use composite. These offer a non negligible gain of weight while respecting the reliability and security requirement of the manufacturer. The obvious composite solutions are the thermoset polymer reinforced with glass or carbon fiber, thanks to their low cost or high performance. However, these composites have some disadvantages in particular in term of recycling or revalorizing. In Europe, two million tons of composites wastes are produced each year.

Today, an alternative appears with the growth of composite reinforced with natural fibers and thermoplastic matrix. These offer mechanical properties close to glass fiber with a positive carbon assessment. Moreover many solutions were developed these previous years to separate the fibers from the thermoplastic matrix, permitting a revalorization of the waste. However these composites are very young, that explain the uncertainty of the manufacturer concerning their reliability, and more studies need to be done before using them at higher scale.

The aim of this internship is to study a new composite made of an acrylic matrix reinforced with short hemp fiber. Several mechanical tests, in static and dynamic load, are performed to validate its use as a structural or semi-structural material.

2 Natural fiber as reinforcement

2.1 Introduction

We can distinguish two class of natural fibers : fibers from plants and from animals.

Many plants can be used to reinforced composites, some of them are listed below : hemp, flax, jute, kenaf, sisal, pineapple, bamboo, rice, etc... A plant is usually preferred to another one principally because of his availability in the region of it utilization, and secondary because of its mechanical properties. Fibers which are the most used (hemp, flax) have strength similar of glass fiber.

Fibers from animals are limited to raw silk and spider web. These materials are known since centuries for their mechanical and thermal properties. Raw silk is the only fiber considered as continuous with a length up to 1200m. However the availability of these is limited because of the small quantity that can be produced by one animal. That is why a mass production of composite reinforced with animal's fibers is not possible.

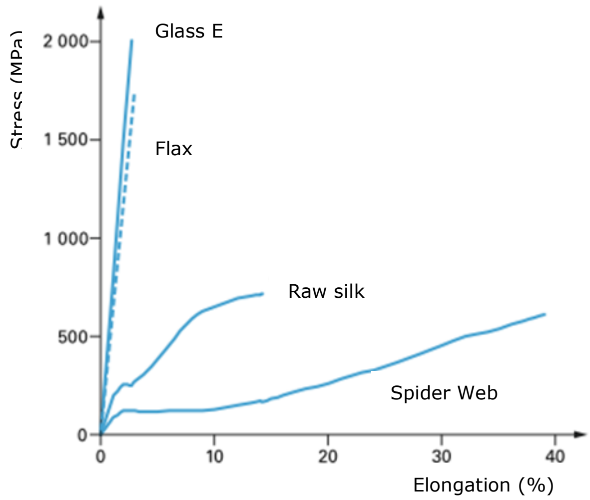

Fig. 1 Natural fibers comportment

Compared to glass or carbon fiber, natural fiber and more exactly vegetal fiber offer substantial reduction of cost, mechanical properties equivalent, a high availability and above all a carbon footprint assessment close to zero.

2.2 Structure of a fiber

A vegetal fiber is easily assimilated to a multilayer composite made of cellulose fibril in a lignin and hemicelluloses matrix (Fig. 2). The first layer is called "primary wall" while the others are called "secondary wall". The primary wall has a low concentration of cellulose (10%) which gives its elasticity. The second secondary wall, which is the thicker one, gives the fiber his strength and rigidity. It is made of a helical structure with and high concentration of cellulose. The helix angle (angle between the fibrils and the fiber axis) influences strongly mechanical properties of vegetal fibers. A high angle will give ductility to the material whereas a low one gives strength.

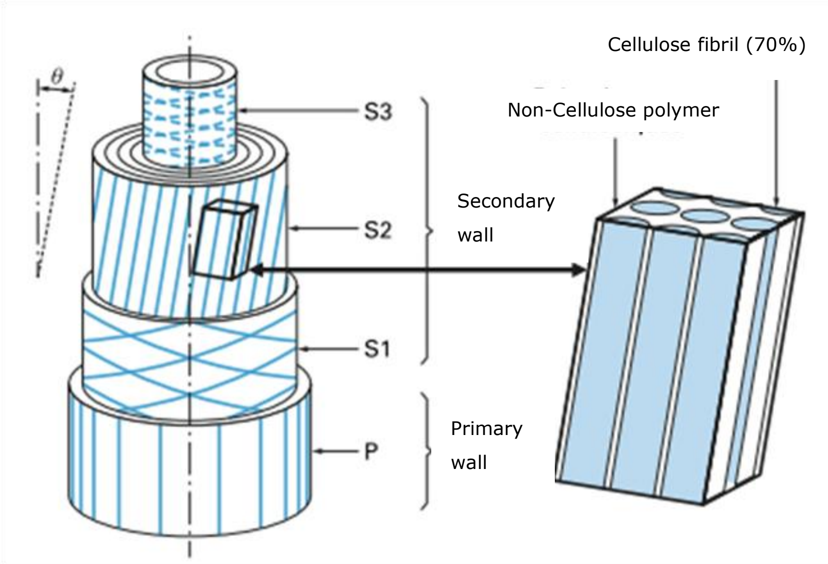

Fig. 2 Vegetal fibers structure

2.3 Problem

2.3.1 Fiber/Matrix interface

The interface (or inter-phase) between the reinforcement and the matrix play a dominating role in the strength of a composite. A solid bonding permits an homogeneous transmission of the effort in the material, whereas a weak one while cause a premature failure of the composite.

Vegetal fibers are hydrophilic while polymer matrixes are usually hydrophobic. These antagonist properties make them difficult to link. Studies proved that the use of a coupling agent like Maleic anhydride polypropylene (MAPP) can enhance the tensile strength by 100% and the Young's modulus by 30%.

2.3.2 Hydrothermal aging

The hydrophilic nature of vegetal fiber makes them absorb humidity of the ambient air, which can cause the lowering of the composite's properties. This phenomenon is called hydrothermal aging and is the major reason why the natural fibers composites are not as used as glass fiber for structural parts.

The first consequence of the aging is the variation of dimensions, weight and appearance of the composite. The second is the degradation of the interface between fibers and matrix and thus the creation of void.

3 Presentation of the studied composite

3.1 Flax fiber reinforced composite

The studied composite was provided by *Pôle de Plasturgie de l'Est* (France). A new acrylic matrix developed by *Arkema* was used to coat non-woven flax fiber. 4.5mm thick plates with a volume fraction of fiber of approximately 30% were manufactured.

We also studied a version of this composite covered by a finishing layer of PMMA and ABS thermoplastic. The PMMA can be colored with a chosen color and the ABS give a brilliant aspect thus no more treatment are mandatory (Fig. 3).

Because all the materials used are thermoplastic, the plate obtained can be re-heated and mold to obtain the right shape.

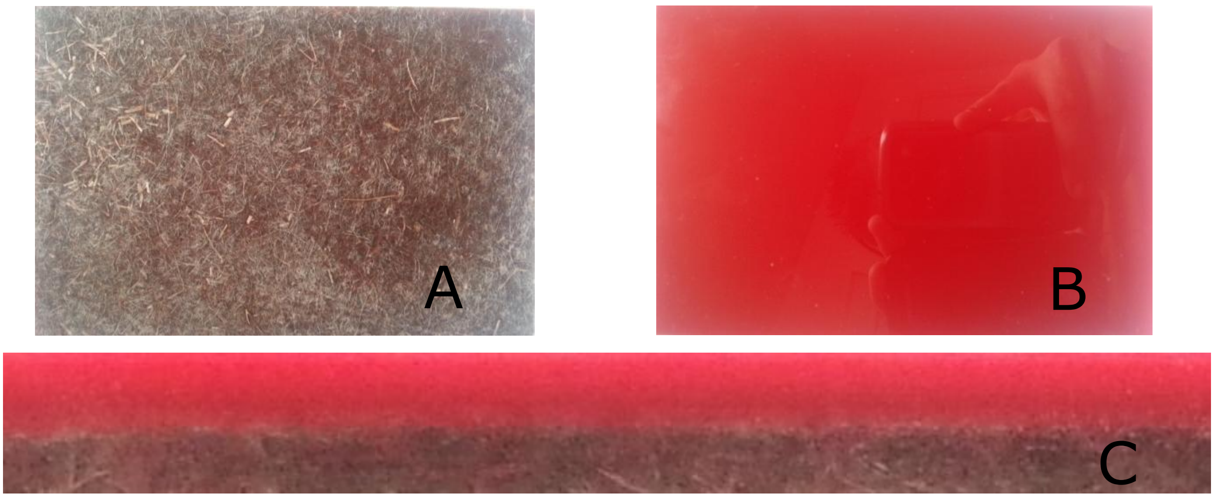

Fig. 3 Picture of finished composite (A) on composite side (B) on PMMA side (C) from the side

3.2 Process

The "Resin Transfer Molding" (RTM) process were used to make the composite plate. This process is the most used to manufacture mass production of thermoset composite but is not adapted of the thermoplastic because of the too high viscosity of these when they are heated. To make up this problem, the liquid precursors to make the acrylic are mixed and injected into the mold thus the polymerization reaction happens after the injection.

For the finished composite, a plate of PMMA is placed in the bottom of the mold, and the dried non-woven flax over it. The resin is injected from the side with 3 bar pressure (Fig. 4.) Because of the similar chemical structure between the PMMA plate and the matrix of the composite, a strong link is created between the two components.

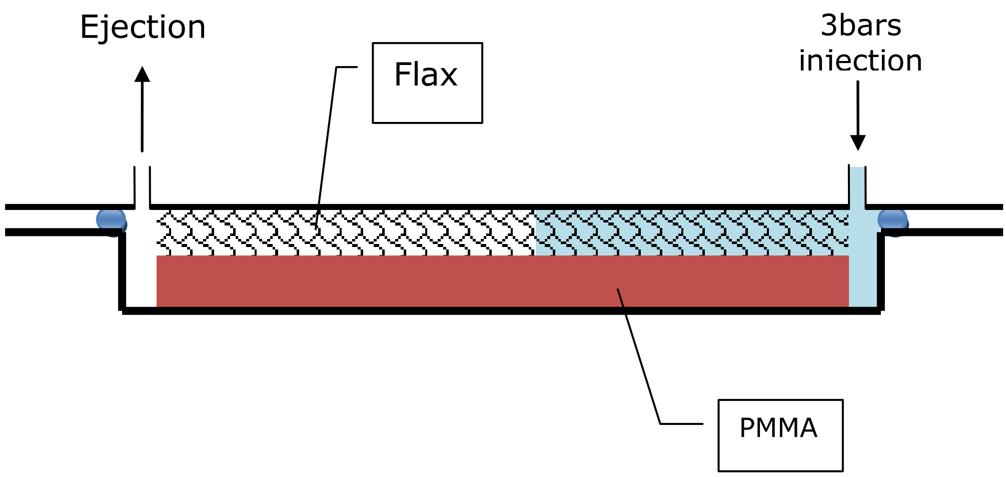

Fig. 4 RTM process with a finishing layer

The specimens tested are machined first with a composite drill, however it caused an overheating of the composite which is at the origin of burned fiber and melted matrix. Then an aluminum drill were used, sharper, it allows a net cut without heating.

4 Experiment and Results

4.1 Imaging

A visual observation of the previous plate allows seeing the presence of defaults on the injection and ejection side of the plate. A white painting is used to highlight this void.

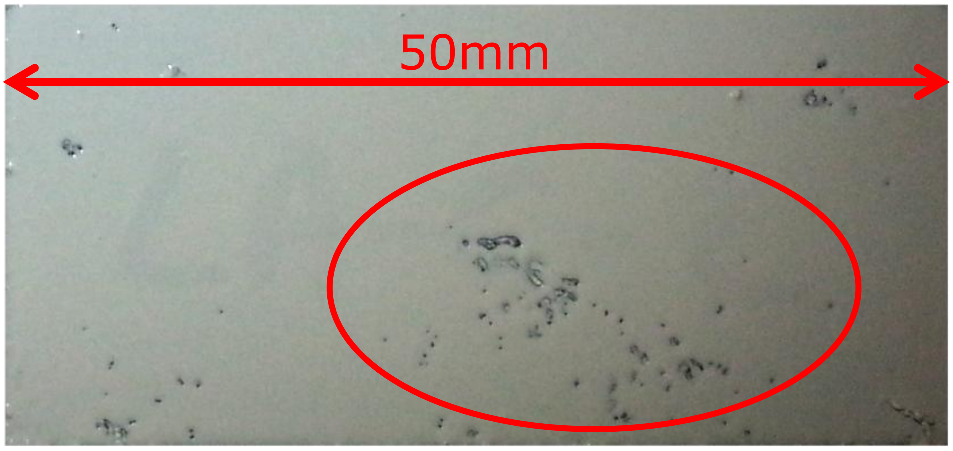

Fig. 5 Picture of external default

Then tomography picture of a small specimen allows us to inspect the composite from the inside. The first objective was to observe the repartition of flax fibers to check the presence of a main orientation. We observed Fig. 6 that fibers are all randomly dispersed in the plan of the plate, we can then expect a transverse isotropic comportment. We also noticed the presence of small defaults that could be water or air bubble.

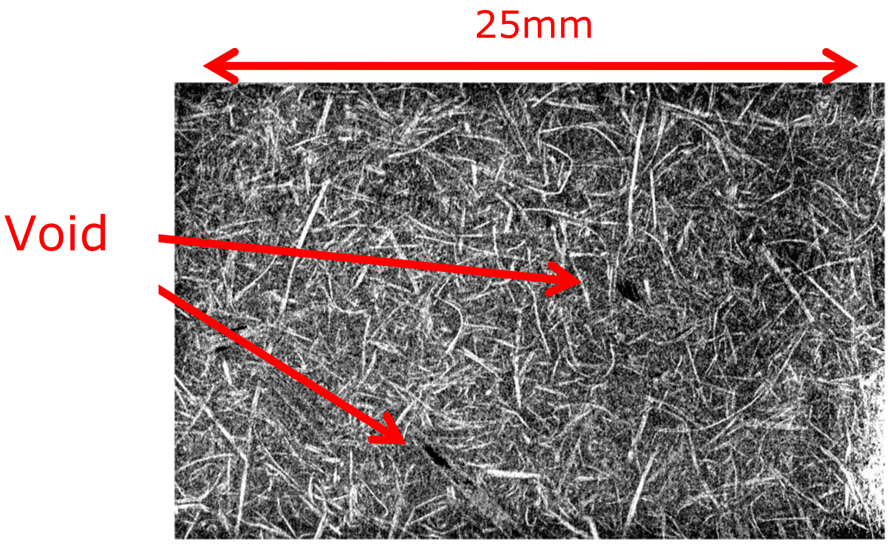

Fig. 6 Tomograph picture of fibers and Acrylic/Flax composite

4.2 Aging

We use speed up hydrothermal aging to analyze the evolution of properties from the flax reinforced composite. Samples are firstly dried in an oven for a week at 80°C and then aged in a climatic chamber for 7 weeks at 80°C and 80% of humidity. At chosen time interval, specimens are taken to be tested.

4.2.1 Weight gain

The weight gain is determined using the EN3615 standard. Six samples from the several plates are weighted with a 0.1mg accuracy at each taking date. The weight gain is assimilated to the humidity absorbed by the composite.

After two weeks of aging we can see in Fig. 7 a fast evolution of the humidity followed by a constant step at 3%HR. Studies proved that the speed of weight gain strongly depends of the condition of aging while the plateau stays at the same value. We also notice a weight lost between the last two measurements, this can be explained by a degradation of fibers. The incoming samples will confirm or not this evolution.

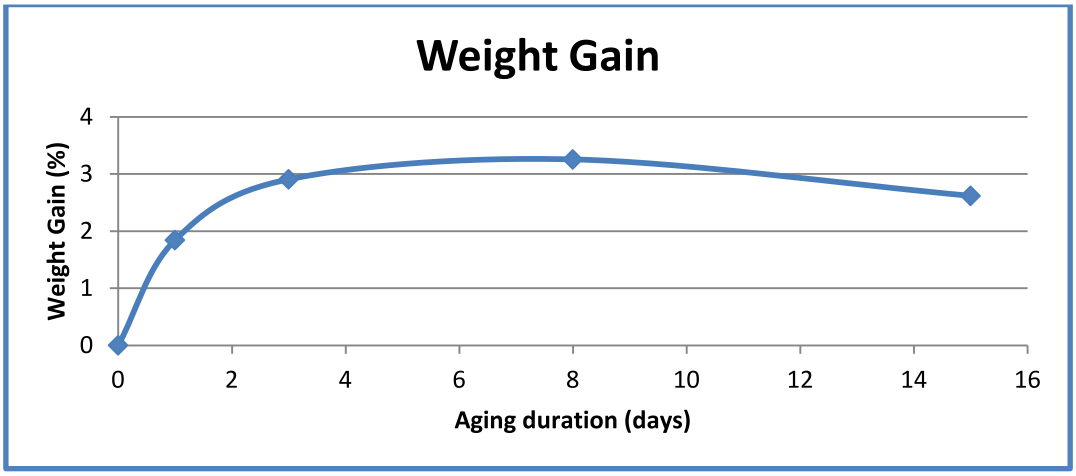

Fig. 7 Graph of the weight gain of the composite during water aging

The evolution of the water concentration depends of the air humidity and the temperature. It is generally modeled by the Fick law of diffusion. We can then determine the coefficient of diffusion D=90days^{-1} at 80°C and 80%RH

$$\frac{Mt}{M\infty} = \frac{4}{L}\left(\frac{D}{\pi}\right)^{0.5} t^{0.5}$$

Mt : Moisture concentration at t time

M∞ : Moisture concentration at equilibrium

L : sample thickness

D : coefficient of diffusion

t : time

4.2.2 Mechanical properties

We used traction test (ASTM D3039 standard) to evaluate the longitudinal properties and V-notched sample (ASTM D 7078 standard) to evaluate shear properties. This allows us to measure the elasticity and strength in quasi-static condition.

We also use compression after impact test (ISO 18352 standard) to determine dynamic properties. This test measures the strength of the material after a small energy impact with the use of a compression test. This test, developed for composite materials, allows us to determine the lowering of mechanical properties after an impact like a pebble on a body part. This test is on-going, no results will be presented in this report.

These tests are performed on samples taken just after manufacturing and after several time of aging

4.2.2.1 Un-aged Material

The results Table 1 are based on the testing of 8 samples in traction and shearing.

Table 1 Mechanical properties of Acrylic/Flax composite

	Traction Test		Shear Test	
	Young Modulus E (MPa)	Tensile Stress σ (MPa)	Shear Modulus G_{12} (MPa)	Shear Stress τ (MPa)
100% Composite	5493 ± 461	60.57 ± 4.09	2440 ± 171	36.91 ± 1.27
Composite + Finish Layer	2977 ± 89	34.53 ± 2.05	1179 ± 76	38.63 ± 1.60

These results are lower than a glass/epoxy non-woven composite, but with his density of 1.2g/cm^3 it is also lighter. We compared the properties of some usual material brought to their density.

Table 2 Comparison of composite and steel properties

	E/ρ	σ/ρ
Acrylic / Flax	4.49	49
Epoxy / Glass	8	85
Steel SAE 1010	27	46

We notice in Table 2 that the Acrylic/Flax properties are two times lower than an Epoxy/Glass composite, his main concurrent. The weight gain objective cannot be achieved with this composite at this step of the research. The properties only match with conventional steel in term of strength. However in a perspective of increasing the recyclability of a product and lowering the environmental impact, the acrylic/flax composite could be an alternative.

4.2.2.2 Aged Material

The aging allows highlighting the modification of the comportment of a material with time or specific climatic condition.

We notice Fig. 8 that the elasticity of the composite in traction and in shearing decreases rapidly until day three where the Shear Modulus and Young Modulus stay constant.

The failure strength is governed by two antagonist phenomenon. The first improves the strength due to the increasing ductility caused by the water in the matrix. The second lowers the strength because of the degradation of the interface and the moisture of the fiber. These explain the increasing strength until day three followed by a fall. The results seem to reach a plateau at this point of the experiment.

All the mechanical properties seem to reach a constant value after a period of aging. This allows us sizing parts for a long life time.

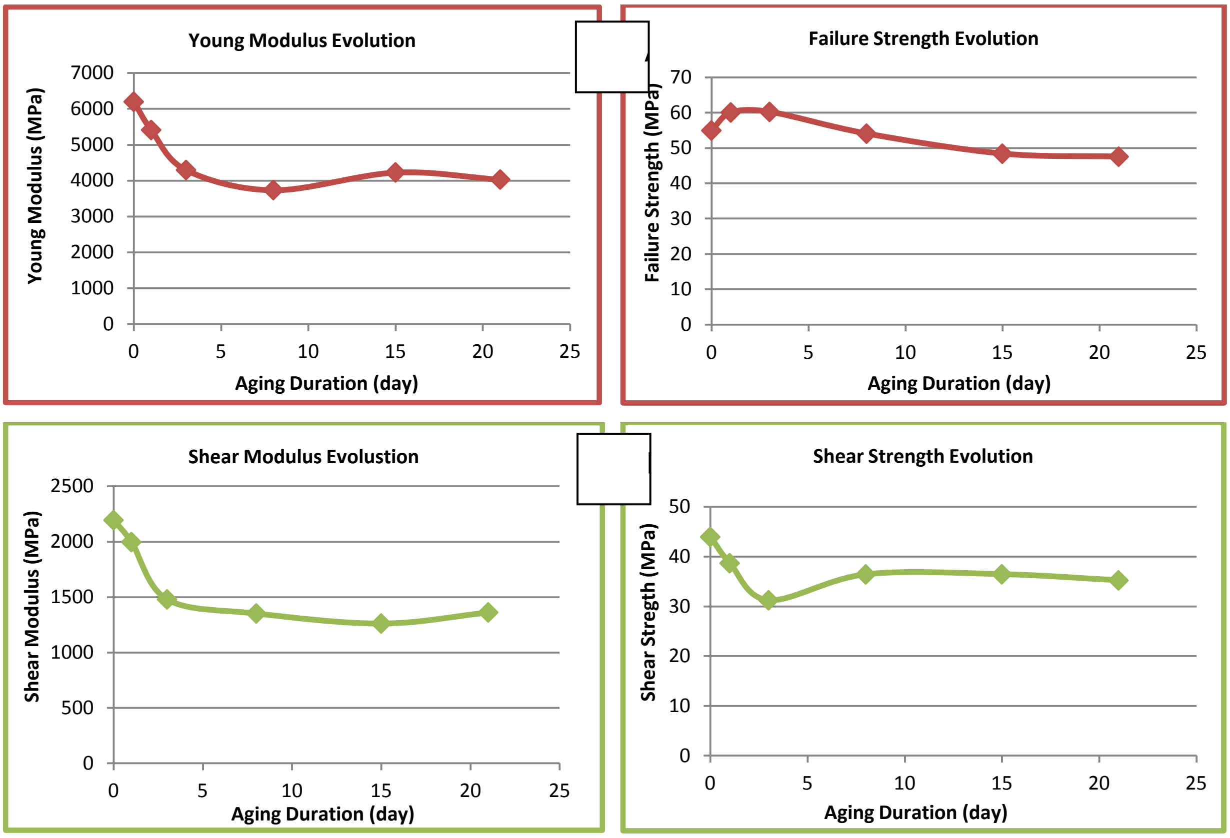

Fig. 8 Evolution of mechanical properties (A) tensile test (B) Shear test, with aging

5 Conclusion

We studied the mechanical properties of a new composite based of natural fiber, flax, and a thermoplastic matrix, acrylic. A new process allows making a colored and bright finished composite in a single step. We checked the benefits and drawbacks of this new material as an alternative for epoxy/glass.

Despite the recyclability of the acrylic/flax composite, the mechanical properties of it seems too low to lighter structures. Indeed his strength per unit of mass is similar to steel with the inconvenient of aging. Some critical points need to be improved like the process or the fiber/matrix interface in order to make this composite viable. We need to pursue the research in this way.