Experimental characterisation of a flax fibre - epoxy resin composite

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Abstract. In the recent years, the scientific community’s attention has gradually started to focus in finding solutions for pollution control and replacement of non-biodegradable products with natural products. In this matter, composite materials with natural fibres are increasingly interesting for different research studies. The present work regards a flax fibre-epoxy resin composite material, which the response is studied under tensile tests until break and repeated loading/unloading cycles. An independent variable under study has been material fibre direction of 0°, 90° and ± 45°, for both type of tests. Samples were extracted from thermo-compression fabricated plates with dimensions following the ASTM D 3039 standard. The experimental setup included an MTS C45 105 Universal Testing Machine, with variable load cells, an extensometer, as well as digital image correlation equipment. The devices allowed the determination of stress-strain curves as well as material constants and Poison ratio. Results show, for the 90° and ± 45° typical responses for composites with these fibre directions, but a particular evolution, bilinear for the 0°.

1. Introduction
The composite materials have long been employed into various fields and the extent of their use is still growing [1, 2]. Since their first use, the goal has been the same: to improve quality and strength. Given their advantages, these materials were implemented in the construction and manufacture of household items (wood and cob) [2], and in the manufacture of battle tools (Mongolian bows, damask swords, or Japanese swords) [3].

Flax fibres use as a material is considerably antique; more than 34.000 years according to a group of archaeologists and paleontologists at Harvard University [4]. Regardless, studies and researches on flax fibres do not completely cover its capabilities.

Flax fibre is distinguished from other composite reinforcement materials, thanks to its high tensile strength and low density [5]. Furthermore, it is a thermal and acoustic insulator and, therefore, it is considered a good replacement for glass fiber [6]. In addition, the plant it is extracted from does not require special conditions for cultivation [7].

Its low density combined with high mechanical properties make it a perfect choice for applications where weight is a critical factor, such as transportation [8]. In the aerospace industry, it is be ideal for lightweight vehicles, such as flying or land drones, ekranoplanes, sometimes used in multiagent systems, which would lead to an increase of the entire system performance.
Tests in the literature frequently disregard viscous effects of the material. Often, they are performed with the machine's crosshead speed control [9-11], an indirect parameter, as the material response is also influenced by the specimen's length. At the same time, sample slippage in the machine's jaws occurs during loading, which disturbs the material response. Thus, high variations in material response might appear, through the neglect of the viscous component.

The aim of this article is to determine specific mechanical properties of this material and to analyze its behavior under load. In order to reach this goal, several UD flax fibre – epoxy resin composite specimens, with the dimensions indicated by the standard ASTM D 3039 [12] were used.

First, tensile tests were performed. Further on, loading/unloading tests at constant speed and progressive loading were carried out.

2. Specimens fabrication and experimental protocol
Composite plates (Fig.1) were fabricated from resin pre-impregnated unidirectional fibres, commercially known as LINEO FLAXPREG T-UD 110 [13], through a cycle of thermocompression. The curing cycle (Fig. 2) is performed at a temperature of 130°C and a 0.3 MPa pressure for one hour. A post curing cycle was performed at 80°C, for 60 min, in an industrial oven. The material samples were cut out of the resulting plates using a band saw.

![Composite plates for the 3 directions of fibre orientation](image1)

**Figure 1.** Composite plates for the 3 directions of fibre orientation

![Curing cycle](image2)

**Figure 2.** Curing cycle

Several samples were fabricated for different types of fibre orientations in order to determine the mechanical properties of this composite. The dimensions of the samples were chosen according to ASTM D 3039, as presented in Table 1.

| Fibre orientation with respect to load direction | 0°     | ± 45°   | 90°     |
|-----------------------------------------------|--------|---------|---------|
| Specimens dimensions [mm]                     | 250 x 25 x 2  | 250 x 25 x 2,5  | 175 x 25 x 2  |

**Table 1.** Specimens dimensions
Aluminum tabs were glued at the extremities of each sample, 50 x 2 mm in size, with the purpose of reducing stress concentrators in the grips. The samples were conditioned in a climatic chamber under a relative humidity of 50% and temperature of 23°C [14]. For humidity control, a saline solution has been prepared by dissolving Potassium Chloride in water and was added in the chamber. The specimens were weighted daily and, while it was found that their weight stabilized in the 3rd day, they were kept for two more days to ensure hygrothermal equilibrium.

The experiments were performed using an MTS C45 105 Universal Testing Machine. It is electromechanically controlled and allows for changeable load cells, a desired feature when characterizing composites with different fibre orientations. Thus, for the directions used, the maximum loads of the cells were: 2.5 kN for 90° samples, 10 kN for ± 45° samples and 100 kN for 0° samples.

The deformation was recorded with a stereo-image correlation camera, for the mechanical characterization and an extensometer, for the cyclic loading tests. For the stereo-image correlation procedure, a surface of each sample was covered with a layer of white paint and black speckles (Fig. 3).

**Figure 3.** Sample prepared for the stereo-image correlation procedure

3. Results

The tensile tests in the three layouts were carried out with the main objectives of obtaining tensile curves in all directions (Fig. 4), defining the maximum material strength, calculating the modulus of elasticity and identifying their damage evolution with stress.

![Stress-Strain curves](image)

**Figure 4.** Stress-Strain curves: (a) 0°, (b) ± 45°, (c) 90°
By analyzing the results, it can be concluded that at ± 45° fibre orientation, the ruptures propagated along the fibre direction; at 0° fibre orientation, the specimen yielded in the grip area, with crack propagations along the length; at 90° fibre orientation, the breaks were sudden, parallel to the transversal section of the sample, with no obvious break region.

The data was analyzed following the norms ASTM D3039, ASTM 3518 [15] for the shear properties and, for the particular bilinear behavior of the composite in the 0° fibre orientation, the method proposed by Shah [16].

The determined tensile stress and elastic moduli are presented in Table 2.

| Fibre orientation | Tensile strength [MPa] | Elastic modulus [GPa] |
|-------------------|------------------------|-----------------------|
| 0°                | 278.5 ± 29             | 38.2 ± 2.5            |
| ± 45°             | 36.3                   | 1.53                  |
| 90°               | 9.7 ± 0.4              | 2.91 ± 0.14           |

For the variable loading tests, tensile tests were carried out in the 3 directions under the constant cross speed of 2 mm/min with different loading levels. Although the loading was achieved by controlling the displacement speed, the deformations are measured by the extensometer.

3.1. Specimens at 0°

Various samples were tested under progressive repeated loading, up to 160 MPa and unloading to 0 MPa.

The evolution of the strain determined by the extensometer as a function of time (Fig. 6) reveals that, from 80 MPa onward, residual deformations appear. These deformations can have a viscoelastic and/or plastic nature. This residual strain also appears in the stress-strain curve.

An interesting fact is that, by eliminating the remaining deformations before each cycle, the loading phases of the curves overlap perfectly. The response is nonlinear, approximated by a bilinear evolution (Fig. 7), which allows the identification of two tangent modules of elasticity. The first module $E_1 = 37.14 \text{ GPa}$ is calculated up to the stress of 50 MPa and the second module $E_2 = 28.06 \text{ GPa}$ is calculated from this stress level onwards.
Figure 6. Strain – Time function for a 0° sample

Figure 7. Stress – Strain function depending on the load for the first 0° sample

Figure 8. Strain – Stress function for two samples at 0°

Note that the hysteresis loops were almost of the same magnitude for the several tests. This loop depends on the magnitude of the applied stress. Another remark is that all the curves overlap during the loading phase. Residual deformation can be observed starting from 45 MPa (Fig. 8).

3.2. Specimens at 90°

In a similar fashion, several samples with fibre orientation of 90° were subjected to the cyclic loading-unloading. While the one presented in Fig. 9 was tested up to 50% of the failure stress, the loading phases are purely linear, and no residual deformations were recorded at the end of any of the loading cycles.
For the second sample, three more load levels were added, up to 80% of the failure stress. In this case, at the additional loading cycles, a viscous response was indeed recorded, through the appearance of hysteresis loops and residual deformations at the end of the cycles (Fig. 10).

3.3. Specimens at 45°
For the ±45° fibre orientation, only one sample was submitted to cyclic loading-unloading, as it was considered enough, by analyzing the previous fibre orientation sample variations. Furthermore, the sample was tested until failure, which occurred at approximately 36 MPa. The sample response in deformation is presented in a time-strain graph in Fig. 11, and in stress-strain coordinates in Fig. 12. A similar response as the sample at the 90° specimens was recorded: an elastic deformation up to 20 MPa (approximately 50% of failure stress) and the appearance of viscoplastic deformations for load levels above this limit.

In the same way as above, a few minutes were waited before the next loading cycle, with the sample mounted in the testing machine. Fig. 11 shows that the residual deformation starts from 20 MPa, which corresponds to approximately 50% of the breaking load of the material.
Figure 11. Stress - Time function for a 45° specimen

Figure 12. Stress - Strain function depending on the load for a 45° specimen

The loading curves in Fig. 12 have the same evolution, despite the plasticization of the material. There is no loss of modulus of elasticity up to 35 MPa.

4. Conclusions
In this article, two types of tests were carried out, tensile tests to failure and cyclic loading-unloading tests with varying maximum load. The specimens used were fabricated from a resin pre-impregnated unidirectional fibre, with three types of angle orientation, 0°, 90°, ± 45°, and conducted and analyzed according to ASTM D 3039, ASTM 3518 or using the method proposed by Shah [16].

The tensile tests up to failure allowed the determination of the maximum tensile strength in all the three directions as well as the elastic modulus and the linear portion of the characteristic curve. The elastic limit in all directions was determined through the appearance of residual deformations in variable load tests and observed to be 20% for the 0° fibre direction and 50% for the 90° and ± 45° ones.

Results show a complex behavior of the material, in all fibre directions, with a viscous component added to the elastic one. While this behavior has been noticed for other polymer-based composites with off axis fiber directions, it is specific for the 0°, where synthetic fibre composites behave purely linear.
The work was aimed at presenting the flax fiber epoxy resin composites behavior. It opens the way for future research, regarding the quantification of the viscous response in various loading scenarios as well as modeling of the behavior, with the purpose of implementing the material in structural applications.

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