A METHOD FOR THE CHARACTERIZATION OF THE ELASTIC ANISOTROPY OF SISAL FIBRES USED IN REINFORCING COMPOSITE MATERIALS

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ABSTRACT

In this work, the manufacturing and characterization of unidirectional Sisal/Epoxy composite plates are thoroughly achieved. Tensile tests are performed to measure the axial and off-axes Young's modulus. An analytical modelling based on the composite cylinders assembly is further outlined in view to optimize the determination of sisal fibres [1] elastic properties. The data from Sisal/Epoxy composites offers especially a curve shape consistent with the expected results, although the overall results should be viewed with all proper reserves.

1. INTRODUCTION

Plants are hierarchically organized in a way that their macroscopic properties emerge from their micro- and nanostructural level. The complex nature of primary and secondary cell walls presumably suggests that fibres may exhibit a great anisotropy. A much better comprehension of this distinctive aspect of their behaviour is thus of considerable importance because of their increasingly widespread use. However, micromechanical tests on the entire structure cannot provide exact values for their properties. In terms of testing methodology, in situ methods that combine micromechanical testing with structural and chemical analyses are particularly well suited for the study of the basic structure-property relationships in fibre design.

Generally, the literature on the identification of anisotropic properties of plant fibres is not abundant. The first attempts to characterize the elastic anisotropy of cellulose fibres originate from the seminal work of Toll and Mänson [2] on a planar fibre network under transverse compression. The basic assumption is that of uniform deformation considered to be equal to the average strain. The direct idea is that, when the network is compressed, more fibre contact points are created and the beam segments providing resistance to compression become shorter and stiffer. Each fibre establishing a given number of contacts is regarded as a continuous bending beam. The beam segment of the fibre is taken as the basic deformable unit. This approach was applied to a wood fibre mats [3] but only lead to a prediction of the Young's modulus of fibres and did not eventually predict the other transverse properties. Cichocki et al. [4] applied an approach based on a simplified representative volume of UD-composites made up of jute fibres considered to be transversely isotropic. They estimated the 5 elastic constants of these fibres while resorting to semi-empirical equations. The programming method used by the authors to derive the solution is not clearly outlined.

This work outlines how well the manufacturing, characterization and modelling of unidirectional Sisal/Epoxy composite plates can be thoroughly combined with the view to determine the fibre's properties. The case is that of a uniaxially-reinforced natural fibre composites in which transversely isotropic fibres are bonded with an isotropic matrix. A brief description of the method used herein is given below.

2. CHARACTERIZING THE ELASTIC ANISOTROPY OF SISAL FIBRES

A UD-composite consists of parallel fibres embedded in a matrix (Figure 1). This type of material is the basic configuration of man made-fibre composites, and is of importance when studying composite materials' behaviour [5]. The main objective here is to take advantage of the elastic properties of a ply expressed in terms of constituents' properties to derive the fibre-phase elastic constants.

Figure 2 shows the 2 coordinate systems used to analyse the ply elastic behaviour. In a pure tensile test about the x-direction, the Young's modulus E_{xx} is given by Eq. 1.

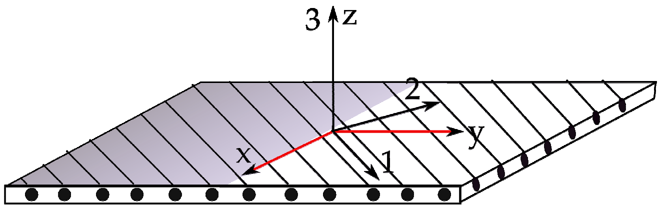

Figure 1: Material axes $(1, 2, 3)$ and off axes (x, y, z) of UD-laminate.

- (x, y, z) : off-axis Cartesian coordinates (reference coordinate system)
- $(1, 2, 3)$: material axes of the unidirectional composite.

$$\frac{1}{E_{xx}} = \frac{1}{E_{11}}\cos^4\theta + \frac{1}{E_{22}}\sin^4\theta + \left(\frac{1}{G_{12}} - 2\frac{\nu_{12}}{E_{11}}\right)\cos^2\theta\sin^2\theta \quad (1)$$

Where E_{xx} is a function of θ and the engineering constants E_{11}, E_{22}, G_{12}, ν_{12} with reference to the material axes.

The reciprocal problem posed here is as follows: knowing E_{xx} (experimental measurements) in a certain number of directions θ, can we determine the properties of the constituents (fibres for instance)? The answer is yes provided that there are analytical relationships of the ply properties in Eq. 1 expressed in terms of constituents' properties.

For a transversely isotropic material, E_{22} is given by Eq.2.

$$E_{22} = \frac{4E_{11}G_{23}K_{23}}{E_{11}K_{23} + E_{11}G_{23} + 4\nu_{12}^2 G_{23}K_{23}} \quad (2)$$

Inserting Eq.2. in Eq.1. gives

$$\frac{1}{E_{xx}} = \frac{1}{E_{11}}\cos^4\theta + \left(\frac{1}{4G_{23}} + \frac{1}{4K_{23}} + \frac{\nu_{12}^2}{E_{11}}\right)\sin^4\theta + \left(\frac{1}{G_{12}} - 2\frac{\nu_{12}}{E_{11}}\right)\cos^2\theta\sin^2\theta \quad (3)$$

Analytical representation of the five ply material properties (E_{11}, K_{23}, G_{12}, G_{23} and ν_{12}) in terms of fibre-phase properties, the matrix-phase properties and the volume fraction of each phase are now to be derived. A geometric model of the composite material must be introduced to accomplish this task.

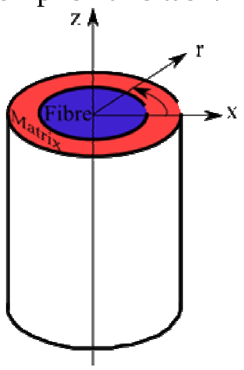

Figure 2: 2-phase composite Cylindre assemblage (CCA).

The most commonly used model is that of the composite cylinder model (Figure 2) introduced by Hashin and Rosen [6, 7, 8]. This model had been widely studied over the year by various authors, see e.g. reference [9] by Wagner et al. for details. The most important results for the properties needed herein are given below.

2.1 Analytical expressions of the composite effective properties

The generalised Hooke's law, considered in cylindrical coordinates $(x_1 = 1 = r,\ x_2 = 2 = \theta,\ x_3 = 3 = z)$, of a transversely isotropic [(r, θ) being the plane of isotropy] material is

$$\begin{Bmatrix} \varepsilon_r \\ \varepsilon_\theta \\ \varepsilon_z \\ 2\varepsilon_{\theta z} \\ 2\varepsilon_{rz} \\ 2\varepsilon_{r\theta} \end{Bmatrix} = \begin{bmatrix} \frac{1}{E_r} & -\frac{\nu_{r\theta}}{E_r} & -\frac{\nu_{rz}}{E_r} & 0 & 0 & 0 \\ -\frac{\nu_{r\theta}}{E_r} & \frac{1}{E_r} & -\frac{\nu_{z\theta}}{E_z} & 0 & 0 & 0 \\ -\frac{\nu_{rz}}{E_r} & -\frac{\nu_{z\theta}}{E_z} & \frac{1}{E_z} & 0 & 0 & 0 \\ 0 & 0 & 0 & \frac{1}{G_{\theta z}} & 0 & 0 \\ 0 & 0 & 0 & 0 & \frac{1}{G_{rz}} & 0 \\ 0 & 0 & 0 & 0 & 0 & \frac{1}{G_{r\theta}} \end{bmatrix} \begin{Bmatrix} \sigma_r \\ \sigma_\theta \\ \sigma_z \\ \sigma_{\theta z} \\ \sigma_{rz} \\ \sigma_{r\theta} \end{Bmatrix} \qquad (4)$$

where

- ν is the Poisson's ratio
- E is the Young's modulus.

In the following, the subscript m and f refer to matrix and fibre respectively.

The effective axial modulus E_{11} is expressed as

$$E_{11} = E_m \phi^m + E_z^f \phi^f - \frac{2\nu_{zr}^f - (\nu^m)^2 \phi^m}{\Delta} \qquad (5)$$

with

$$\Delta = \frac{\phi^m}{\phi^f}\left[\frac{(\nu_{zr}^f - 1)}{E_r^f} + \frac{(2\nu_{zr}^f)^2}{E_z^f}\right] - \frac{\phi^f + \nu^m \phi^m - 2(\nu^m)^2 \phi^f + 1}{E^m \phi^f} \qquad (6)$$

- ϕ^f and ϕ^m represent the fibre and matrix volume fractions respectively.

The effective Poisson's ratio ν_{12} is given by

$$\nu_{12} = \nu^m - \frac{2\left(1-\left(\nu^m\right)^2\right)}{E^m}\frac{\nu_{zr}^f - \nu^m}{\Delta} \qquad (7)$$

Next, the plane strain bulk modulus K_{23} is

$$K_{23} = \frac{E^m \phi^f \left(K_1 K_4 - K_2 K_3\right)}{2\left[-\left(1-\nu^m\right)\phi^f\left(K_4\lambda_1 - K_2\lambda_2\right) + \left(1+\nu^m\phi^m\right)\left(K_4\lambda_1 + K_2\lambda_2 - K_1K_4 + K_2K_3\right) - \nu^m\phi^m\left(K_1\lambda_2 - K_3\lambda_1\right)\right]} \qquad (8)$$

where

$$\begin{cases} K_1 = 2\left(\dfrac{\nu_{zr}^f\phi^m}{E_z^f\phi^f} + \dfrac{\nu^m}{E^m}\right) & K_2 = -\left(\dfrac{\phi^m}{E_z^f\phi^f} + \dfrac{1}{E^m}\right) & K_3 = \dfrac{\nu^m\phi^m + \left(1+\phi^f\right)}{E^m\phi^f} + \dfrac{1-\nu_{r\theta}^f}{E_r^f\phi^f}\phi^m \\ K_4 = -\left(\dfrac{\nu^m}{E^m} + \dfrac{\nu_{zr}^f\phi^m}{E_z^f\phi^f}\right) & \lambda_1 = -\dfrac{2\nu_{zr}^f}{E_z^f\phi^f} & \lambda_2 = -\left(\dfrac{1-\nu_{r\theta}^f}{E_r^f\phi^f} + \dfrac{1+\nu^m}{E^m\phi^m}\right) \end{cases} \qquad (9)$$

and G_{12} is expressed as

$$\frac{G_{12}}{G^m} = \frac{G^m\phi^m + G_{rz}^f\left(1+\phi^f\right)}{G^m\left(1+\phi^f\right) + G_{rz}^f\phi^m} \qquad (10)$$

2.2 In-plane shear modulus G_{23}

Hashin and Rosen [9] reported that for hollow fibres in a matrix, the determination of the exact solution for G_{23} would result in obtaining its lower bound in a stress-type posed problem. Christensen and Lo [10] developped a generalised self-consistent method for a 3-phase composite with plain isotropic cylindrical fibres to derive an exact analytical solution for G_{23}. This method has been generalised to a n-phase composite cylinder assembly by Lagoudas and al. [11]. It is worth noting that the application of Christensen's method to the preceding four properties yields results identical to those obtained from Lagoudas' generalised n-phase assembly, with n equal to 2. In reference [6], Christensen suggests using

$$\frac{G_{23}}{G^m} = 1 + \frac{\phi^f}{G^m\left(G_{r\theta}^f - G^m\right) + \dfrac{k^m + \frac{7}{3}G^m}{2km + \frac{8}{3}G^m}} \qquad (11)$$

with

$$G_{r\theta}^f = \frac{E_r^f}{2\left(1+\nu_{r\theta}^f\right)} \quad G^m = \frac{E^m}{2\left(1+\nu^m\right)} \quad k^m = \frac{E^m}{3\left(1-2\nu^m\right)} \qquad (12)$$

Equations (5), (7), (8), (10) and (11) that incorporate the properties of the composite and matrix may be used in an appropriate optimization process to estimate the elastic properties of the fibre.

3. COMPOSITE MANUFACTURING AND CHARACTERIZATION

3.1 Resin preparation and characterisation

The matrix used herein is an epoxy resin SR5550 marketed by the company Sicomin Composites (CHATEAUNEUF LES MARTIGUES, France), of density 1.145 g/cm^3 with 20 °C (Pycnometer), recommended for use in wood/Epoxy systems. The epoxy resin was mixed with hardener SR5503 of density 1 g/cm^3 (recommended for the stratification and bonding); a resin-to-hardener volume dosage of 1:3, as recommended by the manufacturer, yields a mixture of density 1.109 g/cm^3.
In order to obtain the elastic constants of this epoxy resin 5 specimens were moulded and submitted to the tensile test. The data thus obtained yielded the statistic values $\overline{E^m} = 4.5$ GPa, $\overline{\nu^m} = 0.388$ with standard deviation values 0.371 and 0.006 respectively.

3.2 Alkaline surface treatment of the fibres

On the basis of the results presented elsewhere in reference [1], sisal fibres with about 22 cm in length were treated in 2% sodium hydroxide (NaOH) solutions (fibres-to-solution weight concentration 1:66). The 2% concentration was used to obtain a better compatibility between the fibre surface cleansing and potential mould release problems. The sodium hydroxide solutions were prepared with pre-boiled demineralised water in a stainless steel container that was finally placed in an oven equipped with an automatic temperature control system. The temperature was maintained at 80 °C for 3 hours. Then, the fibres were thoroughly washed with demineralised water and dried at 80 °C for 24 hours (on average).

3.3 Method of impregnation

The main objective is to obtain UD-composite specimens with preferential orientations of 0°, 15°, 30°, 45°, 60°, 75° and 90°. To this end, paper sketches of different orientations were made using the CAD Autocad and stuck to a rectangular glass plate. The glass being transparent, it was thereby easy to manually wrap fibres on the opposite face of the glass plate following the predetermined orientations. Figure 3a shows the paper sketch just described.
The impregnation method used here is similar to that formerly described in reference [4], with some slight but appropriate changes to our situation. The glass plate rolled up of sisal fibres is placed on a wooden shelf fitted in a 500 ml hand mixing bowl filled with epoxy resin mixed with the hardener. A wooden plate with air vents is next used to close the mixing bowl which is further introduced into a vacuum plastic pocket. The unit is then introduced into a vacuum machine and a vacuum of 5 mbar is applied. The vacuum machine used here allows automatic closing of the vacuum pocket by hot plasticization when the required pressure is reached. While the glass-fibres wrapping remains on the shelf, the mixing bowl is slightly inclined allowing the glass-fibre plate to be immersed in the resin bath. The vacuum is then released by boring the vacuum pocket thus enabling better penetration of the resin into fibres in the absence of air bubbles removed by vacuum. Figures 3(a)-4(i) feature the above method.

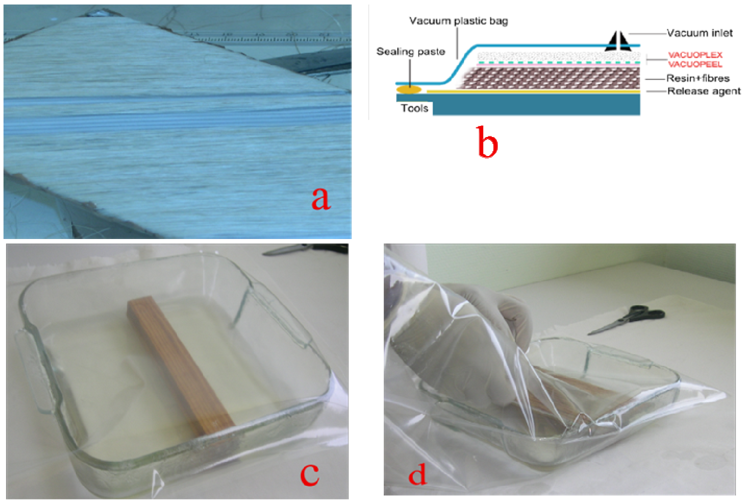

Figure 3: (a) Wrapping fibres on a glass plate (b)-(d) Preparation of the fibre plate to vacuum.

Care has been taken to estimate the fibre content as each rolled up glass-fibre plate was weighed on a precision electronic balance before impregnation and after curing of the resin. It should be noted that the traditional method consisting in dissolving the resin of the composite to determine the fibre rate is not applicable to plant fibres. This is in particular due to the very high risk of total degradation of these fibres during handling. Some authors [4] have reported on a method based on the water uptake of the composite but, it could not be tested in this work.

3.4 Preparation of specimens

3 to 5 specimens of average size 150x20x2 mm^3 (refer to figure 5(i)) were cut from each composite plate, according to NF IN ISO 527-4 standard, using a circular diamond saw mounted on a GRAVOGRAPH VARGA 1 machine. Tabs were used at the ends of the coupons in order to have efficient gripping conditions.

Figure 4: (e)-(h) Vacuum setting stages and 4(i) Sisal/Epoxy specimens.

3.5 Mechanical testing

To enable the determination of the Young's modulus and the Poisson's ratio, specimens were provided, as needed, with strain gauges. For specimens with a 45° orientation in particular, the longitudinal shear modulus G_{xy} could also be evaluated when an additional transversely fixed strain gage is used. Tensile tests were performed on a Zwick ZmartPro UTS20k tensile test machine. The results of measurements for both composite materials are shown in table 1.

4. RESULTS AND COMMENTS

Property	Angles									
	0°			15°			30°			
E_p (mm)	1.149	1.21	1.233	2.25	2.357	2.213	2.103	2.033	2.01	1.95
ϕ^f	0.390	0.390	0.390	0.241	0.241	0.241	0.334	0.334	0.334	0.334
E_{xx}(Gpa)	6.92	7.42	6.29	3.51	5.07	4.93	3.56	3.41	3.09	3.56

	45°				60°			75°		90°	
E_p (mm)	2.72	2.807	2.61	2.73	2.177	2.093	2.557	2.223	2.24	1.257	1.307
ϕ^f	0.321	0.321	0.321	0.321	0.294	0.294	0.294	0.27	0.27	0.364	0.364
E_{xx}(Gpa)	2.71	2.60	2.42	2.85	2.14	2.30	2.59	2.74	2.26	2.89	2.38
ν_{xx}(Gpa)	0.333	0.513	0.439	0.395							
G_{xy}(Gpa)	2.03	1.72	1.68	2.04							

Table 1: Tensile tests data of Sisal/Epoxy composites specimens

Figure 5 shows the evolution of the Young's modulus as related to the orientation angle of fibres in the composite plates Sisal/Epoxy. One obviously expects the module to decrease with an increase in the angle.

These results should be viewed with all proper reserves. The scatter on these data might be ascribable, at least in part, to the specimens' manufacturing conditions. It is well known that the tensile tests of UD-composite specimens produce better results when the fibres are all rectilinear and parallel in the matrix. Indeed it appeared during processing that the Sisal fibres used were less prone to buckling while being wrapped on glass plates.

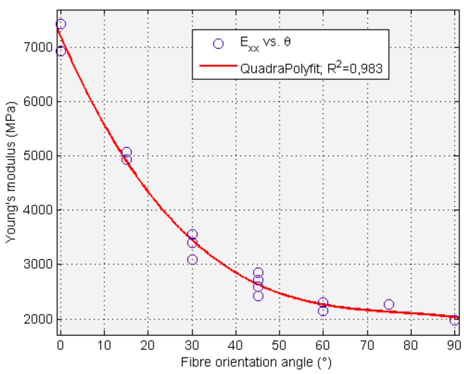

Figure 5: Composites Si/Epoxy Young's modulus.

5. CONCLUSIONS

This study reveals one possible identification method of the elastic anisotropy of natural fibres from their related composite materials. Once again it is obvious that special care should be taken to the fabrication of composites plates. The data from Sisal/Epoxy composites presented herein offers especially a curve shape consistent with the expected results, although the overall results should be viewed with all proper reserves. Further investigations aim at introducing these data in an optimization computing procedure to estimate the fibre elastic constants. To this end, a routine based on the Levenberg-Marquardt's method, known to achieve nonlinear convergence, is to be used.

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