Estimation of the elastic anisotropy of sisal fibres by an inverse method

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Received: 23 February 2008 / Accepted: 1 August 2008 / Published online: 26 August 2008 Springer Science+Business Media, LLC 2008

Abstract This paper is concerned with an inverse method for the characterization of the elastic anisotropy of plant fibres. A good knowledge of the properties of composites reinforced with these fibres is essential for the safe design of the related structures. In this work, experimentation and analytical modelling were thoroughly combined to optimize the determination of plant fibre properties from their related composites. The experimental work focused on the manufacture and characterization of unidirectional (UD) sisal/epoxy composites. Tensile tests were performed to measure the axial and off-axes stiffness of these composites. Tests' data were eventualy used in an optimization process based on a micromechanical model to estimate the fibres' elastic constants. Sisal fibres used herein exhibited a high degree of elastic anisotropy.

Introduction

The anisotropic behaviour of plant fibres is obvious at different scale levels, from the cellulose microfibrils to the fibre bundle made up of unit cells. A much better comprehension of this distinctive aspect of their behaviour is thus of considerable importance because of their increasingly widespread use. Our current knowledge of engineering materials stems from laboratory experiments

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carried out whenever possible. However, the minute cross sections of fibres, of approximately 0.1 mm, make direct measurement of the transverse fibre elastic properties a very difficult task. To date, the only properties directly measured are the axial Young's modulus E_{11} by simple tension and axial shear modulus G_{12} by torsion pendulum. When one deals with such micromaterials, it is difficult to obtain all their properties by traditional means. Nevertheless, most mass-produced materials such as injectionmoulded and sheet-moulded composites are very sensitive to the overall properties of the fibres. Various approaches are used to circumvent this issue. One of these, the ''macromicro'' one (literally from the largest to the smallest), can be used to a great advantage. In this paper dealing, amongst other things, with unidirectional (UD) composites, care has been taken to provide experimental data to feed such an approach for a more complete identification of the elastic properties of sisal fibres selected for this work. A plant fibre is a complex composite structure made up of layers of different anisotropic materials and different principal directions $[1-3]$. It also contains pores, which are essential for the plant, but which can be regarded as defects when mechanical modelling of a single fibre is considered.

As this work deals with estimation of the fibres' properties at the constituents level, the objective is to derive a simple model of the fibre which does not explicitly take into account the variables mentioned above. The model should, however, still be realistic enough to be able to make use of the existing experimental knowledge about fibres and the results from micromechanical modelling.

In this work the fibres are assumed to be linear elastic materials and both homogeneous and transversely isotropic. 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

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originated from the seminal work of Toll and Månson [[4\]](#page-7-0) on a planar fibre network under transverse compression. The basic assumption is that of uniform deformation, which is considered to be equal to the average strain. The 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 wood fibre mats [\[5](#page-7-0)] but only led to a prediction of the Young's modulus of the fibres and did not eventually predict the other transverse properties.

Cichocki and Thomason [[6\]](#page-7-0) applied an approach based on a simplified representative volume of UD composites made up of jute fibres, which are considered to be transversely isotropic. They estimated the five 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 paper is concerned with Levenberg–Marquardt's method based on a comprehensive and analytical micromechanical model. The case is that of a uniaxially reinforced natural fibre composite in which transversely isotropic fibres are bonded with an isotropic matrix. A brief description of the method used herein is given below.

Basics

A UD composite consists of parallel fibres embedded in a matrix (Fig. 1). This type of material is the basic configuration of man-made fibre composites and is of importance when studying composite materials' behaviour [\[7](#page-7-0)].

The main objective here is to take advantage of the elastic properties of a ply expressed in terms of its constituents' properties to derive the fibre-phase elastic constants.

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

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

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

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

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

The reciprocal problem can be posed 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 (1) expressed in terms of the constituents' properties.

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

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

Inserting Eq. 2 in 1 gives

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

$$
+ \left(\frac{1}{G_{12}} - 2\frac{v_{12}}{E_{11}}\right) \sin^2 \theta \cos^2 \theta \tag{3}
$$

We now seek solutions for the effective properties of fibrereinforced composite material. It is found from Eq. 3 that there are five independent properties to be determined $(E_{11},$ v_{12} , K_{23} , G_{23} and G_{12}). Our objective is to obtain an analytical representation of the five properties in terms of fibre phase properties, the matrix phase properties and the volume fraction of each phase. It is to be recalled that such representations found in the literature are usually concerned with isotropic fibres [\[7](#page-7-0)].

A geometric model of the composite material must be introduced to accomplish this task. The most commonly used model is that of the composite cylinder model (Fig. [2\)](#page-2-0), introduced by Hashin and Rosen [\[8–10](#page-7-0)]. This model had been widely studied over the year by various authors, see e.g. Wagner and Nairn [[11](#page-7-0)] for details. The most important results for the properties needed herein are given below.

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

$$
\begin{Bmatrix}\n\varepsilon_r \\
\varepsilon_\theta \\
\varepsilon_\varepsilon \\
\varepsilon_\varepsilon \\
2\varepsilon_{\theta z} \\
2\varepsilon_{r\theta}\n\end{Bmatrix} = \begin{bmatrix}\n\frac{1}{E_r} & -\frac{v_{r\theta}}{E_r} & -\frac{v_{r\pi}}{E_r} & 0 & 0 & 0 \\
-\frac{v_{r\theta}}{E_r} & \frac{1}{E_r} & -\frac{v_{z\theta}}{E_z} & 0 & 0 & 0 \\
-\frac{v_{r\pi}}{E_r} & -\frac{v_{z\theta}}{E_z} & \frac{1}{E_z} & 0 & 0 & 0 \\
0 & 0 & 0 & \frac{1}{G_{\theta z}} & 0 & 0 \\
0 & 0 & 0 & 0 & \frac{1}{G_{r\pi}} & 0 \\
0 & 0 & 0 & 0 & 0 & \frac{1}{G_{r\theta}}\n\end{bmatrix}\n\begin{Bmatrix}\n\sigma_r \\
\sigma_\theta \\
\sigma_z \\
\sigma_z \\
\sigma_r \\
\sigma_{r\theta}\n\end{Bmatrix}
$$
\n
$$
(4)
$$

Fig. 2 Two-phase composite cylinder assemlage (CCA)

where ν is the Poisson's ratio and E is the Young's modulus.

Analytical expressions of the composite effective properties

In the following, the subscripts m and f refer to matrix and fibre, respectively. The effective axial modulus E_{11} is expressed as

$$
E_{11} = E^{m} \phi^{m} + E_{z}^{f} \phi^{f} - \frac{2(v_{zr}^{f} - v^{m})^{2} \phi^{m}}{\Delta}
$$
 (5)

with

$$
\bullet \quad \Delta = \frac{\phi^{\mathrm{m}}}{\phi^{\mathrm{f}}} \left[\frac{v_{r\theta}^{\mathrm{f}} - 1}{E_r^{\mathrm{f}}} + \frac{2 \left(v_{zr}^{\mathrm{f}} \right)^2}{E_z^{\mathrm{f}}} \right] - \frac{\phi^{\mathrm{f}} + v^{\mathrm{m}} \phi^{\mathrm{m}} - 2 (v^{\mathrm{m}})^2 \phi^{\mathrm{f}} + 1}{E^{\mathrm{m}} \phi^{\mathrm{f}}}
$$

- \bullet ϕ^f : fibre volume fraction
- $\phi^m = 1 \phi^f$: matrix volume fraction.

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

$$
v_{12} = v^{\rm m} - 2 \frac{1 - (v^{\rm m})^2}{E^{\rm m}} \frac{v_{zr}^{\rm f} - v^{\rm m}}{\Delta}
$$
 (6)

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

where

$$
K_1 = 2\left(\frac{v_{z'}^{\text{f}}\phi^{\text{m}}}{E_z^{\text{f}}\phi^{\text{f}}} + \frac{v^{\text{m}}}{E^{\text{m}}}\right) \qquad K_2 = -\left(\frac{\phi^{\text{m}}}{E_z^{\text{f}}\phi^{\text{f}}} + \frac{1}{E^{\text{m}}}\right) K_3 = \frac{v^{\text{m}}\phi^{\text{m}} + (1+\phi^{\text{f}})}{E^{\text{m}}\phi^{\text{f}}} + \frac{1-v_{z'}^{\text{f}}\phi}{E_z^{\text{f}}\phi^{\text{f}}} \phi^{\text{m}} \qquad K_4 = -\left(\frac{v^{\text{m}}}{E^{\text{m}}} + \frac{v_{z'}^{\text{f}}\phi^{\text{m}}}{E_z^{\text{f}}\phi^{\text{f}}}\right) \lambda_1 = -\frac{2v_{z'}^{\text{f}}}{E_z^{\text{f}}\phi^{\text{f}}} \qquad \lambda_2 = -\left(\frac{1-v_{z\theta'}^{\text{f}}}{E_z^{\text{f}}\phi^{\text{f}}} + \frac{1+v^{\text{m}}}{E^{\text{m}}\phi^{\text{m}}}\right)
$$
(8)

and G_{12} is expressed as

$$
\frac{G_{12}}{G^{m}} = \frac{G^{m}\phi^{m} + G_{rz}^{f}(\phi^{f} + 1)}{G^{m}(\phi^{f} + 1) + G_{rz}^{f}\phi^{m}}
$$
\n(9)

In-plane shear modulus G_{23}

Hashin and Rosen [\[9](#page-7-0)] reported that for hollow fibres in a matrix, the determination of the exact solution for G_{23} would result in obtaining its lower bound in a stress-type posed problem. Christensen and Lo [[12\]](#page-7-0) developed a generalized 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 generalized to a n -phase composite cylinder assembly by Lagoudas and Seidel [\[13](#page-7-0)]. It is worth noting that the application of Christensen's method to the preceding four properties yields results identical to those obtained from Lagouda's generalized *n*-phase assembly, with *n* equal to 2.

In Ref. [[8\]](#page-7-0), Christensen suggests using

$$
\frac{G_{23}}{G^{m}} = 1 + \frac{\phi^{\text{f}}}{G^{m}(G_{r\theta}^{\text{f}} - G^{m}) + (k^{m} + \frac{7}{3}G^{m})/(2k^{m} + \frac{8}{3}G^{m})}
$$
\n(10)

with

$$
G_{r\theta}^{\text{f}} = \frac{E_r^{\text{f}}}{2(1 + v_{r\theta}^{\text{f}})} \qquad G^{\text{m}} = \frac{E^{\text{m}}}{2(1 + v^{\text{m}})} \qquad k^{\text{m}} = \frac{E^{\text{m}}}{3(1 - 2v^{\text{m}})}
$$

Equations 5, 6, 7, 9 and 10 that incorporate the properties of the composite and matrix may be used in an optimization process to estimate the elastic properties of the fibre.

Problem formulation and method of its solution

The results obtained in Sect. [2](#page-1-0) for the expressions E_{11} , G_{12} , v_{12} , G_{23} and K_{23} of Eqs. 5, 6, 7, 9 and 10 are inserted in Eq. [3,](#page-1-0)

$$
K_{23} = \frac{E^{\mathfrak{m}}\phi^{\mathfrak{f}}(K_{1}K_{4} - K_{2}K_{3})}{2[-(1-\nu^{\mathfrak{m}})\phi^{\mathfrak{f}}(K_{4}\lambda_{1} - K_{2}\lambda_{2}) + (1+\nu^{\mathfrak{m}})\phi^{\mathfrak{f}}(K_{4}\lambda_{1} - K_{2}\lambda_{2} - K_{1}K_{4} + K_{2}K_{3}) - \nu^{\mathfrak{m}}\phi^{\mathfrak{m}}(K_{1}\lambda_{2} - K_{3}\lambda_{1})]}
$$
(7)

thereby yielding a relationship between E_{xx} , θ and the fibre properties (the unknowns $X = \{E_r^f, E_z^f, v_{r\theta}^f, v_{rz}^f, G_{rz}^f\}$). The final objective is to determine the properties characterizing the elastic anisotropy of the fibres considered given the properties of the matrix (E^m, v^m) , the Young's modulus E_{xx} of the ply in several directions θ_i and the fibre volume fraction ϕ^f , the measurements of which are to be provided in Sect. [5.](#page-4-0) These are to be obtained through, at least, five main directions of fibre orientations. Seven values of fibre orientation were considered in this work.

The problem therefore posed is to solve the oversized system (more data than the unknowns) of nonlinear equations formed. It is a nonlinear data-fitting problem, actually an optimization problem, that could be solved following the methods for nonlinear least-squares problems. To this end, a routine based on Levenberg–Marquardt's method is introduced and outlined below.

First of all, $E_{\text{xxi}}^{\text{cal}}$ is computed for a set of parameters X using Eqs. [5,](#page-2-0) [6,](#page-2-0) 7, [9](#page-2-0) and [10](#page-2-0). Then we define a residual value $R_i(X) = E_{xxi}^{\text{mea}} - E_{xxi}^{\text{cal}}$ where E_{xxi}^{mea} is the measured data of E_{xx} for the fibre orientation θ_i .

The error function (or cost function $C(X)$) to be minimized is the sum of squares of the residual values.

$$
C(X) = \frac{1}{2} \sum_{i=1}^{m} (R_i(X))^2 = \frac{1}{2} ||R(X)||^2 = \frac{1}{2} E(X)^T R(X)
$$
 (11)

The key to inverse calculation is to find the value X^* of unknown parameters that produce the smallest cost function $C(X)$, that is to say

$$
X^* = \operatorname{argmin}_X C(X) \tag{12}
$$

Levenberg–Marquardt's method achieves better than linear convergence, sometimes even quadratic convergence, even though it does not need implementation of second derivatives.

In the description of the method in this section we shall need formulas for derivatives of C . Provided that R has continuous second partial derivatives, we can write its Taylor expansion as

$$
R(X+h) = R(X) + J(X)h + O(||h||2),
$$
\n(13)

where $J \in \mathbb{R}^{m \times n}$ is the *Jacobian*, the matrix containing the first partial derivatives of the function components, $(J(X))_{ij} = \frac{\partial R_i}{\partial X_j}(X)$, $m = 7$ (number of fibre orientation value) and $n = 5$ (number of unknown parameters) in this study.

As regards $C: \mathbb{R}^n \to \mathbb{R}$, it follows from the first formulation in (11) that

$$
\frac{\partial C}{\partial X_j}(X) = \sum_{i=1}^m R_i(X) \frac{\partial R_i}{\partial X_j}(X) \tag{14}
$$

Thus, the gradient of C is

$$
C'(X) = J(X)^T R(X) \tag{15}
$$

We shall also need the Hessian of C . From Eq. 14 we see that the component in position (i, k) is

$$
\frac{\partial^2 C}{\partial X_j \partial X_k}(X) = \sum_{i=1}^m \left(\frac{\partial R_i}{\partial X_j}(X) \frac{\partial R_i}{\partial X_k}(X) + R_i(X) \frac{\partial^2 R_i}{\partial X_j \partial X_k}(X) \right),\tag{16}
$$

showing that

$$
C''(X) = J(X)^T J(X) + \sum_{i=1}^m R_i(X) R_i''(X)
$$
\n(17)

Levenberg (1944) and later Marquardt (1963) suggested to use a damped Gauss–Newton method. The step h_{lm} is defined by the following modification to $(J^T J) h_{gn} = -J^T E$,

$$
(JT J + \mu I)hlm = -g,
$$
\n(18)

with $g = J^T R$ and $\mu \ge 0$.

Here, $J = J(X)$ and $R = R(X)$. The damping parameter influences both the direction and the size of the step, that's why we use a method without a specific line search. X_0 being the initial value of X, the choice of initial value of μ should be related to the size of the elements in $A_0 = J(X_0)^T J(X_0)$, e.g. by letting

$$
\mu_0 = \tau \cdot \max_i A_{ii}^0,\tag{19}
$$

where τ is chosen by the user. During iteration μ can be updated as described in Refs. $[14–16]$ $[14–16]$. The updation is controlled by the gain ratio

$$
\varrho = \frac{C(X) - C(X + h_{lm})}{L(0) - L(h_{lm})},\tag{20}
$$

where the denominator is the gain predicted by the linear model,

$$
L(0) - L(h_{lm}) = -h_{lm}^T J^T R - \frac{1}{2} h_{lm}^T J^T J h_{lm}
$$

=
$$
\frac{1}{2} h_{lm}^T (\mu h_{lm} - g)
$$
 (21)

The stopping criteria for the algorithm should reflect that at a global minimizer we have $C'(X^*) = g(X^*) = 0$, so we can use

$$
\|g\|_{\infty} \leq \varepsilon_1 \tag{22}
$$

where ε_1 is a small, positive number, chosen by the user. Another relevant criterion is to stop if the change in X is small,

$$
||X_{\text{new}} - X|| \le \varepsilon_2 (||X|| + \varepsilon_2). \tag{23}
$$

This expression gives a gradual change from relative step size ε_2 when ||X|| is large to absolute step size ε_2^2 if X is close to 0. Finally, as in all iterative processes, we need a safeguard against an infinite loop,

 $k > k_{\text{max}}$. (24)

Again ε_2 and k_{max} are chosen by the user.

Composite manufacturing and characterization

Resin preparation and characterization

The matrix used herein is an epoxy resin SR5550, obtained from Sicomin Composites (CHATEAUNEUF LES MAR-TIGUES, France), of density 1.145 g/cm³ with 20 $^{\circ}$ C (Pycnometer), recommended for use in wood/epoxy systems. The epoxy resin was mixed with hardener SR5503 of density 1 $g/cm³$ (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/cm³.

In order to obtain the elastic constants of this epoxy resin, five specimens were moulded and submitted to tensile testing. The data thus obtained yielded the statistical values of $\overline{E^m}$ = 4.525 GPa and $\overline{v^m}$ = 0.388 with standard deviation values of 0.371 and 0.006, respectively.

Alkaline surface treatment of the fibres

On the basis of the results presented elsewhere in Ref. [\[17](#page-7-0)], 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 preboiled demineralized 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 \degree C for 3 h. Then, the fibres were thoroughly washed with demineralized water and dried at 80 °C for 24 h (on average).

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 [3](#page-5-0)a shows the paper sketch just described.

The impregnation method used here is similar to that formerly described in Ref. [\[6](#page-7-0)], with some slight but appropriate changes according 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 the fibres in the absence of air bubbles removed by vacuum. Figure [3a](#page-5-0)–f features the above method.

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 of 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 [\[6](#page-7-0)] have reported on a method based on the water uptake of the composite, but it could not be tested in this work.

Preparation of specimens

About 3–5 specimens of average size $150 \times 20 \times 2$ mm³ (refer to Fig. [3](#page-5-0)f) 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.

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 gauge 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](#page-6-0).

Results and comments

Composites' Young's modulus

Figure [4](#page-6-0) shows the evolution of the Young's modulus as related to the orientation angle of fibres in the composite Fig. 3 Composites manufacturing stages. (a) Wrapping of fibres on a glass plate; (b) vacuum setting: stage 1; (c) vacuum setting: stage 2; (d) vacuum setting: stage 3; (e) vacuum setting: stage 4; (f) preparation of UD-composites specimens

plates Si/epoxy. One obviously expects the module to decrease with an increase in the angle. The data from Si/epoxy composites offer a curve shape consistent with the expected results.

These results should be viewed with all proper reservations. The scatter on these data might be ascribed, 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 was

observed during processing that the sisal fibres used were less prone to buckling while being wrapped on glass plates.

Estimation of fibre elastic constants

A routine for inverse calculation of fibres' properties based on Levenberg–Marquardt's method was written and implemented in MATLAB R2007a. The algorithm finds the minimum of a scalar function of several variables, starting at an initial estimate. This is generally referred to

Property	Angles											
	0°			15°			30°					
$E_{\rm p}$	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°		
$E_{\rm p}$	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	
v_{xy}	0.333	0.513	0.439	0.395								
G_{xy}	2.03	1.72	1.68	2.04								

Table 1 Tensile tests data of sisal/epoxy composite specimens

Fig. 4 Si/epoxy composites' Young's modulus E_{xx} versus fibre angle θ

as unconstrained nonlinear optimization. It returns a structure output that contains performance information about the optimization. The starting guess used herein was $X_{0\text{signal}} = \{2.05\ 11.35\ 0.0379\ 0.21\ 0.6424\}.$ The results are shown in Table 2.

The reliability of calculations performed with optimization algorithms notably depends on the starting guess X_0 , for the solution vector *s*. For E_z^f , the tensile test values reported in Ref. [[17\]](#page-7-0) were used while an estimation of G_{rz}^f was provided by the results on specimens with a 45° fibre orientation. For the remainder, that is E_r^f , $v_{r\theta}^f$ and v_{rz}^f , the

Table 2 Sisal fibre elastic constants

Sisal fibre	Properties									
	$E^{\rm f}$ (GPa)	$E^{\rm t}$ (GPa)	$v_{r\theta}$	r _z	$G_{r z}^{\dagger}$ (GPa)					
Sisal	11.547	1.424	0.174	0.776	0.126					

general trend for transversely isotropic behaviour was followed.

Optimization consists in finding the best solution to a particular problem. In fact, the implementation of the Levenberg-Marquardt routine in Matlab leads to satisfactory results for almost all the fibre elastic constants. Some disturbances for the obtained values could seemingly be attributed to inaccuracies in the determination of the fibre volume fractions applied to this analytical model. Actually, the fibre and matrix volume fractions have a dramatic influence on the properties of a composite material. This, of course, remains a matter of concern for the study of natural fibre reinforced composites (NFRCs) behaviour.

Conclusions

A comprehensive analytical inverse method of characterization of the elastic anisotropic behaviour of natural fibres and their related composite materials has been presented herein. It was found that, despite several decades' use of synthetic fibres in composites, there are still many issues to overcome for a better application of plant fibres in composite materials. Once again, it is difficult to find a convenient approach which is able to account for the more complex behaviour of our composites, namely the 3Danisotropy induced by one or both constituents (for a 2 phase composite). The case of NFRCs, assumed here to be transversely isotropic, confirms the importance of these issues. Plant fibres used for the reinforcement are porous, in particular when the extraction methods used mean that a high degree of fineness cannot be reached. Consequently, the theoretical concepts to be applied when used in composites' reinforcement should be conveniently outlined. The discrepancies found are likely to be the result of the imperfect fibre-matrix interface, the method used to determine the fibre volume fractions and the hand lay-up

manufacturing of our NFRCs. It is expected that these problems will be eliminated in future experimentations involving such fibres. A preliminary but nevertheless important stage has been completed here with a view to optimizing the estimated elastic properties of the UD laminates made of natural fibres from experimental data. The results obtained are satisfactory from the prediction point of view for each constant, and are revealing of the high degree of anisotropy exhibited by sisal fibres.

Acknowledgement Our thanks go to Mr Jacques Lepetit of INRA, Clermont-Ferrand, for his technical support.

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