Single Filament Mechanical Characterisation of Hemp Fibres for Reinforcing Composite Materials

C. Romão [a] , P. Vieira [b] , F. Peito [c] , A. T. Marques [d] & J. L. Esteves [d]

[a] IPV-Institute Polytechnic of Viseu, Viseu, Portugal

[b] INEGI-Institute of Mechanical Engineering and Industrial Management, Leça do Balio, Portugal

[c] IPB-Institute Polytechnic of Bragança, Bragança, Portugal

[d] FEUP-Faculty of Engineering of University of Porto, Porto, Portugal

Version of record first published: 18 Oct 2010

PLEASE SCROLL DOWN FOR ARTICLE

sub-licensing, systematic supply, or distribution in any form to anyone is expressly forbidden.

The publisher does not give any warranty express or implied or make any representation that the contents will be complete or accurate or up to date. The accuracy of any instructions, formulae, and drug doses should be independently verified with primary sources. The publisher shall not be liable for any loss, actions, claims, proceedings, demand, or costs or damages whatsoever or howsoever caused arising directly or indirectly in connection with or arising out of the use of this material.

Taylor & Francis
Taylor & Francis Group

SINGLE FILAMENT MECHANICAL CHARACTERISATION OF HEMP FIBRES FOR REINFORCING COMPOSITE MATERIALS

C. Romão
IPV-Institute Polytechnic of Viseu, Viseu, Portugal

P. Vieira
INEGI-Institute of Mechanical Engineering and Industrial Management, Leça do Balio, Portugal

F. Peito
IPB-Institute Polytechnic of Bragança, Bragança, Portugal

A. T. Marques and J. L. Esteves
FEUP-Faculty of Engineering of University of Porto, Porto, Portugal

The work described in this paper refers to the mechanical characterisation of Portuguese hemp fibres, to be used on the production of composite materials with an epoxy matrix. A single filament mechanical characterisation is made, for fibres with and without a cleaning bath from a solution of sodium hydroxide, with the propose of increasing the adherence fibre/matrix.

INTRODUCTION

Today, the search for new, recyclable and renewable materials is leading the researchers to new ways. Natural products are emerging and some research is starting in this matter. In our work, we are going to characterise mechanically the Portuguese natural hemp fibre. The single filament characterisation is made according to the ASTM D3822 standard. The tests were made with the fibre in is natural state and with a surface treatment called mercerization. The steps of all the work are described in this paper, starting with the sample preparation, the surface treatment, the measuring

of the fibres before and after the tests, and finally, the results of the mechanical characterization and the comparison with the hemp composite.

FIBRE PREPARATION

The fibre is placed, aligned with the longitudinal axis of the cardboard frame as shown in Figure 1. This disposition gives as the possibility of assembling the sample in the testing machine. The connection between the cardboard frame and the hemp fibre was made with cianoacrylate glue. Due to the small length of the monofilament hemp fibre, each fibre was positioned previously with adhesive labels. The distance between the two glue drops defines the distance between the grips, that is to say, the reference length of the traction test. The selection of this length is of extreme importance, because normally the traction resistance decreases, as the length of the fibre increases, as well as the error associated to the results of the traction tests.

MEASURING THE FIBRES

After assembling the hemp fibres in cardboard frames, it was necessary to measure the transverse section for determination of the respective area, in order to determine the tensile strength and the traction Young modulus.

The variability of dimensions and geometry of the hemp fibres also occurs along each fibre. This fact eliminated the hypothesis of being considered a average area and forced us to make individual measurements. These measurements were made along the length of each fibre, more precisely every each 2.5 mm. So, for the samples with reference lengths of 5 mm three measurements (extremities and centre) while were made, for the samples with reference lengths of 10 mm five measures were made (extremities, centre and between the centre and the extremities of the fibres). In spite of the hemp fibres generally possess transverse sections
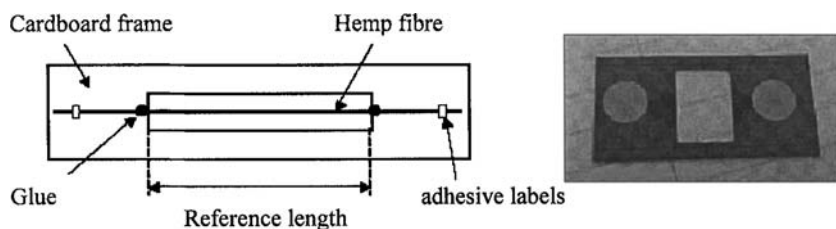

FIGURE 1 Assembling of the hemp fibre in the cardboard frame.

of polygonal geometry, it was considered, for this work, a circular geometry. The diameter measurement was made using two different techniques: the technique of light diffraction and the optical microscopy. The first was, in the beginning, very attractive, due to the high number of samples to be measure. However, after the tests and due to the results obtained, it has been considered the possibility of this method introduced significant errors in the calculated values of the mechanical properties of the fibres. The origin of these possible errors could lie in the fact that the measurements could have been made in places with defects or with superficial sludge's. Because of that, we repeated the whole process using the optical microscopy.

Light Diffraction Technique

As we can see in Figure 2, the cardboard frame with the fibre is placed in the support. After this we project the diffracted light that changes the angle when passing through the fibre. After measuring the distance between the two first nodes of the image projected in the display table, we could, using an Eq. (1), calculate the diameter.

$$\text{If } A >> \frac{\pi^* d_f^2}{4^* \lambda_{He-Ne}} \quad \text{then } d_f = \frac{2^* \lambda_{He-Ne}^* A}{\delta} \tag{1}$$

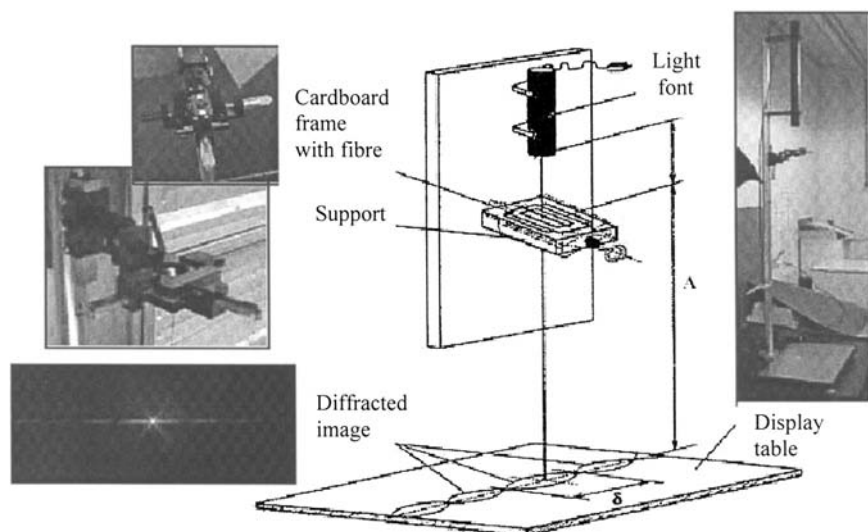**FIGURE 2** Light diffraction equipment.

d_f – Fibre diameter; λ_{He-Ne} – Wave length of the laser beam; A – Distance between the support and the display table; δ – Distance between the two first nodes projected.

This measuring system is quite effective when applied to synthetic fibres. However, when natural fibres are used the location of the interference nodes from the diffracted image in the display table can be difficult to find. The smallest precision in the diffracted image can be the result, as it was already referred, of the presence of defects, of superficial sludge's in the fibres or of the geometry of the same ones.

Optic Microscopy

The second diameter measurements were made using an optic microscopic equipment (Fig. 3). Using this equipment we could confirm (or not) the first measurements made and see the rupture. We could also observe some defects in the fibres. In Figure 4 some of these images can be seen. The
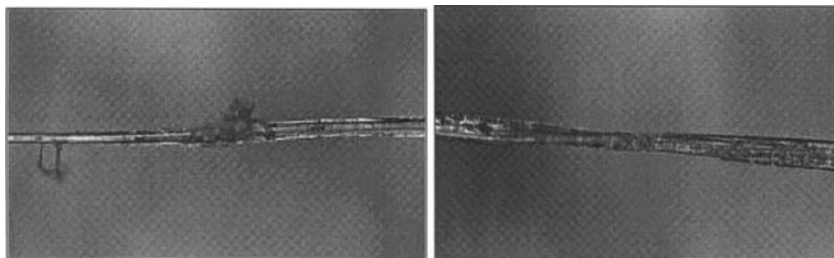

FIGURE 3 The microscopic equipment.

FIGURE 4 Some defects in the fibres.

TABLE 1 Values of the Fibre Measuring

		Fibre diameter (μm)					
		Without treatment			Treatment of mercerization		
		$L_0 = 5\,mm$	$L_0 = 10\,mm$	Total	$L_0 = 5\,mm$	$L_0 = 10\,mm$	Total
Light	Average	23.5	22.0	22.4	21.2	22.1	21.8
diffraction	Stdev.	6.0	5.3	5.6	5.4	4.7	5.0
technique	Minimum	13.2	11.2	11.2	13.4	12.2	12.2
	Maximum	36.5	37.6	37.6	40.7	34.5	40.7
	N° fibres	27	40	67	31	41	72
Optic	Average	26.1	23.5	24.3	22.4	24.5	23.8
microscopy	Stdev.	6.7	5.4	5.9	4.7	5.3	5.2
	Minimum	14.2	12.9	12.9	12.0	12.3	12.0
	Maximum	42.0	38.8	42.0	43.7	41.8	43.7
	N° fibres	27	40	67	31	41	72

results of the measurements made are shown in Table 1. We can see in this table that the Stdev (Standard deviation) is very high, approximately 23.5%. This is due to the variability in dimensions of the hemp fibres. We can see that the mercerization treatment did decrease the diameter. It would be expected that, due to the removal of substances, the medium diameter of the fibres decreased, which was not always verified. This fact should result of the great variability in dimensions that characterize the vegetable fibres.

SURFACE TREATMENT

With the hemp fibres a surface treatment has been done to increase the fibre/matrix adhesion. This treatment (mercerization) is made in some steps. First step is to perform an immersion procedure in a bath, two hours in a solution of 8% in volume of Sodium Hydroxide (NaOH) with distilled water. During this process the bath was stirred continuously using a mechanical agitator (Fig. 5). At the end, the solution presented a yellow colour because of the substances removed from the fibre. The next step is the cleaning of the fibres several times in a distilled water bath, until the water is clean. After several baths, a neutralizing solution of 25% in volume of acetic acid is used. Again, more two or three baths with distilled water and the treatment is finished. To dry the fibres we left them 5 days at ambient temperature and, then, six hours at 60°C in an oven (Fig. 6) [3–6].
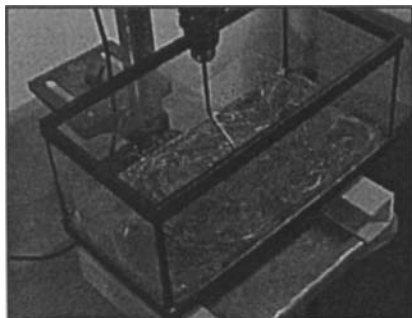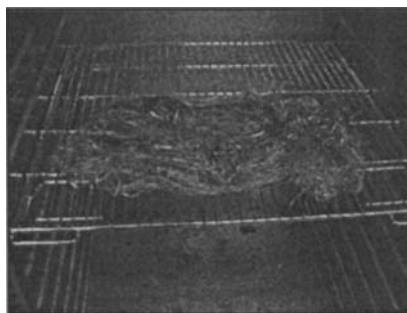
FIGURE 5 Sodium Hydroxide bath.

FIGURE 6 Fibres in the oven.

MECHANICAL TESTS

The testing machine was equipped with a load cell of 2,5 N and with pneumatic grips adapted for the fixation of the carboard frames. The upper grip was freely suspended in the load cell, so that when a tensile force was applied, the grip auto aligns itself with the longitudinal axis of the fibre. Before beginning each test, the lateral parts of the cardboard frame were cut through an incandescent metallic wire, so that only the hemp fibre is submitted to the tensile force. Figure 7 shows all process. In agreement with the standard ASTM D3822, the test speeds, were of 0.5 mm/min and 1,0 mm/min, respectively, for the samples with lengths of reference of 5 mm and 10 mm. The tests were preformed in an atmosphere under temperature conditions and relative humidity of $21 \pm 1°C$ and $65 \pm 2\%$, respectively. Analyzing through optical microscopy the rupture section of the tested fibres, we could verify that the rupture
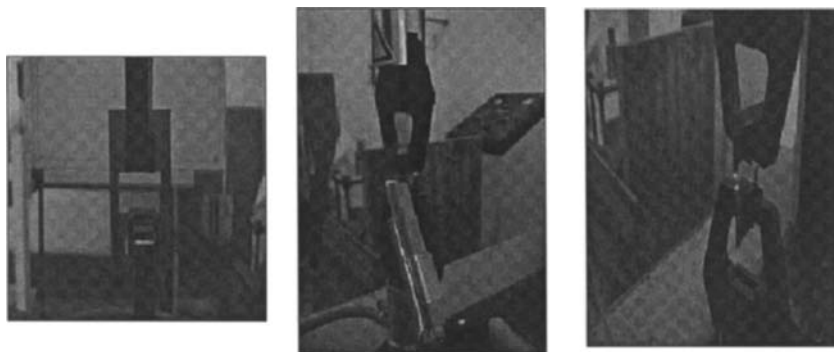
FIGURE 7 Fixation in the pneumatic grips/Cutting the cardboard frame/Ready for testing.
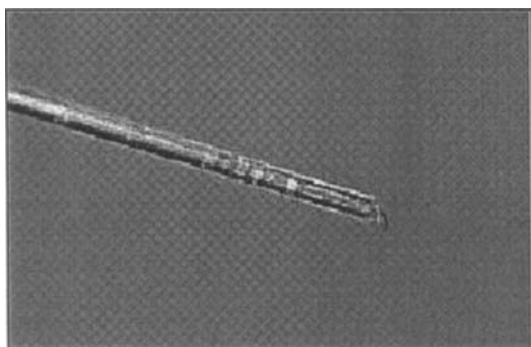

FIGURE 8 Plane surface separation.

happened by forming a plane surface separation (Fig. 8) or by an irregular laceration of the structure (Fig. 9). In Table 2, we present the ruptured diameters, used in the calculation of the tensile strength of the hemp fibres. We used these new values, because the average value calculated measuring the fibre in several points could had an error. Using the ruptured value for tests we eliminate that error. The average rupture diameter is of the same order of magnitudes as the average diameter of the fibres, presented in the Table 1. Although the average values obtained by the two measuring techniques are not very different, it was verified great differences between some fibres rupture diameter and the medium diameter of the respective fibre, originating these situation incorrect values in the tensile strength.
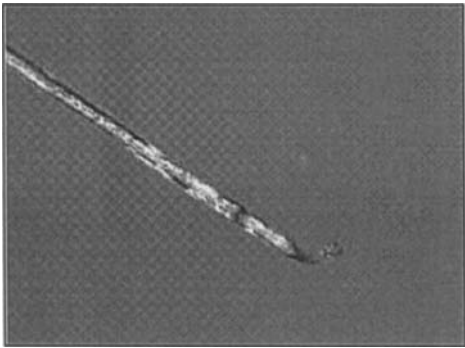
C. Romão et al.

FIGURE 9 Irregular laceration of the structure.

TABLE 2 Values of the Rupture Section

		Fibre diameter (μm)					
		Without treatment			Treatment of mercerization		
		$L_0 = 5\,mm$	$L_0 = 10\,mm$	Total	$L_0 = 5\,mm$	$L_0 = 10\,mm$	Total
Light	Average	23.4	21.0	21.9	20.4	21.2	20.8
diffraction	Stdev.	5.8	4.1	5.0	5.3	4.9	5.1
technique	Minimum	13.2	13.5	13.2	14.5	14.1	14.1
	Maximum	35.5	31.8	35.5	33.6	31.0	33.6
	No. fibres	27	40	67	31	41	72
Optic	Average	24.6	20.9	22.4	21.9	22.2	22.0
microscopy	Stdev.	5.5	4.8	5.4	3.5	4.5	4.1
	Minimum	14.2	10.4	10.4	15.4	14.4	14.4
	Maximum	34.7	29.6	34.7	29.8	35.8	35.8
	N° fibres	27	40	67	31	41	72

MAKING AND TESTING A COMPOSITE

To manufacture a plate we used the compression moulding technique. The mould has a cavity of $150 \times 100 \times 4\,mm3$ and is made of aluminium. The first step is to clean the surface and then apply the mould release agent QZ13 from Ciba. After the preparation of the resin, we introduce it in the mould. The mat is then placed in and the mould closed. At least everything is placed in the hot plate press, where the combination of pressure and temperature makes the plate. After 1 hour in the press at ambient temperature and with a post cure of 6 hours at $60°C$, the mould has been open and the trimming of the plate was made.

For the mechanical tests we used an INSTRON 4208. We made the tests according to the ISO 527–4 standard. We used a 100 kN load cell and a 2 mm/min traction speed.

RESULTS

When represented graphically, the tension/displacement values measured during the single filament test present a typical aspect shown in Figure 10 (fibres without treatment) and in Figure 11 (fibres with mercerization treatment).

In the beginning of the tests and in both curves we verified that, the force supported by the fibres varies in a non linear way with displacement. This initial behaviour is due to the fact that the fibres are not perfectly aligned, being necessary to make a correction in the displacement of the mobile grip so that we can determine the effective displacement, during the test. To accomplish this correction it was necessary to extrapolate the linear portion of the experimental curve to intersect the abscissas axis.
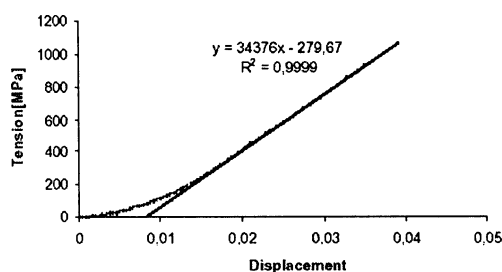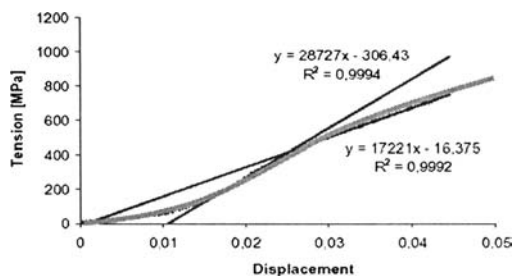

FIGURE 10 T/D without treatment.

FIGURE 11 T/D With mercerization treatment.

Starting from the analysis of the previous figures, it was possible to verify that the strength-displacement curve corresponding to the treated fibres has two linear portions. For these fibres two modules of elasticity were determined corresponding, one to the first linear portion, and the other to the second linear portion.

After the tests made, we used the following formulas to calculate the tensile strength (2) and the Young Modulus (3).

$$\sigma_r = \frac{F_r}{A_r} \tag{2}$$

σ_r – Fibre tensile strength; F_r – Traction force at rupture; A_r – Rupture section of the fibre.

$$E = \frac{\Delta\sigma}{\Delta\varepsilon} = \frac{\Delta F^* L_0}{A^* \Delta L} \tag{3}$$

E – Young Modulus; $\Delta\sigma$ – Tensile strength variation; $\Delta\varepsilon$ – Displacement variation; ΔF – Traction force variation; L_0 – Reference length; A – Average of the transversal section of the fibre; ΔL – Displacement variation*.

Tables 3 and 4 show us the tensile strength and the Young modulus for the fibres tested.

In both cases we tested 27 fibres without treatment and 31 with the mercerization treatment. The standard deviation is very high. The number of tests should increase to solve this problem. The fibre variability is definitely influencing these results, and so the number of tests that we need to do in natural fibres should increase drastically. We can see that, by increasing the reference length, the properties worsened, and that is logic. What we didn't expect is that the mercerization treatment decreases the tensile strength in 25% and the Young modulus in 48%. Further study should be made to better understand this. Other significant analysis is the fact that the optic microscopy decreases the results in about 10%. This is due to the improvement in calculating the fibre section.

The composite results can be seen in Table 5. The results are far from good, because the tensile strength decreases 8% without treatment and with the treatment decreases 24%. The Young Modulus increases 200% without treatment and 140% with treatment. Comparing this results with the mechanical characterization of the filaments, we can see that the influence of the treatment has a similar behaviour. The Tensile strength of both composites is very low. If we compare it to a glass fibre composite

*The displacement values were corrected taking into consideration the system compliance.

TABLE 3 Tensile Strength

		Tensile strength [MPa]			
		Without treatment		Treatment of mercerization	
		Average	STDV	Average	STDV
Light	$L_0 = 5$ mm	948	403	868	290
diffraction	$L_0 = 10$ mm	943	498	718	240
technique					
Optic	$L_0 = 5$ mm	1110	409	722	226
microscopy	$L_0 = 10$ mm	970	502	638	206

TABLE 4 Young Modulus

		Young modulus [GPa]					
				Treatment of mercerization			
		Without treatment		Average**			
		Average	STDV	Initial	Secondary	STDV	
Light	$L_0 = 5$ mm	66	20	35	16	12	5
diffraction	$L_0 = 10$ mm	57	13	33	16	12	5
technique							
Optic	$L_0 = 5$ mm	55	18	26	14	9	4
microscopy	$L_0 = 10$ mm	50	13	24	13	8	4

**The two modulus shown in this table are of the two lines explained in Figure 11. Only the initial modulus was taken in consideration for the rest of the appreciation.

TABLE 5 Composite Results

Composite	% Fibre [%]	Tensile strength [MPa]	Modulus [GPa]	Deformation [%]
REAPOX WOOD RX8	25	45.6 ± 0.9	5.4 ± 0.4	0.98 ± 0.03
+	30	45.1 ± 6.3	6.2 ± 0.8	0.90 ± 0.22
Hemp without treatment	35	47.1 ± 3.6	6.7 ± 0.5	0.81 ± 0.15
REAPOX WOOD RX8	25	37.9 ± 1.9	3.8 ± 0.3	0.69 ± 0.02
+	30	38.3 ± 3.9	4.4 ± 0.6	0.64 ± 0.16
Hemp with treatment	35	40.7 ± 3.5	4.6 ± 0.3	0.56 ± 0.10
REAPOX WOOD RX8		50	3.0	7

C. Romão et al.

that has more or less 190 MPa, 46 MPa is less than 25%. This should increase. When talking about Young modulus, the difference is less, about 80% of the glass fibre value. This of course without the mercerization treatment. With the treatment the values should decrease more 25%.

CONCLUSIONS

In this study, in which we characterize the hemp fibres and compare them with mercerized treated ones, we arrived to several conclusions. Natural fibres are in reality difficult to characterize. The results obtained are far from good, the standard deviation, in some cases, being greater than 50%. The section of the fibre is very difficult to calculate, because there is no geometric pattern associated to it. We calculate the section as if the fibres were cylindrical. The results using optic microscopy are more accurate, but the cylindrical simplification is probably still giving some errors to the process.

It is necessary to point out the research in the cleaning and in the surface treatment of the fibre. Only with good surface treatment we can obtain good mechanical properties. To evaluate the influence of the alteration of the chemical composition and of the morphology of the cellular wall in the properties of the fibres in study, took place a treatment of mercerization to a group of hemp fibres.

That treatment proved to be inappropriate, because it didn't improve the mechanical properties, but instead it decreases those properties. The acid damaged the cellular wall, and that was the reason for this problem. Future works should be made to find a treatment that doesn't change the mechanical properties of the fibres and increases the adhesion fibre/matrix. We are going to start with removing the acid bath.

REFERENCES

[1] Romão, C. (2003). Study of the Mechanical Behaviour of Composite Polymeric Materials Reinforced with Natural Fibres, Dissertation presented for obtaining a Master's degree in Mechanical Engineering, from the Engineering Faculty in the University of Porto.
[2] Project SAPIENS POCTI/40201/EME/2001, Mechanical behaviour of Composites of Polymeric Matrix Reinforced with Natural Fibres, Report of Annual Progress, 1° year of activity, 2002.
[3] Peters, R. H. (1963). *Textile chemistry I: the chemistry of fibres*, Elsevier Publishing Company.
[4] Peters, R. H. (1967). *Textile chemistry II: impurities in fibres, purification of fibres*, Elsevier Publishing Company.

[5] Rowell, R. M. (1995). Chemical Modification of Agricultural Fibres for Property enhanced Composites, Cap. V, Project: Modification of Lignocellulosics for Advanced Materials and New Uses, http://www.fppl.fs.fed.us

[6] Vieira, P., Romão, C., Marques, A. T., & Esteves, J. L. (2003). Mechanical Characterisation of Natural Fibre Reinforced Plastics, MATERIALS 2003, II International Materials Symposium April, 14–16.