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Micromechanics of hemp strands in polypropylene composites

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A B S T R A C T

The present paper investigates micromechanics of hemp strands. The main objective of the present work has been the determination of the intrinsic strength of hemp strands. Hemp strands have been used as reinforcement of Polypropylene composites. Different percentages of hemp strands and coupling agents (MAPP) have been tested to obtain a map of the mechanical properties of that kind of composites and the effect of the components on the final properties. Mechanical properties of the different specimens have been tested using standard experimental methods and equipment. Micromechanics of the strands have been obtained using Hirsch model, Bowyer–Bader methodology and Kelly-Tyson model.

1. Introduction

The research of the macromechanical properties of the composite materials with hemp strands as reinforcement is a current issue [1–3]. Some years ago the research group LEPAMAP studied the tensile, flexural and impact macromechanical properties, for hemp strands/polypropylene composites [4,5]. However there is little bibliography about micromechanics of composite materials with natural fibers or strands as reinforcements [6–11].

Usually for the composite materials, and particularly for the ones using Hemp strands as reinforcement it is important to evaluate the orientation factor, the interfacial shear strength and the intrinsic tensile strength of the hemp strands inside the polymeric matrix. Most of the published researches on the intrinsic tensile strength of hemp strands are based on single fiber pull out test and experimental measurements of the ultimate tensile strengths of the strand [6,12–14]. A revision of the available data and the results showed the possibility to use the experimental data to study the micromechanical properties of hemp strands based on theoretical models [10,15].

The modified rule of mixtures, $\sigma_t^C = f_c \cdot \sigma_t^F \cdot V^F + (1 - V^F) \cdot \sigma_t^{m*}$, could predict, with enough precision, the behavior of composite materials with natural fibers or natural strands as reinforcements. In the equation σ_t^C and σ_t^F are the ultimate tensile strengths of composite and strand, respectively. σ_t^{m*} is the matrix tensile stress at the failure point of the composite. V^F is the volume fraction of the reinforcement and f_c is the compatibility factor. For an interface optimized composite f_c takes values around 0.2 [16]. The main problem involves the estimation of the value of the intrinsic tensile strength of the fiber (σ_t^F) needed to predict σ_t^C. Due to the dimension of the majority of the lignocellulosic fibers from agro-forestry (corn stalks, rape stalks, hemp core fibers...), the measurement of their strength is practically impossible. In the case of hemp strands, due to its major length, it is possible and there is bibliographic data, for the determination of the characteristic for natural strands (hemp, abaca, sisal, jute...) [9,17]. However depending on the article the values had very high deviations with high standard deviations. In addition strands from annual plants present an internal lumen that collapses during compounding [18]. That change on the structure of the strand could cause a change on the intrinsic properties, and the σ_t^F measured could change once the strand is compound.

Initially the value of σ_t^F is a function of the strand typology and the magnitude of the coupling between matrix and reinforcement. The experience points out that the main factor that determines the value σ_t^F inside the composite is the degree of adhesion of the strand with the polymer [10].

In the present work the intrinsic tensile strength of the hemp strands is evaluated from composite materials reinforced with a 20, 30, 40 and 50 wt.% [5].

Hirsch model has been used to obtain the intrinsic Young modulus of the strands, Bowyer–Bader methodology to compute the

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orientation factor (χ_1) and the interface shear strength (τ), and the Kelly-Tyson equation to calculate the intrinsic tensile strength (σ_t^F).

2. Materials and methods

2.1. Materials

The untreated hemp strands where provided by Agrofibra S.L. (Puigreig, Spain). The initial hemp strands length was between 20 and 30 cm. The polymeric matrix used was polypropylene ISPLEN® 090 G2M (Repsol-YPF, Spain). A modified maleic anhydre-grafted polypropylene (MAPP) coupling agent was used: Epolene® G3015 from Eastman (Netherlands).

2.2. Methods

2.2.1. Composite preparation and characterization

The hemp strands were chopped to a nominal length of 10 cm in a blade mill and dried in a Dycometal oven at 80 °C for 24 h before its use as reinforcement. A heated roll mixer from IQAP LAB, S.L. (Roda de Ter, Barcelona, Spain) was used to blend the Polypropylene (PP), hemp strands, and MAPP. The process took 10 min at 180 ± 5 °C. Composite materials comprising 20, 30, 40 and 50 wt.% of hemp strands and 0, 2, 4, 6 or 8 wt.% of MAPP with respect to the fiber content were obtained. The blends were cut down to pellets in a blade mill from Agrimsa (St. Adrià del Besos, Barcelona, Spain). The samples for the tensile test were produced with a steel mold in accord to ASTM D3641 standards in an injection-molding machine (Meteor 40, Mateu&Solé). For each composite blend were obtained 10 test specimens.

2.3. Micromechanics

2.3.1. Intrinsic tensile modulus

The intrinsic tensile modulus of the hemp strands was determined using the Hirsch model [10,17,19,20]. With a factor β, that determines the stress transfer between fiber and matrix, with a value of 0.4 [21].

2.3.2. Determination of the interfacial shear strength (IFSS) and the fiber orientation factor (τ)

With current standard processing techniques, perfect fiber alignment is almost impossible, and the orientation factor (τ) must be taken in account. The calculation of τ can be accomplished through the Kelly-Tyson modified equation (Eq. (1)) [15,22].

$$\sigma_t^C = \chi_1\left(\sum_i\left[\frac{\tau\cdot l_i^F\cdot V_i^F}{d^F}\right]+\sum_j\left[\sigma_t^F\cdot V_j^F\left(1-\frac{\sigma_t^F\cdot d^F}{4\cdot\tau\cdot l_j^F}\right)\right]\right) + (1-V^F)\cdot\sigma_t^{m*} \quad (1)$$

where σ_t^C and σ_t^F represent the ultimate tensile strength of the composite and the reinforcing fibers. σ_t^{m*} is the contribution of the matrix at failure. d^F and $l_{i,j}^F$ represent respectively the fiber diameter and the length, and V^F is the volume fraction of reinforcement in the composite and the characteristics of the reinforcing fibers: strength (σ_t^F), orientation (χ_1), IFSS (τ), diameter (d^F), and length distribution (l^F). In order to solve the equation Bowyer–Bader methodology has been used [10,15,19,23] evaluating χ_1 and τ.

2.3.3. Determination of the intrinsic tensile strength (σ_t^F)

Once the intrinsic tensile modulus, τ and χ_1 are known, applying the data for the ultimate failure point in Eq. (1) the intrinsic tensile strength could be calculated by means of bisection numerical methods starting with a lower limit (x_l) and an upper limit (x_u) for σ_t^F.. The values are based on the bibliography, ensuring a solution and a fast convergence, inside the defined interval: $x_l = 350$ MPa and $x_u = 800$ MPa.

3. Results and discussion

The objective was to determine the value of the mean intrinsic strength (σ_t^F) of hemp strands (HS) inside the composite. The first steep was the optimization of the interface HS/PP. To do so different percentages of MAPP were tested. In these formulations, the quantity of coupling agent is related to the fiber content. The optimum percentage of MAPP was found to be at 8 wt.%, with respect to the fiber content, for composites at 20% by weight, while it was about 4 wt.% for composites at 30–50 wt.%. For higher reinforcement percentage, lower quantity of MAPP coupling agent was needed to attain the maximum efficacy of the stress transfer at the fiber–matrix interface.

Fig. 1 shows the evolution of the weighted strand size and aspect ratio in function of the percentage of reinforcement.

The evolutions on the lengths clearly show the effects of the shear efforts experienced by the composite when the percentage (wt.%) of reinforcement is increased. The weighted length experiences a decrease of 61% when the percentage of reinforcement evolves from 20 to 50 wt.%. The diameter of the strands (Table 2) could be considered as approximately constants and independents of the wt.% with a mean value of 31.64 μm and a standard deviation of 1.01. So l^F/d^F diminishes as the same time as the wt.% is increased. In Fig. 2 it could be observed that the mean fiber length (l^F) decreases as the wt.% of reinforcement increases. The diminution of the l^F could be caused by the attrition happening during the composite fabrication [24,25]. This is more relevant for coupled composites as the fiber is better tied to the matrix [10].

Fig. 2 and Table 2 show the stress–strain curves and data for the different percentages of reinforcement tested, reflecting the change of the σ_t^C in relation with the deformation ε_t^C. The different curves correspond with the experimental outcome that is closest to the mean σ_t^C value. It is verified that when σ_t^C increases ε_t^C decreases as a consequence of the rise in rigidity of the material.

The matrix tensile strength function at any strain is obtained by fitting the equation to the experimental stress strain result. The mean result is reflected by a polynomial 4th grade regression which is $\sigma_t^m = -0.0159\varepsilon^4 + 0.3712\varepsilon^3 - 3.3674\varepsilon^2 + 14.895\varepsilon + 0.0493$ in this case.

The theoretical intrinsic Young's modulus for the hemp strands, using Hirsch model, is evaluated to be 27.3 GPa as a mean value, very similar to 24.8 ± 16.3 obtained by Beckerman and Pickering [6]. E_t^C for the different composites with a 20, 30, 40 and 50 wt.% has a respective value of 3, 3.85, 5.2 and 6.35 GPa.

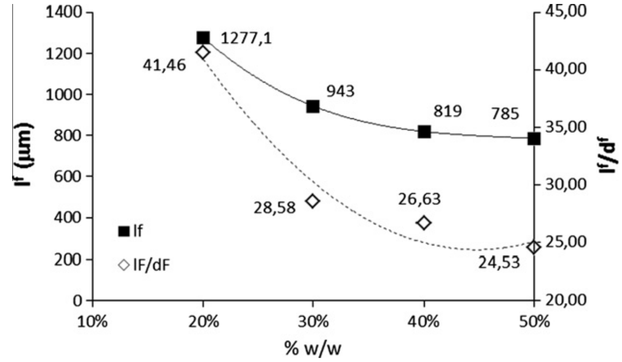

Fig. 1. Evolution of the strand length and of the aspect ratio l^F/d^F vs. reinforcement percentage.

Table 1
Composite properties. Stress–strain input data and parameters.

Reinforcement content (%)	20%	30%	40%	50%
Reinforcement volume content (v/v)	0.132	0.206	0.288	0.378
Weighted average length (μm)	1277	943	819	785
Average diameter (μm)	30.8	33.0	30.8	32.0
Composite strength (MPa)	36.2	43.4	48.8	57.1
Composite modulus (GPa)	3.00	3.85	5.20	6.35
Strand modulus (GPa)	27.3	27.3	27.3	27.3
Elongation at break (%)	3.9	3.7	3.5	3.3
Strain level 1 analyzed (%)	0.97	0.92	0.88	0.82
Composite stress at strain level 1 (MPa)	31.9	23.5	23.5	26.9
Strain level 2 analyzed (%)	1.95	1.85	1.76	1.65
Composite stress at strain level 2 (MPa)	19.1	37.5	29.3	43.4
Matrix stress at strain level 1 (MPa)	11.7	11.2	10.8	10.2
Matrix stress at strain level 2 (MPa)	18.8	18.2	17.7	17.0
Matrix stress at break (MPa)	25.3	24.9	24.5	24.0

The experimental data values required to apply Bowyer–Bader methodology are summarized in Table 1.

The composite shows a typical tensile stress–strain curve for a treated fiber composite. The elongation at break is 3.5%. Strain levels at ¼ and ½ of the breaking point have been chosen as reference levels to perform the calculations. The values of stress at level 1 and 2 (Table 1) are deduced from the experimental data [5].

The Bowyer–Bader model, as an approximation, assumes: that the stress transfer at the interface increases linearly from zero at the fiber end to a maximum value, fiber–matrix debonding does not happen, χ_1 is independent of strain and constant for all fiber lengths, interfacial shear stress is independent of loading angle, porosity in the composite is negligible and fiber and matrix stress vs. strain curves is linear.

From the input data and applying the methodology some results have been obtained (Table 2).

The mean value of the orientation factor (χ_1) for the different composites (20, 30, 40 and 50 wt.%) is 0.286. That value is very similar to the one obtained for a composite of PP and Stone ground wood [10]. The obtained orientation factor implies a mean orientation angle of 43° taking in account that $\chi_1 = \cos^4\theta$. The value is also compatible with the one obtained by [6], 0.34 that represents a mean orientation angle of 40.2°. There are big controversies with regard on those values among different authors [15,26]. Khamsehnezhad uses as orientation factor (χ_1) the modular orientation factor (η_0), and Thomason also uses typical values of the modular orientation factor (η_0).

The critical fiber length is calculated by: $l_c^F = d^F \cdot \sigma_t^F/2\tau$.

The value of the mean interface shear strength (IFSS) obtained is 14.45 MPa. The value stands in the interval of 15.95 MPa and 13.8 MPa derived from the application of the Von Misses and Tresca criteria respectively [15,27], considering the σ_t^m of the polypropylene. That value of the IFSS could be denoted as near to the optimum, given that the value of σ_t^C is meaningful when compared with another strands or fibers [6,10,16,17].

Also could be observed that l_c^F increases as the wt.% of reinforcement increases. If the equation is examined and as d^F and τ are almost constant, l_c^F depends approximately lineally of σ_t^F.. However in all the cases the mean weighted length in the composite is bigger that the critical length ($l^F > l_c^F$) (Fig. 3). The l^F is function of the transformations to which the strands are subject to during the preparation of the composite [28]. If ($l^F > l_c^F$) there will be supercritical fibers, and as bigger is the difference the better because the contribution of supercritical fibers will increase. Fig. 3 shows that the distance between critical and supercritical fibers decreases and so it is predictable that the contribution of supercritical fibers will diminish in percentage.

Table 2
Output data.

Reinforcement content (%)	20%	30%	40%	50%
Orientation factor $-\chi1$	0.28	0.315	0.27	0.28
Interface shear strength (MPa) – τ	14.95	13.05	14.25	15.60
Fiber's tensile strength at max. stress σ_t^F (MPa)	472.0	527.7	616.5	606.5
Critical length (μm) – l_c^F	486	667	665	622

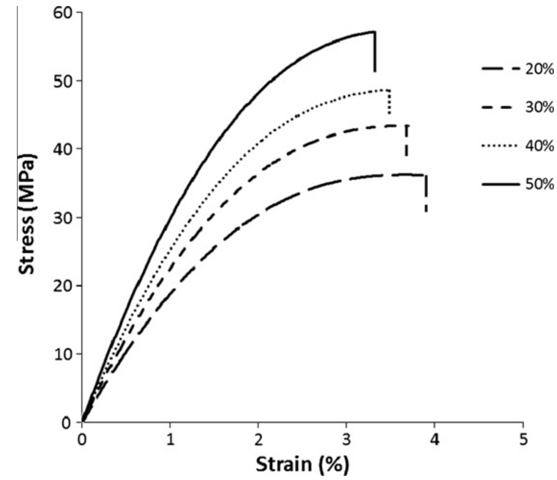

Fig. 2. Stress strain diagrams for the different %w/w of reinforcement.

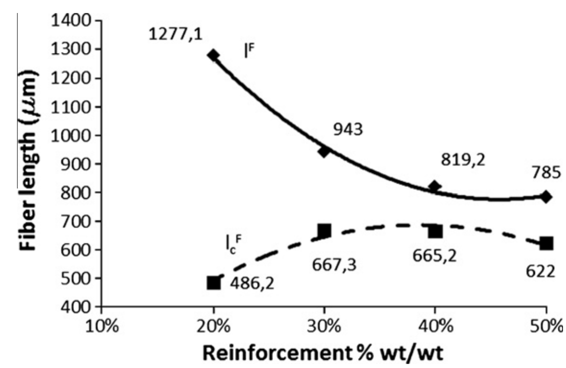

Fig. 3. Mean weighted length vs. critical length.

Once obtained the values for χ_1 and τ we used Kelly-Tyson modified equation (Eq. (1)) to obtain a value of σ_t^F for all the tested composites (Table 2). The mean value of σ_t^F for 20, 30, 40 and 50 wt.% composites is 555.7 MPa ± 68.5, and it is compatible with other publications [12,29,30]. However the standard deviation takes a value of 68.5 showing the involved large range of values.

Anyway the range of values for the fibers tensile strength present in the literature is very wide taking values from 347 ± 107 to 947 ± 245, and taking in account a lot of treatments and boundary conditions. The majority of the measurements are made by single fiber tensile testing using elementary hemp strands separated from the fiber bundles. The strands are subject to structural transformation in the compounding process (lumen collapse, diameter changes, length diminution…). All that facts tend to consider that a measurement of the fibers tensile strength based on a model that takes in account the real contribution n of the fiber to the composite as more accurate. The value obtained by Kelly-Tyson modified equation has as inputs the experimental measurement made to the composite and from that values is able to evaluate σ_t^F.

One of the assumptions made by the Kelly-Tyson model is the way the fibers are loaded. The model assumes that at the end of

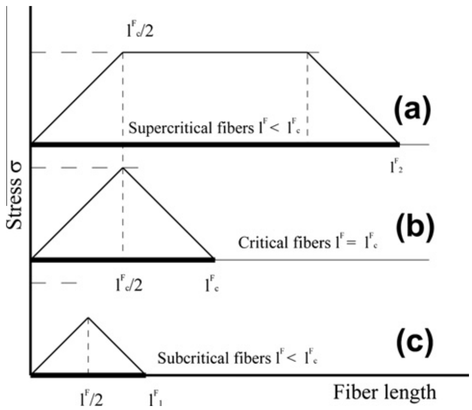

Fig. 4. Axial load diagrams for (a) subcritical, (b) critical and (c) supercritical length fibers.

the fibers the load is null and it increases along its length. The axial loads are transmitted to the fiber in form of shear loads along its surface, and then transformed in axial loads inside the fiber. Three different cases must be differentiated depending on the critical length (l_c^F). In the case of subcritical fibers ($l^F < l_c^F$) it is impossible to fully charge them, so the load increases until half its length and then decreases to the other end (Fig. 4a). The critical length fiber (l_c^F) is able to increase the load until its saturation point and then decrease (Fig. 4b). In the case of fibers larger than the critical, at ½ of l_c^F the axial load of the fiber remains constant and the response is symmetrical from ½ of its length (Fig. 4c). Assuming that the area below the load diagram represents the amount of energy that the fiber is able to dissipate it is clear that a square area increases that capability in greater quantity than a triangular one does. That explains in part the assumption that subcritical fibers contribution to σ_t^C is less important than the contribution of supercritical fibers.

Values for X, Y and Z were calculated from Eqs. (2)–(4), respectively, and are derived from Eq. (1). To estimate the final contribution to the composite X and Y must be multiplied by χ_1.

$$X = \sum_{i}^{l_i^F < l_c^F} \frac{\tau \cdot l_i^F \cdot V_i^F}{d^F} \tag{2}$$

$$Y = \sum_{j}^{l_j^F > l_c^F} \sigma_t^F \cdot V_j^F \left(1 - \frac{\sigma_t^F \cdot d^F}{4 \cdot \tau \cdot l_j^F}\right) \tag{3}$$

$$Z = (1 - V^F) \cdot \sigma_t^{m*} \tag{4}$$

Table 3 shows the contribution of; X: subcritical fibers, Y: supercritical fibers, and Z: polymeric matrix to σ_t^C. The contribution of the subcritical fibers remains minimum, but not negligible for the composites with bigger amount of reinforcement. Thus for the case of 50 wt.% it represent the 13.5% of the total and ½ of the contribution of the polymeric matrix. In the 30 wt.% case the contribution represents an 11.7% contribution and more or less 1/3 of the contribution of the matrix. The contribution of subcritical fibers in the cases of 30% and 20 wt.% composites is really minor (2.8% and 0.02%) in comparison with the other two elements.

Table 3
Contribution of the different elements to σ_t^C.

	20%	30%	40%	50%
$X \cdot \chi 1$	0.020	1.218	5.691	7.806
$Y \cdot \chi 1$	14.27	22.47	25.44	34.42
Z	21.91	19.71	17.67	14.88

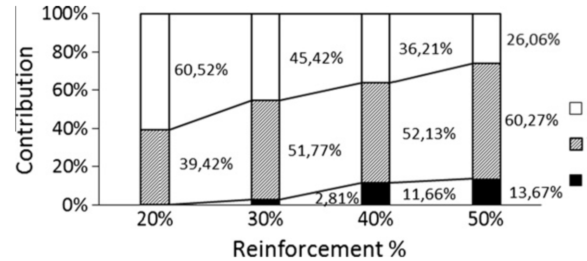

Fig. 5. Percentage contributions of X, Y, Z to σ_t^C.

Fig. 5 represents the cumulative contribution of the different element to σ_t^C.. In all the cases it is clear the contribution of supercritical fibers, representing respectively the 39.5%, 51.77%, 52.13% and 60.27%. Taking in account the cumulative contribution of the fibers ($X + Y$) the amount increases respectively to 39.48%, 54.58%, 63.8% and 74%.

4. Conclusions

The micromechanical properties of hemp strands have been investigated. The hemp strands Young modulus has been obtained by means of Hirsch model, and the final mean value is 27.6 ± 2.6 GPa, a number that is rather dissimilar to past bibliographic values but stands in line with recent publications.

The Bowyer–Bader methodology has allowed the evaluation of the orientation factor and the interfacial shear strength (IFSS). The orientation factor takes values around 0.3. That value is very different, in absolute value, from the orientation modular factor values. Moreover the mean IFSS is evaluated to be 14.45 MPa, and it is positioned inside the range of values predicted by Von Misses and Tresca criteria.

Entering the orientation factor and the IFFS data in the Kelly-Tyson modified equation it is possible to obtain a value for the intrinsic fiber tensile strength. The found figure is 555.7 ± 68.5, in line with other publications.

Finally the use of the Kelly-Tyson modified equation allows the determination of the contribution of subcritical fibers, supercritical fibers and matrix to the final tensile strength of the composite.

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