DETERMINATION OF CORN STALK FIBERS' STRENGTH THROUGH MODELING OF THE MECHANICAL PROPERTIES OF ITS COMPOSITES

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Worldwide cultivation of corn is expanding, due in part to the increasing production of bioethanol. In consequence, huge amounts of corn stalks residues are been produced. Instead of incineration, we transformed the corn stalks into a semichemical pulp and successfully applied it as reinforcement in polypropylene composites. PP composites reinforced with 40% wt corn stalk single fibers were prepared, and their mechanical properties were evaluated. Through mechanical properties modeling of the composites, the intrinsic tensile strength of the cellulosic fibers that constitute the corn stalk have been determined.

Keywords: Intrinsic tensile strength; Corn stalks; Composites; Modeling

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INTRODUCTION

The determination of the intrinsic mechanical properties of natural strands (hemp, sisal, jute, etc.) as well as lignocellulosic fibers from wood and agro-forestry (wood pulp, thermomechanical pulp, semichemical pulp, recycled fibers, etc.) is a matter of controversy. Bibliographic data for the tensile strength of most lignocellulosic fibers presents big disparities. For instance, the tensile strength of flax fibers has been reported to be between 345 and 1100 MPa (Bledzki and Gassan 1999; Oksman et al. 2009). In the case of mechanical pulps, to our knowledge, there is only one report establishing its tensile strength as 750MPa (Sanadi et al. 1994), although it does not mention the true nature of the pulp (hardwood, softwood) or type (ground wood, thermomechanical pulp, etc.). Meanwhile, the tensile strength of a kraft-bleached fiber has been reported by some researchers to be in the neighborhood of 300 MPa (Beg and Pickering 2008), whilst other reports suggest a tensile strength of over 1000 MPa (Michel and Willis 1978).

For fibers obtained from crop plants, the disparity in the tensile strength values reported can be easily understandable due to the influence of climatic conditions on plants. Besides, tensile strength of fiber strands can be empirically measured be means of single fiber analysis and, due to the naturally occurring imperfections on the fiber structure, the strength at break of the cellulosic fibers diminishes with fiber length. For this reason, typical protocols imply the analysis at variable gauge length. Then, the intrinsic strength of the fiber is extrapolated to a hypothetical zero gauge length, thus

minimizing the effect of any possible fiber imperfections. On the other side, one must also take into consideration that, due to their size, single fiber analysis is not an option for all types of wood fibers (as well as many nonwood fibers).

Nevertheless, the knowledge of ultimate tensile strength of the reinforcing fibers is a key element during the analysis of a composite efficiency and the study of the phenomena occurring at the fiber-matrix interface. Thus, quantitative estimations of tensile (and flexural) strength of reinforcing fibers have been performed thorough several mathematical models.

Starting from the analysis method of Bowyer-Bader (1972), based on the Kelly-Tyson model, Thomason developed a methodology that allowed the determination of the tensile strength of fiberglass from the mechanical properties of its composites (Thomason 2002). Tensile strength values obtained through this mathematical approach showed good agreement with typical single fiber analysis values.

In contrast to synthetic fibers such as fiberglass, when included into a composite formulation, the tensile strength provided by the reinforcing fibers does not correspond with their ultimate tensile strength. This is due to the changes suffered in their structure (lumen collapse) during compounding (Shibata et al. 2008).

In this study, we have obtained high yield fibers from corn stalks that have been subsequently used as reinforcement of polypropylene. Materials with up to 40% wt fiber content were prepared, and their mechanical properties have been evaluated. An accurate analysis of the fibre-matrix interface has been conducted through common theoretical models. This has allowed an estimation of the tensile and flexural strength of the lignocellulosic fibres that constitute corn stalks.

MATERIALS AND METHODS

Materials

Corn stalks were provided by Fundació Mas Badia (La Tallada d'Empordà, Spain). The polymeric matrix used was polypropylene ISPLEN® 090 G2M (Repsol-YPF, Spain). In order to improve the compatibility between the polymer matrix and the reinforcing fiber, a maleic anhydre-grafted polypropylene (MAPP) coupling agent was used: Epolene® G3015 from Eastman (Netherlands). According to manufacturer, this material has an acid number of 17.4 mg KOH/g and number average and weight average molecular weights of Mw = 47,000 and Mn = 24,800.

Methods

Corn stalk single fibers (CSF)

Corn stalks were submitted to a sodium hydroxide:antraquinone (AQ) cooking (12.5% NaOH: 0.1% AQ). The liquor-to-fiber ratio was 4:1. Fibers were kept at 160ºC for 30 minutes. Afterwards, the pulped material was rinsed profusely with water, passed thorough a Sprout-Waldrom single-disk refiner, and oven dried. In order to ease fiber individualization during composite preparation, corn stalk single fibers were dispersed in a water:diethyleneglycol dimethyl ether (diglyme) (1:3) mixture, filtered, and oven dried. The presence of diglyme in the previous step does not prevent but limits the formation of

hydrogen bonds between the cellulosic fibers. This eases the final fiber individualization, which was carried out in a mill for 5-10 seconds. Fibers length and diameter distributions were determined by means of a Kajaani FS300 instrument.

Composites preparation and characterization

Polypropylene (PP), corn stalks single fibers, and MAPP were blended at different wt/wt ratios using an intensive melt mixer, a Brabender® Plastograph. The mixing was carried out at 180ºC for 10 min at 80 rpm in order to ensure a well dispersed material. In the formulations containing MAPP this was added into the plastograph mixed with the PP pellets. The blends obtained were cut down to pellets, dried, and stored at 80ºC for at least 24 h before injection. Tensile and flexural test specimens were obtained according to the ASTM D638 standard protocols by means of an injection-molding machine (Meteor-40, Mateu&Solé). A schematic flowchart is presented in Fig. 1. Uncoupled and MAPP coupled composites with 40% wt reinforcement content were prepared and their tensile and flexural properties evaluated using an INSTRON testing machine (model 1122) following ASTM D638 and ASTM D790 standard methods. All the results were taken as the average of at least five samples.
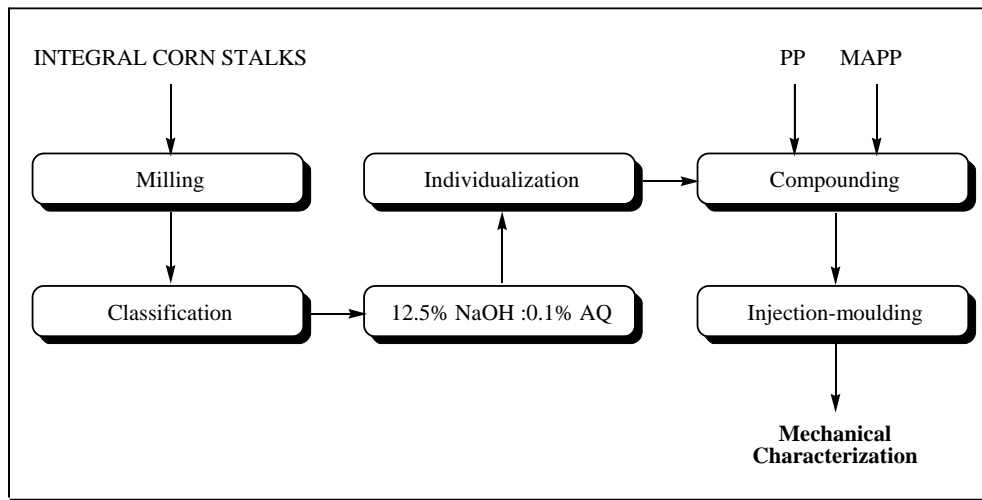
Fig. 1. Schematic flowchart of composites preparation and characterization

Determination of interfacial shear strength (IFSS) and fiber tensile load

Calculation of IFSS (τ) starts with the Kelly-Tyson equation. This model was designed for the prediction of the tensile strength of a material with a perfect fiber alignment.

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

In this equation σ_t^c and σ_t^F represent the tensile strength of the composite and the reinforcing fibers; $\sigma_t^{m,*}$ corresponds to the contribution of the matrix at failure; d^F and l^F represent the fiber diameter and length respectively, and V^F is the volume fraction of

reinforcement in the composite.

However, this is not a real case. With current standard processing techniques, perfect fiber alignment is almost impossible, and an orientation factor (χ_1) must be taken into consideration. The calculation of τ can be accomplished throughout a model proposed by Bowyer-Bader and derived from the modified Kelly-Tyson equation:

$$\sigma_t^c = \chi_1\left(\sum_i\left[\frac{\tau\, l_i^F V_i^F}{d^F}\right] + \sum_j\left[\sigma_t^F V_j^F\left(1 - \frac{\sigma_t^F d^F}{4\tau\, l_j^F}\right)\right]\right) + \left(1 - V^F\right)\sigma_t^{m,*} \tag{2}$$

Considering the low plastic deformation of cellulosic fibers, the tensile strength of the fiber can be represented as a function of its elongation (ε_t^f) and modulus (E_t^f) according to: $\sigma_t^F = E_t^f \cdot \varepsilon_t^f$. Moreover, within the first part of the stress-strain curve of the composite (especially at moderate to high crosshead speed), one can assume a low fiber slippage from the polymeric matrix and in consequence consider the elongation of the composite to be equal to that of the fiber ($\varepsilon_t^f = \varepsilon_t^c$). Thus the Kelly-Tyson expression can be reformulated as in (3).

$$\sigma_t^c = \chi_1\left(\sum_i\left[\frac{\tau\, l_i^F V_i^F}{d^F}\right] + \sum_j\left[E_t^F \varepsilon_t^c V_j^F\left(1 - \frac{E_t^F \varepsilon_t^c d^F}{4\tau\, l_j^F}\right)\right]\right) + \left(1 - V^F\right)\sigma_t^{m,*} \tag{3}$$

Although it looks complex, this equation can be simplified to $\sigma_t^c = \chi_1 (X + Y) + Z$, where X and Y represent the contribution of the reinforcing fibres and Z represents the contribution of the polymeric matrix. Note that the contribution of the fibers to the strength of the composite is split into two factors (X and Y). This is do to the fact that the reinforcing fibres are split between those larger and shorter than a length that is considered as critical (shorter fibres are considered to be inefficient). In the simplest model, the critical length is defined as:

$$l_c^F = \frac{d^F \sigma_t^F}{2\tau} \tag{4}$$

However, a more accurate approach suggested critical length is a function of the stress-strain curve of the composite. Thus, given any composite deformation (ε_t^c), the critical length can be calculated according to:

$$l_c^F(\varepsilon_t^c) = \frac{E_t^F \varepsilon_t^C d^F}{2\tau} \tag{5}$$

In order to solve the modified equation of Kelly-Tyson, it is necessary to know or estimate the values of E_t^F, E_t^m, and the characteristics of the reinforcing fibres: strength (σ_t^f), orientation (χ_1), IFSS (τ), diameter (d^F), and length (l^F). By previous extraction

from the polymeric matrix, the fiber distribution can be empirically obtained. However, usually σ_t^f, χ_1, and τ are unknown.

The Bowyer-Bader model suggests that these parameters can be calculated following the following expressions:

$$\text{R} = \frac{\sigma_t^{c1} - Z_1}{\sigma_t^{c2} - Z_2} \; ; \; \text{R}^* = \frac{\chi_1(X_1 + Y_1)}{\chi_1(X_2 + Y_2)} = \frac{X_1 + Y_1}{X_2 + Y_2} \tag{6}$$

From the equations above, the strength of the composite at two different deformations (σ_t^{c1}; σ_t^{c2}) can be calculated from the experimental stress-strain curve of the composite. Similarly, Z_1 and Z_2 refer to the strength of the polymeric matrix at the same deformations and are also quantifiable from the stress-strain curve of the neat matrix. The value of τ can be determined by iteration until $R = R^*$ (Bowyer and Bader 1972).

RESULTS AND DISCUSSION

Characterization of Corn Stalk Fibers

The raw material was composed of 74% corn stalks and 26% leaves. The integral corn stalks were used as raw material, including the "core", which accounted for 23% of the dry mass. Table 1 presents the main characteristics of fibers obtained from the integral corn stalks.

Table 1. Characteristics of the Corn Stalk Fibers Obtained

Yield (%)	Uncooked (%)	Kappa number	ºSR	Diameter (μm)	Length* (μm)	Fines** (%)
65.6%	4.6%	15	37	16.8	950	54.8%
* Length-weighted fiber length						
** % fines in length (fraction of fibers shorter than 200μm)						

Considering its yield, the corn fiber can be considered to be a semichemical pulp. The kappa number is in concordance with yield, since the lignin content is relatively high, and the pulp contains a low percentage of uncooked material. The Shopper–Riegler (ºSR) obtained is equivalent to 335 CSF (Canadian Standard Freeness) and can be considered high in comparison to that of chemical wood pulp. In fact, for an unrefined wood pulp like the one prepared one would expect a higher CSF (ca. 650 ml). The reason for the high ºSR (low CSF) of the pulp prepared has to be found in the high percentage of fines. In opposition, for highly refined wood fibers high ºSR are mostly related with the high swelling capacity of the fibers and their surface fibrillation.

The average fiber length of the corn stalk pulp is in the same order of magnitude to those obtained from hardwood. The high difference between the weighted (950 μm) and the arithmetic average length (470 μm) of the fibers is indicative of the high amount of fines in the pulp (>50%). The average diameter of the fibers indicates that mechanical defibrillation allowed obtaining a pulp with good fiber individualization. The high aspect ratio (length/diameter) of the fibers is a very interesting parameter for the application of

the corn pulp as reinforcement during the production of composite materials. The breaking length of a standard paper sheet made of corn stalks fibers (prepared according to Tappi T205 SP-95 and determined following ISO 1924-2:1994 standard protocols) was 4572m, which is comparable to that made of a typical unbleached eucalyptus pulp (4528m) and bigger than those obtained from recycled fluting (3161m).

Optimization of coupling agents

For most natural fiber-reinforced polypropylene composites it has been found that the optimum coupling agent content ranges between 3 to 5% of the weight of reinforcement, depending on the selected coupling agent. However, our previous experiences (Vilaseca et al. 2008, 2010) with MAPP E-3015 showed that the maximum mechanical properties of the composites were reached when the coupling agent was added in a 4 to 6% wt. For this reason, a preliminary optimization of the coupling agent content was performed.
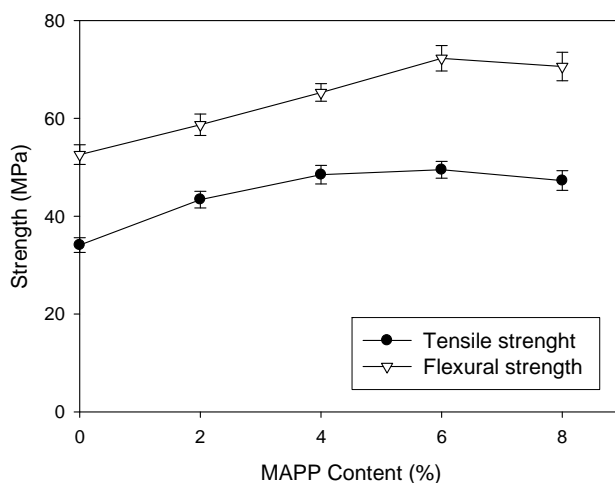

Fig. 2. Effect of MAPP content on tensile and flexural strength of 40% corn stalk fiber–reinforced PP composites

Composites with 40% corn stalk pulp with increasing amounts of MAPP E-3015 (from 2 to 8% wt.) were prepared and analyzed. Figure 2 shows the variation in the tensile and flexural strength of the composites at different MAPP content. Paying attention to tensile strength, one can appreciate that the maximum reinforcing effect was reached at 4 to 6% MAPP content, with no significant differences between these two values. As a consequence, from an industrial point of view, a 4% MAPP content could be considered the optimum. However, flexural data showed a clear strength enhancement when MAPP content is increased up to 6%. Although addition of MAPP offers a clear enhancement in the main mechanical properties of the composites even at 2%wt, we can conclude the optimum coupling agent content was 6%.

The effect of MAPP is attributed to the reactivity of its anhydride groups towards hydroxyl groups on the surface of the cellulosic fibers (Fig. 3), whilst its main polypropylene chain is mixed with the polymeric matrix, forming random entanglements. In order to further support this hypothesis, pulp fibres were treated with alkyl ketene

dimer (AKD), a common chemical in pulp and paper industry to provide paper with hydrophobic character. AKD is known to react and/or block the hydroxyl groups on the surface of the fibers, thus suppressing or limiting its reactivity (Mutje et al. 2006). When AKD-treated pulp was used as reinforcement, the mechanical properties of its composites remained unaffected by the presence of MAPP.
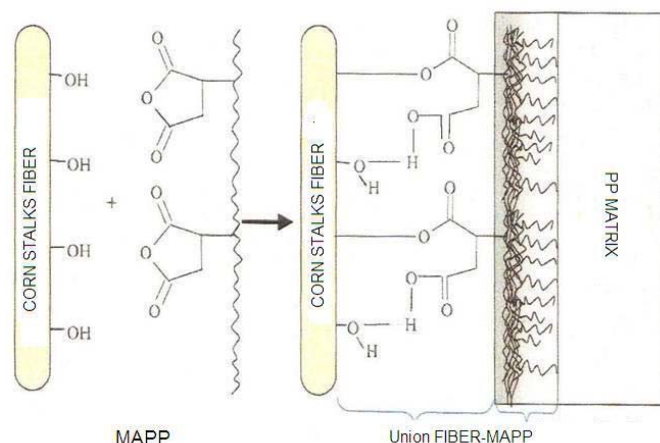

Fig. 3. Hypothetical interactions provided by MAPP at the interface of a cellulosic fiber – reinforced polypropylene composite

General trends in composites' mechanical properties

Following the general trends of natural fiber-reinforced composites, the mechanical strength of materials with no coupling agent showed small differences when compared with those of the plain matrix (Table 2). The tensile strength of the composite was 23% higher than that of PP, whilst its flexural strength increased 35%. In clear contrast, when MAPP was added to enhance the fiber-matrix interface, both tensile and flexural strength increased by almost 80%. Concerning the modulus, they both experienced a big increase (almost 300%) due to the higher rigidity of the material when it had reinforcing fibers in its formulation. However, the data provided evidence that the Young's modulus was independent on MAPP content, and in consequence, independent on the quality of the fiber-matrix interface. In addition to modulus increases, the higher rigidity of the material was further demonstrated by the reduction of the capacity of the material to sustain plastic deformation. However, it must be pointed out that MAPP did have a significant effect on the plasticity of the material, since the deformation at break of composites coupled with MAPP was higher that for uncoupled.

Table 2. Mechanical Properties of Corn Pulp-Reinforced Polypropylene

Material	Tensile strength (MPa)	Youngs' Modulus (GPa)	Elongation at break (%)	Flexural strength (MPa)	Flexural modulus (GPa)
Neat PP	27.6 ± 0.5	1.5 ± 0.1	9.3 ± 0.2	40.2 ± 1.0	1.1 ± 0.1
Composite without MAPP	34.1 ± 0.7	4.3 ± 0.2	2.1 ± 0.2	54.1 ± 1.6	3.2 ± 0.1
Composite with 6% MAPP	49.5 ± 0.9	4.3 ± 0.1	4.3 ± 0.4	72.3 ± 0.7	3.0 ± 0.1

Modeling of Mechanical Properties

From the evaluated mechanical properties one must suppose that corn stalk fibers are well dispersed in the polymeric matrix and they possess a big enough aspect ratio to efficiently reinforce PP. Furthermore, increases in composites' strength suggest that MAPP enhanced the fiber-matrix stress interface, whilst higher capacity to sustain deformation also suggests a better stress transfer. In order to determine whether or not these observations were correct, the mechanical properties of the composites prepared were evaluated following common theoretical models.

For short fiber composites it is accepted that tensile strength can be modeled according to the modified rule of mixtures,

$$\sigma_t^C = f_c \sigma_t^f V^F + \left(1 - V^F\right) \sigma_t^{m,*} \tag{7}$$

where σ_t^c, σ_t^f, and $\sigma_t^{m,*}$ represent tensile strength of the composite, the reinforcing fiber, and the matrix respectively. The parameter V^F is the volume fraction of the reinforcement, and fc is the efficiency factor. This efficiency factor represents the effectiveness of the reinforcement and can be decomposed into two factors ($fc = \chi_1 \chi_2$), where χ_1 is evaluates the fiber alignment (orientation factor) and χ_2 is evaluates the fiber-matrix interface and the reinforcing fiber aspect-ratio.

In contrast to single strands from natural resources (hemp, abaca, jute, etc.) the tensile strength of corn stalk fibers (σ_t^f) is almost impossible to be empirically measured and one must appeal to analytical methods to accurately estimate its value. As a consequence, the coupling efficiency factor fc cannot be determined directly.

The easiest way to determine the value of σ_t^f is to solve the equation proposed by Kelly-Tyson for the composite, following the methodology of Bowyer-Bader, as previously described. This procedure allows the determination of χ_1 and τ (interface shear strength = IFSS), but it requires the knowledge of E_t^f and $\sigma_t^{m,*}$. The contribution of the matrix to the composites strength ($\sigma_t^{m,*}$) can be obtained from the stress-strain curve of neat PP (Fig. 4), but the Young's modulus of the reinforcing fiber cannot be empirically determined, and the application of the Hirsch model (Equation 8), another approach based on the combination of parallel and series models of the rule of mixtures, is required:

$$E^C = \beta(E^F V^F + E^m(1 + V^F)) + (1 - \beta)\frac{E^F E^m}{E^m V^F + E^F(1 - V^F)} \tag{8}$$

In this case, β is a parameter that determines the stress transfer between the fiber and matrix. It is assumed that β is determined mainly by fiber orientation, fiber length, and stress amplification effect at the fiber ends (Li et al. 2000). It has been reported that theoretical and experimental values can fit when the value of β is 0.4 (Kalaprasad et al. 1997). Subsequently, the theoretical intrinsic Young's modulus for the fibers from the corn stalk pulp would be 20.5 GPa.
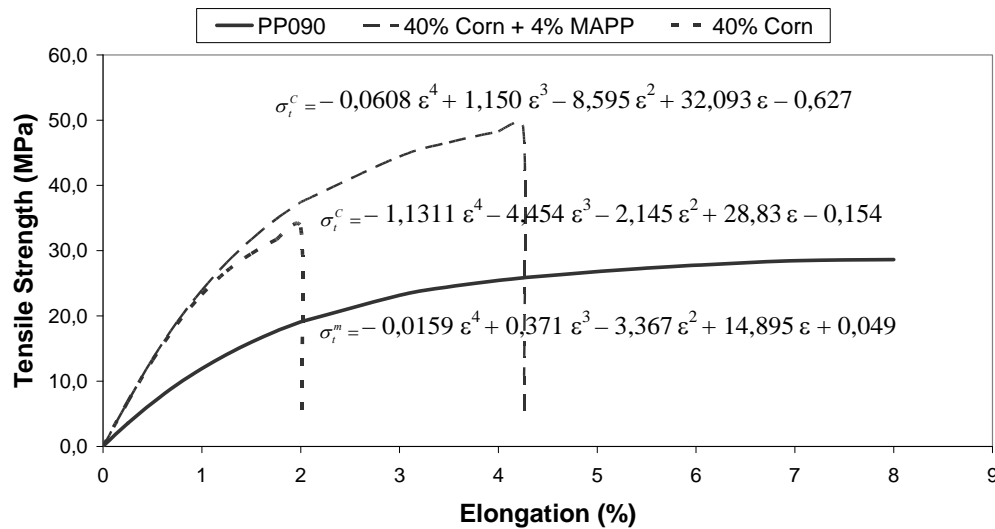
Fig. 4. Experimental curves and mathematical fitting of tensile strength of PP and corn stalk – reinforced PP with and without MAPP coupling agent content
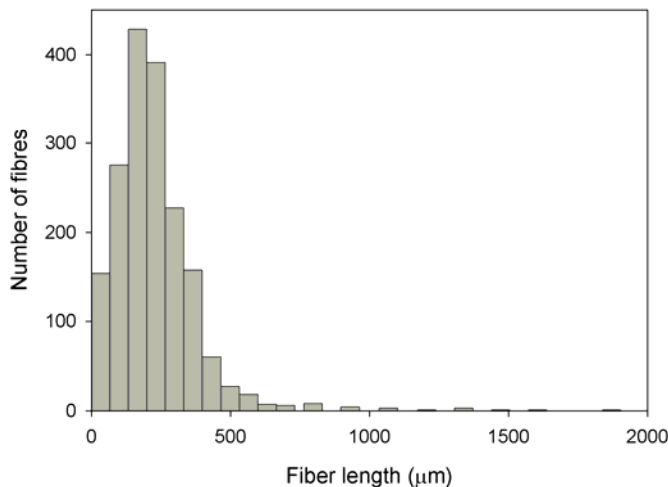

Fig. 5. Length distribution of corn stalk fibers inside a composite with 0%MAPP

Knowing the fiber length distribution (Fig. 5) and the intrinsic Young's modulus of the reinforcing fiber, the methodology of Bowyer-Bader can be applied, and the orientation factor (χ_1) and IFSS (τ) calculated. Experimental data values required to apply the Bowyer-Bader methodology are summarized in Table 3.

The IFSS obtained for composites coupled with 6% MAPP ($\tau = 15.5$MPa) is in good agreement with the value predicted according to Von Misses criterion ($\tau = \sigma_t^m / \sqrt{3}$) (Pegoretti et al. 1996). Meanwhile, if attention is centered on the orientation factor, one can observe that this was bigger for uncoupled composites. The differences observed with respect to fiber orientation are attributed to the higher viscosity of materials with MAPP in their formulation, which would reduce the mobility of the fibers and thus limit its orientation.

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Table 3. Composite Properties, Stress-Strain Input Data and Parameters Obtained from Equations 6-8.

Coupling agent content	6%	0%
Reinforcement weight content	40%	40%
Reinforcement volume fraction	29%	29%
Average length (μm)	226.8	222.3
Weighted average length (μm)	332.9	341.6
Average diameter (μm)	16.1	16.1
Weighted average diameter (μm)	17.1	17.1
Composite strength (MPa)	49.5	34.1
Composite modulus (GPa)	4.3	4.3
Fiber modulus (GPa)	20.5	20.5
Elongation at break (%)	4.35	2.1
Strain levels analyzed (%)	0.834 and 0.417	1.4 and 0.7
Composite stress at strain level 1 (MPa)	20.8	28.35
Composite stress at strain level 2 (MPa)	11.35	17.95
Matrix stress at strain level 1 (MPa)	10.4	15.25
Matrix stress at strain level 2 (MPa)	5.7	8.95
Parameters obtained from equations 6-8		
Orientation factor – χ_l^{BB}	0.318	0.358
Interface shear strength – τ^{BB} *(MPa)*	15.5	7.9
Fibers' tensile strength at maximum stress – σ_t^f *(GPa)*	670	460

Finally, χ_l and τ can be applied in the Kelly-Tyson equation to determine the tensile strength of the reinforcing fiber at maximum stress (σ_t^f) (Table 3). It must be remembered that the fibers' tensile strength at maximum composite stress (σ_t^f) does not represent the ultimate tensile strength of the reinforcing fiber unless perfect fiber-matrix bonding is achieved. Considering this, it is not surprising that the maximum strength at break (σ_t^f) of corn stalk fibers is higher for composites containing MAPP coupling agent than for uncoupled materials. Such behavior is attributed to an enhanced stress–transfer from the matrix to the reinforcing fibers, and that is supported by the values obtained for IFSS.

The methodology here applied also enables the determination of a minimum value for the intrinsic flexural strength of the reinforcing fibers (σ_f^f). Hashemi suggested that one could assume the flexural strength of fibers (σ_f^F) to be the same as in tension (σ_t^F) (Hashemi et al. 2008). However, as he reminded, this assumption might not necessarily be correct. Thus, given the difficulty to establish the correct value of σ_f^F, researchers can calculate what has been denominated as tensile (TSF) and flexural strength factors (FSF):

$$TSF = \left(\frac{\sigma_t^C - \left(1 - V^F\right)\sigma_t^{m*}}{V^F} \right); \; FSF = \left(\frac{\sigma_f^C - \left(1 - V^F\right)\sigma_t^{m*}}{V^F} \right) \qquad (9)$$

Assuming that the efficiency factors are independent of stress direction and as a consequence f_c and f_a^F are in the same order of magnitude, the difference between TSF and FSF would be proportional to the intrinsic properties of the reinforcing fiber.

Table 4. Tensile and Flexural Strength Factors and Ultimate Strength of the Corn Stalk Fibers Reinforcing Polypropylene Composites

MAPP content (%)	TSF	FSF	FSF/TSF	σ_t^f	σ_f^f
0	69.4	97.2	1.46	460	645
6	106.9	160.0	1.58	670	1005

For the case analyzed, this would imply that the relationship between the intrinsic tensile and flexural strength (σ_f^F / σ_t^F) of the corn stalk fibers is around 1.5 (average FSF/TSF). This difference can be attributed to the fact that whilst in tension the entire specimen is under tensile stress, in flexure only a fraction of the fiber is subjected to tensile stress. Considering the FSF/TSF ratio, the strength sustained by the reinforcing fibers at maximum flexural strain has been evaluated (Table 4). Again, the maximum strength sustained by corn stalk fibers increased when a coupling agent (MAPP) was added to the formulation.

CONCLUSIONS

1. The analysis of the mechanical properties of fiber-reinforced polypropylene composites through the Bowyer-Bader methodology has allowed the determination of the tensile load on reinforcing fibers at composite failure. The load bearing the reinforcement can be considered as a minimum value for the reinforcement tensile strength.
2. The protocol followed in this work might be useful for the determination of the intrinsic mechanical strength of reinforcement in a hypothetical composite with perfect fiber-matrix stress transfer.
3. This methodology allows a deeper knowledge of phenomena occurring at the interface of fiber-reinforced composites, which is a fundamental aspect for an accurate prediction of the mechanical properties of composite materials.

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