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Mechanical Properties of PP/Jute and Glass Fibers Composites: The Statistical Investigation

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Abstract: A systematic and statistical approach to evaluate and predict the properties of random discontinuous natural fiber reinforced composites was studied. Different composites based on polypropylene and reinforced with natural fibers (flax and glass) have been made and their mechanical properties are measured together with the distribution of the fiber size and the fiber diameter. The values obtained were related to the theoretical predictions, using a combination of the Griffith theory for the effective properties of the natural fibers and the Halpin-Tsai equation for the elastic modulus of the composites. The relationships between experimental results and theoretical predictions are statistically analyzed using a probability density function estimation approach based on neural networks. The results show a more accurate expected value with respect to the traditional statistical function estimation approach.

Key words: Composites, micro-mechanic, mechanical properties, natural fiber, statistical

INTRODUCTION

Staple conventional fibers (glass, aramid, carbon, etc.) have been extensively used over the last decades as reinforcements of thermoplastic polymeric matrices. They are incorporated into plastics with the main objective of improving the mechanical properties of the polymer reducing the cost of the final products (Bolton, 1994; Sanadi *et al*., 1994; Chen and Porter, 1994) with respect to long fiber composites. The growing interest in using natural vegetable fibers as a reinforcement of polymeric based composites is mainly due to their renewable origin, relative high specific strength and modulus (Biagiotti *et al*., 2004), light weight and low price. Recent developments in natural fibers (Hornsby *et al*., 1997; Semsarzadeh, 2004; Roe and Ansell, 1985) such as jute, sisal, coir, flax, banana, etc., have shown that it is possible to obtain (Mukherjee *et al*., 1983; Gassan and Bledzki, 1997) well performing materials, using environment friendly reinforcements (Gassan and Bledzki, 1999). The mechanical properties of natural fiber-reinforced composites can, in fact, be further improved by chemically promoting a good adhesion between the matrix and the fiber (Stamboulis *et al*., 2001; Bos *et al*., 2002). Other advantages of utilizing natural fibers are related to their cycle of production that is economical and their ease of processing which demands minor requirements in equipment and safer handling and working conditions with respect to glass fibers. In any case, the most interesting feature coming from the employment of natural fibers is the extremely favorable environmental impact, due to the fact that natural fibers are produced from a renewable source and are biodegradable (Baley, 2002). Furthermore, natural fiber composites can be easily recycled or burned allowing clean energy recovery and avoiding damping at the end of their life cycle. Therefore, lingo-cellulosic natural fibers represent an interesting alternative as substitutes for traditional synthetic fibers (Morton and Hearle, 1993) in composite materials. On the other hand, low thermal stability, high moisture uptake and limited fiber lengths, represent some of the disadvantage related to the utilization of natural fibers composites.

Another noticeable drawback of these fibers lays in their intrinsic variability. The behavior and the properties of natural fibers depend, in fact, on many factors such as harvest period, weather variability and quality of soil and climate of the specific geographic location (Barkakaty, 1976) as well as preconditioning (Ray *et al*., 1976). The variability of the properties of natural fibers is so high, that it can also be observed in fibers belonging to the same plant (Lewin and Pearce, 1985; Zadorecki and Lodin, 1986). Moreover, it has been shown that fibers coming from the plant stems have different properties with respect to those taken from the leaves (Bisanda and Ansell, 1992).

In contrast with synthetic fibers, whose properties can be easily and univocally determined, natural fibers are characterized by a large dispersion of their characteristics. Such features make it necessary to utilize a more systematic statistical approach to define their properties and those of their relative composites.

In order to describe the characteristics of natural fiber reinforced composites in the present study, the use of statistical representations based on probability density functions of the quantities of interest is proposed. It is possible to relate the mechanical performances of the composites to the properties of their constituents with this method. In particular, the modulus of the composites studied can be analytically correlated to the geometrical and mechanical characteristics of the fibers and to the mechanical properties of the polypropylene matrix using the Halpin-Tsai equations (Mallick, 1993; Halpin and Kardos, 1976). On the other hand, the variation of the elastic modulus and the tensile strength of untreated natural fibers with the diameter size can be predicted using the model proposed by Griffith (Griffith, 1921). Then, the statistical approach presented here utilizes the distribution of the geometric properties of the fibers measured over a post-processed composite to obtain a statistical distribution of the mechanical response of the composite through the non-linear equations arising from the combination of the two models previously reported. The true distribution functions are explicitly estimated by the help of semi-parametric algorithms, drawn from the neural network literature. These functional estimates allow easy visualization of results and make it possible to perform a more accurate interpolation from missing data.

Estimations of the probability density functions: The Probability Density Function (PDF) of a random variable describes the distribution of its possible values (or determinations) within its range in terms of the expected number of times that a single value will fall in a very small numerical interval when observing the variable. PDFs are frequently used in physics and chemistry to describe complex phenomena that cannot be characterized in a deterministic way (e.g., Johnson and Levy, 1974; Zucker and Shulz, 1982; Coppens, 1993; Kuhs, 1992). A typical problem in the PDF theory is the approximation of the probability density function describing a physical phenomenon by sample analysis from incomplete data, which may be regarded as a constrained functional approximation problem (Cover and Thomas, 1991). It concerns the estimation of the PDF of a signal when some particular features of the true PDF are observed (measured) or signal samples are obtained through measures. Several techniques are available in scientific literature to solve this problem and they may be classified mainly in parametric, non-parametric and semi-parametric techniques. The parametric techniques assume a specific functional form for the density model that contains a number of parameters; such parameters are optimized by fitting the model to the available data. The main drawback of this approach is that the chosen parametric function might not be suitable to provide a good representation of the density. In the non-parametric techniques, the functional form of the probabilistic model is not specified in advance, but is only dependant on the data. The main disadvantage of non-parametric approaches is that the complexity of the model grows with the number of available observations, which cannot be kept too small because the estimation ability would degrade. In order to combine the advantages of parametric and non-parametric methods, semi-parametric ones have been developed, which are not restricted to specific functional forms and the size of the model only grows with the complexity of data-space structure, not simply with the amount of available data. Classical semi-parametric models for PDFs are given by artificial neural networks (Bishop, 1995), which possess the necessary model flexibility and learning ability to match the available data. In the present work we utilize a neural algorithm for PDF estimation from incomplete data based on unsupervised information-theoretic neural structures, known as adaptive-activation function neurons (Fiori, 2001) (FAN), which proved useful in asymmetric probability density function approximation in the presence of little data.

The Griffith and Halpin-Tsai models: The mechanical properties of fibers as a function of their diameter were studied on metal and alloy wires by Karmasch (1934) in the early 1800s and revised by Griffith (1921). These studies brought the following empirical expression:

$$E_f(d_f) = A + \frac{B}{d_f} \tag{1}$$

Where:
$E_f(d_f)$ = Analyzed property
A and B = Constants
d_f = Fiber diameter

Although, Griffith theory has been mainly applied to the fiber tensile strength following the simple observation that thinner fibers contain less strength reducing flaws, the experimental observations indicate that also the elastic modulus has a similar dependence on the fiber diameter.

Tsai and Pagano (Gibson, 1994) related the value of the modulus of randomly oriented discontinuous fiber composites to the corresponding oriented module, according to the following equation which arises as the result of an averaging process:

$$E_{ramdom} = \frac{3}{8}E_{11} + \frac{5}{8}E_{22} \tag{2}$$

Where, E_{11} and E_{22} are the longitudinal and transverse modulus of a unidirectional discontinuous fiber composite having the same volume fraction of fibers. Indeed, Eq. 2 applies to randomly in plane oriented short fiber composites. However, it is very well known that high shear rate flow conditions induces fiber orientation in injection molded show fiber composites with eventual presence of out of plane fibers. In our approach we have neglected fiber orientation for simplicity and following experimental observation discussed in the following section.

The values of the composite modulus E_{11} and E_{22} can be derived by using the Halpin-Tsai model as follows:

$$E_{11} = \frac{1 + 2(l_f / d_f)\eta_L V_f}{1 - \eta_L V_f} E_m \quad (3)$$

$$E_{22} = \frac{1 + 2\eta_L V_f}{1 - \eta_L V_f} E_m \quad (4)$$

In these equations, the parameters η_L and η_T are given by:

$$\eta_L = \frac{(E_f(d_f)/E_m) - 1}{(E_f(d_f)/E_m) + 2(l_f / d_f)} \quad (5)$$

$$\eta_T = \frac{(E_f(d_f)/E_m) - 1}{(E_f(d_f)/E_m) + 2} \quad (6)$$

Where:

E_m = Elastic modulus of the PP matrix
$E_f(d_f)$ = Elastic modulus of the fiber as a function of the fiber diameter
l_f and d_f = Length and diameter of the fiber, respectively
V_f = Fiber volume fraction

Glass and carbon fibers used in traditional composites are characterized by a very narrow distribution of fiber diameter leading to a very low uncertainness in the values of the elastic modulus of the fiber used in the model equations. However, natural fibers have a very broad distribution of the diameter and the mechanical properties of the fibers are widely distributed as a function of their dimensions. Then, we propose in this approach to introduce the variation of the modulus with the diameter of the fiber, expressed using the Griffith model, in the above equations. In this study, therefore, the values of the module of the composites are not directly calculated using a single geometrical parameter in the Halpin-Tsai equations, but the modal values of statistical distribution are considered, which allow one to take into account the different possible combinations of fiber aspect ratios and module.

MATERIALS AND METHODS

Table 1 shows the characteristics of the raw materials utilized in this study. A commercially available grade of isotactic polypropylene (iPP) (MFI: 2.9 dg min^{-1} at 190°C and 5 kg), kindly supplied by Solvay, under the trade name of Eltex-P HV-200, was used in this work. Natural flax fibers provided by Finflax were used as a reinforcing agent. The common flax plant, Linum Usitatissimum is a member of the Linaceae family, which is widely distributed in Europe and other areas of the world. In this study, the variety Belinka, which was cultivated in 1995 in Tyrnävä (Oulu, Finland) were used. Fibers are extracted from the plant by biotechnical retting and dried at 55°C. The length of a technical fiber is in the range of 30-90 cm, but the fibers are previously cut to an average length of 1 cm, before processing. A technical E-glass fiber, kindly supplied by Vetrotex, whose properties are also shown in Table 1, was adopted for a comparative estimation in our study.

Fiber characterization: The mechanical properties of the fibers were measured using the single-filament tensile test carried out at room temperature on a Lloyd dynamometer mod. LR 30K, according to ASTM D3379-75. The measurements were performed over fifty fiber samples having gage length of approximately 30 mm at a crosshead speed of 1 mm min^{-1}. The data obtained on the mechanical properties of the fiber were 6 represented by a two-parameter Weibull equation (Weibull, 1951), which expresses the cumulative density function of the elastic modulus of the fibers as:

$$F(E_f) = 1 - \exp\left[-\left(\frac{E_f}{E_o}\right)^{\alpha}\right] \quad (7)$$

Where:

α = Dimensionless shape parameter
E_0 = Location parameter

Composite preparation: The compounds were prepared by means of hot-rolls, at a temperature of 180°C, which is above the melting point of the thermoplastic matrix, for 30 min. The natural fibers were previously cut to their initial length, about 10 mm in size and dried in an oven at 70°C for 12 h. Once the polymer was melted, the appropriate percentage of fibers was added to the

Table 1: Main characteristics of the materials utilized

Materials	PP	Flax fiber	Glass fiber
Manufactured	SOLVAY	FINFLAX	VETROTEX
Designation	Eltex-P HV-200	Retted flax fiber	P 368
Density (g cm^{-3})	0.9	1.5	2.5
Initial length (mm)	-	10	7
Diameter (μm)	-	36-450	13

polymer. The same weight fraction of flax and glass fibers was used for simplicity, taking in consideration that the objective of this study is not the comparison between natural fiber and glass fiber composites but the development of advanced statistical tools to describe the mechanical properties of these materials. Immediately after mixing, the material was extruded into pellets and then injection molded in a Sandretto Micro30 injection-molding machine to obtain standardized dog-bone specimens. To avoid thermal degradation of the fibers, the temperatures in the three main zones of the equipment were carefully selected at 182, 184 and 186°C, respectively.

A mold temperature of 25°C and a specific injection pressure of 1700 bar were used. The time-intervals for the packing and cooling stages were 30 and 25 sec, respectively.

Composite characterization: Tensile tests were performed at room temperature on a Lloyd mod. LR 30K, according to ASTM D638M standards. The tests were carried out at a cross-head speed of 5 mm min^{-1} and the dimensions of test samples were 150×10×4 mm.

In order to estimate the probability distribution function curves of the geometrical characteristics of fibers in the composites, samples were melted in a Mettler FP-82 HT automatic hot-stage thermal control to improve the visibility of the fibers included. Samples were sandwiched between microscope cover glasses, melted and maintained at 200°C for the test duration, the lengths and diameters of the fibers were measured using a Hund Weztlar H600 optical polarizing microscope.

RESULTS AND DISCUSSION

Figure 1 shows the characteristic stress-strain curves obtained on the studied fibrous reinforcements. As expected, the glass fibers presented higher values of elastic modulus and tensile strength compared to flax fibers. The mechanical characteristics of the reinforcements, in terms of elastic modulus and tensile strength, were measured and registered to be used in the statistic analysis. In this sense, Fig. 2 shows the ability of the Weibull model to describe the mechanical behavior of both fibrous systems. The Weibull parameters α and E_0 were computed from the fitted curves and reported in Table 2. Again, the flax fibers manifest a lower performance both in terms of mechanical properties and scattering of data. The calculated location parameter, E_0, which represents an average value of the measured property, is lower for flax fibers. Furthermore the second Weibull parameter, the shape factor, α, which is related to the dispersion of data, confirmed a wider scattering for flax fibers. As a consequence, the glass fibers exhibit a more reliable behavior with elevated mechanical performance.

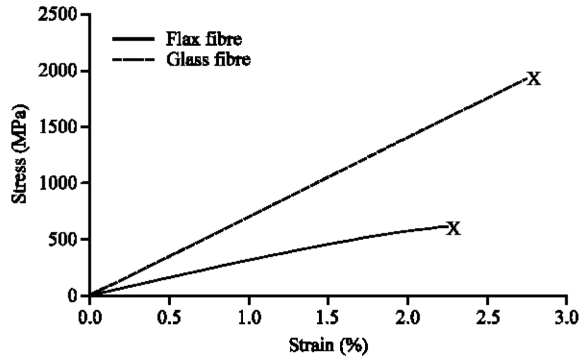

Fig. 1: Typical tensile curves for flax and glass fibers used in this research

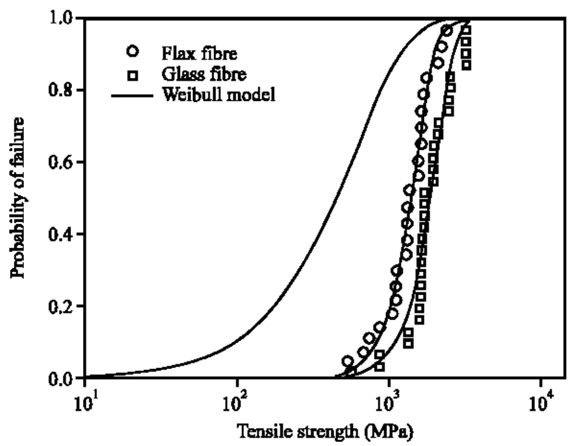

Fig. 2: Weibull distributions for Young's modulus of fibers used in this research

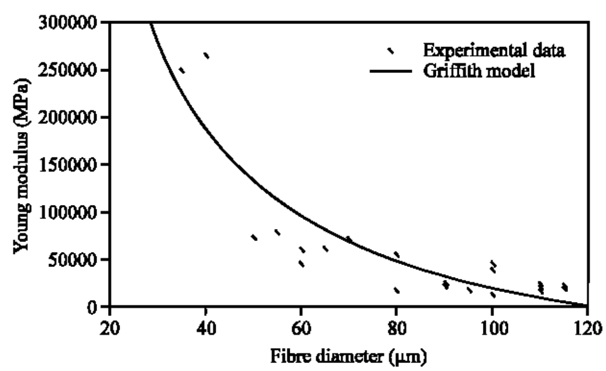

Fig. 3: Young's modulus versus diameter plot for the flax fibers used in this research

Figure 3 shows the Young's modulus as a function of the diameter size for flax fibers. It is possible to observe that the modulus value is strongly dependent on diameter size and furthermore, a wide range of diameters is present

Table 2: Mechanical parameters of the fibers used in this research

	Young's modulus				Tensile strength			
	Weibull model parameters		Griffith model parameters		Weibull model parameters		Griffith model parameters	
Fiber	α	E_0 (MPa)	A (MPa)	B (MPa mm)	α	E_0 (MPa)	A (MPa)	B (MPa mm)
Flax	1.59	48798	3023	2674	1.22	601	110	166.6
Glass	3.46	72706	-	-	3.52	2093	-	-

in the same bunch of fibers; such a variation is a typical drawback of natural fibers. The real stresses on the flax fibers were calculated 11 measuring the equivalent diameter from optical microscopy after each tensile test. The variation of the modulus of the flax fiber with the diameter size shown in Fig. 3 was therefore modeled using the Griffith model which captures the inverse proportionality between the Young modulus and fiber diameter clearly exhibited by the measured data. On the other hand, with synthetic fibers, where the steep distribution of diameters allows the utilization of an average value without introducing substantial errors, there was no need to use the Griffith equation. In our study, only the Weibull location parameter was adopted to characterize the mechanical performance of glass fibers.

Concerning the application of the Halpin-Tsai equations to the natural fiber composite, a more accurate modeling of the composite mechanical properties can be obtained by using the measured values of the geometrical dimensions and the predicted values of the respective module. Such operation can be completed with the help of statistical distribution of the properties of the composites, which, by taking into account the above expressed property variations, is able to embody the rich information content of the measured data.

To accomplish this task we started with the estimation of the distribution of the geometrical characteristics (the length l_f and the diameter d_f) based on unsupervised information-theoretic neural structures, known as adaptive-activation function neurons. These structures allow one to estimate the probability density function of every uni-variate random variable from a reduced set of available measurements through an information-theoretic criterion. As opposed to classical methods, such as the one relying on histogram computation, this approach produces a smooth function that avoids granularity effects and that can also be used to predict the values of the distribution, even in those range of values where no data was observed. The effects of mastication during processing on the fiber dimensions for both flax and glass fibers were monitored by means of a series of measurements of the geometrical characteristics of post processed composites, which were used to build up their PDFs curves.

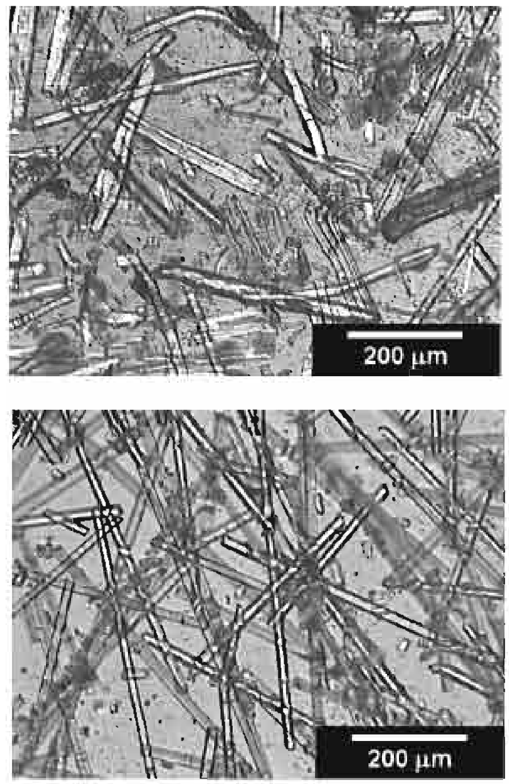

Fig. 4: Images of flax (a) and glass (b) melted composites for determining fiber dimensions

Figure 4a and b show typical micrographs utilized for determining final fiber dimensions. A more irregular shape of natural fillers with respect to the glass fibers is detected, which confirms what was previously stated regarding the calculation of the diameters of the reinforcements used. While only differences in length are observed in glass fibers composites, a large variation of sizes in terms of both length and equivalent diameter characterize flax fibers. Furthermore Fig. 4a and b, evidence a predominant random in plane orientation supporting the use of Eq. 2 as a first approximation of the composite properties.

The length and diameter distribution curves for the studied systems are shown in Fig. 5a and b. The use of

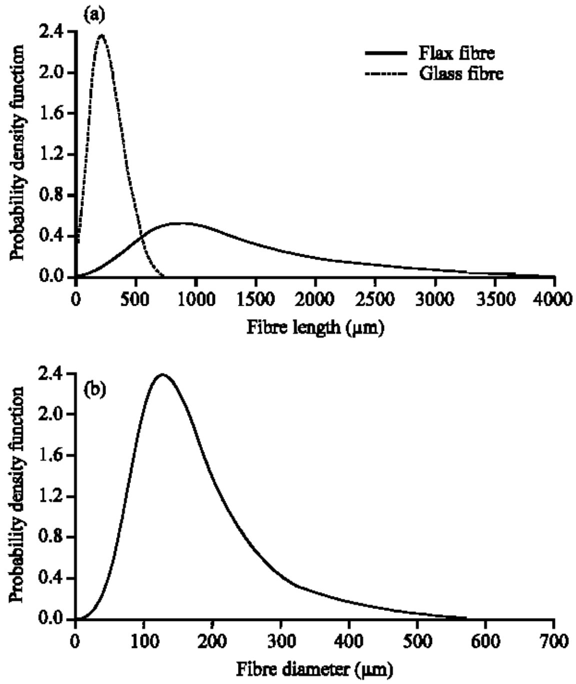

Fig. 5: Probability density function (PDF) curves of geometrical characteristics of fibers: lengths (a) and diameters (Flax) (b)

Table 3: Geometrical characteristics of post-processed flax and glass fibers

Fiber	Modal length (μm)	Modal diameter (μm)
Flax	860	127
Glass	209	13

Table 4: Theoretical and experimental mechanical properties of flax and glass fiber composites

	Young's modulus		Tensile strength
Composite material	Theoretical (MPa)	Experimental (MPa)	Experimental (MPa)
PP	-	1049±72	30.0±0.9
PP+20% wt. flax fiber	1851	1502±102	17.9±1.4
PP+20% wt. glass fiber	2727	2347±75	52.4±1.4

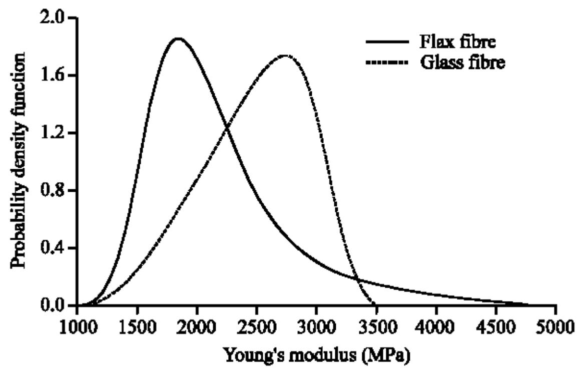

Fig. 6: Theoretical PDF curves of Young's modulus for natural and glass fibers composites

the aforesaid PDFs estimation technique produces asymmetrical curved shapes for both kinds of composites; the curves show that the most probable values (i.e., the curve peak) of the distribution are shifted towards the lower values. Furthermore, it is possible to notice that the natural fibers present a larger distribution of geometrical dimensions which is due to their characteristic natural dispersion. The modal values obtained using the curves of Fig. 5 for the lengths and for the diameters of the studied fibers are shown in Table 3. It is important to point out that the measured values of the diameters of natural fibers present a lot of uncertainty because the displayed fiber diameters consist of a bundle of elementary fibers, which of course may separate during testing. This fact makes a precise characterization of the aspect ratio for natural fibers more difficult, justifying the use of a more accurate distribution function.

Table 4 shows the matrix properties experimentally determined which were used in the Halpin-Tsai equation for the determination of the composite moduli. The fiber-dependent properties in the Halpin-Tsai equation were calculated in the following way: the modulus E_f, as a function of the measured diameter and described well with the use of the Griffith model (Fig. 3) and the aspect ratio were determined for each observed fiber in the composite. Each measured length and diameter produced a single value of the modulus of a hypothetical composite with a fiber volume fraction of $V_f = 0.13$ for natural fibers and $V_f = 0.08$ for glass fibers and having all the fibers the same measured geometrical dimensions. Each value of the composite elastic modulus, obtained by applying the Halpin-Tsai model to every available pair (d_f, l_f), was considered as an observed realization of the elastic modulus variable, thought of as a random variable. With the help of the mentioned PDF estimation technique, the statistical distribution of the elastic modulus values was then constructed.

Figure 6 shows the PDF curves obtained in this way for the Young's modulus of the studied composites. The most important feature of the PDF curves shown is their asymmetrical shape that is more representative of the complex distributions of fiber properties. Representing and predicting the behavior of short natural fiber reinforced composites with such curves gives a more appropriate estimation of their mechanical features because they embody the main fiber class characteristics. Furthermore, such a model better represents the variability introduced by the mastication during processing.

However, it is possible to notice that, in all cases, the theoretical modal values of the mechanical properties

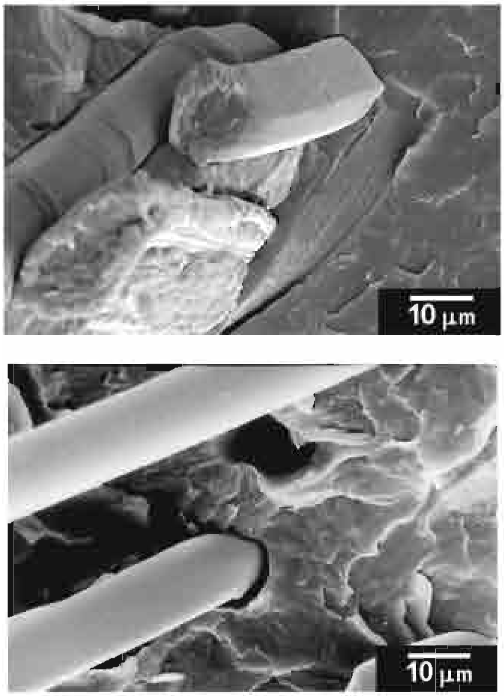

Fig. 7: Fracture surface of composites based on PP reinforced with flax (a) and glass (b) fibers

described are slightly greater in comparison to the measured experimental values. These differences can be probably explained in terms of the assumptions introduced in the approach. In particular, a deeper consideration of the following questions should improve the accuracy of the approach applied here: the accurate determination of the η_T parameter in the Halpin-Tsai equations, the possibility of utilizing the term ξ of the explicit Halpin-Tsai equation as a fitting parameter, the inclusion of in plane and out of plane fiber orientation. Furthermore, the deviation of the real composite features from the basic assumptions of the theoretical approach, where a perfect adhesion at the fiber/matrix interface and an absence of voids are required (Holister and Thomas, 1966), can also contribute to the slight disagreement between experimental data and the model. Then, the lower experimental values highlight the poor adhesion between the polypropylene and the pair of fibers utilized in this work, also confirmed by the SEM micrographs illustrated in Fig. 7a and b.

A preliminary application of the model to the prediction of the tensile strength has produced inconsistent results probably because the high anisotropy of natural fibers, which present strong differences between the longitudinal and the transverse modulus. Furthermore the length of the flax fibers was always shorter than the critical length, limiting in this way the reinforcing effect of the fibers in the matrices. The different effect of the two kinds of reinforcement on the tensile strength of the composite shown in Table 4 confirms what was stated earlier.

CONCLUSIONS

A systematic statistical approach to evaluate and predict the properties of random discontinuous natural fiber reinforced composites was developed. The proposed model was applied to different composites based on polypropylene matrix reinforced with natural fibers and short glass fibers. The validity of the proposed statistical approach was verified experimentally. It was observed that the theoretical elastic modulus predicted was close to the experimental value. The relatively small differences between the expected values of the module were attributable to imperfections, in terms of fiber/matrix adhesion and voids, in the analyzed composites. This kind of modeling allows a better characterization of natural fiber composites which mechanical properties are strongly affected by the broad distribution of fiber dimensions. With the proposed method, in fact, one of the typical problems of the natural system, like the dispersion of properties, can be approached utilizing a more accurate semi empirical methodology, which can be useful in the design and the optimization of the processing of natural fiber reinforced composites. Further investigations are in progress in order to extend the proposed approach with other varieties of natural fibers.

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