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Prediction of tensile properties of injection moulding flax fibre reinforced polypropylene from morphology analysis

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Abstract. The Young modulus and tensile strength of flax fibre reinforced polypropylene were determined and compared with the micromechanical models usually used in the case of short glass fibre reinforced composites. The fibre length and fibre diameter distributions of the injected reinforced of 2, 4, 8 and 22vol% compound were determined and used to the models in order to evaluate the expected properties of the composites. The mechanical properties were interpreted on the base of real fibre content, fibre orientation, fibre length and diameter distributions and morphology of the composites. The Kelly-Tyson's model of the tensile strength prediction has been modified to take in consideration the fibre property variability due to the large distribution of fibre shape ratio induced by the process. Finally matrix modulus has been adjusted to take into account the change of crystallinity with fibre content.

Introduction

Nowadays, natural fibre composites have emerged as a realistic alternative to glass-reinforced composites in many applications [1,2]. Vegetal fibres are low cost fibres with low density and specific properties comparable to those of other fibres used for reinforcements, biodegradable and non-abrasive unlike other reinforcing fibres. Among vegetable fibres, flax is probably the oldest one used in many textile applications and consequently the most cultivated. In recent years, the rigorous control of plant culture, fibre extraction and roving manufacture make a technical fibre capable of a wide use in the technical composite applications [3,4].

Because of the heterogeneous microstructure depending on the age of the plant, extraction and preparation methods, chemical composition, its thermal degradation dependence and the hydrophilic nature of the fibres, the injection moulding process/microstructure/mechanical properties relationships are quite different when discontinuous flax fibre reinforced thermoplastics are used [5]. Thus, during the first stage of pellet preparation by extrusion compounding, a considerable fibre breakage occurs giving on one hand both reduction of length and aspect ratio and on a second hand flax bundle delaminations leading to the appearance of elementary flax fibres of 10 to 20µm diameter [6,7]. This elementary fibre extraction is accentuated during injection moulding process. This fibre reduction causes a high decrease of composite stiffness and strength [4,5].

The macrostructure induced by the melt flow during the processing reveals also fibre orientations. The fibre arrangement through the thickness shows a multilayer structure more or less similar to this of short glass fibre reinforced composites. However the transition between the layers is more complicated [8]. That's why natural fibres do not behave like glass fibres during injection moulding due to high fibre/fibre and fibre/matrix interactions. It is of prime importance to carefully investigate the relationship between moulding conditions, processing-induced microstructure and mechanical properties of natural fibre-reinforced composites. The fibre/matrix adhesion plays an

important role on mechanical properties because of the transmission of composite loads to the fibres, as well as in its ageing behaviour. Unfortunately, in most of reinforced natural fibre thermoplastic composites, due to the weak compatibility with the cellulose, the fibre/matrix interfaces show bad adhesion in particular when polypropylene is considered. The addition of compatibilizer is often used to obtain a better composite strength [9, 10].

Most of common micromechanical models used to predict tensile properties of short glass fibre reinforced thermoplastics has been evaluated in the case of short flax fibre composites [9, 11-14]. Generally models are unsuitable to predict accurately the experimental data. The first reason puts forward is the complexity to measure fibre Young's modulus because this property changes with both fibre gauge length [15] and fibre diameter [16]. An elementary fibre can present a Young's modulus from 150 to 80 GPa [9] whereas a fibre bundle is assumed around 40 GPa [16]. The fibre Young's modulus or tensile modulus must be adjusted according to the shape ratio of the flax fibre. Another reason comes from the evaluation of the orientation factor and the interfacial shear stress from Bowyer and Bader method [17] used in the tensile strength prediction. Besides, at high fibre content, the increase of fibre-fibre interactions (increase of fibre contact points) during composite loading contributes to a decrease of composite strength due to a decrease of fibre stress transfer surface area. Thus, A.G. Facca & al [12] have shown that the tensile strength of short flax fibre reinforced polyethylene reach a plateau above 30wt% fibre content. A clustered parameter has to be introduced in the predicted model to improve the strength prediction [12].

The aim of the present work is to evaluate the improvement of the prediction of the micromechanical models usually used for short fibre reinforced injected polypropylene taking into account the composites morphology.

Experimental

Material

Compounded flax reinforced polypropylene pellets were provided by AFT Plasturgie (France) under the trade name of PP-LIN. The material has a melt flow index of 14 g/10 min at 230 °C / 5 kg. The pellet contains 30 wt% of flax fibre. It has the density almost equal to that of water. The residual humidity is less than 1% (supplier data). Neat PP, grade B10 FB, from Polichim Industrie with melt flow index of 12 gms/10 min (ASTM D1238) and density of 0,904 g/cm³ was also used to produce compounds with lower flax fibre content (5-10-15wt %), based on the dilution of the 30wt % flax (PP-LIN) with this material.

Processing conditions

Table 1. Moulding conditions for Flax fibre reinforced PP

<i>Processing parameters</i>	<i>Set up value</i>
Mould temperature (°C)	25
Melt temperature (°C)	205
Volume flow rate (cm ³ .s ⁻¹)	25
Holding pressure (Bar)	400
Back pressure (Bar)	50
Screw rotation speed (cm.s-1)	440

The compounded pellets were processed on an 80 ton clamping force injection moulding machine (KM80-160E, Krauss-Maffei, Germany) into standard tensile test specimen. The optimum moulding conditions previously determined by Tagushi DOE to achieve the highest tensile Modulus in the case of 30-wt% of flax fibre reinforced PP injection moulding were used. They are reported in Table 1.

Characterizations

Tensile tests were performed on a tensile machine (Model 1185, Instron) using an extensometer for strain measurement. Tests were conducted on five samples according to ISO 527 standard at 20 mm/min crosshead speed for the yield and ultimate stress measurements and at 1 mm/min for Young's modulus. Samples were stored during 2 days at 23°C, 50% relative humidity before testing.

For the fibre orientation measurements, 10x10 mm samples were cut in the middle of tensile ISOA standard sample and subjected to successive stages of cutting through the thickness by a Leica 4201 microtome. The residual part of cutting sample is then placed under a polarizing microscope (Jenapol, CarlZeiss Jena Switzerland) used in reflection mode and associated to a CCD camera coupled to a computer to get and record suitable images. The experimental procedure described by Bay and Tucker [18] was used to get the components of the orientation tensor. For characterization orientation, a minimum of 150 fibres were measured.

The thermogravimetric analysis (TGA) has been used to evaluate the real fibre content in the composites. The weight fraction of flax fibres in the composites was determined by thermogravimetric analysis (TGA). A TGA Q500 V6.5 from TA Instruments was used at a heating rate of 10° C/min, over the temperature range from 30-600°C, using a constant flow of nitrogen as purging gas. The same program was used to estimate the fibre content along the thickness direction. For that 15µm slices were cut in a microtome Leitz 1401 along the thickness direction of the tensile bar, up to the centre of the specimen. The fibre weight fraction (w_f) was calculated using the following equation [19]:

$$X = a(w_f) + b(1 - w_f) \quad (1)$$

where X is the percentage weight of TGA residue; a is the percentage of residual weight when 100% fibre is degraded and b is the percentage of residual weight when 100% neat resin is degraded. All results collected through TGA analysis is presented in Table 2.

Table 2. The measured flax fibre content in the composites from TGA analysis.

Materials	Real flax content (%)	
	Weight	Volume
Flax fibre	-	-
PP	-	-
PP/5wt% flax	5.4	4
PP/10wt% flax	9.1	6
PP/15wt% flax	11.4	8
PP/30wt% flax	28.9	22

Fibre length and fibre diameter are measured by Optical Microscopy analysis (OM). To image the composite microstructure, in bright field and polarized light modes, a BH2 Olympus transmission microscope coupled with a digital camera LEICA and a software Leica Qwin Pro was used. Thin films of 15µm thickness were cut in a microtome Leitz 1401. The films were then placed between a glass slide and cover glass, with Canadian balsam. The number-average fibre length L_n (Eq. 2a), the number-average fibre diameter D_n (Eq. 2b) and the corresponding standard deviation on the sample were calculated according to the following equation:

$$\begin{aligned}
 \text{a) } L_n &= \frac{\sum_i L_i n_i}{\sum_i n_i} & \text{b) } D_n &= \frac{\sum_i D_i n_i}{\sum_i n_i}
 \end{aligned} \tag{2}$$

where n_i is the number of fibres and L_i the fibre length or D_i the fibre diameter.

A PERKIN-ELMER DSC-7 was used to determine the degree of crystallinity of the composites. Tests were run at the temperature range between 25°C to 200°C, at 10°C/min, with nitrogen as purging gas. The degree of crystallinity was determined using the equation described below, considering the heat of fusion of 100% crystalline (ΔH_0) PP to be 148 J/g [11, 12]. ΔH_m is the enthalpy of melting estimated by the DSC curves and wf is the fibre weight fraction in the composite.

$$X_c = \frac{\Delta H_m}{\Delta H_0(1 - wf)} \tag{3}$$

Results and discussion

Young's modulus evaluation

In the case of short fibre composites, not all the fibres are not oriented in the same direction and have the same length. In addition, the usual rule of mixture applied for a unidirectional reinforced composite does not give the expected accuracy. The most common model used to predict young's modulus in the case of short fibre reinforced polymers is the Halpin-Tsai equations [21]. These equations are especially attractive since no experimental measurement is required. The Halpin-Tsai equations are a set of empirical relationships that enable the property of a composite material (E_c) to be expressed in terms of the matrix (E_m) and reinforcing phase (E_f) properties together with their proportions and geometry:

$$E_c = E_m \cdot \left(\frac{1 + \zeta \eta X_{vf}}{1 - \eta X_{vf}} \right) \quad \text{Where } \eta = \frac{\frac{E_f}{E_m} - 1}{\frac{E_f}{E_m} + \zeta} \tag{4}$$

ζ -depending of fibre shape ratio and arrangement, for circular fibre shape $\zeta = 2 \left(\frac{l}{d} \right)$ [21]

E_c – composite modulus;

X_{vf} - Volume fraction of the fibres;

E_f – fibre modulus;

d – fibre average diameter;

E_m – matrix modulus;

l – fibre average length;

Tensile strength evaluation

The tensile stress is more complicated to predict than Young's modulus because the material is no longer in the lower strain states, it evolved from its initial state to plastic strain, rearrangement and damage. In a composite, the failure can occur in the fibres or at the fibre-matrix interface. In the case of a slipping matrix/fibre load transfer, the Kelly Tyson's model [22] is commonly used to evaluate the composite strength from the equation:

$$\sigma_{cu} = \left[\eta_0 \cdot \sum_{l_i < l_c} \frac{\tau_u \cdot l_i \cdot X_i}{2 \cdot r_f} \right] + \left[\eta_0 \sum_{l_j > l_c} \sigma_{rf} \cdot X_j \cdot \left(1 - \frac{l_c}{2 \cdot l_j} \right) \right] + \left[\sigma'_m (1 - X_{vf}) \right] \tag{5}$$

X
Y
Z

- σ_{rf} – The ultimate fibre stress
- σ_{cu} – The ultimate composite stress (stress at break)
- l_i et l_j – The subcritical and supercritical fibre length to the critical fibre length l_c
- X_i et X_j – The volume fraction of l_i and l_j fibre lengths
- σ'_m – The stress supported in the matrix at composite failure strain ($\sigma'_m = E_m \cdot \epsilon_c$)
- l_c – The fibre critical length
- σ_{rf} – The ultimate fibre stress
- τ_u – The shear stress at the interface due to fibre/matrix friction
- η_0 – The virtual orientation factor

Thomasson [23] suggested an experimental method called IFSS (Interfacial Shear Stress) to estimate the matrix and fibre contributions. From the equation 5, the model can be written to a simplified expression: $\sigma_{cu} = \eta_0 \cdot (X + Y) + Z$; where Z is the matrix contribution, X is the subcritical fibre contribution and Y is the supercritical fibre contribution.

Morphological composite evaluations

The average fibre length of the composites is reduced of almost 40% compared to the initial fibre length in the pellet (Table 3). At high fibre content the fibre breakage decreases to about 30%. At the same time, the average fibre diameter tends to decrease of 15 to 20% up to 8vol% fibre content (Table 3) and maintains its initial fibre diameter at high fibre content (22vol%). For lower fibre content, the fibre rotating movements during flowing [24] induce both fibre breakage and fibre defibrillation. For higher fibre content, the increase of the shear stresses in the melt tends to decrease rotational movements of the fibres and thus to decrease fibre breakage. Besides, as revealed by N. Le Moigne & al [6], the coexistence between fibre breakage and fibre defibrillation induces no really significant changes in aspect ratio (length on diameter). In the present study, the aspect ratio is calculated respectively of 11.7, 12.5, 14.3 and 11.5 for 4, 6, 8 and 22vol% of fibre content.

Table 3. Macrostructure evaluations of the flax fibre reinforced composites

Fibre volume content	Average diameter (μm)	Average fibre length (μm)	l_c (μm)	τ_u (MPa)	Virtual orientation factor η_0	Orientation factor η_0 (measured Fig 1)
4 vol%	46	533	904	30	0.001	0.694
6 vol%	44	549	1569	18	0.199	0.735
8 vol%	43	621	1750	17	0.189	0.749
22 vol%	51	642	1776	16	0.360	0.580

The orientation factor can be deduced from the measured orientation profiles taken through the thickness direction of the sample. These results are presented in the figure 1. Up to 8vol% fibre content, the composites show a quite similar orientation profile with a high oriented layer just at the cavity wall due to the fountain flow, an increase of orientation under the skin occurring by the shear flow during filling stage and an oriented core layer depending on the flax fibre content (lower oriented layer for higher flax fibre content) due to extensional flow during filling stage. This core orientation achieved during the filling stage of the mould seems to be not affected by the holding pressure. Excepted for the higher fibre content (22vol%) that reveals a random fibre orientation in the middle of the part (about 40% of the whole thickness), looking like to a short glass fibre reinforced composite orientation profile. The microscopy observations of the sample cross section areas have revealed a fibre planar orientation.

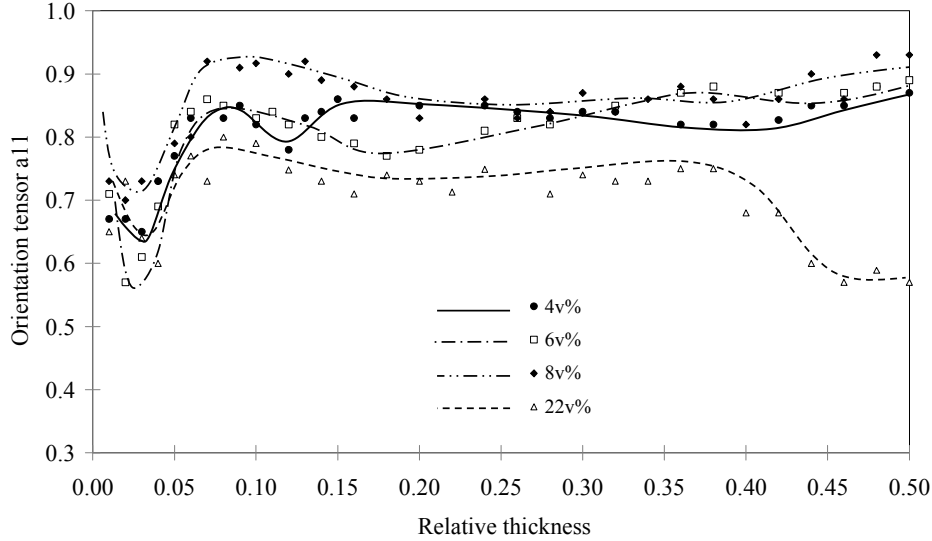


Fig. 1: Orientation tensor in the flow direction through the thickness for the injected composites

Mechanical properties and tensile strength model confrontation

The Kelly-Tyson's model (Eq.5) has been used to evaluate the tensile strength of the flax fibre reinforced composites from the Interfacial Shear Stress (IFSS) method. The predicted tensile strength has been calculated using a flax fibre strength σ_f of 1.5 GPa and a flax fibre modulus E_f of 50 GPa [9]. The flax fibre diameter of each composite came from the experimental measurements (Tab.3). The matrix contribution Z has been determined from tensile test on pure polypropylene to calculate R ratio and the stress supported by the matrix when the fibres break (Eq.5). Using the measured fibre length distribution, the interfacial shear strength τ_u and the virtual orientation factor η_0 can be calculated and They are presented in table 3.

The calculated interfacial shear stresses are lower than the common value found in the literature for a elementary flax fibre in a PP compatibilized matrix [26], but these value are in accordance with other author for short flax fibre injected compounds (15 MPa for 40 vol% to 32 MPa for 20 vol%) [9, 27]. The interfacial shear stress decreases as the fibre content increases in the composite, revealing thus a lower matrix adhesion around the fibres. The virtual orientation factors are all very low with regard to experimental evaluation from orientation through the thickness (Fig 1), probably due to the low strains values chosen for all the composites ($\varepsilon_1=0.5\%$ and $\varepsilon_2=1\%$) that decrease the contribution of the fibre orientation at low stress level in particular for a low fibre content. The critical fibre length is in accordance with the range of literature values [9]. The average fibre length of each composite is widely under the critical fibre length meaning at the failure, the tensile strength should be dependent on fibre orientation and fibre length in the composites [17, 28].

The Kelly-Tyson's model predictions presented in Figure 2 tends to over evaluate the tensile strength for all composites even if the tendency seems to be in accordance with experimental. The origins of this over evaluation are multiple. The first one is the over evaluation of flax fibre mechanical properties, contrary to the glass fibres, flax fibre is a natural reinforcement with a large spectra of properties depending of the physical composite-like structure coming from flax fibre preparation (scutched, hackled fibre) and composition (bundle, technical fibre, elementary fibre or micro-fibril) [27]. Thus, flax fibre modulus and flax fibre strength can be adjusted according to their diameter [16, 29]. From about 10 μ m mean diameter (elementary fibre) to 30 μ m (technical fibre), the modulus vary on average from 65 to 30 GPa and strength from 1.5 to 0.5 GPa , in the case of bundles, young modulus can decrease up to 20 GPa [30]. The second reason of the over evaluation

could come from the estimation of the stress supported by the matrix when the composites break (σ'_m ; Z term in Eq.5) commonly considered as the product of the matrix modulus (E_m) and the strain at which the composite fail (ϵ_c). This consideration suggests that the matrix has a linear behaviour at the moment of the break. However, due to the polypropylene ductility most of breakage occurred in the non-linear deformation domain in particular for the lowest fibre content when the composite failure comes long away from the yield point. Besides, in most of cases, the composite failure comes from matrix cavitation around macro-defaults composed by fibre agglomerates that decrease strongly the composite strength. More over the strength of the matrix is widely influenced by its crystalline and the trans-crystalline structures induced by both fibre content and processing conditions.

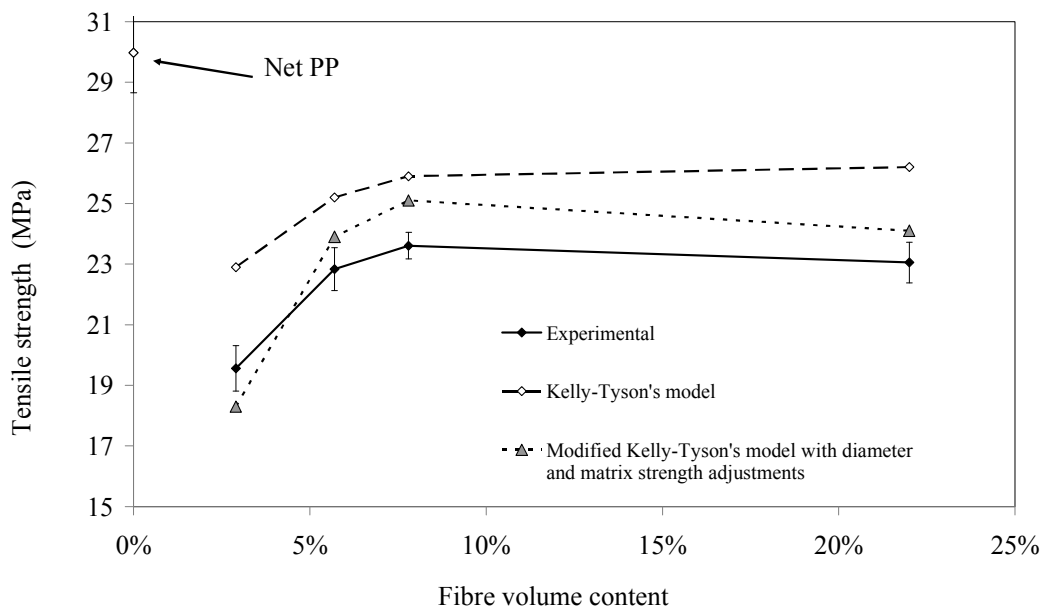


Fig.2: Comparison of tensile strength estimations and experimental data for short flax fibre reinforced polypropylene

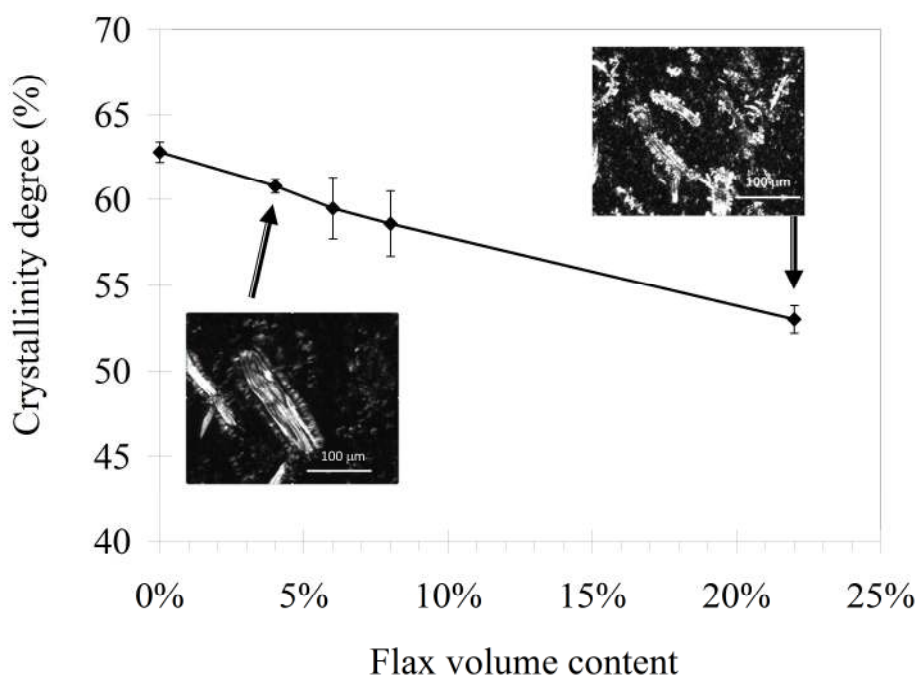


Fig.3: Evolution of crystallinity degree as a function of flax fibre content [20]

A previous study [20], has shown a decrease of 10% in the degree of crystallinity between the net polypropylene and 22vol% flax fibre reinforced polypropylene moulded with the same injection moulding parameters and a decrease of trans-crystalline structures with an increase of fibre content (Fig.3). The decrease in the crystallinity degree induces a variation of matrix modulus in the range of 1.5 to 0.7 GPa for 65 to 40% of crystallinity degree [31], the tensile strength is also affected in a same range.

With these considerations, the Kelly-Tyson strength model can be modified in order to take in account the fibre properties variability (σ_{ff}) with flax fibre diameter (r_{ff}). The relation between fibre defibrillation (fibre diameter) and fibre breakage (fibre length) shown in the literature [6], has been established in order to estimate flax fibre young's modulus as a function of its diameter as revealed by some authors [8, 29]. Fibre strength has been adjusted on average to 0.8 GPa according to [29]. The second modification concerned the matrix strength estimation. Taking in account the bulk crystallinity degree variations with fibre content previously revealed [20] and thus matrix modulus, it has been considered justly in a first approximation that the matrix strength at the composite failure is the composite strength reduced of the volume fibre content according to a rule of mixture: $E_m = (E_c - E_f X_{vf}) / (1 - X_{vf})$. With the adjustments, the matrix young's moduli were in the range of 0.78 to 1.23 GPa from 22vol% to 4vol% flax fibre content. These values were in accordance with the literature for the polypropylene modulus-crystallinity degree relationship [31]. Finally, the composite strength calculations give better accuracy with the experimental data (Fig.2).

Mechanical properties and elastic models confrontation

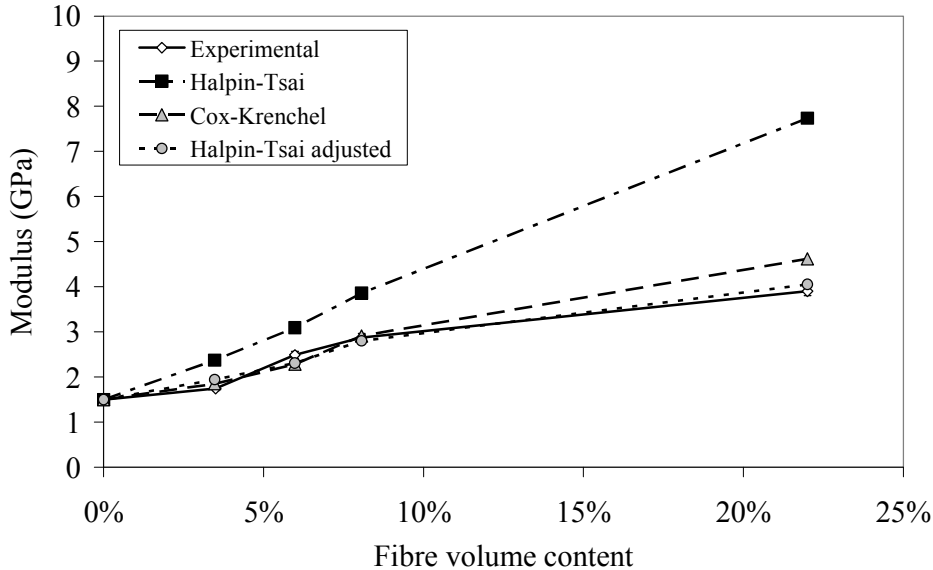


Fig.4: Young's modulus estimation from Halpin model in comparison with experimental data and matrix modulus value adjustment

The Young's modulus calculations from Halpin-Tsai model presented in figure 4 give an overestimation of 25 to 98% according to composite fibre contents. The difference between experimental values would come from the lack of fibre length correction factor based on fibre shape ratio and defined for instance in the common Cox-Krenchel model [25, 5], or by wrong estimation of fibre and matrix properties depending on fibre degradation and matrix morphology. In any cases, an adjustment of matrix young's modulus previously presented gives a good prediction of experimental (Fig.4).

Conclusion

The prevision of the mechanical properties from the common micromechanical models usually used for short glass fibre reinforced thermoplastics has shown a good representation in terms of tendencies of the mechanical behaviour of the composites with the increase of fibre content but most of them over evaluate the experimental data. For the young's modulus prediction, the Cox-Krenchel's model including both the measured fibre orientation and fibre ratio factors gives an improvement of the calculated values with regard to the experimental data, but a better definition of fibre properties linked with fibre shape ratio associated to an adjustment of matrix properties permitted to improve significantly the modulus evaluation by the Halpin-Tsai model.

For the tensile strength prediction, the usual Kelly-Tyson model over evaluates the composite strengths in less proportion than for the modulus. The adjustments of the model taking into account physical structure of flax fibre resulting from the processing (fibre breakage, fibre defibrillation) and giving a large spectra of mechanical properties as well as a better evaluation of matrix strength depending of the crystallinity rate and trans-crystalline morphology, have permitted to evaluate the composite strength with a better accuracy.

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