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Tensile and fracture behaviour of PP/wood flour composites

Ezequiel Pérez [a], Lucía Famá [a], S.G. Pardo [b,c], M.J. Abad [b], Celina Bernal [a,*]

[a] Advanced Materials Group, INTECIN (UBA-CONICET), Department of Mechanical Engineering, Engineering Faculty, University of Buenos Aires, Av. Paseo Colón 850, C1063ACV Buenos Aires, Argentina

[b] Grupo de Polímeros, Univ. A Coruña (UDC), Av. 19 de Febrero, s/n, 15405 Ferrol, Spain

[c] Centro Galego do Plástico (CGaP), A Cabana s/n, 15590 Ferrol, Spain

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ABSTRACT

Polypropylene/wood flour composites with different fibre content were prepared. The effect of composition and the incorporation of maleinated polypropylene on the materials tensile and fracture and failure behaviour was investigated. Reliable fracture toughness data that will be useful for structural applications were obtained. In unmodified composites an increase in Young's modulus was found with the addition of wood flour to PP, whereas tensile strength, strain at break and fracture toughness were observed to decrease as fibre content increased. The presence of MAPP was beneficial to tensile strength and ductility and had no significant effect on fracture toughness, as a result of enhanced fibre dispersion within the matrix and improved interfacial adhesion. Although reduced ductility and toughness were observed for the composites respect to the matrix, in the case of modified composites, environmentally friendly stiffer materials were obtained with cost saving without sacrificing strength.

1. Introduction

Over the recent years, reinforcing polymers with natural fibres has become very attractive mainly because of the good mechanical properties that can be obtained at relatively low cost. Natural fibre reinforced thermoplastics have light weight, adequate strength and stiffness, and low cost and can be easily produced by conventional plastics processing techniques. The use of natural fibres as an alternative of glass fibres is also driven by ecological reasons and because natural fibres have several advantages. They are low cost fibres, highly available and renewable, with low density and high specific properties as well as biodegradable and less abrasive to processing equipments. However, their potential use as reinforcement is greatly reduced due to their incompatibility with the hydrophobic polymer matrix, their poor resistance to moisture and their tendency to form aggregates during processing. Poor mechanical and physical properties of natural fibre reinforced composites are frequently attributed to a weak fibre–matrix interface. Therefore, different additives able to react with the fibre and the matrix are frequently added in the formulations.

Poly(propylene) (PP) is a useful commodity polymer with outstanding properties such as low density, sterilizability, good surface hardness, very good abrasion resistance, excellent electrical properties, as well as good mechanical and barrier properties to water. It also has low cost, worldwide production, simplicity of processing, capability to burn without producing toxic emissions, working security, and recyclability. Its blends and composites find wide applications in home appliances, automotive parts, extruded profiles, packaging industry, construction, etc. [1,2]. In particular, natural fibre reinforced PP composites are commonly used in the automotive and building industries mainly in structural applications such as fencing, decking, outdoor furniture, window parts, roofline product, door and panels [3]. For these applications, in addition to high stiffness and mechanical strength, adequate fracture toughness is often needed. Although significant efforts have been devoted to the mechanical and fracture behaviour of PP and its blends and composites [3–16], many aspects of this behaviour are still unclear, and to our knowledge, only scarce fracture toughness data are available in the open literature in the case of natural fibre reinforced PP [17,18]. These data are required for design purposes when these materials are used in structural applications.

The full potential of natural fibre reinforced PP will not be achieved unless their mechanical properties, especially fracture resistance, are optimised and this will only be possible if all the factors (chemical, physical and mechanical) that control the fracture mechanisms and toughness are clearly understood.

In this work polypropylene/wood flour (WF) composites with different fibre content were prepared. The effect of composition and the addition of maleic anhydride grafted PP (MAPP) on the

* Corresponding author.
E-mail addresses: sgarcia@udc.es (S.G. Pardo), cbernal@fi.uba.ar (C. Bernal).
URLs: http://www.grupolimeros.com (S.G. Pardo), http://www.fi.uba.ar (C. Bernal).

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materials tensile and fracture behaviour was investigated. The failure mechanisms occurring in the composites were also indentified. The aim was to obtain reliable fracture toughness data useful for design purposes as well as to correlate the morphology with the materials tensile and fracture behaviour. The knowledge of the structure–property relationship in these materials is a critical step towards tailoring the required mechanical properties of the end product.

2. Materials and methods

2.1. Materials and sample preparation

A commercial isotactic PP (ISPLEN® PP 070 G2 M) provided by Repsol YPF, with a density of 0.902 g/cm^3, and a melt flow index (230 °C – 2.16 kg) of 10 g/10 min was used as the matrix of the composites. Wood flour from red pine (WF) was used as filler (average size = 200 µm). 10% of maleic anhydride functionalised iPP (Fusabond® MD 511 D, Dupont) with a melt flow index of 22,5 g/10 min (190 °C/2, 16 kg), was incorporated as a coupling agent. Prior to the mixing process, wood flour was dried for 24 h at 100 °C. Composites with different wood flour content (10, 20 and 30 wt.%) were prepared in an internal mixer at 180 °C and 45 rpm for 10 min. Then, composite plaques (3 mm) were compression moulded at 190 °C under a pressure of 50 bar for 10 min.

2.2. Mechanical characterisation

Uniaxial tensile tests were performed in an Instron dynamometer at a crosshead speed of 1 mm/min for neat PP and the different composites by following ASTM D638-02 standard recommendations. From these tests, stress–strain curves were obtained and from these curves, Young's modulus (E), tensile strength (σ_u) (maximum stress) and strain at break (ε_b) were determined.

Quasi-static fracture tests on deeply double edge-notched (DENT) specimens were also carried out at 1 mm/min for all materials in the testing machine [19]. Sample dimensions were: length (L) = 50 mm and depth (W) = 20 mm. The distance between grips was 30 mm. Two properly aligned sharp notches of 5 mm were introduced by sliding a fresh razor blade into machined slots with the help of a specially designed device. The J-Integral approach was adopted to characterise fracture toughness. It was obtained from the whole area under the load–displacement curve (U_{tot}) as follows:

$$J_c = \frac{\eta U_{tot}}{B(W-a)} \quad (1)$$

where U_{tot} is the overall fracture energy, B is the thickness and η is a geometrical factor defined by [20]:

$$\eta = -0.06 + 5.99\left(\frac{a}{W}\right) - 7.42\left(\frac{a}{W}\right)^2 + 3.29\left(\frac{a}{W}\right)^3 \quad (2)$$

All mechanical tests were performed at room temperature. A minimum of five replicates were tested for each system and the average values with their deviations were reported.

2.3. Fracture surface analysis

Fracture surfaces of specimens broken in tensile and fracture tests were analyzed by Scanning Electron Microscopy (SEM) after they had been coated with a thin layer of gold.

3. Results and discussion

3.1. Uniaxial tensile behaviour

Fig. 1a and b shows typical tensile stress–strain curves for the PP matrix and the PP/WF composites with different fibre content without and with MAPP, respectively.

As it can be observed in Fig. 1, most materials displayed plastic deformation beyond maximum load with a precipitous drop of load to zero at a certain point in the stress–strain curve. The increase in fibre content led to a more brittle behaviour. In addition, none of the materials presented necking before fracture.

The incorporation of MAPP in the composites formulation did not qualitatively change the materials tensile behaviour.

Table 1 lists Young's modulus, tensile strength and strain at break values for neat PP and the different PP/WF composites investigated. It was found that, irrespectively of the presence of the coupling agent, the material stiffness slightly increased with fibre content as expected from the incorporation of the more rigid fibres into PP. On the other hand, tensile strength and strain at break values were found to decrease as fibre content increased for the composites without MAPP. The decrease of the maximum stress indicates that filler particles debonded from the matrix prior to or at the start of plastic deformation. As a result of debonding, the strain constraint is released and hence, tensile yield stress is lowered.

The presence of the coupling agent was slightly detrimental to the material stiffness but beneficial to tensile strength (it allowed retaining the matrix tensile strength) and led to a slight recovery of ductility for most compositions. Similar results have been

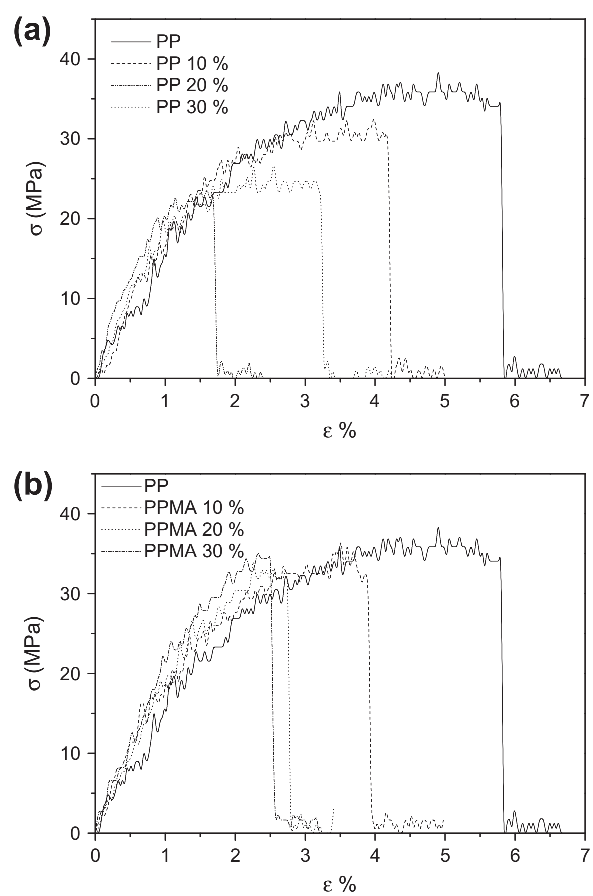

Fig. 1. Typical tensile stress–strain curves for the PP matrix and the PP/WF composites with different fibre content. (a) Without MAPP. (b) With MAPP.

Table 1
Tensile and fracture parameters values for the matrix and the different composites investigated.

System	Young's modulus E, (MPa)	Tensile strength, σ_u (MPa)	Strain at break, ε_b (%)	J-Integral, J_c (kJ/m^2)
Neat PP	1965 ± 70.70	34.63 ± 0.16	6.66 ± 1.23	50.46 ± 6.19
PP/10 wt.% WF	2615 ± 134.35	34.78 ± 7.57	2.68 ± 0.70	21.27 ± 2.26
PP/20 wt.% WF	2405 ± 176.78	24.08 ± 0.09	2.87 ± 0.36	15.94 ± 0.44
PP/30 wt.% WF	2760 ± 84.85	20.54 ± 3.51	1.44 ± 0.21	8.06 ± 2.53
PP/MAPP/10 wt.% WF	2095 ± 63.64	34.48 ± 0.68	3.74 ± 0.27	24.27 ± 3.82
PP/MAPP/20 wt.% WF	2160 ± 296.99	32.59 ± 0.44	2.68 ± 0.04	14.39 ± 1.77
PP/MAPP/30 wt.% WF	2130 ± 113.14	34.67 ± 0.49	2.30 ± 0.15	9.85 ± 2.04

previously reported in the literature [18] for sawdust/recycled-PP composites and have been attributed to the enhancement in fibre dispersion and interfacial interaction promoted by MAPP.

3.2. Fracture behaviour

Typical load–displacement curves for DENT samples of the matrix and the different PP/WF composites without and with coupling agent are given in Fig. 2a and b, respectively.

All materials displayed quasi-brittle fracture behaviour characterised by non-linear load–displacement records with a precipitous drop of load to zero (unstable crack growth) before maximum load or around this point. In addition, macroscopically, none of the samples exhibited plastic damage during the tests.

Furthermore, the area under the load–displacement records decreased as the fibre content increased. However, load–displacement curves did not qualitatively change with fibre loading or with the incorporation of MAPP.

As all materials exhibited non-linear fracture behaviour and unstable crack growth at a certain point in the load–displacement curve, J-integral parameter at instability (J_c) was adopted to characterise the materials fracture behaviour [19]. In Table 1, average values of the J_c parameter are also summarized. A decreasing trend of fracture toughness with fibre loading was observed, in agreement with the qualitative analysis of load–displacement records suggesting that wood flour fibres acted as critical-sized flaws that induced premature failure in our composites.

It should also be noted that the J_c values obtained here are reliable values of fracture toughness that can be used for design purposes.

In addition, no significant differences were observed between fracture toughness values for the composites with and without MAPP. The introduction of the coupling agent is expected to improve wettability of wood fibre by the matrix polymer, lowering the fibre to fibre interactions, improving fibre dispersion, and enhancing the adhesion between wood fibre and the polypropylene matrix. Improved adhesion usually leads to decreased fracture resistance [17], but in our composites, this effect would have been

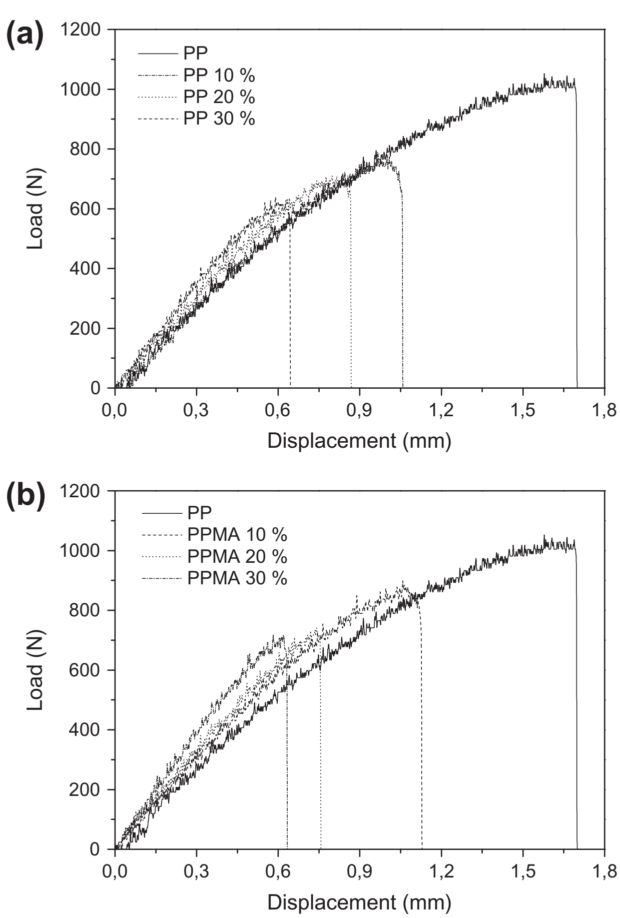

Fig. 2. Typical load–displacement curves for DENT samples of the PP matrix and the PP/WF composites with different fibre content. (a) Without MAPP. (b) With MAPP.

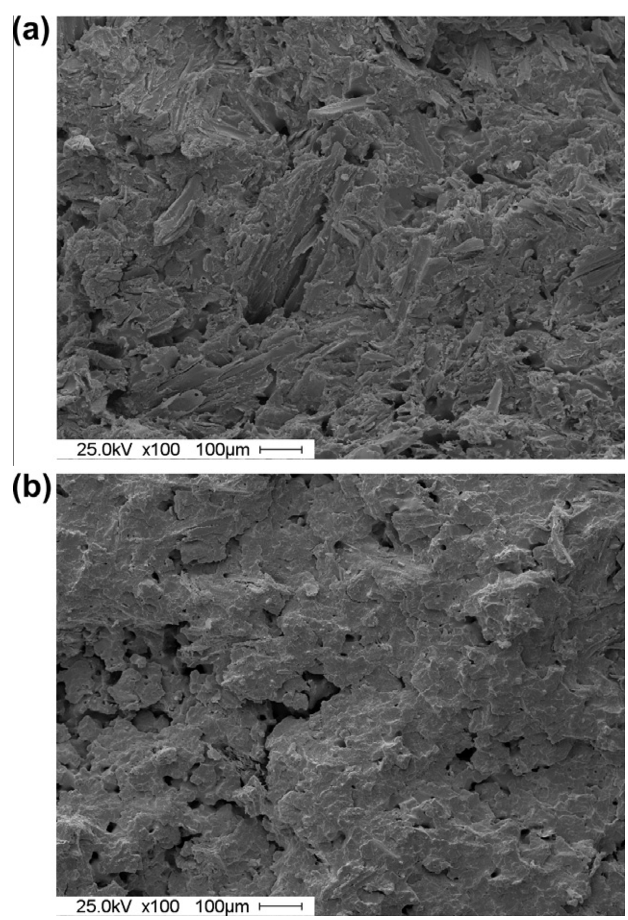

Fig. 3. Typical fracture surfaces of tensile specimens of the composite with 30 wt.% wood flour. (a) Without MAPP. (b) With MAPP.

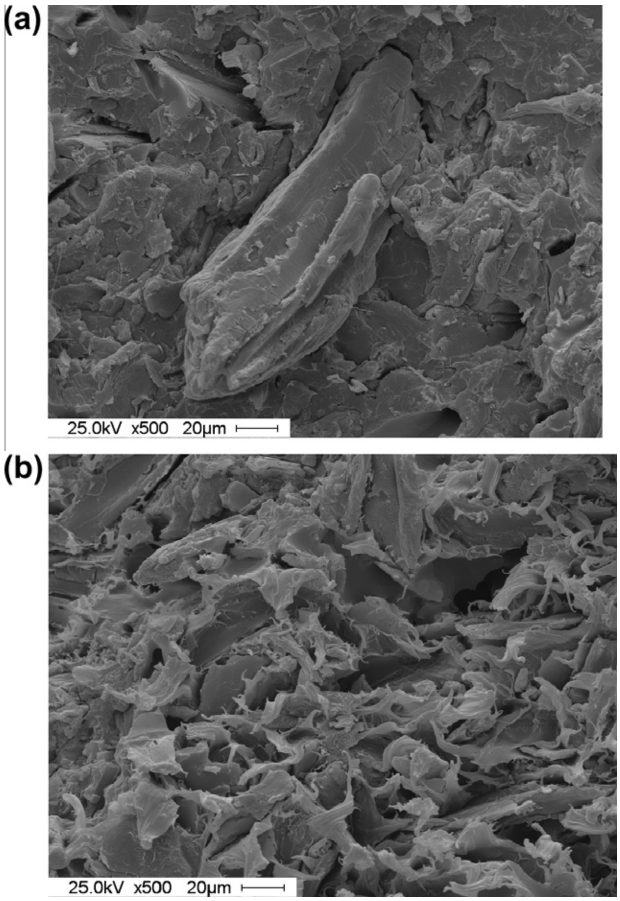

Fig. 4. Closer views of brittle and ductile zones of Fig. 3a. (a) Brittle zone. (b) Ductile zone.

counteracted by the beneficial effect of better fibre dispersion on the materials fracture behaviour.

3.3. Fracture surface analysis

Fig. 3a and b shows typical fracture surfaces of tensile specimens of the composite with 30 wt.% wood flour without and with MAPP, respectively. It can be observed in Fig. 3, that the fracture of the composites occurred within the PP matrix without noticeable plastic deformation which was suppressed by the constraint imposed by the rigid fibres. Materials in front of the crack tip in these circumstances were subjected to plane strain condition. The crack propagated through the PP matrix with little plastic deformation.

In unmodified composites (Fig. 3a) fibres were debonded and pulled out from the PP matrix with smooth and clean surfaces because of the poor interfacial adhesion. In modified composites (Fig. 3b), on the other hand, fibre fracture was the dominant failure mechanism. Furthermore, in the micrograph of Fig. 3a, wood flour fibres were much more distinguishable than in Fig. 3b suggesting better interfacial adhesion in modified composites. As a result of improved adhesion, fibres on the crack path were split into two pieces rather than being debonded and pulled out from the matrix. The fracture toughness in these composites was mainly caused by the fracture energy of wood flour fibre and matrix, which is relatively low [21]. However, fracture toughness values did not show significant differences between unmodified and modified composites. The incorporation of MAPP in the composites formulation is expected to be detrimental to the material toughness, as a result of the concomitant effect of the reduced molecular weight of the PP-MAPP blend [22,23] and an increased interfacial adhesion promoted by the coupling agent [17]. The beneficial effect of the improved dispersion of wood fibres in PP seems to be responsible for the observed fracture behaviour, as mentioned above.

In addition, although fracture surfaces globally presented a brittle appearance, they also exhibited zones of localised ductile pattern. Fig. 4a and b and Fig. 5a and b are closer views of brittle and ductile zones of Fig. 3a and b, respectively. The increased adhesion of modified composites is also evident in these micrographs. Besides, ductile tearing of the PP matrix is clearly observed in Figs. 4a and 5b.

Finally, Fig. 6a and b are SEM fractographs of DENT samples of the composites with 30 wt.% wood flour without and with maleated PP, respectively. Once again, these micrographs show enhanced adhesion in the presence of MAPP. While clean and pulled-out fibres as well as many free spaces around the fibres are clearly seen in the unmodified composite, wood flour fibres are completely coated by the polymer matrix and hardly distinguishable in the composite with MAPP. In addition, the typical failure mechanism of natural fibre reinforced composites of fibre splitting into elementary fibres is also observed in Fig. 6a.

As stated before, low interactions between hydrophobic polymer chains and hydrophilic fillers hinder the good dispersability level and thus could negatively affect some mechanical properties of the composites. To overcome this difficulty, functional oligomers with polar groups like maleic anhydride grafted PP are generally added to cause the affinity for polar fillers. So that MAPP, can serve as a coupling agent between the filler and the matrix [24]. MAPP is formed by the reaction of maleic anhydride (MA) with PP in the presence of an initiator to obtain PP chains with pendant groups.

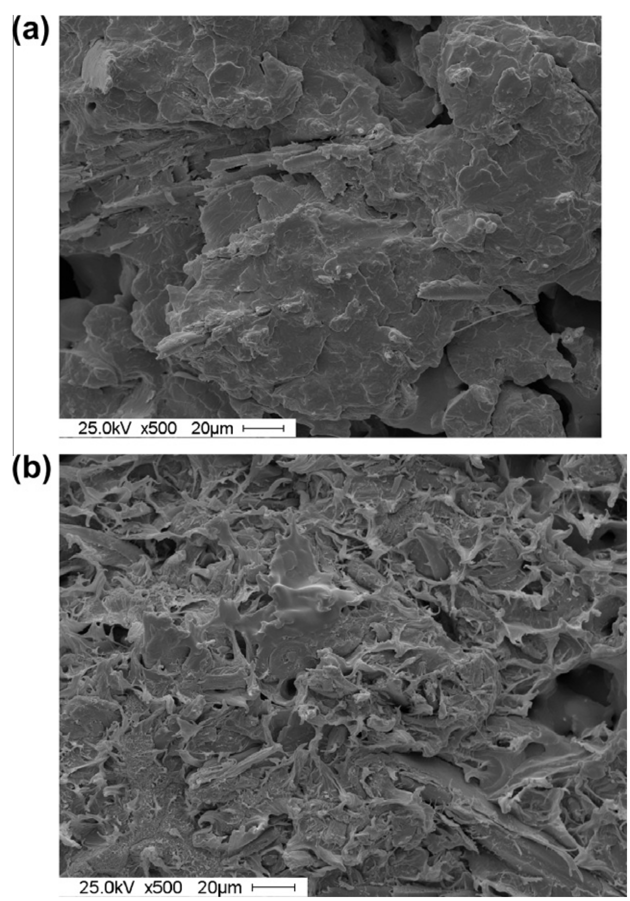

Fig. 5. Closer views of brittle and ductile zones of Fig. 3b. (a) Brittle zone. (b) Ductile zone.

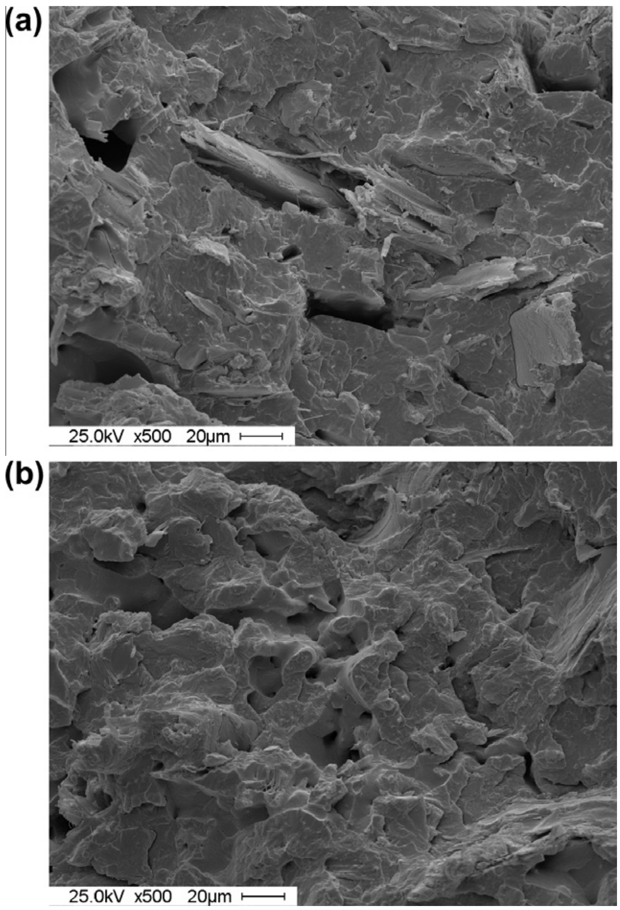

Fig. 6. SEM fractographs of DENT samples of the composites with 30 wt.% wood flour. (a) Without MAPP. (b) With MAPP.

In PP composites, it is expected that the PP portion of MAPP entangles and cocrystallises with the unmodified PP and the reactive maleic anhydride groups of MAPP react with the active surface groups of polar fillers, such as wood flour fibres. Generally, chemical bonds between the active –OH groups of cellulose and the functionalised polymer are formed [3]. The weak bonds of the fibres are destroyed reducing the number and size of aggregates hence, leading to better dispersion of the fibre in the matrix [25]. The results obtained in this work, confirm that MAPP acted as an effective coupling agent in our composites, leading to improved dispersion of the wood flour fibres within the PP matrix and to increased interfacial adhesion between phases.

3.4. Modelling of strength

Pukánszky et al. [26] have developed a simple model for the quantitative evaluation of the composition dependence of tensile strength in reinforced polymers which also allows detecting the appearance and effect of aggregation. According to this model, the tensile strength of composites is determined by three components: the matrix tensile strength, the effective load-bearing cross-section and the interaction between phases:

$$\sigma_u = \sigma_{uo}(1 - v_f)/(1 + 2.5v_f)\exp(Bv_f) \quad (3)$$

where σ_u and σ_{uo} are the tensile strength of the composite and the matrix, respectively, v_f is the fibre volume fraction and B is a parameter related to the load-bearing capacity of the reinforcement. Parameter B can be determined form the linear form of Eq. (3).

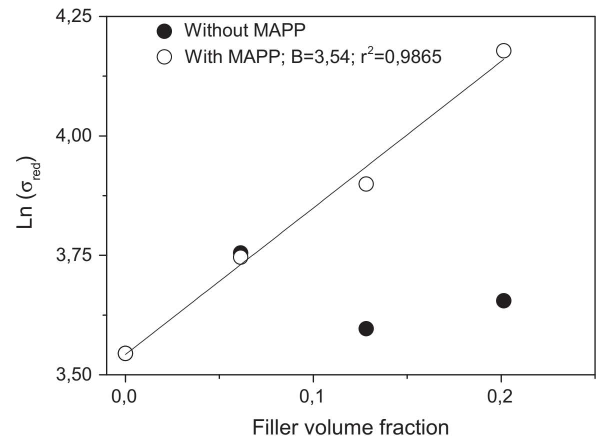

Fig. 7. Reduced tensile strength values as a function of fibre volume fraction for the PP/wood flour composites investigated.

Reduced tensile strength values can be expressed by rearranging Eq. (3) as follows:

$$\sigma_{ured} = \sigma_{uo}(1 + 2.5v_f)/(1 - v_f) = \exp(Bv_f) \quad (4)$$

and its natural logarithm should depend linearly on composition with a slope of B. Apart from calculating B, the linear plot allows checking the validity of the model and the presence of aggregation. Any deviation from a straight line indicates the presence of structural effects.

Fig. 7 shows the plot of reduced tensile strength values as a function of fibre volume fraction for the PP/wood flour composites investigated. As it can be observed in this figure, a very good linear fit was obtained in the case of the data for the composites with MAPP without any deviation from the straight line for the composition range explored. For unmodified composites, in contrast, points deviate from the linear correlation at fibre volume fractions higher than 0.06 indicating the early appearance of some structural effect, which are assumed to be aggregation [27]. This suggests that the shear forces achieved in the internal mixer were not enough to break down fibre aggregates during the time of mixing in the unmodified composites [2]. However, good dispersion of the wood flour fibres in the PP matrix was predicted for the composites with coupling agent.

The results obtained from the application of the model of Pukánszky et al. [26] for the composite tensile strength are in good agreement with the analysis of fracture surfaces and tensile and fracture results.

4. Conclusions

Polypropylene/wood flour composites with different fibre content were prepared by intensive mixing and subsequent compression moulding. The effect of fibre content and the incorporation of maleic anhydride grafted polypropylene (MAPP) on the materials tensile and fracture and failure behaviour was investigated under quasi-static loading conditions by means of uniaxial tensile and fracture mechanical tests.

Reliable fracture toughness data were obtained for different PP/WF composites. They are useful for design purposes when these composites are used in structural applications.

An increase in Young's modulus was obtained with the addition of wood flour to PP as expected, whereas tensile strength, strain at break and fracture toughness were found to decrease as fibre content increased. The presence of MAPP was beneficial to tensile strength and ductility and had no significant effect on fracture toughness.

From SEM analysis of fracture surfaces, the main failure mechanisms occurring in our composites were identified. In the unmodified composites, debonding and fibre pullout were found to be the main energy absorbing mechanisms, whereas in the composites with MAPP fibre breakage was dominant as a result of an increased interfacial adhesion.

The improved fibre dispersion promoted by the coupling agent and initially detected from tensile and fracture results as well as fracture surface analysis, was also confirmed from the application of a simple model for strength.

From the results obtained in this work, it can be concluded that although ductility and toughness were reduced for the composites respect to PP, environmentally friendly stiffer materials were obtained by using MAPP as a coupling agent. These materials present a significant reduction in the amount of the non-biodegradable material (PP) and also a concomitant cost saving without sacrificing strength.

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