Recycled Polypropylene Reinforced with Curaua Fibers by Extrusion

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ABSTRACT: Curaua fibers were studied as reinforcing agents for postconsumer polypropylene. The composites were processed by extrusion. The composite properties were investigated by mechanical tests, thermal methods, melt flow index, surface morphology, and water uptake. The variables studied were as follows: fiber contents (10 to 40 wt %), fiber surface treatment, initial fiber length, and modification of the polypropylene matrix. The treatment of the fiber with 5 wt % NaOH aqueous solution did not improve fiber-matrix adhesion and the composites using 20 wt % of untreated curaua fibers presented the better mechanical properties. Feeding the extruder with fibers having shorter lengths (0.01–0.4 mm) produced better fiber dispersion, improving the mechanical properties of the composites. Composites prepared using fibers without surface treatment with postconsumer polypropylene and with polypropylene modified with maleic anhydride showed mechanical properties and water uptake similar to composites using the same polymer reinforced with other lignocellulosic fibers. The extrusion process caused also partial fibrillation of the fibers, improving their aspect ratio. © 2009 Wiley Periodicals, Inc. J Appl Polym Sci 112: 3686–3694, 2009

Key words: natural fibers; curaua fibers; polymer-matrix composites; extrusion

INTRODUCTION

Synthetic fibers, like fiberglass, carbon fibers, and metallic fibers, are used by the industry as reinforcing agents in polymeric composites producing materials with improved mechanical properties. However, these synthetic fibers present disadvantages as follows: their production consumes energy; they are abrasive to the processing equipment; and they are not degradable on a short term. These fibers also produce residues after thermal recycling of the composite. The use of vegetal fibers as reinforcement agents for polymers is not a new practice[1] but, due to economical and environmental concerns, there is renewed interest in developing these composite materials.[2]

Presently, different car makers are using thermoformed composites with natural fibers in panels. The most used natural fibers are as follows: wood chips, jute, coir, sugar cane bagasse, flax, hemp, coconut husks, and sisal. After disposal, even when burned, the plant fibers produce small amounts of residue and return less carbon dioxide to the atmosphere than they used during plant growth. Glass fibers have a density of 2500 kg/m³ and natural fibers have a density from 810 to 1450 kg/m.[3] In terms of volume, this represents two to three times more material.[3] Besides, renewable raw materials have unlimited availability with very low energy consumption. The abrasivity of natural fibers is much lower compared with that of glass fibers, which leads to advantages with regard to recycling or processing of the composite materials.[1,2,4]

One of the drawbacks of natural fibers is their hydrophilicity, lowering their adhesion to hydrophobic polymer matrices like polypropylene, reducing the interfacial interactions between the surface of the fibers and polymer, and affecting negatively the mechanical properties of the composites. Improvement of fiber-polymer adhesion can be done through modification of either the fiber surfaces or the polymeric matrix. Another drawback is the low temperature of thermal degradation of these fibers, precluding mixture with polymers requiring processing temperatures above 220°C.[5]

The curaua fibers used in this work are extracted from the leaves of the *Ananas erectifolius* L. B. Smith plant (hydrophilous species), which is cultivated in semiarid regions of the Amazon. Its properties have been extensively studied.[6] Earlier studies using curaua fibers as reinforcement showed improved mechanical properties for the composites.[7,8]
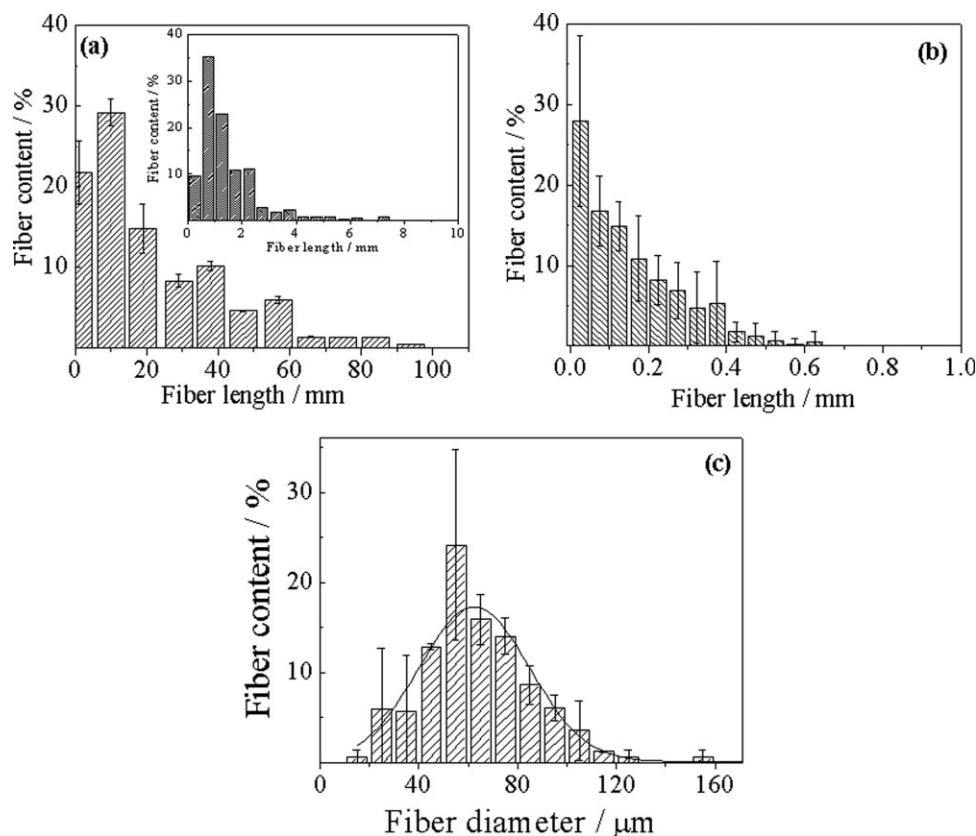
Figure 1 Curaua fiber length distribution after milling: (a) long fibers (insert is the fiber length distribution after processing); (b) short fibers; and (c) fiber diameter distribution.

Most reported studies of thermoplastic composites reinforced with natural fibers use virgin polymers.[9–14] Using a postconsumer polymer to produce reinforced composites has economic and environmental importance. Presently, it is possible to obtain on a large-scale postconsumer polypropylene, pcPP, with a controlled melt flow index. The pcPP is partially aged and contain carbonylic groups, originating from its thermal or photochemical oxidative degradation and this may improve the interaction between the fibers and the polymer matrix.

In this work, we compare the influence of fiber surface modification, matrix modification, fiber content, and fiber length distribution, on the mechanical properties of pcPP composites reinforced with curaua fibers.

EXPERIMENTAL

Materials

Postconsumer polypropylene, pcPP, (melt flow index, MFI = 5.5 g/10 min) was supplied by Proceplas (Campinas, Brasil). Maleic anhydride grafted polypropylene (MAPP, Licomont AR 504, Mn = 3500 g/mol, acid value of 37–45 mg KOH/g) was supplied by Clariant (São Paulo, Brasil) and was used as coupling agent.

Curaua fibers supplied after harvesting and decordication at POEMA (Poverty and Environment in Amazon Program, Belém, Brasil) were used. The fibers were washed in a conventional washing machine at 80°C for 1 h with tap water, rinsed with distilled water, and then dried in air for 48 h. The fibers were milled using two rotating knife mills (Rone, model NFA 1533, and Thomas Scientific, ED-5) obtaining fibers with two fiber-length distributions, long fiber and short fiber, respectively, Figure 1(a,b). The length distribution was analyzed using the Image-Pro Plus® 3.0 software for about 8000 fibers. The diameter of 120 fibers was determined using a micrometer (Mitutoyo, ±0.001 mm) at three points of 5 cm long samples and the smaller value was considered. Figure 1(c) displays the fiber diameter distribution, using the average frequency in relation to each interval.

Fiber surface treatment (NaOH treatment)

Long fibers were treated with 5 wt % aqueous NaOH solution (solution: fibers weight ratio = 15 : 1) for 2, 4, 6, 8, 12, 24, and 48 h at 23°C. The treated
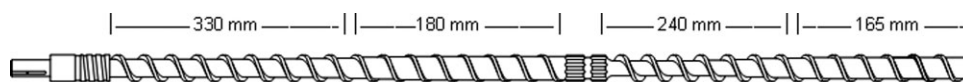
Figure 2 Maddock mixing screw design used in this work.

fibers were washed with water, with dilute acetic acid solution (10% v/v), and distilled water up to pH = 7. These materials were air dried, in the shadow, for 48 h and in an oven at 60°C for 12 h.

Compounding and processing

Different composite batches were prepared, as follows:

a. Using exclusively long curaua fibers, and contents from 10 to 40 wt % (**PP10-l, PP20-l, PP30-l,** and **PP40-l**). These fibers were washed only with water and dried.
b. Using 10 wt % of long curaua fibers treated with NaOH (**PP10-l-NaOH**).
c. Using 20 wt % short curaua fibers, with 3 wt % of MAPP (**PP20-s-MA**) or without MAPP (**PP20-s**).

In all cases fibers and MAPP were fed together with pcPP into the extruder hoper without previous mixing.

Before processing, the fibers were further dried for 4 h at 100°C. Composites were processed in a Wortex single-screw extruder (screw design shown in Fig. 2), screw diameter 32 mm, screw length/diameter ratio (L/D) = 30. The temperature profile used was 170, 180, 185, 200, and 210°C, from feed to die, and the screw rotational speed was 102 rpm. The extruded strands were water cooled, cut in the form of pellets, and then dried.

The pellets were injection molded (Arburg All Rounder M-250) as test samples according to ASTM D 638-02, D790-02, and D 256-02 with a temperature profile of 185, 195, 200, 210, and 200°C, a mould temperature of 20°C and a mould cooling time of 20 s.

Characterization

Thermal analyses of pcPP and their composites were made by differential scanning calorimetry, DSC (TA Instruments 2100) from −50 to 200°C at 10°C/min and inert atmosphere, argon. Thermogravimetry, TGA (TA Instruments 2050) was carried out from 25 to 800°C at 10°C/min (under inert atmosphere, argon, 50 mL/min).

Melt flow indices, MFI, were measured according to ASTMD1238 in a DSM plastometer. The reported values are an average of ten measurements.

The tensile and flexural strength of injection-molded test samples were measured according to

ASTM D 638-02 and D 790-02 standards, respectively, in an EMIC model DL2000 equipment. The impact resistances of the pcPP and the composites were measured (ASTM D 256, method A) with a Tinius Olsen 92T instrument. Ten composite specimens were tested for each set of samples.

The fracture surfaces of cryo-fractured (liquid N_2) injection-molded test samples were sputter-coated with gold, and their surface morphologies were investigated by scanning electron microscopy, SEM, using a JEOL JSM-6360LV (acceleration voltage of 25 kV) instrument.

The water uptake of the composites (sample dimensions = 3.2× 12 × 50 mm) were determined according to ASTM D570. The samples were dried for 1 h at 110°C, cooled to room temperature in a desiccator, weighted, and then immersed into distillated water. Change of weight was checked after: 24 h, 1 week, and 2 weeks; until reaching constant weight. Before weighing, the samples were surface dried using paper tissue.

Results are presented as percent water absorption in relation to the dry weight of the specimens.

RESULTS AND DISCUSSION

Effect of fiber loading

Mechanical properties

The tensile modulus, tensile strength, elongation at break, and Izod impact strength of injection-molded composites using long fibers are illustrated in Figure 3. Observing the test samples, it is possible to verify that the curaua fibers were oriented along the melt flow direction in the injection-molded samples as expected for single gated molds. The addition of 10 to 40 wt % long curaua fibers causes a linear increase of tensile modulus compared with pure pcPP. The composite loaded with 40 wt % of long curaua fibers shows 70% increase in the tensile modulus, Figure 3(a). Similar results were reported for other unmodified lignocellulosic fiber-reinforced composites.[15] Tensile strength presents a small increase (11%) at up to 20 wt % of fiber load, and after that a decrease occurs, Figure 3(b). This behavior can be assigned to the weak adhesion between the neat curaua fibers and the pcPP matrix, which is required for a composite to achieve an improvement in these mechanical properties, precluding an efficient stress transfer at the interface. The reduction of tensile strength above 30 wt % of fiber content can be also assigned to the increasing number of fiber
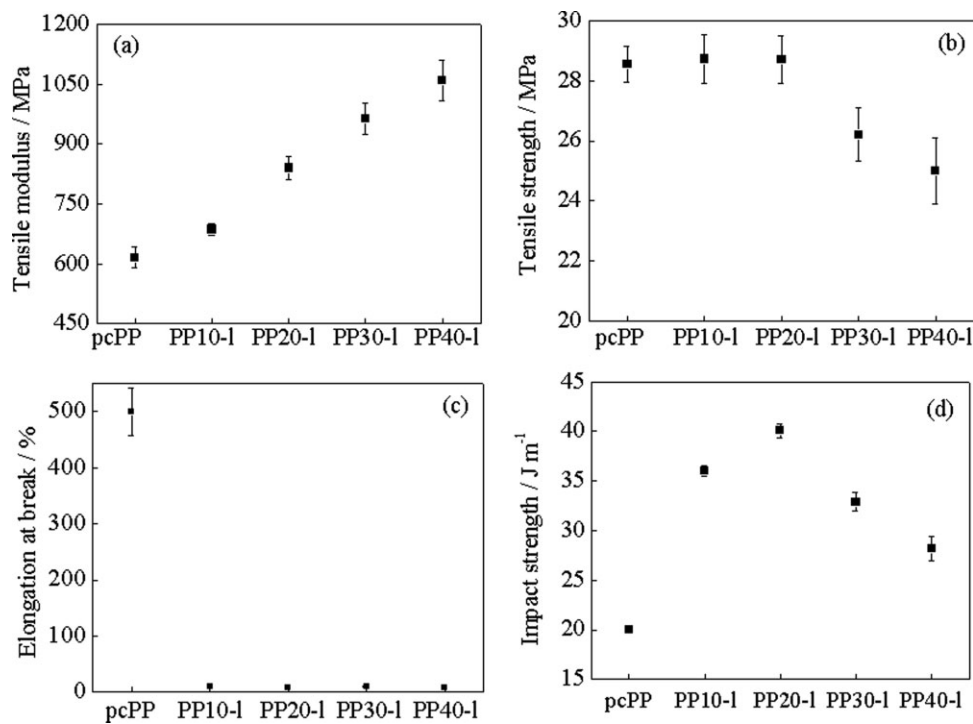
Figure 3 Variation of: (a) tensile modulus, (b) tensile strength, (c) elongation at break, and (d) Izod impact strength, as a function of long fiber content for injection molded test samples.

ends (low aspect ratio) in the composites, which could serve as loci for crack initiation and subsequent failure at low stress. Elongation at break is drastically lower in the composites, when compared with pure pcPP, Figure 3(c), and there were no significant changes in the composites with increase of fiber content. This behavior is expected because lignocellulosic fibers have low-elongation at break and restrict polymer molecules flow.[15–17]

Despite the linear improvement of the tensile modulus, the impact strength of the composites presents a linear increase until a loading of 20 wt % of fibers. Above this value, the impact strength decreases. This can be related to a change from duc-

tile to brittle fracture behavior with the increase of fiber content, thus providing sites for crack initiation. Besides, the probability for fiber agglomeration also increases at high fiber content, creating regions of stress concentration that require less energy to initiate or propagate a crack, Figure 3(d).[14,18]

Thermal properties and melt flow index

TGA curves were used to evaluate the thermal stability of the curaua fibers and the composites. For the fibers, Figure 4 (a) shows an initial weight loss at 75°C (onset temperature), attributed to water loss, and three other processes as follows: the first from
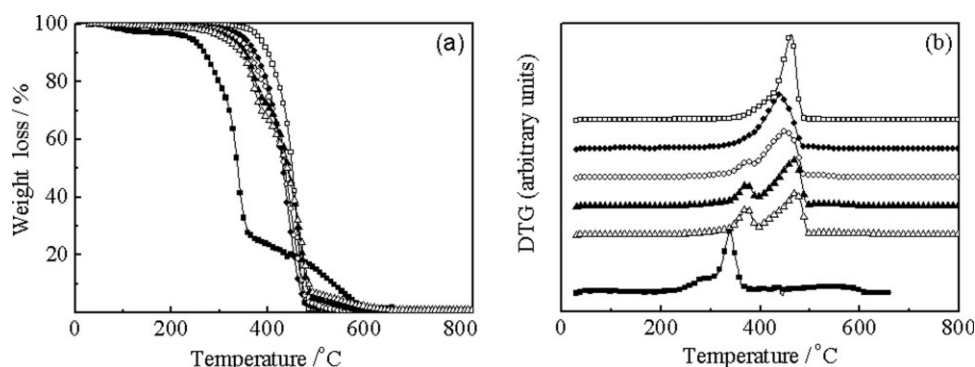

Figure 4 (a) Thermograms (10°C min^{-1} and inert argon atmosphere) and (b) first derivative of the curves: (■) curaua fibers; injection molded test samples of composites with (□) 0, (●) 10, (○) 20, (▲) 30, and (△) 40 wt % of long curaua fibers.
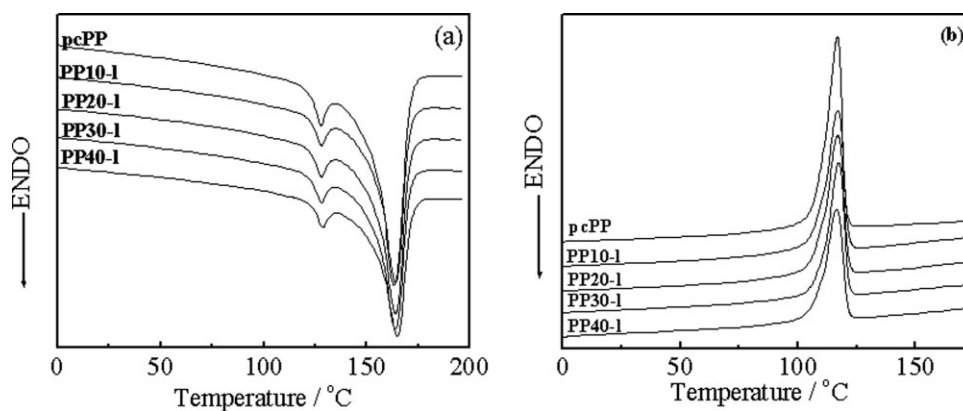
Figure 5 (a) DSC heating curves and (b) DSC cooling curves (10°C min^{-1}) for pcPP, PP10-l, PP20-l, PP30-l, and PP40-l.

236 to 297°C, with its maximum rate at 268°C, attributed to the decomposition of hemicellulose present in the curaua fibers, the second, from 298 to 366°C, with maximum rate at 339°C attributed to the cellulose degradation and, the decomposition of lignin at 439°C.[6] Figure 4(a) also shows the TGA curves for pure pcPP and the composites. These curves show two degradation processes, as evidenced by the first derivative curves, shown in Figure 4(b). The first process can be correlated to cellulose degradation of the curaua fibers (369–372°C) and the second to the pcPP degradation (439–471°C). The temperature where the maximum degradation rate occurs (T_{deg}) was 463°C for pure PcPP. In the composites, this temperature is shifted to 439, 452, 468, and 471°C, as a function of fiber contents from 10 to 40 wt %, respectively. For cellulose, T_{deg} is 339°C and for the composites, this temperature is shifted to 369, 374, 372, and 372°C, as a function of fiber contents from 10 to 40 wt %, respectively.

The shift of the weight loss rate maxima, in the first derivative curves, Figure 4(b), indicates that there is an interaction between the thermal degradation mechanisms of the fibers and of the thermoplastic. The T_{deg} of the fiber shifts to higher temperatures for all composites, whereas T_{deg} corresponding to the polymer shifts to lower temperature for 10 and 20 wt % and to slightly higher temperatures for 30 and 40 wt %. Previous reports suggest that the hindered phenols present in lignin may act as free radical scavengers, retarding the thermal degradation of the polymer.[19]

Analysis of the onset temperature, $T_{1\%}$ (the temperature at which 1% of weight loss occurs), show that these values for the decrease from 398°C for pure pcPP to 293 (pcPP10-l) and 215°C (pcPP40-l). Compared with the pristine fibers ($T_{1\%} = 73$°C), there is a large shift, indicating that the polymer precludes the thermal degradation of the fibers. Similar results were observed by Lei et al.[20] However, the

shifts are significantly larger than those described in the literature.[11]

Melting, T_m, and crystallization, T_c, temperatures (164 and 117°C, respectively) and crystallinity degree of the polymer matrix, obtained by DSC, do not change with fiber content, Figure 5. The decrease of the melting enthalpies of the composites (calculated from the 2nd heating curves) is proportional to the fiber content, within the experimental error. This indicates that the fibers are not acting as nucleating agent. The endotherm observed at 125°C can be assigned to melting of polyethylene, present as contamination of the pcPP.

The MFI of the composites decrease as a function of fiber contents (1.66; 0.90; 0.33; and 0.27 g/10 min), from 10 to 40 wt %, respectively. These values are low, compared with pure pcPP (5.5 g/10 min). The presence of the fibers hinders the mobility of the polymer matrix, reducing the MFI of the composites.

Effect of fiber surface modification using NaOH solution: mechanical properties and morphology

The values of tensile strength, Young modulus, and impact strength obtained for injection molded test samples of the composites with curaua fibers treated with NaOH solution are shown in Table I. These results can be analyzed using for comparison the pure pcPP. For the composites using curaua fibers treated with alkali, an improvement in impact strength is observed, however, no significant improvement of tensile strength and Young modulus occurred. This is an indication of poor adhesion between the phases of the composite. SEM micrographs of the composites prepared with curaua fibers treated with alkali solution confirmed that fiber-matrix adhesion was not improved with the alkali treatment, Figure 6.

TABLE I
Tensile Strength (σ), Young Modulus (E), and Notched Impact Strength (IS) for Injection Molded Test Samples of pcPP and Composites Using Fibers Treated with NaOH Solution

Material	σ (MPa)	E (MPa)	IS (J m^{-1})
pcPP	28.55 (± 0.6)	616 (± 26)	20 (± 0.7)
PP10-l-NaOH-2h[a]	27.6 (± 0.4)	753.2 (± 18.2)	35.8 (± 3.7)
PP10-l-NaOH-4h[a]	27.4 (± 0.4)	746.6 (± 18.8)	37.8 (± 3.4)
PP10-l-NaOH-6h[a]	29.5 (± 0.7)	778.1 (± 21.7)	40.6 (± 3.2)
PP10-l-NaOH-8h[a]	29.8 (± 0.5)	752.5 (± 15.8)	40.6 (± 3.0)
PP10-l-NaOH-12h[a]	30.1 (± 1.2)	748.5 (± 37.7)	45.3 (± 5.5)
PP10-l-NaOH-24h[a]	27.8 (± 0.4)	730.0 (± 14.1)	33.8 (± 5.5)
PP10-L-NaOH-48h[a]	29.3 (± 0.4)	746.0 (± 28.9)	37 (± 1.5)

[a] Treatment time of curaua fiber with NaOH solution in hours.

Effect of fiber length

According to the results presented above, in relation to fiber loading and treatment, the composite using 20 wt % of fibers without treatment showed the best mechanical properties, Figure 3. Consequently, composites with 20 wt % of long and short fibers were prepared to analyze the effect of fiber length on the mechanical properties and water uptake of the composites. The use of long fibers (PP20-l) causes no changes in the tensile strength of the composite comparing to pure pcPP results. On the other hand, when the same content of short fibers was used, a 10% increase was observed, Figure 7(a). For flexural strength, a similar behavior was noted; the use of 20 wt % of long (PP20-l) fibers caused a decrease of 23% in the flexural strength, whereas the short fibers (PP20-s) caused an increase of 35%, when compared with pure pcPP, Figure 7(a).

Several factors affect the mechanical properties of the composites; among them, the dispersion and the interaction between the phases are very important. When the interaction is weak, lack of stress transfer from the matrix to the fibers occurs. The use of long fibers causes no improvement in tensile and flexural strength, which can be related to weak fiber-matrix adhesion. The reduction of fiber length (PP20-s) promotes an increase of tensile and flexural strength and this can be related to more efficient fiber dispersion in the matrix. Fiber length also influences in the flexural modulus; an increase of 53 or 97%, was observed when long or short fibers were used, respectively.

By SEM, we observed that the composites prepared with long fibers (PP20-l), Figure 8(a) show poor dispersion and the fibers were pulled out. The composite prepared with short fibers (PP20-s) present good dispersion and more uniform surface, Figure 8(b), with little fiber pull-out. It is also possible to observe in Figure 8(c) that fibrillation of the fibers is occurring and that the micro fibrils have a diameter of about 3.5 μm.[5] Fibrillation may cause an increase in the aspect ratio [see insert in Fig. 1(a)], improving the reinforcement effect.

Effect of coupling agent

Cellulose presents polar hydroxyl groups on its surface and corresponds to the main component of the lignocellulosic fibers, being incompatible with a nonpolar matrix like polypropylene. Consequently, the composites tend to present weak fiber-matrix adhesion. Different strategies have been used to improve fiber-matrix adhesion as follows: modification of fiber surface or modification of the polymeric matrix.[2] The modification of the polymeric matrix has been reported using different coupling agents, including maleic anhydride.[10,15,18,21]

In this work, 3 wt % MAPP was used as coupling agent for composites prepared with 20 wt % of short curaua fibers (PP20s-MA). This composite presented increases of 30 and 51% in the tensile and flexural strength, compared with pure pcPP, Figure 7(a). In contrast to the results of tensile and flexural
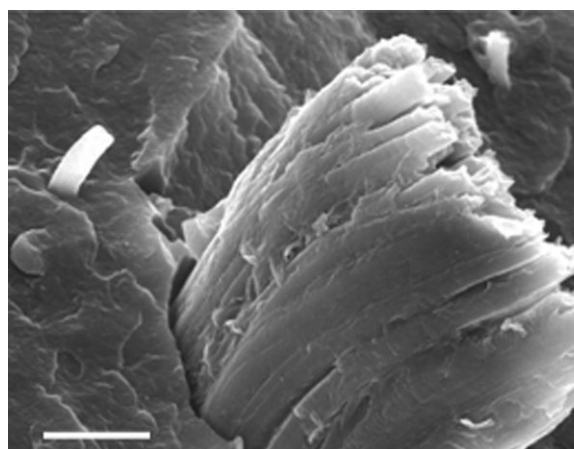

Figure 6 SEM image of pcPP/20 wt % composite prepared with curaua fiber treated for 12 h with NaOH solution. Scale bar: 15 μm.
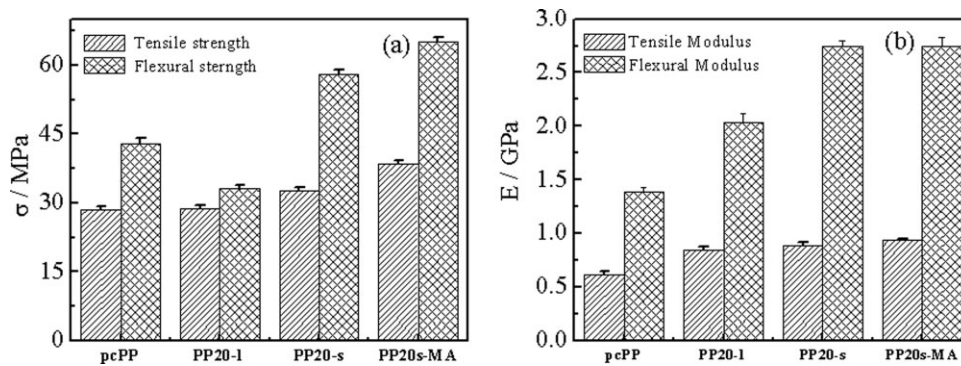
Figure 7   Effect of fiber nature on the mechanical properties of the composites: (a) tensile and flexural strength and (b) tensile and flexural modulus, for pcPP and composites using 20 wt % of curaua fibers.

strength, the tensile modulus is not affected by the presence of the coupling agent. Generally, Young modulus reflects the capability of both fiber and matrix material to transfer the elastic deformation in the case of small strains without interface fracture. Consequently, the tensile modulus is less sensitive to variations of interfacial adhesion than the tensile strength, which is strongly associated with interfacial failure behavior.[18]

The results of the increase of the tensile strength indicate that the coupling agent (MAPP) interacts with the fibers and the matrix creating a favorable interaction between them. A hypothetical model of the interface between MAPP and the hydroxyl groups of the curaua fibers is shown in Figure 9. This model considers that the acidic maleic anhydride groups of MAPP interact with the basic hydroxyl groups of the cellulose, resulting in both covalent interaction (cyclic ester bond) and hydrogen bonds, promoting an anchoring of the coupling agent to the fiber surface. Besides, the PP chain of MAPP diffuses into the PP matrix through interchain entanglements.[18,22]

The values of tensile and flexural strength obtained for the composite of pcPP with 20 wt % of curaua fibers using 3 wt % of MAPP as a coupling
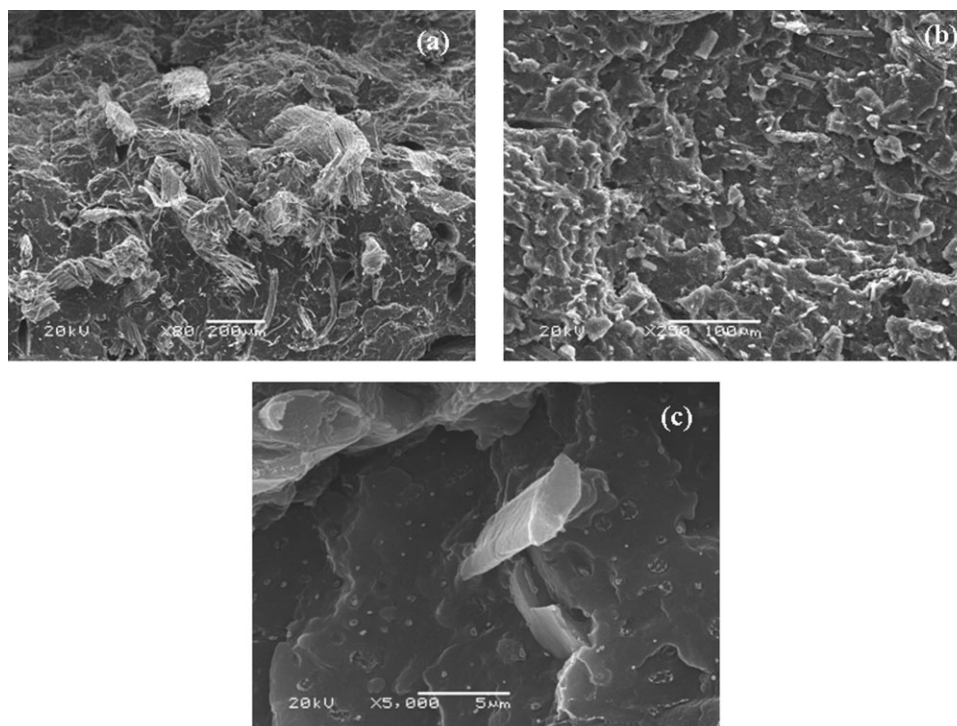

Figure 8   SEM images of pcPP composites containing 20 wt % of curaua fibers: (a) long and (b) short fibers. (Scale bars: 100 μm) and (c) short fibers (scale bar: 5 μm).
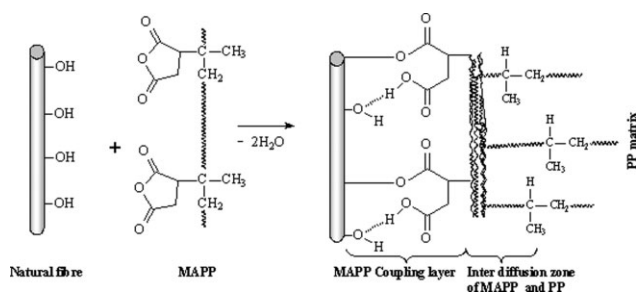
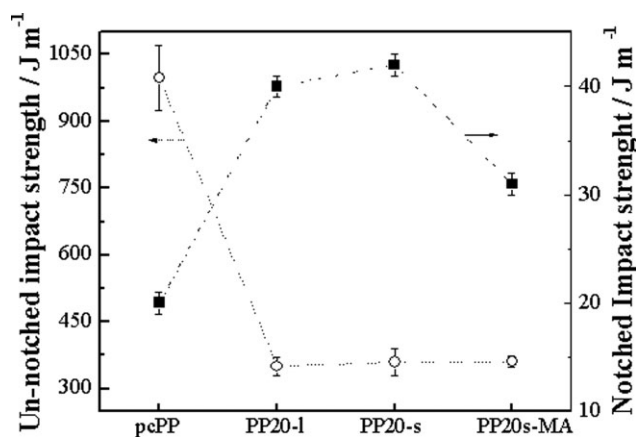**Figure 9** Hypothetical model of the interface between MAPP and hydroxyl groups of the curaua fibers.

Figure 10 Un-notched and notched impact strength of pcPP and composites using 20 wt % of long (PP20-l) or short (PP20-s) curaua fibers and matrix modified with maleic anhydride (PP20s-MA).

agent is similar to composites made using virgin polypropylene matrix reinforced with other lignocellulosic fibers.[11,14–16,18,23,24]

The main goal in the development of reinforced polymers is to improve the stiffness combined with sufficient impact resistance. The pcPP shows an unnotched impact strength of 998 J/m, and it decreases to 350 J/m when 20 wt % of curaua fibers (short or long) are added, Figure 10. The un-notched impact behavior is controlled to a considerable extent by fracture initiation processes that, in turn, are controlled by stress concentration at defects in the system. The presence of curaua fibers in the matrix provides sites for crack initiation. Consequently, there is a little difference between the composites using or not the matrix modified by maleic anhydride.[14]

The notched impact strength is lower than the corresponding un-notched impact strength due to the additional energy required for crack initiation in the un-notched specimen. Notched impact behavior is controlled by factors which affect the propagation of fracture initiated due to stress concentration at the notch tip. Hence the fiber pull-out effect is more distinctly visible in case of notched impact resistance.[14,25,26] The addition of 20 wt % of long or short curaua fibers in the pcPP matrix causes an increase of 100 and 110%, respectively, in the notched Izod impact energy compared with pure pcPP. When the matrix modified with MAPP was used in a composite with 20 wt % of curaua fibers, an improvement of interfacial adhesion occurred and the notched impact strength increased only 55%. This interaction in the interface inhibits polymer mobility and, thereby, reduces the ability of the system to absorb energy during fracture propagation.[27]

Water uptake measurements

Water absorption by the fibers can have undesirable effects on the mechanical properties and the dimensional stability of the composites.[9,28,29] Moisture penetration into composites can occur by three different

mechanisms as follows: diffusion of water molecules inside the microgaps between polymer chains, capillary transport into the gaps and flaws at the interfaces between fibers, and polymer and transport by micro cracks in the matrix, formed during the compounding process.[28]

Figure 11 shows that water is continuously absorbed by each composite up to a certain time. Composites with and without maleic anhydride present similar curves from 0 to 6 months. However, the composites processed with maleic anhydride present lower water absorption in this period, indicating an esterification of hydroxyl groups of cellulose present on the fibers surface, consequently reducing water adsorption. The percentages of water uptake for the composites of pcPP using 20 wt % of
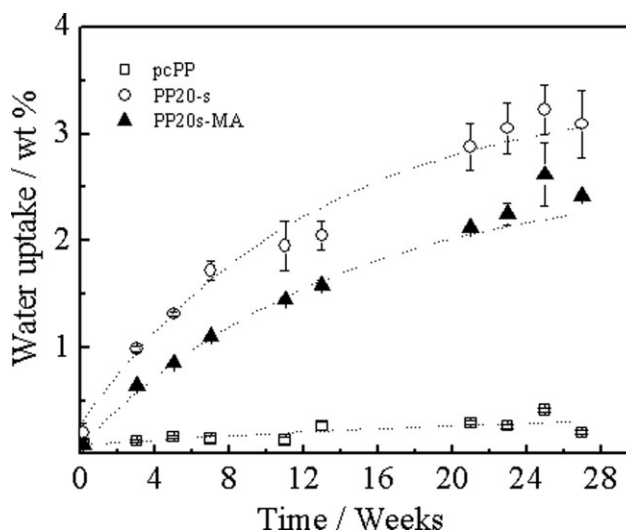

Figure 11 Water uptake as a function of time for pcPP and composites.

curaua fibers and MAPP were lower than the values for composites of polypropylene using other fibers.[30] The water uptake can be seen as an indirect evidence of interfacial adhesion.

CONCLUSIONS

The reinforcement of pcPP with curaua fibers is an alternative to add value to this material, especially when using a continuous processing method like extrusion. The properties of these composites are similar to those of composites using virgin polypropylene. pcPP is partially oxidized and contains polar carbonyl groups, favoring the adhesion of the fibers to the matrix. Concerning the mechanical properties, it is observed that the Young modulus increases linearly as a function of the fiber content. However, the composites are hard and fragile. Improvements of the composite strength properties were observed using the maleic anhydride-grafted polypropylene as coupling agent and using shorter fibers. It is also demonstrated that alkali treatment of the fibers prior to processing does not improves fiber-matrix adhesion. Finally, the mechanical properties of the prepared composites offer reasonable properties, justifying its use in the automotive, appliance, and building construction industries. Injection molding also allow producing composites with complex shapes.

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