Investigation on the Use of Coir Fiber as Alternative Reinforcement in Polypropylene

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ABSTRACT: Polypropylene/coir fiber composites were prepared according to an experimental statistical design, in which the independent variables, coir fiber, and compatibilizer content, were varied. The compatibilizer used was maleic anhydride grafted polypropylene (PP-g-MA). Compatibilizer free composites were also prepared. Composites were processed in a corotating twin-screw extruder and submitted to mechanical and morphological analyses. The effects of the independent variables on the mechanical properties were assessed through tensile strength, elongation at break, flexural modulus, and impact strength. The morphological properties were assessed by scanning electron microscopy (SEM). The results indicated the need for using compatibilizers in the composites due to the incompatibility of PP and coir fiber. The variable with the strongest effect on the properties was coir content, whose increase caused increase in tensile strength, impact strength and elastic modulus, and decrease in elongation at break. The presence of PP-g-MA was fundamental to achieving the aforementioned results. The effect of increasing compatibilizer content was only observed for the elastic modulus. © 2010 Wiley Periodicals, Inc. J Appl Polym Sci 118: 2841–2848, 2010

Key words: natural fiber composites; interface/interphase; statistical properties/methods; mechanical testing

INTRODUCTION

In recent years, there has been considerable increase in research on natural or lignocellulosic fibers to reinforce polymers, due to several positive contributions of these fibers to the composites, such as reduction in density and cost.[1–4] In addition, they present low abrasiveness when compared to synthetic fibers, are recyclable, biodegradable and may be incorporated at high contents, resulting in considerable increase in stiffness. Several types of natural fibers have been studied as thermoplastic reinforcement, including curauá,[5,6] jute, sawdust,[7–9] sisal,[10,11] and coir.[12–19,20–24] The combination of interesting mechanical and physical properties along with an environmental appeal has motivated several industrial sectors, especially the automobile industry, to consider these fibers potential substitutes for glass fibers in environmentally safe products.

Brazil shows great potential as producer of natural fibers, among which coir, which can be extracted either from the green or ripe fruit. Coir consists of lignocellulosic fibers, obtained from the mesocarp of the coconut fruit and makes up about 25% of the nut. Some characteristics of coir are that the fibers are not fragile like glass fiber, not toxic and can be chemically modified.[25]

The increasing consumption of green and ripe coconuts and the industrialization of coconut water have led to increasing production of solid wastes formed by the fibrous outer covering. The use of these fibers may lead to the production of low-cost material and help reduce solid wastes.

Composites of polymer and coir fibers present processing limitations due to the low degradation temperature of cellulose of about 200°C. Therefore, polymers that can be used for composite manufacture should have melt temperature below the degradation temperature of cellulose. One of the most used polymers is polypropylene, because of its intrinsic properties and low melt temperature (165°C), which enables composite processing at temperatures below 200°C, avoiding degradation of the lignocellulosic fibers.[26]

The use of coir fiber as reinforcement in polymers has been the object of several works. The most commonly used matrixes include polypropylene,[12–14,16–19] natural rubber,[20,21] and polyester resins.[22–24]

The effects of coir fiber content and mixing conditions on morphology and mechanical properties of PP/coir fiber composites have been investigated by

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some authors.[13,14] It was reported that incorporation of coir fiber in PP did not result in significant increase in mechanical properties, at the implemented processing temperatures and rotations.

One of the main difficulties encountered in the preparation of polymer composites with lignocellulosic fibers stems from the chemical differences between the phases. The commonly used thermoplastic matrixes consist of nonpolar polymers whereas the fibers consist of polar cellulose, hemicellulose, and lignin. These differences result in poor adhesion between the phases, with high surface tension, rendering an incompatible system. To improve adhesion for adequate stress transfer, compatibilizers, or treatment with coupling agents are required. Another alternative is to promote chemical attack of the fiber surface, rendering it more irregular to facilitate adhesion to the polymer matrix.

In the aforementioned works, the coir fibers were incorporated in the polypropylene matrix without the aid of any compatibilizer or surface treatment of the fiber. However, investigations carried out with other types of lignocellulosic fiber based composites (e.g. sawdust) showed the need for improving adhesion to achieve adequate properties of the composite.[9,27–31] One of the most commonly used compatibilizer in PP/sawdust composites is maleic anhydride grafted polypropylene (PP-g-MA).

The effect of lignin as compatibilizer in PP/coir fiber composites has also been investigated by Rozman et al.[12] This investigation showed that the presence and increase in lignin content increased flexural properties; however no significant increase was seen in tensile properties when compared to the composites without this compatibilizer. For comparison sake PP-g-MA was also used as compatibilizer in this work and the composites compatibilized with PP-g-MA showed better mechanical properties than those compatibilized with lignin.

The objective of this work was to study the effect of coir content and of the presence and content of maleic anhydride grafted polypropylene compatibilizer, on morphology and mechanical properties of PP/coir fiber composites. Composites were prepared in a twin-screw extruder according to a central composite experimental design.

EXPERIMENTAL

Materials

PP powder was supplied by Quattor (Mauá, Brazil), under code HP550K, with melt flow index (MFI) of 4.0 g/10 min. Antioxidant and lubricant incorporated to the formulations were Irganox B215 and calcium stearate, respectively. Irganox B215 is a mixture of a phenolic antioxidant (Irganox 1010) and a phosphite (Irgafos 168) supplied by Ciba Especialidades Químicas (São Paulo, Brazil).

The compatibilizer used was PP grafted with maleic anhydride (PP-g-MA), under code Polybond 3200 (MFI = 110 g/10 min at 190°C and 2.16 kg), supplied by Crompton-Uniroyal Chemical (São Paulo, Brazil).

Green coir fiber with length of 12 to 14 cm was supplied by Projeto Coco Verde (Rio de Janeiro/RJ).

Methods

Grinding of the coir fiber

The green coir fiber was first cut to 6 cm using a guillotine. Next, the fibers were dry ground at 1750 rpm in a MAK-250 grinder from Kie Máquinas e Plásticos (Louveira/SP).

Grinding was performed in two steps. In the first step a 3-cm screen was used and in the second a 0.5-cm screen.

Analysis of the coir fiber

Determination of insoluble lignin content was performed according to ASTM D1106-96. The procedure involved digestion of the fibers in hot water (100°C) for 4 h, followed by drying to remove moisture. After drying, the fibers were transferred to an Erlenmeyer and 15 mL of a 72% sulfuric acid solution was added and maintained under stirring at room temperature for two more hours. Next, 560 mL distilled water were added to dilute the acid solution and the samples were submitted to reflux for 4 h. The solution was filtered in a ceramic crucible and placed in an oven for 18 h at 100°C to dry. The procedure was performed in triplicate.

Preparation of the PP/coir fiber composites

Prior to extrusion, the fibers were dried in an oven at 100°C for 24 h and conditioned in aluminized bags containing moisture and light barrier.

Composite components were weighed and premixed in a Mecanoplast mixer (Rio Claro, Brazil). Formulations were according to a central composite experimental design, where the variables fiber content and compatibilizer content (PP-g-MA) were varied. The experimental design is shown in Table I.

For comparison sake three composites were also prepared without addition of compatibilizer containing 20, 30, and 40% coir fiber (formulations 13 to 15), as well as a formulation of pure polypropylene (formulation 12) and another with maximum concentration of compatibilizer used in the experimental design (formulation 16), as shown in Table II.

The extruder used was a corotating twin-screw extruder model ZSK26P10.6 (year of construction

TABLE I
Central Composite Design

Experiment	C_{CF} (%)	C_C (%)
1	20	4
2	40	4
3	20	8
4	40	8
5	30	6
6	30	6
7	30	6
8	15.86	6
9	30	8.83
10	44.14	6
11	30	3.17

C_{CF}, coir fiber concentration; C_C, compatibilizer concentration.

2006), from Coperion Werner & Pfleiderer GmbH & Co., with diameter of 26 mm, L/D = 44 and degassing zone.

Composite extrudates were then cooled in air and ground in a P1001 FDR Primotécnica grinder (Mauá, São Paulo). The process conditions in the extruder were:

Temperature profile: 170, 175, 180, 180, 180, 180, 185, 185, 190, 190, 190°C.

Screw frequency: 250 rpm.

The ground composites were injection molded into tensile, bending, and impact specimens according to ASTM D638, D790, and D256, respectively, using a TM 750/210 Battenfeld Injection Molding GmbH molding machine at a pressure of 80 bar, with temperature profile of 190, 190, 190, and 195°C in zones 1, 2, and 3 and nozzle, respectively.

Analysis of the extruded composites

Tensile and bending tests were conducted in a 5565 Instron Universal Testing Machine (Norwood, U.S.A.), according to ASTM D638 and ASTM D790, at test speeds of 5 mm/min and 1.3 mm/min, respectively.

Fractured surfaces of the tensile tests were coated with platinum and analyzed in a LEO Stereoscan 440 scanning electron microscope, SEM, (Cambridge, U. K.) under high vacuum (5×10^{-6} torr).

Impact tests were carried out according to ASTM D256 in a Monitor/ Impact Tester, model 43-02, from Testing Machines Inc.

Coir fiber size analysis after processing

Extruded and subsequently injected specimens were submitted to the following methodology to determine fiber length (L) and diameter, and consequently aspect ratio (L/D): tensile specimens were solubilized in xylene at 120°C under reflux for 1 h.

Next, the samples were filtered in a preheated Büchner funnel (to prevent precipitation of PP). The filtered coir fibers were then vacuum dried at 80°C for 1 h. The dried coir fibers were then analyzed using a LEO Stereoscan 440 scanning electron microscope, SEM, (Cambridge, U.K.) under high vacuum (5×10^{-6} torr). Two hundred measurements were made of fiber length and thickness.

The coir fibers are not perfectly circular, as shown by SEM, and hence diameter measurements refer to the smallest dimension normal to the fiber axis.

RESULTS AND DISCUSSIONS

Analysis of the coir fibers

The various types of lignocellulosic fibers (coir, curuauá, sisal, sawdust, etc) present significantly different contents of lignin, cellulose, and hemicellulose, rendering very different adhesion and mechanical properties to the composites, as fibers containing higher lignin content tend to present improved compatibility towards nonpolar polymers, as lignin is the most nonpolar constituent of the fiber. Climatic and soil conditions also tend to affect fiber properties.[25] It was therefore decided to determine lignin content of the coir fibers used in this study.

Average insoluble lignin content, determined by the previously described methodology, was found to be 32.3% with standard deviation of 0.32 (data were obtained in triplicate). This value is inferior to those encountered in the literature, i.e., ranging from 36 to 47%.[20,22,32,33] These differences may be attributed to fiber nature, which is a consequence of cultivation region, climatic and irrigation conditions, plant age, and coconut species.

Average fiber length may significantly affect the mechanical properties of the composite. Works performed with PP/coir fiber composites[14] showed that elevated amounts of fibers with lengths inferior to 1 mm resulted in low mechanical strength of the noncompatibilized composites. Aspect ratio of the coir fibers in the composites was obtained from SEM analysis.

Figures 1 and 2 contain results of length and aspect ratio of the fibers extracted from injected composite specimens, as previously described. Average fiber length was 827 ± 365 μm and average aspect

TABLE II
Reference Formulations

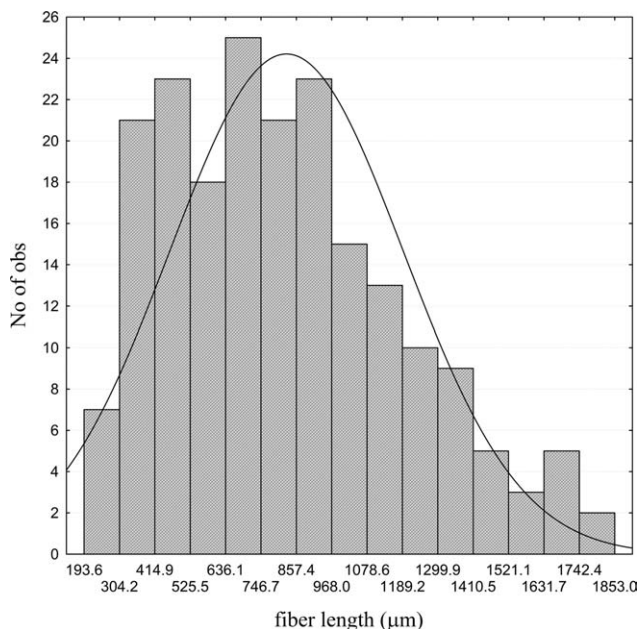| Experiment	C_{CF} (%)	C_C (%)
12	–	–
13	20	–
14	30	–
15	40	–
16	–	8.83

Figure 1 SEM analysis of fiber length, obtained from the injection molded specimens.

ratio 5.2 ± 2.2, indicating short fibers with large dispersion in length and aspect ratio.

Micrographs of the coir fibers are shown in Figure 3. The micrographs also show nonuniformity in fiber size and flattened (i.e., nonspherical) shape of the fibers.

Analysis of the PP/coir fiber composites

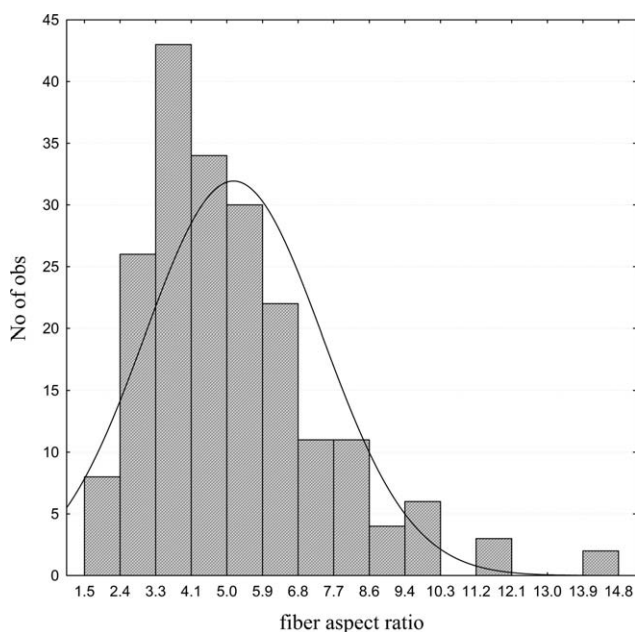The tensile, bending, and impact test results of the PP/coir fiber composites, are shown in Table III.

Figure 2 SEM analysis of fiber aspect ratio, obtained from the injection molded specimens.
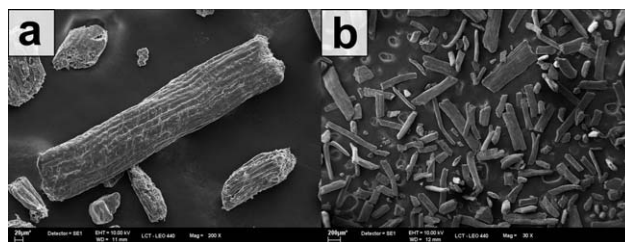
Figure 3 SEM micrographs of the coir fibers.

First, the effect on the mechanical properties when incorporating coir fiber in PP without compatibilizer will be assessed. Experiments 12 to 15 (Table III) show that incorporation of the coir fiber resulted in decrease in tensile strength and elongation at break. This behavior can be explained by the fact that as the coir fibers are polar by nature and PP is a nonpolar polymer, there is no adhesion at the interface and the fibers act as stress concentrator in the composite due to the difficulty to transfer loads during the tensile test. This fact is corroborated by SEM images shown in Figure 4(a,b), where it can be seen that the fibers are detached from the matrix and were pulled out during the test, because of the lack of adhesion at the interface.

In addition, these data corroborate studies by Ishizaki et al.,[14] as average fiber length of the composites was smaller than 1 mm (827 ± 365 μm), leading to reduction in tensile strength, in the absence of compatibilizer.

It can be seen that incorporation of the coir fibers as well as increasing fiber content increases elastic modulus, because mobility in the amorphous region becomes increasingly restrained as the fibers are stiffer than the polymer matrix.

Increase in coir content is seen to increase composite impact strength, corroborating observations of Wambua et al.[13] However, only at coir content above 30% composite impact strength exceeds that of pure PP.

To increase adhesion between the phases and reduce interfacial tension and consequently achieve higher tensile strength and elastic modulus, maleic anhydride grafted polypropylene was added to the composites as compatibilizer.

The data presented in Table III were submitted to multivariable analysis and allowed to obtain the equations that describe the behavior of each one of the independent variables (fiber and compatibilizer content) on the investigated responses (tensile strength, elongation at break, flexural modulus, and impact strength), for significance level of 5% using Statistica® software.

Table IV lists the coefficients of the equations fitted by the multivariable analyses. These coefficients enabled construction of the surface responses

TABLE III
Results of the Tensile, Flexural, and Impact Tests for the Investigated Samples

Experiment	C_{CF} (%)	C_C (%)	Tensile strength (MPa)	Elongation at break (%)	Flexural modulus (MPa)	Impact strength (J/m)
1	20	4	36.40	5.15	2134.0	19.6
2	40	4	43.14	3.22	2846.1	25.3
3	20	8	36.63	5.94	2139.0	16.8
4	40	8	42.34	4.08	2737.9	25.8
5	30	6	39.60	4.66	2485.9	23.7
6	30	6	39.81	3.73	2478.1	22.1
7	30	6	39.86	4.3	2471.4	20.9
8	15.86	6	35.15	6.45	1969.9	19.3
9	30	8.83	40.09	3.76	2456.7	24.1
10	44.14	6	45.59	2.96	2988.7	25.6
11	30	3.17	39.10	4.21	2408.8	24.3
12	--	--	32.04	508.31	1599.4	24.4
13	20	--	28.26	4.43	2097.8	20.6
14	30	--	27.72	2.79	2435.2	25.2
15	40	--	26.48	1.68	2833.7	28.4
16	--	8.83	31.89	500.31	1570.2	26.5

graphs. The fitted coefficients from the multivariable analysis show that the independent variable, coir fiber content, affects all dependent variables, i.e. all investigated properties. However, the only dependent variable affected by compatibilizer concentration was flexural modulus.

Figures 5 and 6 present the surface responses of tensile strength and elongation at break, when the independent variables, coir content, and compatibilizer content, were varied.

It can be seen in these figures that in the presence of compatibilizer an increase in coir fiber content leads to an increase in tensile strength and a reduction in elongation at break and that the increase in amount of compatibilizer (PP-g-MA) does not alter

Figure 4 SEM micrographs of the fractured surfaces in the tensile tests of the samples: F14, PP/coir fiber: 70/30 (magnification 50 X (a) and magnification 150 X (b)); F5, PP/coir fiber/PP-g-MA: 64/30/6 (magnification 50 X(c) and magnification 150 X (d)).

the investigated properties. This analysis shows the importance of the addition of the compatibilizer in reducing interfacial tension, with consequent increase in adhesion between the phases. When the coir fibers were added to PP without the presence of compatibilizer (experiments 13–15), tensile strength was seen to reduce with the increase in fiber content. So, although the effect of the independent variable compatibilizer content was not observed, its presence is necessary. It is also verified that for the investigated coir fiber contents and processing used the lower level of PP-g-MA content would be sufficient to achieve good adhesion. The incorporated compatibilizer (PP-g-MA) presents a polypropylene backbone and short branches of maleic anhydride.[34] In this way, the main chain will be compatible with PP and the polar branches may interact or even react with the polar groups of the coir fibers.

To corroborate this behavior, SEM analyses are shown in Figure 4, where samples a and b present PP composites with 30% coir fiber, without compatibilizer, and samples c and d present the composites compatibilized with 6% PP-g-MA. This figure clearly shows the adhesion provided by the compatibilizer (c, d); the fibers are seen to be well adhered to the PP matrix, ruptured flush with the polymer surface, showing no disengagement, whereas fibers are sticking out from the PP in the noncompatibilized composites, indicating poor fiber–matrix adhesion (a and b).

The micrographs, shown in Figure 7, suggest that increase in compatibilizer content did not alter adhesion, for the investigated levels, indicating that the lowest level of PP-g-MA would be sufficient to compatibilize the investigated composites.

It is worth noting that the increase in tensile strength obtained with the coir fibers, in the presence of compatibilizer, was very significant. In
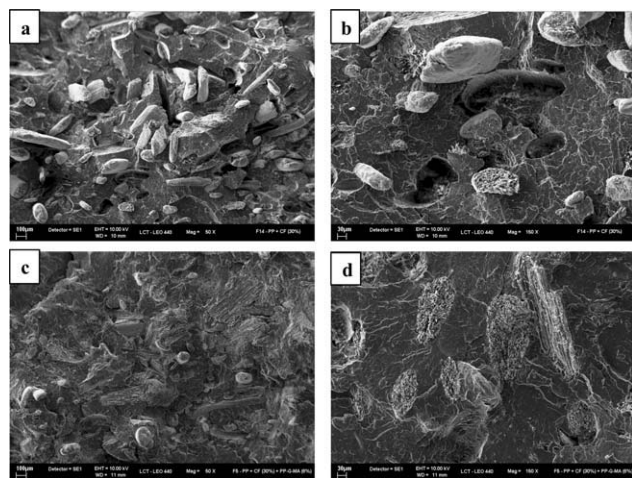
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TABLE IV
Coefficients of the Fitted Equations Obtained from the Experimental Design to Construct Response Surface Plots of the Composites[a]

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Property	R^2	Linear parameters			Quadratic parameters			Cubic parameters	
		a_0	a_1	a_2	a_3	a_4	a_5	a_6	a_7
TS	0.97190	29.58642	0.34018	–	–	–	–	–	–
%E	0.79448	7.67755	−0.10907	–	–	–	–	–	–
Modulus	0.99928	−677.281	125.5492	649.7755	−1.41435	−2.91762	−101.5543	0.032418	5.342875
IS	0.75492	13.67284	0.294027	–	–	–	–	–	–

[a] Polynomial equation: $a_0 + a_1C_{cf} + a_2C_c + a_3C_{cf}C_c + a_4C_{cf}^2 + a_5C_c^2 + a_6C_{cf}^3 + a_7C_c^3$ TS, Tensile Strength; %E, % Elongation at Break; Modulus, Flexural modulus; IS, Impact Strength.

relation to pure PP, the increase was 34.6% and, in relation to the noncompatibilized composite, at the same level of coir fiber, the increase attained 62.8% (sample 2 in relation to sample 15). Taking into account that in countries like Brazil where consumption of coconut water is very high and where the abundantly disposed coconut husks are becoming an environmental problem, the use of coir fibers as reinforcement has become a feasible potential. Even though the production of coir fibers from coconut husks involves costs, these may be offset by the abundant availability of the coconut husks, which are currently considered residues to be disposed of.

The tensile test results obtained in the present investigation are quite high when compared to those reported in other works[12,13,18,19] for similar fiber contents and even compared to those using PP-g-MA as compatibilizer. This fact might be attributed to the rigorous control of the composites processing conditions, which include: drying of the fibers before processing; extrusion in corotating twin-screw extruder with high length to diameter ratio (L/D = 44), which enables adequate mixing; drying of the

extrudates with air instead of water; and the use of injection molded specimens instead of compression molded ones. Several previous investigations analyzed properties of composites obtained in either internal mixers or single-screw extruders,[12,14,16–19] in which mixing tend to be less efficient than in twin-screw extruders. As a consequence, due to the low amount of material produced, test specimens were obtained by compression molding. Adequate processing is therefore of great importance for efficient action of interface agents

Figure 8 shows the response surface graph of the response flexural modulus when coir fiber and compatibilizer (PP-g-MA) content were varied. Both coir content and compatibilizer content are seen to affect flexural modulus, at a significance level of 5%. The increase levels were very proximate for both the compatibilized and noncompatibilized composites. Despite the fact that the statistical analysis indicated that compatibilizer content affects elastic modulus of the composite for a significance level of 5%, it was fitted to a third order model which showed that
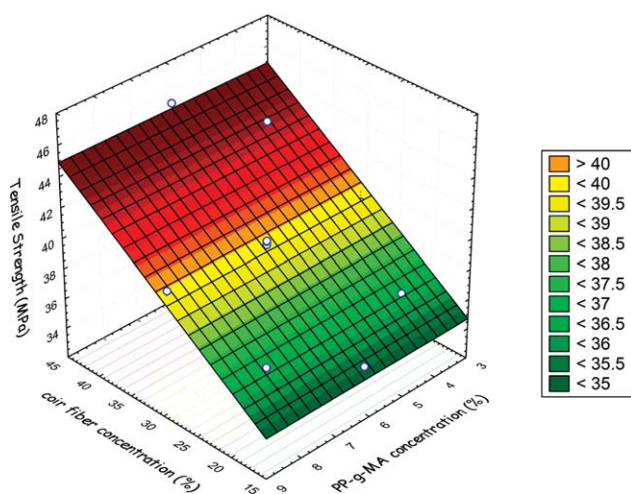

Figure 5 Effect of the variables fiber content and compatibilizer content (PP-g-MA) on the tensile strength of the composites. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]
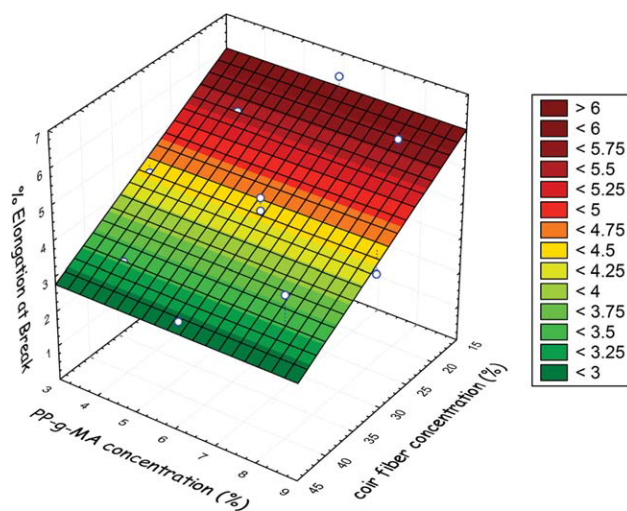

Figure 6 Effect of fiber and compatibilizer content (PP-g-MA) on % elongation at break of the composites. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]
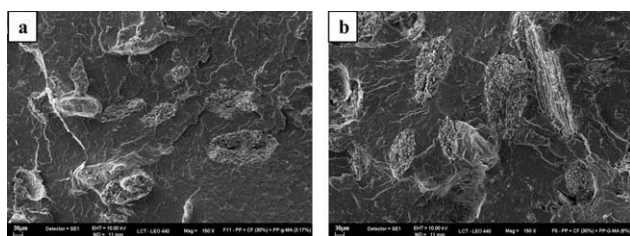
Figure 7 SEM micrographs of the fractured surfaces in the tensile tests of the samples: (a) F11: PP/coir fiber/PP-*g*-MA: 66.83/30/3.17; (b) F5: PP/coir fiber/PP-*g*-MA: 64/30/6.

elastic modulus was less affected by compatibilizer content than by coir fiber content.

Incorporation of 20 wt % coir fibers into PP, without compatibilizer, initially causes reduction in impact strength, however at higher contents of 30–40% impact strength increases (experiments 12–15, Table III). The increase in impact strength might be explained by an increase in the dissipation of energy brought about by fiber debonding and pull-out, causing reduction in tensile, yet increase in apparent impact strength. It should be mentioned that during fiber pull-out from the matrix energy may be dissipated by mechanical friction.[35]

On analyzing impact strength in the presence and absence of compatibilizer, it can be seen that for the same fiber content, the presence of compatibilizer reduces impact strength. For instance the composite containing 40 wt % fiber without compatibilizer (experiment 15, Table III) exhibits impact strength of 28.4 J/m, whereas composites containing the same amount of fibers but with compatibilizer (experiments 2 and 4, Table III) exhibit values of 25.3 and 25.8 J/m, respectively. This fact indicates that the amount of energy dissipated through the previously mentioned mechanisms decreased, suggesting increased adhesion between the fibers and the ma-
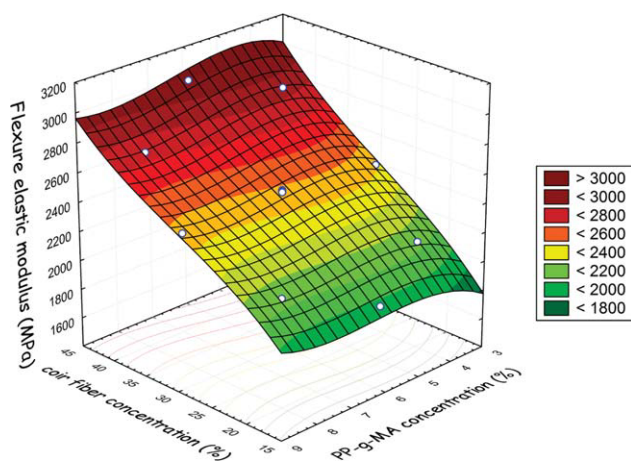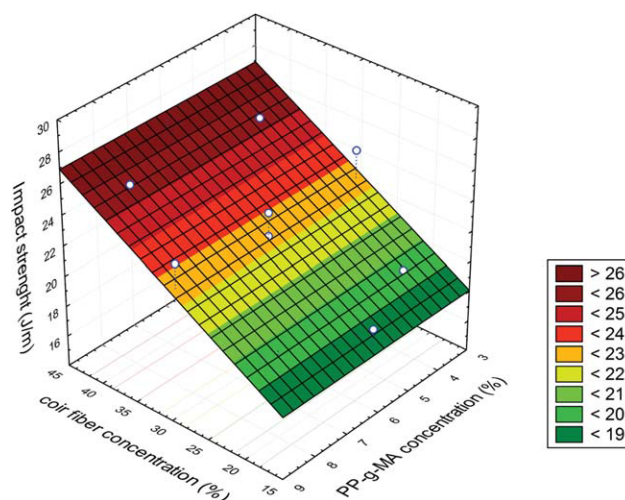

Figure 9 Effect of the variables fiber and compatibilizer content (PP-*g*-MA) on impact strength of the composites. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

trix. However analysis of Figure 9 shows that increase in compatibilizer content does not alter impact strength whereas fiber content increases this property. This increase is a result of both the debonding and fiber pull-out process; however, due to the lower increase in relation to that observed in the absence of compatibilizer, it can be asserted that the presence of the compatibilizer causes improved adhesion at the composite interface.

Hence, incorporating coir fibers in PP in the presence of compatibilizer may result in composites with improved tensile strength and elastic modulus, and reduced impact strength.

CONCLUSION

Incorporation of coir fibers in polypropylene showed to be feasible from the viewpoint of processing, morphology, and mechanical properties.

Composites processed without the presence of compatibilizers present poor adhesion of the fibers to the matrix due to the polarity difference between the phases, causing reduction in tensile strength.

However, addition of the PP-*g*-MA compatibilizer showed to be adequate and necessary for obtaining PP/coir fiber composites with high tensile strength.

The presence of PP-*g*-MA increased tensile and reduced impact strength of the composites. However, the increase in PP-*g*-MA concentration did not cause variation in these properties, indicating that the lowest concentration used was sufficient to compatibilize the composites.

The high tensile strength values of the compatibilized PP/coir fiber composites indicate the importance of using a twin-screw extruder and of rigorous control of the manufacture process.

Figure 8 Effect of the variables fiber and compatibilizer content (PP-*g*-MA) on flexural modulus of the composites. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

The main conclusion is the feasibility of using natural fibers, obtained from a renewable source, which is considered waste from consumption and industrial processes, as polymer reinforcement.

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