Key Engineering Materials Vol. 668 (2016) pp 28-38
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doi:10.4028/www.scientific.net/KEM.668.28

Submitted: 2015-04-30
Accepted: 2015-06-09

Preparation of Polypropylene Composites with High Levels of Coir Short Fiber for use in Products by Injection

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Keywords: fibers, coir, polypropylene, composites.

Abstract. The main objective of this investigation was to study the properties of composites of polypropylene (PP) containing different proportions (20, 40 and 60% wt%) of coir short fiber (natural vegetable fiber) without treatment of fibers, for use in products by injection with applications in the automotive industries, construction and other segments. Samples were prepared in a only stage using a high intensity thermokinetic mixer (K-Mixer). Additives were used in the mass fraction of 3 wt% compatibilizer (PP-g-MA), 2.2 wt% processing additive and 0.12 wt% thermal antioxidant. The composites were characterized by tensile test according to ASTM D638-10. The surface properties of the polymeric matrix with additives were studied by determining the contact angle (CA) in a sessile drop tensiometer and the carbonyl index (CI) by Fourier-transform infrared spectroscopy (FTIR). Thermal properties of the PP and the composition were evaluated by thermogravimetric test, and the interface of the fiber and the matrix in the composites were evaluated using images from scanning electron microscopy (SEM). The CA analysis showed that the PP matrix with additives has become less hydrophobic and the FTIR and the CI that there was a better stabilization of the PP with additives. There was an increase in thermal stability of the composites for all fiber content, which was up to 15 °C above PP for coir fiber composites. In the Young's modulus values showed that the inclusion of fibers reinforced the polymeric matrix and increased the stiffness of the composites, especially in composites containing 60% (wt%) in which the values were ~1.7 times greater than the polypropylene. Images of micrographs showed the interaction of the fiber in the matrix and that despite the hydrophilic character of the fibers and hydrophobic character of the PP, the composites showed non-homogeneous interfaces. These findings confirm the feasibility of using high level of coir fiber in polypropylene composites even without pretreatment of the fibers and the preparation of samples by injection.

1. Introduction

In the last 20 years there has been considerable progress in the development of new polymeric materials, particularly composites reinforced with natural fibers and minerals, for applications in aero-naval, petrochemical, marine and automotive industries, as well as in construction [1-6].

Composites are considered to be better than single component polymeric matrices because of their improved ability to distribute stress throughout the matrix. This is achieved by incorporating a second dispersed phase that produces a higher tensile modulus, resulting in enhanced mechanical properties, especially the elasticity (stiffness) and resistance to breakage. This reinforcing effect can be achieved by adding natural or synthetic fibers to obtain materials with excellent structural performance (mechanical strength), low weight and low cost [7]. Natural fibers can be obtained from wood, vegetable fibers, animal fibers and mineral fibers, all of which vary in shape, dimension and morphology [7, 8]. Thermoplastic polymers such as nylon, polypropylene and polyethylene, as well as thermosetting unsaturated polyester and epoxy resin, have been used in composites with natural fibers originating from wood, pineapple, flax, sisal, coir, piassava, wheat straw, palm, jute and curaua [2, 6, 9-17].

The use of natural fibers to reinforce polymer matrices is particularly important because of the environmental, socioeconomic and developmental implications for a sustainable society that increasingly seeks to obtain renewable, biodegradable and non-toxic materials with minimal energy consumption and a reduction in inappropriate waste disposal and recycling [5, 7, 18-20].

Coir or coconut is the fruit of the coconut palm tree (*Cocos nucifera L.*), which belongs to the sub-family *Cocoideae*, family *Palmae* and Monocot Class. The coir fiber is extracted from the mesocarp and the amount of fiber obtained from each fruit is substantial, generally in the neighborhood of 25% by mass. World production of coir fiber in 2011 was 60 million tones. The leading global producers of coconuts that year were Indonesia 19.5 million tones, Philippines (15.3 million tones) and India (10.9 million tones). Brazil produced 2.7 million tones, representing under 0.5% of global output. Nevertheless, Brazil is the leading producer in South America, accounting for 87.6% to the continent’s output. Within the country, the northeastern region is responsible for 70% of coconut production, and that region’s states of Bahia, Sergipe and Ceara account for 50% of national production [21].

Numerous studies have compared various methods for obtaining composites with satisfactory properties. The use of internal mixers, calendar-type, thermokinetic mixers and mono or twin screw extruders [16, 22, 23]. In addition, various studies have examined the effect of using coupling additive or coupling agents to improve the interaction at the fiber–matrix interface, because of the chemical incompatibility between hydrophilic natural fibers and the hydrophobic thermoplastic matrices. The actual mechanism of interaction of this compatibilizers with the fiber/filler is generally assumed that maleic anhydride (MA) grafted polyolefins like polypropylene (PP-g-MA) interact with the natural fiber/filler via ester bonds and/or hydrogen bridges [3, 24, 25]. Other important aspects are the influence of the content, the dimension and the existence of pre treatment of the fibers [3, 23, 24, 26].

To evaluate the use of coupling agents, Leblanc et al. prepared composites of PP reinforced with 30% (wt%) of green coconut fiber of Brazilian origin, by means of melt blending in a Haake mixer, and studied their properties in the melted and solid states using mechanical assays and scanning electron microscopy (SEM). The effect of fiber load with addition of 1 to 3% of PP-g-MA was also investigated. This addition was found to improve the wettability of the fibers by the melted polymer, but the effect was so small that the specific chemical reactions could not be considered efficient. The increase in the flexural modulus confirmed the strengthening effects of the fiber [13].

The search for composites with high fiber content has been desired and in this sense Bledzki et al. [12] compared the properties of polypropylene composites containing 40% (wt%) coir fibers, grain and wood in the absence and presence of 5% (wt%) polypropylene with maleic anhydride (PP-g-MA) as a coupling agent additive. Analysis of the mechanical, chemical, thermal and morphological properties of these composites showed that the tensile strength of a composite containing wheat fiber was 10% higher than that of a composite containing wood fiber. For elongation at break, the inclusion of coir fiber resulted in values 80% higher than for wood, whereas for wheat the increase was only 40% compared to wood fiber. Similar behavior to the foregoing was noted for the Charpy impact strength, although the actual percentage increase was lower (only 20% and 35% higher than wood fiber, respectively). The differences in the properties of these composites, including tensile strength, were largely attributed to variations in the morphological form and surface properties of the fibers.

According to Santos et al. [27], fibers with lengths below 10 mm can be classified as short. In the study, the authors prepared composites of PP with coir fibers and highlighted the difficulties of feeding extruders and injection molding machines with longer fibers. They also observed in processing composites in a twin-screw extruder with the addition of 30% coconut fibers that at higher temperatures there was an increase in the polymer's fluidity and this effect diminished the shear forces on the reinforcing fibers, minimizing their breakage during processing. They theorized that this can make alignment of the fibers during injection easier, producing an in increase in rigidity of the material, i.e., its elastic modulus.

Despite the various studies [11, 12, 26-30] mentioned above, there are few studies comparing composites containing different fiber types, particularly for contents >40% wt%. In this study, we examined the properties of polypropylene composites containing coir fiber at high contents (up to 60% wt%) with no pre treatment of the fibers (fibers in nature) and in the short length. The properties of these composites were compared with those of composites containing similar amounts of calcium carbonate that is a traditional mineral filler.

2. Materials and methods

2.1. Materials

Polypropylene (PP) indicated to compounds and injection molding processing (type CP 202XP; melt flow index: 26 g.10 min^{-1} as determined by ASTM D1238) was supplied in pellet form by Braskem S.A. (Triunfo, RS, Brazil).

The coupling agent polypropylene with maleic anhydride (PP-g-MA) (commercial name - Licocene PP MA 7452 TP, supplied in granular form; Clariant S/A, São Paulo, SP, Brazil). Licolub H12 (Clariant S/A, São Paulo, SP, Brazil) and calcium stearate (Chemson Ltda., Rio Claro, SP, Brazil) were used as external and internal process lubricants, respectively, and Hostanox (Clariant S/A, São Paulo, SP, Brazil) was used as an thermal antioxidant.

2.2. Processing of composites

The composites of PP with fibers coir (in natura) in the contents 20%, 40% and 60%, wt%, as shown in Table 1, were processed using a thermokinetic type K-mixer (model MH-1000 CT, MH Equipamentos Ltda., Guarulhos, SP, Brazil) fitted with a probe temperature control module for generating graphical processing parameters and a system for removing moisture from the materials during processing.

The coir fibers were freshly (untreated) ground in a model TE-625 mobile and stationary slicer (Tecnal Ltda., Piracicaba, SP, Brazil) and subsequently passed through a sieve with a manually set range of <=3 mm (8 mesh).

The samples of composites were prepared in batch reactors (four loads of each composition) and the raw materials were weighed and processed with direct feeding of the materials. The maximum processing temperature was 190 °C, the processing speeds were 3800 rpm and 3000 rpm and the moisture was removed during processing of the composite.

Table 1 – Fractional weight of components used to prepare the polypropylene (PP) composites.

Components	**Proportion of components used in sample preparation**		
	Fiber content or mineral filler (% w/w)	**PP (% w/w)**	**Additives***
PPadded	0.0	94.68	0,2% Calcium stearate 0,12% Antioxidant Hostanox 2% Licoclub H12 3% Licocene PP MA 7452
Coir Fiber	20.0	74.68	
	40.0	54.68	
	60.0	34.68	

*** All composites contained the same percentage of additives.**

2.3. Specimen preparation

After preparation, the composites, in the form of solid mass, were ground in a mill (model P2005G 20CV, Primotécnica Mecânica e Eletricidade Ltda., Mauá, SP, Brazil) fitted with three rotating knives, two fixed knives and a fixed screen with 8 mm of diameter holes. Subsequently, the specimens were processed using an injection molding machines (model PIC-BOY 22, Petersen & Cia. Ltda., Sorocaba, São Paulo, Brazil). The injections were done according to ASTM D638-10 and ASTM D256 Type IV. The total cycle time for injection was 30 s and the temperatures used in zones 1 (nozzle), 2 and 3 were 210 °C, 210 °C and 180 °C, respectively.

2.4. Contact angle

The contact angle static of the polymeric matrix (pure PP, PP after mixing (no additives) and the PP mixing with the additives were determined using the sessile water micro drop technique in an optical tensiometer (Attension Theta, Espoo, Finland) as the average of five measurements.

2.5. Fourier-transform infrared spectroscopy (FTIR)

FTIR spectra of the polymeric matrix (pure PP, PP after mixing (no additives) and the PP mixing with the additives were obtained from the FTIR Frontier Perkin Elmer equipment with scanning from 600 to 4000 cm^{-1} and 10 scans using an ATR accessory and the data processed with Pyris software and Origin 8.

2.6. Thermogravimetric analysis (TGA)

The TGA analysis were run on a device model STA 449 F3 Jupiter from Netzsch under N_2, using heating rate of 10 °C min^{-1} in the range 25-800 °C and the data processed with Proteus software and Origin 8.

2.7. Tensile test

The tensile strength was determined in a universal mechanical testing machine (model 5569, Instron, São José dos Pinhais, Paraná), according to ASTM D638-10, using type IV specimens and a platter speed of 25 $mm.min^{-1}$ with ten determinations [31].

2.8. Scanning electron microscopy (SEM)

The surfaces of the composites were investigated by microscopic observation JEOL model JSM 6010 LA, in injected samples freeze-fractured after 10 minutes in N_2, in the uncoated sample, SEI mode, at 5 mm away and 1kV.

3. Results and discussion

3.1. Contact angle

The influence of additives, especially the compatibilizer (PP-g-MA), on the surface properties of the composites was assessed by determining the contact angle using the sessile micro drop technique. Fig. 1 shown the static contact angle (theta) for pure polypropylene and polypropylene processed with and without additives. Processing and the inclusion of additives reduced the contact angle. This reduction indicated that the presence of additives reduced the hydrophobicity of polypropylene, i.e., there was greater wettability with respect to water. This enhanced hydrophilicity presumably allows greater interaction of the matrix with vegetable fibers containing free hydroxyl groups, thereby resulting in a composite with improved properties. The increased wettability of polypropylene samples processed with additives may also be attributed to the polar groups existing in the compatibilizing agent, stabilizer and lubricants. Similar results were reported by Cato [32], who evaluated the influence of different percentages (1, 3, 5 and 7%) of a coupling agent (PP-g-MA) in a polymer matrix. Up to 3% coupling agent resulted in a gradual reduction in the contact angle whereas at percentages >3% the values increased, which suggested the formation of unreacted residues of groups of the compatibilizer.

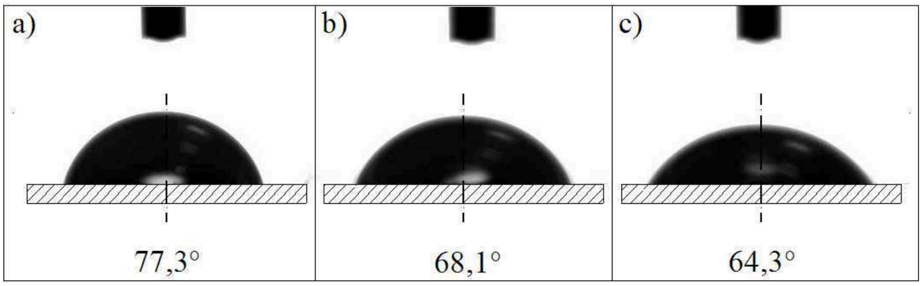


Fig. 1 – The contact angle for the surfaces of (a) pure polypropylene, (b) processed polypropylene and (c) polypropylene with additives.

3.2. Fourier-transform infrared spectroscopy (FTIR)

Fig. 2 presents the FTIR spectra for the pure PP and PP processed with and without additives. In two studies conducted by Albano et al. [33] and [34], the authors found that the peaks in the region of 1450 to 1460 cm^{-1} correspond to the CH_2 bonds, those between 1370 and 1380 cm^{-1} correspond to CH_3 bonds, those between 1670 and 1675 cm^{-1} to C=C bonds, and the peaks between 3000 and 2800 cm^{-1} to C-H bonds, all of which are common in olefins and typical of PP. In the present study, when comparing the spectra of the pure PP samples and those processed with and without additives, it is possible to observe that the changes in intensities of the peaks are probably generated by the presence of the additives, and that these alterations enhance the thermal stability, as evidenced in the PP processed without additives, where the CH_2 and CH_3 peaks were reduced, probably due to degradation by shear of the PP chain. This effect was also evidenced in the analysis of the MFI.

To assess the effect of thermal aging of the PP and the composites, we calculated the carbonyl index (CI) from the spectra obtained, calculated by using the peaks between 1880-1650 cm^{-1}, corresponding to the occurrence of composites containing carbonyl (C=O) [34-36].

These composites for the most part result from oxidation of the polymer, as can be observed in the FTIR spectra in the region between 1680 and 1780 cm^{-1} as superimposed bands, mainly corresponding to acids (1712 cm^{-1}), ketones (1720 cm^{-1}), aldehydes (1730 cm^{-1}), esters (1743 cm^{-1}) and lactones (1785 cm^{-1}). The peak absorbance value of 2722 cm^{-1} is related to the angular molecular vibrations of the CH group and the axial vibrations of the CH_3 group, and was utilized to normalize the IR spectra, because is not sensitive to the oxidative degradation of the PP [35, 37, 38].

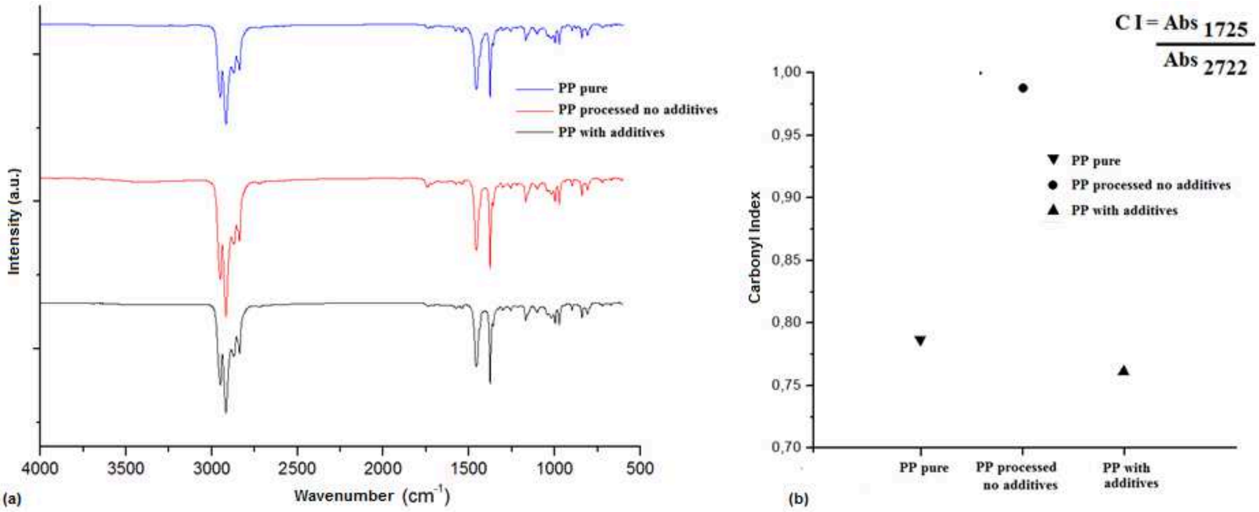


Fig. 2 – FTIR spectra for the surfaces of (a) pure polypropylene, (b) processed polypropylene and (c) polypropylene with additives.

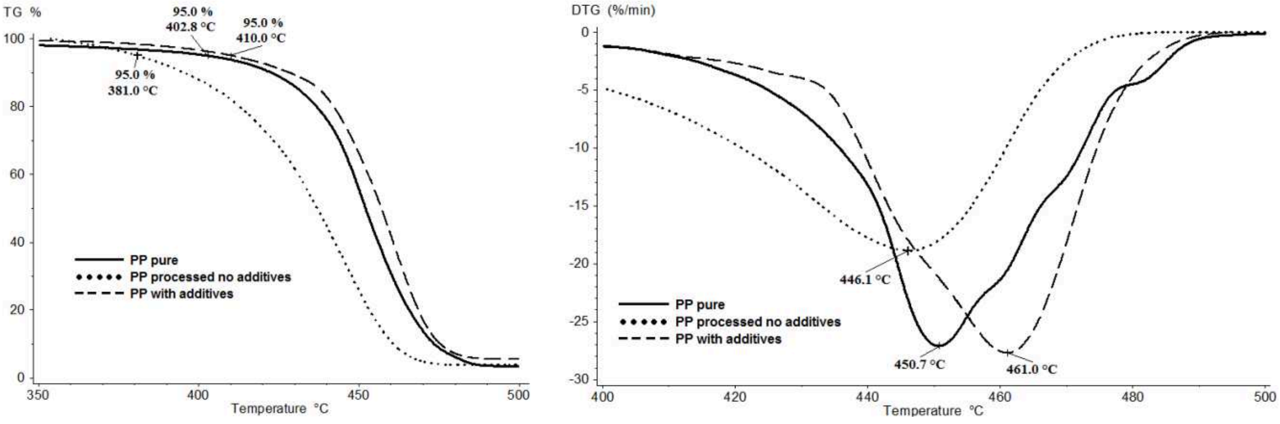
Fig. 2 (b) illustrates the behavior of the carbonyl index (CI) for the samples of PP, PP processed without additives and PP processed with additives. The CI was highest for the PP processed without additives, indicating possible oxidation of the PP during processing. In other words, some alterations occurred in the chemical structure of the polymer matrix and less pronounced oxidation of the PP processed with additives, probably due to the inclusion of the antioxidant agent (Hostanox). Similar behavior was observed by Passatore et al. for the MFI [39], in which for PP without additives, the MFI increased significantly.

3.3. Thermogravimetric analysis (TGA)

The thermal stability of the fibers, the polymer matrix and the composite was evaluated with the curves of weight loss and the first derivative of TG. In Fig. 3 it can be seen that the processing (PP processed no additives) reduces the initial degradation temperature of PP, probably due to breakage of chains as a function of shear occurred during processing [40] and its additives (PP with additives) this increases the thermal stability even after processing with the same process parameters, generated due to thermal stabilization by additives, particularly thermal antioxidant. In the literature, it appears that a single peak in the PP refers to degradation of saturated and unsaturated bonds of carbon atoms and the shift to higher temperatures in the PP processed with additives indicated good dispersion of the additives in the matrix [41, 42].

In Table 2 the temperature of 5% less mass and the degradation temperature onset peaks illustration of the composite in comparison to pure PP are shown, revealing that the composites have not only a step of decomposition as observed for PP, but two distinct peaks. According to studies, the first is the degradation of cellulose and lignin in the fibers and the second of saturated and unsaturated bonds, belonging to the polymer matrix [24, 43].

The temperature of the maximum in the derivative TG curve was in the following order, first the fibers (Table 2), then the PP (Fig. 3b) and finally the composite (Table 2), which indicates a good interaction between the fiber and the polymer matrix [9, 43]. The highest temperature at the beginning of degradation of the composite compared to the fibers is caused by the protection of the fiber for the polymeric matrix (PP), present in the composites [42]. The highest values for the maximum temperature of degradation of composites compared to PP highlight the good fiber-matrix composites interaction that improves their thermal stability, even with higher fiber content in the composites (40 and 60 %, wt%).



(a) (b)

Fig. 3 – (a) Curve of mass loss versus temperature of pure PP, PP processed no additives and PP with additives. (b) Curve of the first derivate of pure PP, PP processed no additives and PP with additives and illustration of the maximum degradation temperature.

Table 2 – Temperatures of pure PP and the composites of PP with coir fiber.

Composites	**Temperature (°C)**		
	Degradation 5% mass loss	**Maximum degradation First peak**	**Maximum degradation Second peak**
PP pure	402.8		450.7
Coir fiber	57.4		321.0
PP + Coir 20% (wt%)	308.1	328.7	465.2
PP + Coir 40% (wt%)	237.0	328.0	464.4
PP + Coir 60% (wt%)	222.8	328.8	463.2

3.4. Tensile tests

There was an increase in Young's modulus (an indication of stiffness) with increasing fiber. In the Fig. 4 shown that, compared with PP containing additive, all composites had a greater Young´s modulus at each of the fiber contents, although this was most marked for 20% (wt%), 40% and 60% (wt%) with coir fiber; there was an increase of 20%, 44% and 71% with coir fiber, respectively, compared to polypropylene with additive. Pimenta et al. [26], who studied composites with 20% (wt%) sisal fiber subjected to different treatments, reported values for Young´s modulus 60-80% higher than for polypropylene; these increases were greater than the 46% observed here for fibers that had not been washed or subjected to any other treatment.

Santos concluded that composites containing coconut fiber, for example, have weaker mechanical properties than composites with other fibers (ramie, curaua, sisal), due to the low cellulose content and high lignin content, despite the favorable aspect coconut fiber represented by the presence of a waxy layer (long-chain fatty acids) on the fiber surface, which can improve compatibility in composites with polymers [44].

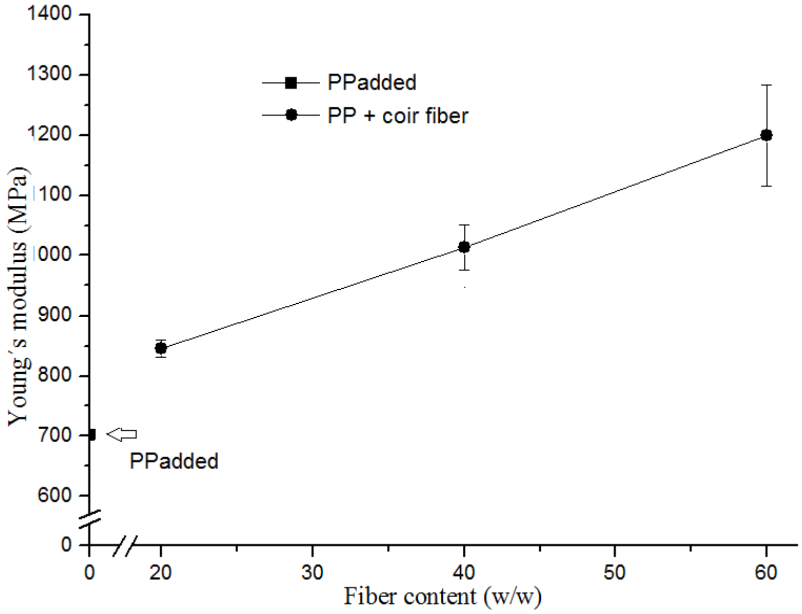
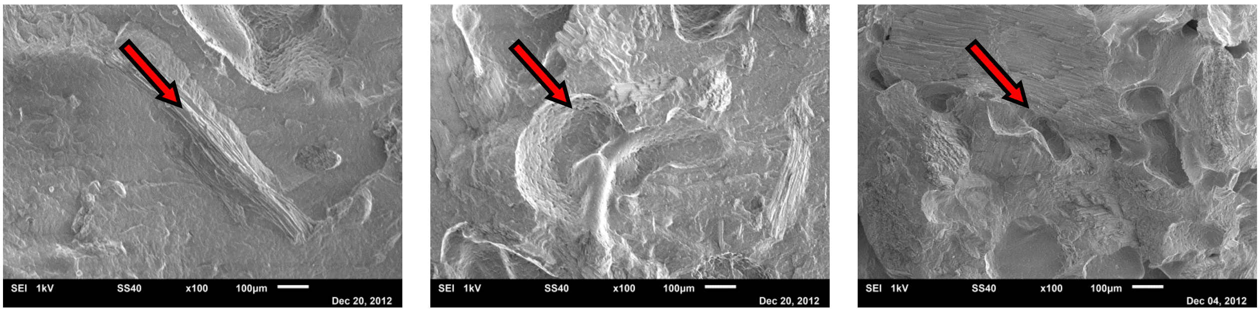


Fig. 4 – Young's modulus for polypropylene containing additive and polypropylene composites according to fiber types and filler. The points are the mean ± standard deviations (SD) of ten determinations.

3.5. Scanning electron microscopy (SEM)

Fig. 5 shown the images of photomicrographs of composite fibers with coir at levels 20, 40 and 60% wt%. Fig. 5 (a) shows more homogeneous surfaces with fibers adhered to the matrix (without protrusions) and without voids at the fiber-matrix interface, indicating good interaction and consequently better adhesion. Fig. 5 (b) and (c) show the pull-out of the fiber from the matrix, indicating weak adhesion in the matrix. Leão (45) observed that coconut fiber has a rough surface covered by layers of waxes and extractives, besides the amorphous components such as hemicellulose and lignin [45]. This surface favors the interaction of the fiber with the polymer matrix and compatibilizers, explaining the good mechanical properties obtained, even in the composites with the highest fiber content (60% wt%). Dwived et al. [11] considered this interaction between fiber and matrix to be normal, especially in untreated fibers. Bledzki et al. [12] found that composites with 40 wt% of fibers without treatment present some effect of pull out, like similar images shown in this work.



20% wt% (a) 40% wt% (b) 60% wt% (c)

Fig. 5 - Images photomicrographs of PP composites with coir (100x).

4. Conclusion

The results of this study demonstrate the feasibility of using high levels of short natural plant fibers to prepare environmentally friendly composites for applications by injection. The additives use in the composites, especially the thermal antioxidant and compatibilizer were adequate and increased thermal stability of the polymeric matrix for the use thereof, in the preparation of composites. There was an increase in thermal stability of the composite in all levels of fibers (20, 40 and 60% wt%), and this increase was up to 15 °C for composite with coir fiber with 20% wt%.

The results for Young's modulus suggest that various concentrations (20, 40 and 60%, wt%) of fibers from different sources can be used. The stiffness values of composites containing coir fiber were superior to polypropylene containing additive. Because of this it was possible to verify in this work that the poor adhesion between the hydrophilic fibers and the hydrophobic polymer matrix did not prevent the incorporation of high levels (60 wt%) of natural vegetable fibers in PP composites with the good.

These results also show that the method of processing was efficient, once there was no need to alter the superficial fibers (with chemical treatment, washing or pre-drying); this lack of intermediate steps reduced the amount of energy needed and facilitated preparation of the good PP+coir composites.

Acknowledgements

This work was supported by the CNPq - National Council for Scientific and Technological Development (grant n°. 447180/2014-2) and FAPESP - São Paulo State Research Foundation (grant n°. 2012/13445-8).

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