Effect of Hybridization and Compatibilization on the Mechanical Properties of Recycled Polypropylene-Hemp Composites

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ABSTRACT: Hemp fibers and particles, with different sizes and contents, were used to make hybrid composites based on recycled polypropylene (PP). In particular, the effect of maleated polypropylene (MAPP) addition on the morphology and mechanical properties is reported. The results show that better adhesion is obtained with MAPP addition. In general, fiber content and size had a substantial effect on the tensile, flexural, torsion, and impact properties of the resulting composites. Although, adding MAPP to the samples improved the impact strength of the composites, the values were always lower than neat PP. © 2011 Wiley Periodicals, Inc. J Appl Polym Sci 124: 2494–2500, 2012

Key words: hemp fiber; recycled polypropylene; hybrid composites; mechanical properties

INTRODUCTION

Natural fiber reinforced polypropylene (PP) has found a growing interest in the past decades. Polyolefin/natural fiber composites show good mechanical properties on a weight basis due to lower density of reinforcements,[1] while other advantages include low cost, renewability, environmental friendly source of fiber, and lower equipment wear.[2–6] In spite of these advantages, natural fiber composites have some drawbacks like low-dimensional stability, high water sorption, and low fiber-matrix interaction because of the different nature of the components: hydrophobic matrix and hydrophilic fiber.[7–10]

Hybrid composites are materials with two or more types of reinforcing phases. Most of the experimental works performed on natural fiber reinforced hybrid composites contain only one natural fiber, while the other reinforcements are usually from manmade sources.[11–13] The main objective to use natural fiber is related to their lower density and weight reduction of final products.[14] In such cases, it is usually reported that incorporation of reinforcements, mostly glass fibers, led to significant increase in mechanical properties of the composites. For example, Panthapulakkal and Sain[15] studied the effect of hybridization of PP/short hemp fiber/glass fiber composites. They reported that the mechanical properties of PP/short hemp fibers increased noticeably after incorporation of even small amounts of glass fibers. In their case, tensile strength increased from 53 to 60 MPa after adding 15% of glass fiber.

Actually, very limited works have been conducted to produce hybrid composites based on two different kinds of natural fibers.[16,17] Idicula et al.[18] performed a study on the properties of short banana/sisal hybrid reinforced polyester composites and reported increased mechanical properties due to hybridization. For example, maximum tensile strength was reported for samples with banana/sisal fibers volume ratio of 3 : 1.

Processing hybrid composites containing both short and long natural fibers of the same kind, on the other hand, provides materials with a wide variety of properties with no need to use different fibers and processing techniques. Gómez et al.[19] investigated the effect of fiber length on the mechanical properties of Agave fiber reinforced composites. They observed that increasing fiber length led to higher Young's modulus, but lower elongation a break. They also reported an optimum in impact strength for a length of 0.81 mm. In other words, incorporation of long and short reinforcing fibers can improves some properties over others. It is thus expected to optimize mechanical properties by producing hybrid composites from both long and short particles of the same origin (self-hybridization).

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Another fast growing research field is the use of recycled polymers as the matrix for natural fiber composites. Especially, products from postconsumer origins have attracted worldwide attention because of economics coupled with environmental concerns.[20,21] For example, Gosselin et al.[22] mixed postconsumer polyethylene (PE) with birch wood and reported increased mechanical properties compared to the unreinforced recycled polymer. They observed that adding 40% of wood fiber increased specific flexural modulus by almost 100%. Rodrigue et al.[23] added agave fibers, which are residues from the Mexican Tequila industries, into recycled PE from milk and juice bottles. They observed that incorporation of 25% agave fibers increased Young's modulus by 70%.

This work combines different aspects of materials processing and structures as to obtain and characterize natural fiber hybrid composites based on recycled polymers. In particular, postindustrial recycled PP samples reinforced by self-hybridization from hemp are produced by injection molding. Two different sizes of hemp was used (fiber and powder) with different ratio to make the hybrid composites. Maleated polypropylene (MAPP) was added as a coupling agent and a complete morphological and mechanical characterization was performed to show the effect of hemp content, hemp fiber/powder ratio, and coupling agent content.

EXPERIMENTAL METHODS

Materials

Hemp from Hempline Canada (now Stemergy) was sieved between 300 and 710 μm to obtain long fibers. Then, size reduction was done by grinding in a rotational knives grinder. The material was sieved again to keep only the part between 45 and 180 μm (hemp powder). Postindustrial polypropylene (PP-RP) was obtained from RECYC-RPM (Quebec, Canada) with a melt flow index of 19.8 g/10 min (ASTM D1238). The coupling agent used in this study was MAPP Epolene PMG-3003 from Eastman Chemicals.

Preparation of composites

First, masterbatches of postindustrial PP-hemp composites were extruded with different fiber/powder (f/p) ratios of 100/0, 80/20, 60/40, 33/67 and 0/100 while the overall hemp concentration was kept at 40% by weight. The compounds were prepared on a Haake TW-100 laboratory scale twin-screw extruder with L/D = 20 and a temperature profile from 150°C at the feeding point to 180°C at die exit. The composites were cooled in a water bath and then pelletized to be used later. In a previous study, Mechraoui et al.[24] showed that optimum adhesion between hemp and PP occurs between 3 and 5 wt % (filler basis) of Epolene G-3003. In our case, three coupling agent contents were selected: 0, 3, and 5%. Table I shows sample compositions with coding.

TABLE I
Sample Compositions and Coding

Sample code (–)	PP (wt %)	Hemp (wt %)	Fiber/Powder ratio	MAPP (wt %)
PP	100	0	–	0
h20f100	80	20	100/0	0
h20f80	80	20	80/20	0
h20f60	80	20	60/40	0
h20f33	80	20	33/67	0
h20f0	80	20	0/100	0
h30f100	70	30	100/0	0
h30f80	70	30	80/20	0
h30f60	70	30	60/40	0
h30f33	70	30	33/67	0
h30f0	70	30	0/100	0
h30f-3	70	30	100/0	3
h30f-5	70	30	100/0	5
h30p-3	70	30	0/100	3
h30p-5	70	30	0/100	5

Prior to injection molding, the masterbatches were diluted with neat polymer to obtain fiber contents of 20 and 30% by weight by a second extrusion under the same conditions as for the initial masterbatch. The diluted compounds were fed in a Nissei PS60E9ASE injection molding machine with a mold temperature of 60°C. All samples were prepared in a rectangular mold cavity with dimensions of 110 × 25 × 3 mm^3. Injection temperatures were kept under 180°C to limit material degradation.

Mechanical properties

For tensile measurements, type V dog bone samples were cut in the injection molded bars according to ASTM D638. Tensile tests were performed at room temperature (23°C) with a crosshead speed of 10 mm/min on an Instron model 5565 with a load cell of 500*N*. Young's modulus, tensile strength, elongation at break, and ductility of the samples are reported considering that ductility is defined as the surface area under the stress–strain curve. A minimum of five samples were tested to get an average and standard deviation for each data presented.

From the molded samples, rectangular samples (75 × 11 × 3 mm^3) were cut to perform flexion test according to ASTM D790. A three point bending geometry with a 60 mm span was used to perform the tests at room temperature (23°C) with a crosshead speed of 10 mm/min on an Instron model 5565 with a load cell of 500*N*. A minimum of three samples were tested to get the average and standard deviation of the flexural modulus. The same samples were used for torsion testing on an ARES Rheometer using the torsion rectangular fixture. Dynamic frequency

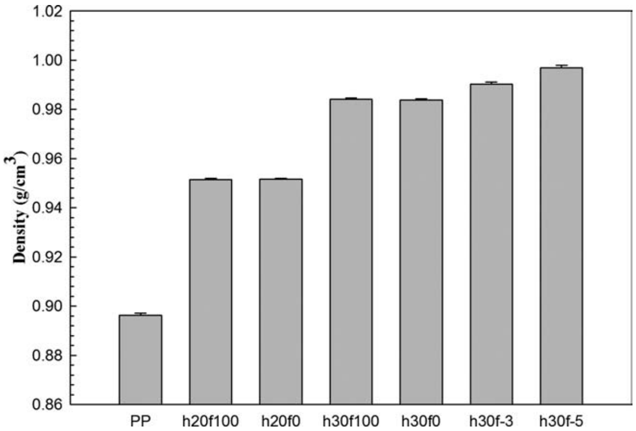

Figure 1 Effect of hemp content and MAPP addition on composities density.

sweeps at 0.05% strain (linear viscoelastic regime) were performed and the dynamic shear modulus reported was determined for a frequency of 1 rad/s.

Finally, the notched impact strength of the composites was obtained from a Tinius Olsen model Impact 104. Charpy impact tests were performed on specimens cut from the injection molded bars (110 × 12 × 3 mm^3) according to ASTM D256. The samples were notched with an automatic notcher device, Dynisco model ASN 120m. A minimum of seven samples were tested to get an average and standard deviation.

Morphology

Surface of cryo-fractured samples, in liquid nitrogen, was used to study the morphology (mainly the interface between the matrix and reinforcement) of the different compounds produced. The exposed surfaces were first coated with gold/palladium and micrographs at different magnifications were obtained from a Scanning Electron Microscope (SEM). The micrographs were taken on a JEOL model JSM-840A under a voltage of 15 kV.

Density

Density was obtained by a gas pycnometer, ULTRAPYC 1200e from Quantachrome Instruments, using nitrogen as the gas phase. The data reported are the average and standard deviation of five measurements.

RESULTS AND DISCUSSION

Density

Figure 1 shows the effect of adding hemp and MAPP to the PP matrix. As expected, density increases with hemp content from 0.896 g/cm^3 to 0.951 and 0.984 g/cm^3 after addition of 20 and 30% hemp, respectively. It can also be seen that density slightly increases after introduction of the coupling agent, indicating better adhesion by removing voids and gaps between the reinforcements and the matrix. The results also revealed that change in the fiber (f)/powder (p) ratio did not influence density of the samples produced.

Morphology

Analyzing the SEM micrographs provides helpful information about distribution and compatibility of the different phases in the composite. Figure 2 shows typical state of adhesion between the hemp fibers and the PP matrix with [Fig. 2(a)] and without [Fig. 2(b)] a coupling agent.

In Figure 2(a), a lack of compatibility is obviously seen. The fibers, indicated by the arrow, are completely clean and showing a gap between hemp and matrix due to low surface interaction between phases. A hole can be seen next to the fiber which indicates fiber pull-out while breaking the sample. In the sample shown in Figure 2(b), on the other hand, good contact between the phases shows that the compatibility has improved substantially. The fiber breakage at its base and complete fiber-matrix contact is a proof of good fiber-matrix surface interaction. This will improve

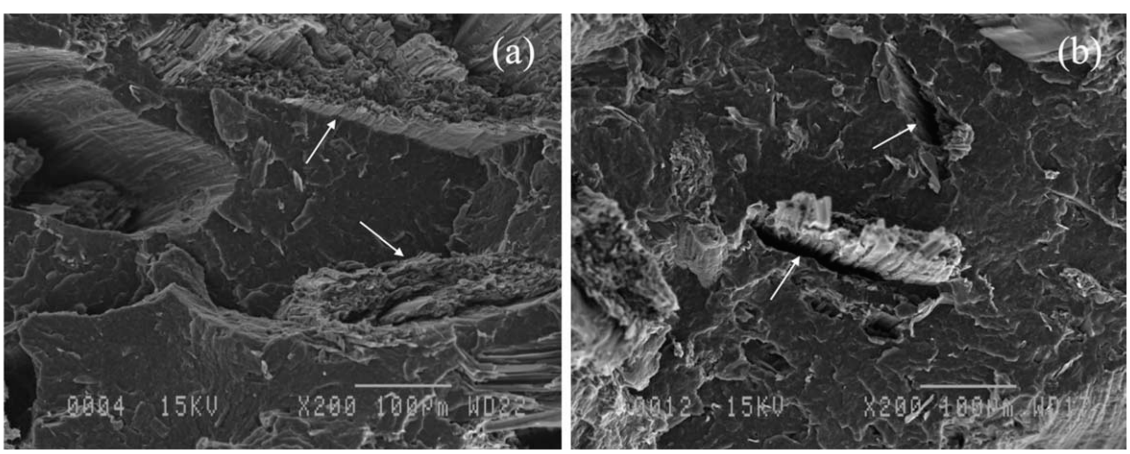

Figure 2 SEM micrographs from sample (a) h30f100 and (b) h30f-3.

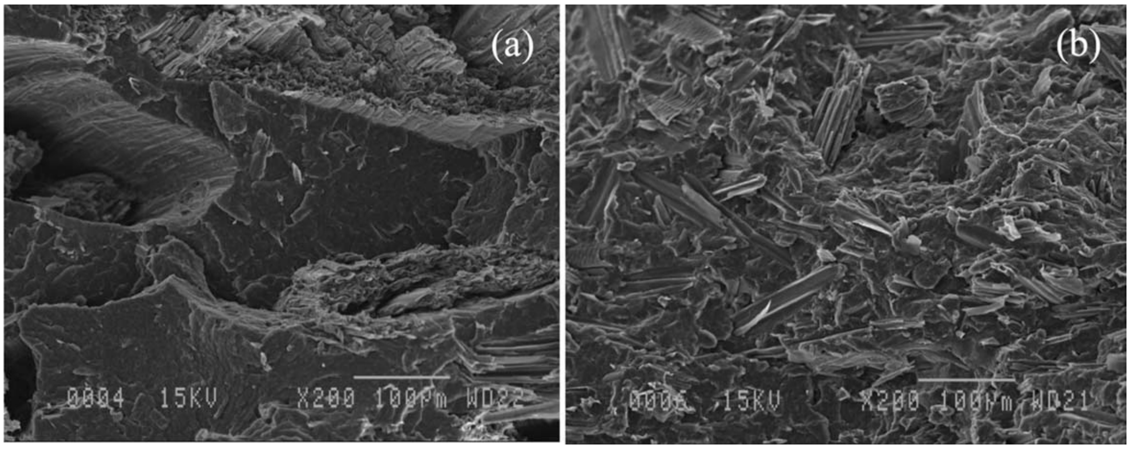

Figure 3 Typical SEM micrographs for (a) h30f100 and (b) h30f0 composites.

substantially the load transfer from the matrix to the fibers (efficiency), thus producing higher mechanical properties as reported later on.

Figure 3 shows the fractured surfaces for the fiber and powder filled composites, h30f100 and h30f0, respectively. It is implied that longer fibers are more oriented than shorter particles, which are more randomly distributed. Better orientation leads to better reinforcing effect of long fibers compared to short particles, depending on the type of mechanical property measured and the direction of applied load. These effects are now discussed in relation with the mechanical properties measured.

Tensile properties

Tensile tests may be considered as the most important and common mechanical characterization performed on polymer composites. Table II presents the mechanical properties for samples with different fiber/powder (f/p) ratios with and without coupling agent.

In both hemp concentrations, it can be seen that samples with 100% hemp fiber show higher tensile strength compared to samples having 100% hemp powder, while a different trend is observed after powder incorporation. For samples with 20% hemp content, a positive deviation is observed with a maximum around a f/p ratio of 80/20. In this case, the tensile strength of h20f80 is 25.4 MPa compared to 23.9 MPa for sample h20f100. Furthermore, h20f80 has the highest tensile strength between all the samples, except for samples with coupling agent, produced even compared with compounds with 30% hemp. This increase in tensile strength can be related to combined reinforcing mechanisms in hybrid materials, with smaller reinforcements being able to accommodate between larger ones to produce higher and more efficient specific surface area between the matrix and all the hemp particles present; i.e., good

TABLE II
Mechanical Characterization of the Composites

Sample	Tensile strength (MPa)	Tensile modulus (MPa)	Elongation at break (%)	Ductility (MJ/m^3)	Flexural modulus (MPa)	Impact strength (J/m)	Torsion modulus (MPa)
PP	21.6 (0.5)	334 (37)	–	–	1454 (33)	28.3 (2.0)	477 (5)
h20f100	23.9 (0.5)	472 (21)	25.4 (4.8)	3.02 (0.18)	1746 (50)	18.5 (0.6)	620 (8)
h20f80	25.4 (0.6)	518 (26)	29.9 (4.6)	3.51 (0.14)	2273 (92)	16.4 (0.9)	713 (26)
h20f60	24.3 (0.7)	515 (17)	19.0 (1.5)	3.86 (0.23)	2076 (120)	17.5 (1.2)	654 (17)
h20f33	23.9 (1.6)	488 (23)	18.2 (2.2)	2.59 (0.14)	1897 (78)	15.3 (1.6)	596 (21)
h20f0	21.2 (0.8)	425 (40)	16.9 (1.9)	2.64 (0.24)	1856 (45)	11.9 (0.7)	624 (4)
h30f100	25.3 (0.8)	551 (34)	18.3 (2.6)	2.45 (0.21)	2575 (70)	16.5 (1.3)	754 (34)
h30f80	22.6 (1.0)	575 (31)	18.8 (3.1)	2.38 (0.12)	2138 (60)	15.7 (1.3)	793 (23)
h30f60	21.7 (1.0)	591 (40)	20.0 (1.9)	2.30 (0.26)	2142 (114)	14.7 (1.6)	740 (9)
h30f33	21.6 (0.8)	569 (28)	16.9 (1.7)	2.00 (0.21)	2138 (163)	15.2 (1.2)	738 (14)
h30f0	22.2 (1.1)	571 (11)	16.0 (1.4)	1.96 (0.19)	2049 (98)	11.0 (0.8)	719 (5)
h30f-3	25.9 (0.3)	612 (16)	17.9 (1.2)	2.51 (0.09)	2658 (126)	18.0 (0.9)	765 (20)
h30f-5	27.0 (0.9)	603 (20)	13.5 (0.5)	2.39 (0.14)	2682 (93)	18.3 (1.2)	770 (28)
h30p-3	23.7 (2.1)	590 (12)	15.0 (1.1)	1.97 (0.12)	2241 (228)	13.2 (0.8)	731 (16)
h30p-5	26.0 (0.7)	592 (14)	14.4 (1.8)	1.99 (0.28)	2192 (173)	12.7 (0.5)	730 (26)

Numbers in parentheses represent the standard deviations.

dispersion and orientation without particle–particle contact.

For samples with 30% hemp, the tensile strength has a negative deviation with respect to the f/p ratio and seems to be controlled more effectively by the powder. This may be related to more particle–particle interaction and contacts between the particles may be responsible for this observation. These results indicate that hybridization seems to be more effective for composites at low filler contents.

The effect of coupling agent addition on the tensile strength of samples with 30% hemp content is also presented in Table II. Considerable increase in tensile strength is observed after incorporation of MAPP to composites with powder reinforcement. For example, the tensile strength of 100% powder (h30f0) increased from 22.2 to 26.0 MPa (17% increase) after incorporation of 3% MAPP (hemp basis). The maleic anhydride groups from MAPP can bond with the hydroxyl groups of the cellulose in the hemp fibers, while the hydrophobic part of MAPP creates good compatibility with the polymer matrix leading to better interaction between the fillers and recycled PP. As a result, better stress transfer from the matrix to the reinforcements increases the mechanical properties of the produced materials. For the samples with fibers, adding 3% coupling agent led to slightly improved tensile strength, 25.9 MPa (h30f-3) compared to 25.2 MPa for the composite without coupling agent (h30f100). Adding more coupling agent (5%) increased tensile strength to 27 MPa (h30f-5).

The tensile modulus data of the samples present that composites with lower filler content (20% hemp) have positive deviation due to hybridization, the optimum being again at a f/p ratio of 80/20. In this case, the tensile modulus of h20f100 increased from 472 to 518 MPa for 20% powder. On the other hand, hybridization was not very effective at 30% reinforcement (almost constant values), but no negative deviation as for tensile strength was observed. The tensile moduli of h30f100 and h30f60 are 551 and 591 MPa, respectively.

Both fiber and powder filled samples show improved characteristics after incorporation of coupling agent. Tensile modulus of sample h30f-3, with 3% coupling agent, is 612 MPa compared to 573 MPa for sample h30f100. Adding more coupling agent led to negligible variation in tensile modulus for both fiber and/or powder reinforced samples. For commercial applications, the optimum concentration seems to be around 3% in this case.

The effect of hybridization on tensile elongation at break is presented in Table II. Elongation at break results shows a positive deviation due to hybridization. Although, the effect is negligible at 30% hemp, elongation at break for sample h20f100 improved from 25% to more than 30% for sample h20f80 with a f/p ratio of 80/20. Nevertheless, all the samples have lower elongation at break compared with neat PP (176%). Strain at break decreases with hemp content because of lower homogeneity caused by adding more rigid particles in the PP matrix.

On the other hand, the results shows that strain at break always decreases with coupling agent addition. Sample h30f-3 with 3% coupling agent has 17.9% elongation at break compared to 18.5% for sample h30f100 without coupling agent. Adding more MAPP decreased even more the elongation at break: down to 13.5% for sample h30f-5. The same trend is observed for samples filled with hemp powder. As expected, more rigid materials (higher modulus and strength) produced by adding coupling agents leads to lower elasticity, thus decreased strain at break.

Ductility is defined as the energy absorbed by the specimen undergoing tension failure. The higher the ductility, the more energy the material can absorbs before rupture under tensile stresses. This information is important for PP based composites due to their low ductility, especially at low temperatures.[25]

Ductility measurements show that the ductility of PP-hemp composites at 20% hemp has a positive deviation due to hybridization. In this case, the optimum f/p ratio is around 60/40. Adding hemp powder improved ductility from 3.02 MJ/m^3 for sample h20f100 to 3.86 MJ/m^3 for sample h20f60. Again, hybridization did not affect significantly the results at 30% hemp content.

The effect of coupling agent content on ductility is also investigated. Once again, there is no significant difference between the samples with different MAPP content. This can be explained by the fact that adding a coupling agent increases the modulus and the strength of the composites, but decreases the elongation at break leading to similar values of the area under the stress–strain curves.

Overall, a comparison between the data presented so far shows that different tensile properties are affected differently by hybridization. In our case, different behaviors (positive, negative, or no deviation) are observed with hemp content where the optimum f/p ratio is function of the measured property itself. The results for other types of deformation (loading) are presented next.

Flexural properties

The flexural modulus of the samples with different f/p ratios was measured. The trends are similar to the tensile modulus data reported before. Again, hybridization led to increases properties at 20% hemp fiber (positive deviation) with the optimum at 80/20 fiber to powder ratio. In this case, the flexural modulus increased from 1750 to 2270 MPa, a 30% difference between h20f100 and h20f80. For 30% hemp, almost no effect of the f/p ratio is observed. Once again, hybridization at higher hemp concentrations does not give improved properties which are mostly controlled by the smaller particles (powder phase). Nevertheless, higher hemp content produced higher flexural modulus in general.

As mentioned earlier, coupling agent addition should improve stress transfer at the hemp-PP interface, but the results are not significantly modified for the range of parameters studied. The flexural modulus of 30% hemp fibers increased from 2570 for sample h30f100 to 2682 MPa after adding 5% of MAPP. For hemp powder, the addition of 3% MAPP increased the flexural modulus from 2049 to 2241 MPa. Clemons[26] also reported the same trend after adding MAPP to PP-wood flour composites. Adding 3% MAPP to PP/wood flour with 30% wood content did not make any substantial change in their flexural modulus (3.13 to 3.16 GPa). It can be concluded that MAPP addition has a negligible effect on both fiber and powder based composites.

Impact strength

The notched impact strengths for the samples with different f/p ratio are presented in Table II. As expected, the impact strength for all specimens is lower than neat PP. Adding a second or a third rigid phase to the PP matrix causes lower homogeneity in the material leading to lower ductility. Among all the samples, it can be seen that specimens with 100% hemp fiber have higher impact strength. Over 55% improvement is observed after replacing hemp powder with hemp fiber. The impact strength of sample h20f100 is 18.5 J/m compared to 11.9 J/m for sample with 100% powder reinforcement (h20f0). Adding more reinforcement to the matrix led to lower ductility: the impact strength of h30f100 being 16.5 J/m, while sample h20f100 has impact strength of 18.5 J/m. Lower homogeneity coupled with higher hemp rigidity compared to neat PP are responsible for this behavior. In this case, hybridization did not have any significant effect on the results; i.e., a continuous decrease of impact strength with decreasing f/p ratio.

Although, fibers are more effective than powders, the results show that adding 3% MAPP can improve the results at 30% hemp fiber or powder, but adding 5% do not modify significantly the impact strength. Thus 3% MAPP is again the optimum content.

Torsion modulus

Table II shows the torsion modulus of samples with different hemp contents and f/p ratios. In this case, the results are similar to the tensile and flexural modulus. It is clearly shown that adding hemp to PP increased the torsion properties of the specimens significantly. For example, the torsion modulus of sample h30f100 shows a 58% improvement over neat PP. Hybridization had a significant effect on the 20% hemp composite. Again, the optimum f/p ratio was found to be 80/20 with the torsion modulus increasing from 620 MPa for sample h20f100 to 713 MPa for sample h20f80 and down to 624 MPa for h20f0. Hybridization for samples with 30% hemp did not produce significant variations with f/p ratio.

Torsion moduli of samples with coupling agent show a slight increase over other compounds. For example, adding 3% MAPP increased the torsion modulus of sample h30f100 from 754 to 765 MPa for sample h30f-3. Less effect was observed after adding more coupling agent; i.e., increasing the concentration from 3 to 5% did not make any noticeable change for both fiber and powder reinforced composites.

CONCLUSIONS

The mechanical and morphological properties of hemp powder/hemp fiber hybrid composites reinforced PP of postindustrial origin have been investigated for different hemp concentrations and fiber/powder ratios. Also, different MAPP contents were studied to determine the effect of hemp/PP adhesion on the tensile, flexural, impact, and torsion properties of the composites. From the results obtained, several conclusions can be obtained.

First, SEM micrographs cleared that adding MAPP as a coupling agent to PP/hemp composites increased substantially the compatibility of the compounds produced. Better adhesion also led to higher density confirming that holes and voids were eliminated at the hemp/PP interface.

As expected, the mechanical properties of the composites depended on hemp concentration. In general, tensile strength, as well as tensile, flexural, and torsion moduli increased with hemp content. On the other hand, tensile elongation at break, ductility, and impact strength decreased with increasing hemp content.

Interesting results were obtained for hybrid composites. In general, a positive deviation was observed at low hemp content (20%), while negative or no deviations were observed at higher concentrations (30%). This result can be explained in terms of smaller particles being able to accommodate themselves between larger ones leading to less particle–particle interactions (contact), thus more efficient stress transfer between the matrix and all the reinforcement. In general, f/p ratio around 80/20 gives the optimum properties for 20% total hemp content.

Finally, optimum coupling agent content was found to be around 3% weight based on hemp content since results at 5% did not show any significant differences from the latter.

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