

Mechanical Properties of Injection-Molded Isotactic Polypropylene/Roselle Fiber Composites

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ABSTRACT: Natural fiber-thermoplastic composite materials, based on their cost-effectiveness and environmental friendliness, have attracted much interest both scientifically and technologically in recent years. Other advantages of natural fibers are good specific strength, less abrasion, and less irritation upon inhalation (in comparison with some common inorganic fillers). In the present contribution, roselle (*Hibiscus sabdariffa* L.) fibers were chosen and used as reinforcing fillers for isotactic polypropylene (iPP) for the first time, due mainly to the cost-effectiveness and natural abundance on Thai soil. Processibility and mechanical properties of the resulting composites were investigated against the type and the mean size of the fibers. The results showed

that the highest mechanical properties were observed when roselle bast fibers were incorporated. When whole-stalk (WS) fibers (i.e., the weight ratio of bast and core fibers was 40 : 60 w/w) were used, moderate mechanical properties of the resulting composites were realized. The optimal contents of the WS fibers and the maleic anhydride-grafted iPP compatibilizer that resulted in an improvement in some of the mechanical properties of the resulting composites were 40 and 7 wt %, respectively. © 2006 Wiley Periodicals, Inc. *J Appl Polym Sci* 101: 3291–3300, 2006

Key words: composite; isotactic polypropylene; roselle; mechanical properties; compatibilizer

INTRODUCTION

Isotactic polypropylene (iPP) has entertained global commercial utilization since its inception because of the balance in its properties and cost-effectiveness. However, any type of insurgence in oil price could result in fluctuation in the price of downstream petroleum-based products, notwithstanding iPP. The industry has coped with this problem by simply compounding iPP with cheaper, inorganic fillers, such as CaCO₃, talcum, and mica. In recent years, use of natural, cellulosic fibers as reinforcing fillers for commodity plastics has received much attention because of a number of advantages over traditional, inorganic ones such as good specific strength, high toughness, good thermal insulation, less abrasion, minimal dermal and respiratory irritation, biodegradability, and natural abundance. Despite these advantages, some disadvantages of natural, cellulosic fibers are, for examples,

high water absorption, low thermal resistance, poor processability, and, especially, inconsistency in the properties of the raw materials.

Many types of naturally-derived cellulosic fibers have been explored as reinforcing fillers in commodity thermoplastics, some of which are fibers from rubber wood,¹ beech wood,² sisal,³ hemp,⁴ jute,⁵ kenaf,⁶ and ramie.⁷ Uses of these fibers satisfy both economic and ecological interests. Apart from these various fibers, roselle (*Hibiscus sabdariffa* L.), a close relative of jute (*Corchorus capsularis* L.), has not yet been explored as a reinforcing material for a commodity thermoplastic. However, Thai farmers have used roselle bast fibers (after removal of lignin and pectin) to make sacks for stocking their produces for a long time. Like a number of other natural plants, roselle comprises core (the inner part of the plant that is composed mainly of short fibers and is a highly porous material) and bast (the outer part of the plant that is composed mainly of long, continuous fibers). Commonly, the composition of bast and core materials in roselle plants is about 40 × 60 w/w.

The present contribution reports utilization of roselle (*H. sabdariffa* L.) fibers as reinforcing fillers for iPP for the first time. The roselle fiber-reinforced iPP composites were prepared in a self-wiping, corotating twin-screw extruder. The composites were later injection-molded into specimens for mechanical tests. The effects of type, size, and content of roselle fibers and

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TABLE I

Effects of type and size of fibers		Effect of fiber content (WS 20–40 content, wt %)	Effect of compatibilizer (MAPP content, wt %)
Type of roselle fibers	Grade of iPP		
Without roselle fibers			
BF PM4			
BF PM6			
WS 20–30	HP500N	0	0
WS 30–50		10	1
WS > 50		20	3
WS 20–40	CS23	30	5
WS 20–50		40	7
WC 20–30	EP548S	50	10
WC 30–50			
Core 20–30			
Core 30–50			
Selected WS 20–40 and HP500N for further studies		Selected 40 wt % WS 20–40 HP500N for further studies	

the presence and content of a commercial compatibilizer on mechanical properties of the as-prepared composites were investigated and reported.

EXPERIMENTAL

Materials

Three commercial grades (i.e., HP500N, CS23, and EP548S, respectively) of isotactic polypropylene (iPP) were donated by HMC Polymers Co., Ltd. (Thailand). HP500N was a homopolymer grade with density and melt-flow rate (MFR) values of about 0.90 g cm^{-3} and 12 dg min^{-1} , respectively; CS23 was a homopolymer grade with density and MFR values of about 0.90 g cm^{-3} and 25 dg min^{-1} , respectively; and EP548S was an impact-modified copolymer grade with density and MFR values of about 0.90 g cm^{-3} and 44 dg min^{-1} , respectively. A commercial grade (i.e., Polybond 3200; hereafter PB3200) of maleic anhydride-grafted iPP (MAPP) with the maleic anhydride content of 1.0 wt %, used as the compatibilizer, was purchased from Crompton Corp. (USA). The density and MFR of PB3200 were 0.91 g cm^{-3} and 115 dg min^{-1} , respectively.

Roselle, after removal of lignin and pectin, is composed of 40 wt % of bast and 60 wt % of core materials. After being processed into fibers of specific sizes, bast and core fibers were gravitationally separated in water. Because of the high aspect ratio of bast in comparison with that of core fibers, bast fibers were less processable than core ones. The higher aspect ratio of the bast fibers should also impart better properties to the as-prepared composites. To investigate the effect of the fiber type on mechanical properties of the as-prepared composites, four types of roselle fibers were prepared and incorporated in iPP: they were (1) bast fibers (BF; the weight ratio of bast/core fibers = 100/

0), (2) core fibers (Core; the weight ratio of bast/core fibers = 0/100), (3) whole-stalk fibers (WS; the weight ratio of bast/core fibers = 40/60), and (4) core-added whole-stalk fibers (WC; the weight ratio of bast/core fibers = 20/80). It should be noted that WC was prepared to systematically observe the effect of fiber composition (between bast and core fibers) on the mechanical properties of the resulting composites. To investigate the effect of the size of fibers on mechanical properties of the as-prepared composites, bast fibers of various sizes were prepared by milling the raw bast fibers in a home-made pin mill with different numbers of the mill (i.e., 4 or 6), while both WS and core fibers of various sizes were prepared by passing corresponding fibers, which were processed in a hammer mill through sieves of varying mesh number (i.e., 20, 30, 40, or 50).

Preparation of iPP/roselle fiber composites

In the present work, two separate experiments were planned out. The first experiment was to investigate the effects of type (i.e., BF, Core, WS, and WC, respectively) and size (i.e., reported as the number of the mill for bast fibers or the mesh number for both WS and core fibers) of roselle fibers on mechanical properties of the resulting iPP composites. All the three iPP resins were used in this experiment and the fiber content was fixed at 30 wt %. The second was to investigate the effects of roselle fiber content (i.e., 0–50 wt %) and the presence and content (i.e., 0–10% by weight of the compound for a fixed fiber content of 40 wt %) of MAPP on mechanical properties of the resulting iPP composites. In this experiment, only HP500N resin and WS (20–40) (i.e., WS fibers that had the size between 20 and 40 mesh) were studied. Table I summarizes the formulation for all as-prepared composites.

TABLE II

Sample	Range of size (mm)	Average size (mm)	SD
Diameter of BF fibers	0.130–0.460	0.230	0.060
Diameter of Core fibers	0.290–0.700	0.450	0.090
Length of Core, WS, or WC fibers (mesh 20)	0.054–4.497	2.407	0.805
Length of Core, WS, or WC fibers (mesh 20–30)	0.054–3.475	1.990	0.568
Length of Core, WS, or WC fibers (mesh 30–50)	0.047–3.743	1.201	0.521
Length of Core, WS, or WC fibers (mesh <50)	0.030–2.330	0.817	0.372

Preparation of composite samples started with pre-mixing iPP and dried (60°C, overnight) roselle fibers of a specific type and content in a tumble mixer for 10 min to prepare iPP/roselle fiber compounds. In some cases, MAPP of a specified amount was also added. The mixed compounds were then fed into a Collin ZK25 self-wiping, corotating twin-screw extruder, operating at a screw speed of 100 rpm and using a temperature profile (from the feed zone to the die) of 90, 170, 175, 180, and 185°C, respectively. The composite extrudates were pelletized using a Planetrol 075D2 pelletizer and the pellets were dried again at 60°C overnight, prior to being shaped into specimens for mechanical tests, according to the ASTM D 638–91, ASTM D 256–90b, and ASTM D 790–92 standard test methods, using an ARBURG Allrounder® 270M injection molding machine. The temperature settings (from the feed zone to the nozzle) were 150, 160, 170, 180, and 185°C, respectively. The injection pressure was 1700 bar and the dwelling pressure was 700 bar. Prior to the mechanical tests, all test specimens were conditioned in ambient conditions for 7 days.

Characterization

Size and its distribution of the roselle fibers were characterized by image analysis using a Sony Cyber-Shot DSC-P73 digital camera and Scion Image Beta 4.02 software. Thermal degradation behavior of the fibers was characterized using a Perkin–Elmer TGA7 thermal gravimetric analyzer (TGA) over a temperature range of 25–600°C using a heating rate of 10°C min⁻¹ in a nitrogen atmosphere.

The tensile strength at yield, elongation at yield, and Young's modulus for all as-prepared iPP/roselle fiber composites were measured on an Instron 4206 universal testing machine according to ASTM D 638–91 standard test method using a 100 kN load cell, a 50 mm min⁻¹ crosshead speed, and a 50 mm gauge length. Izod impact resistance of these composites was determined on a Swick 5113 impact tester according to ASTM D 256–90b standard test method with the original size of each specimen being about 27 × 62 × 4 mm³, using a 2.7 Joule pendulum and a 124.4° release angle. The flexural strength and flexural modulus of the composites were determined on test pieces cut

from the molded dumbbells according to the procedure described in the ASTM D 790–92 standard test method, using the three-point loading fixture of the Instron 4206 universal testing machine. All mechanical measurements were carried at room temperature and the results were reported as averages of the data taken from at least 10 specimens.

A JEOL JSM-5200 scanning electron microscope (SEM) was used to observe the microstructure of the impact-fractured surface of selected specimens obtained after the impact testing. Each selected specimen was cut about 2 mm below the fractured surface and the cut piece was stuck onto an aluminum stub. Prior to observation under SEM, each sample was gold-coated to enhance the conductivity of the surface.

RESULTS AND DISCUSSION

Characterization of roselle fibers

To evaluate the effect of fiber size on mechanical properties of the as-prepared roselle fiber-reinforced iPP composite materials, both diameters and lengths with corresponding average values for all fiber types investigated were determined and the results are summarized in Table II. Figure 1 shows examples of histograms of fiber sizes that were used to arrive at the numerical values reported in Table II. The diameter of pure roselle bast fibers ranged between about 0.13 and 0.46 mm, with the average value being about 0.23 mm, and that of pure roselle core fibers ranged between 0.29 and 0.70 mm, with the average value being about 0.45 mm. Obviously, the diameters of the bast fibers were smaller than those of the core fibers. Both core and WS fibers of various lengths were separated by sieves of different mesh numbers (i.e., 20, 30, 40, or 50), while bast fibers of various lengths were separated by different numbers of the pin mill used (i.e., either 4 or 6). Generally, both core and WS fibers that passed through a sieve of a smaller mesh number had greater aspect ratios than those that passed through a sieve of a greater mesh number, while bast fibers prepared with a number-4 pin mill (i.e., BF PM4) had greater aspect ratios than those prepared with a number-6 pin mill (i.e., BF PM6). Figure 2 shows selected SEM images of both pure roselle bast and core fibers at two

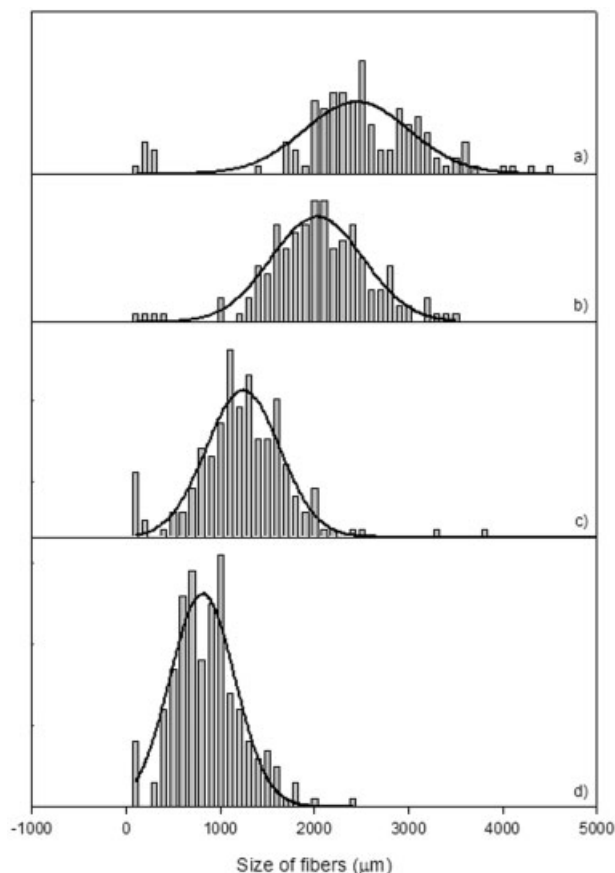


Figure 1 Lengths of as-prepared roselle (core, WS, or WC) fibers: (a) mesh >20, (b) mesh 20–30, (c) mesh 30–50, and (d) mesh <50.

different magnifications. Clearly, the bast fibers had a better physical structure than that of the core fibers, which showed a porous structure.

Figure 3 shows derivative TGA curves for all fiber types investigated. Clearly, only two degradation peaks around 295 and 350°C were observed. These peaks corresponded to the degradation of hemicellulose and cellulose, respectively. However, during processing, degradation of both hemicellulose and cellulose can occur at a much lower temperature, due largely to the elevated pressure conditions and the presence of oxygen. This is quickly recognized from changes in both the color and the odor of the composite products.

Characterization of as-prepared composites

To investigate the effects of type (i.e., BF, Core, WS, and WC, respectively) and size (i.e., reported as the number of the mill for bast fibers or the mesh number for WS, WC, and core fibers, respectively) of roselle fibers on mechanical properties of the as-prepared iPP/roselle fiber composites, three grades of iPP (i.e., HP500N, CS23, and EP548S, respectively) were used

as the base matrix and the content of the fibers incorporated was fixed at 30 wt %.

Figure 4 shows tensile strength at yield, elongation at yield, and Young's modulus for all as-prepared roselle fiber-reinforced iPP composites. Basically, for tensile strength at yield, only the bast fiber-reinforced composites showed the property values greater than that of the pure polymer, while, for Young's modulus, all as-prepared composites showed the property values greater than that of the pure polymer. Interestingly, for CS23, incorporation of WS (20–40) fibers resulted in the composite that had the tensile strength at yield greater than that of the pure polymer and, at the same time, showed the highest Young's modulus. The increased rigidity of the as-prepared composites (i.e., the increase in the Young's modulus) resulted in a marked decrease in the elongation at yield.

Furthermore, among the composites reinforced with any type of roselle fibers, both the tensile strength at yield and the Young's modulus generally increased with increasing aspect ratio of the fibers, or *vice versa*. Interestingly, for the composites reinforced with WS fibers, such property values decreased when the lengths of the fibers decreased from 20 to 30 mesh numbers to >50 mesh number. However, when the content of the fibers with shorter lengths was increased (comparing between composites reinforced with WS (20–30) and WS (20–40) fibers), such property values increased tremendously. Further increase in the fibers with shorter lengths (comparing between composites reinforced with WS (20–40) and WS (20–50) fibers) resulted either in an increase or a decrease in the property values of the resulting composites. Comparatively, the composites reinforced with WS (20–50) showed much better property values than those reinforced with WS (>50), possibly a result of the presence of the fibers with high aspect ratios. In addition, comparison among the composites reinforced with WS, WC, and Core fibers of similar aspect ratios suggested that incorporation of core materials resulted in a deterioration of both the tensile strength at yield and Young's modulus, or, in other words, such property values of the composites increased with increasing bast fiber content.

Figure 5 shows Izod impact resistance for all the as-prepared composites. For HP500N and CS23 composites, incorporation of most of the fiber types (except for WS (>50) and WC (30–50) fibers) resulted in an increase in the impact resistance of the resulting composites, when comparing with that of the pure polymer, while, for EP548S composites, incorporation of most of the fiber types (except for BF6 and BF4 fibers) resulted in a decrease in the impact resistance of the resulting composites. For most of the as-prepared composites, roselle fiber-reinforced EP548S composites showed the highest impact resistant values, most likely a result of the fact that EP548S was an

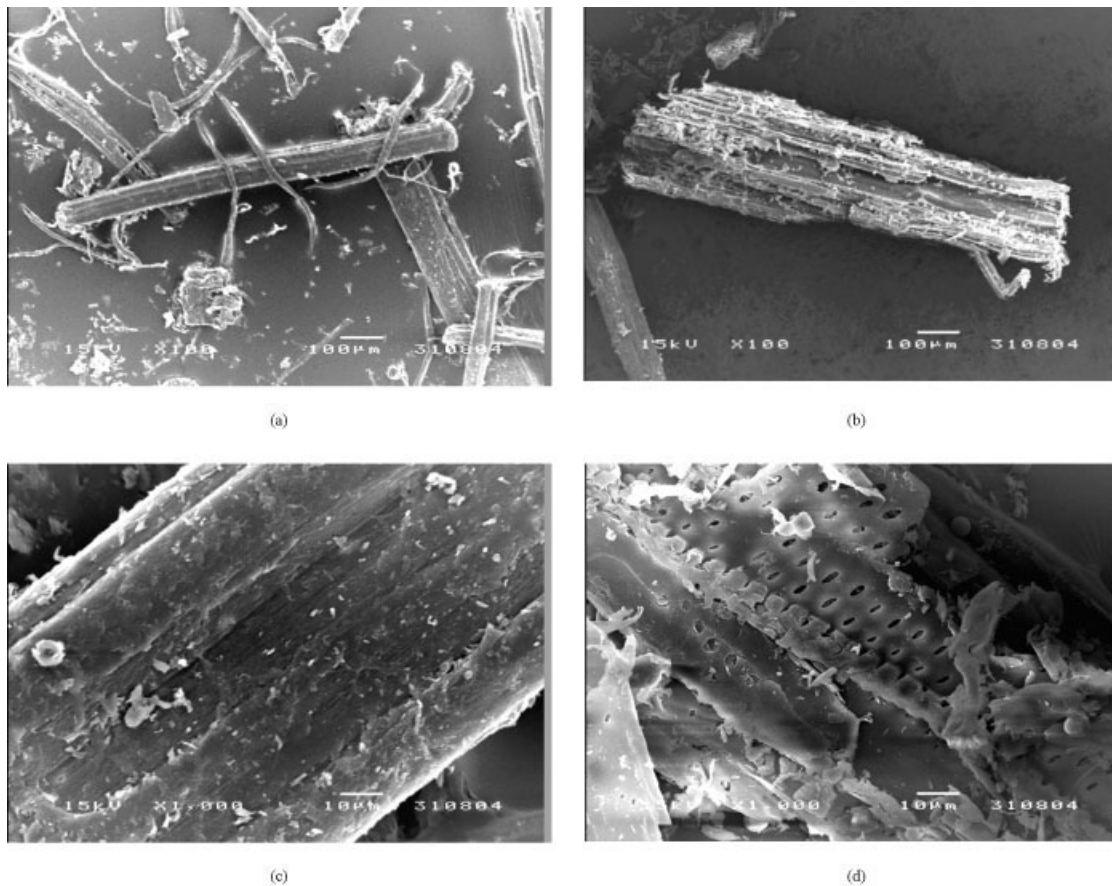


Figure 2 SEM images of (a) bast (×100), (b) core (×100), (c) bast (×1000), and (d) core fibers (×1000).

impact-modified copolymer grade. Generally, impact resistance was an increasing function of the lengths (hence, aspect ratios) of the fibers, most likely a result of the increased surface area that could absorb more impact energy.

Fractured surface of some selected impact specimens was observed under a SEM. All SEM images

showed no particular adhesion between the surfaces of the roselle fibers and the iPP matrix, as evidenced by the rather clean surface of the fibers (see Fig. 6). This is quickly recognized, since cellulosic fibers are naturally hydrophilic, while iPP is hydrophobic. The chemical incompatibility between the reinforcing material and the matrix caused no appreciable improvement in the mechanical properties of the as-prepared composites. As a result, the observed improvement in the impact resistance of some of the as-prepared composites (especially for those with high bast fiber contents) should depend on size and shape of the fibers alone. Despite the fact that the adhesion between the fiber surface and the polymer molecules was poor, the presence of fibers of high aspect ratios could help absorb more impact energy that was needed to overcome the mechanical friction that occurred during the pull-out of the fibers from the matrix.

Flexural properties such as flexural strength and flexural modulus of the as-prepared roselle fiber-reinforced iPP composite materials were investigated and the results are shown graphically in Figure 7. In general, the flexural properties for all the composite materials investigated showed a similar behavior to the tensile properties earlier reported. Obviously, addi-

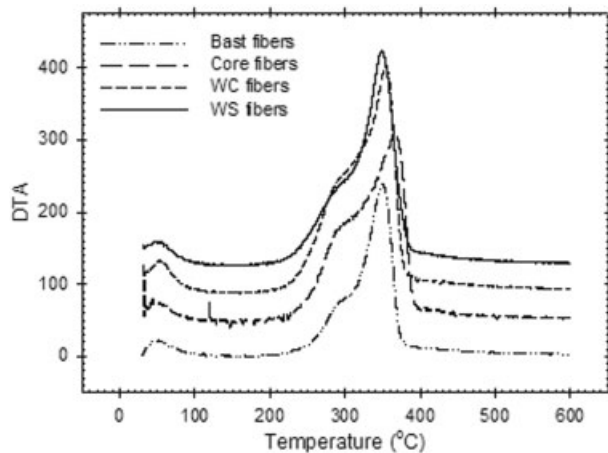


Figure 3 Derivative TGA spectra of roselle bast, WS, WC, and core fibers.

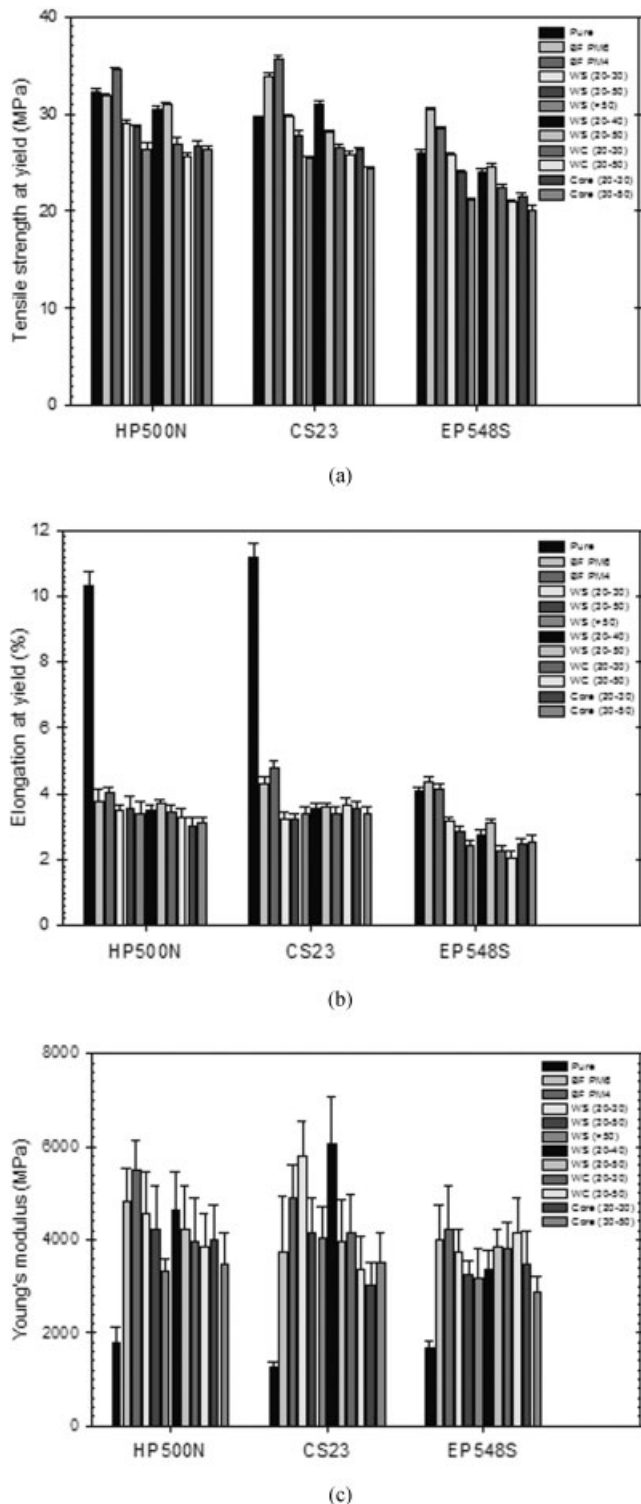


Figure 4 (a) Tensile strength at yield, (b) elongation at yield, and (c) Young's modulus for all the as-prepared roselle fiber-reinforced iPP composite materials at a fixed fiber loading of 30 wt %.

tion of the fibers of all types did not improve much the flexural strength of the composites, but it played an important role in the appreciable increase in the flex-

ural modulus of these composites over that of the pure polymer. Similar to both the tensile strength at yield and the Young's modulus of these composites, an increase in the lengths or the aspect ratios of the fibers resulted in a much improvement in the flexural modulus.

On the basis of the results obtained earlier, WS (20–40) fibers were chosen as the model fibers for further investigating the effects of roselle fiber content (i.e., 0–50 wt %) and the presence and content (i.e., 0–10% by weight of the compound for a fixed fiber content of 40 wt %) of MAPP on mechanical properties of the resulting iPP composites. Only HP500N resin was used. Figure 8 shows tensile strength at yield, Young's modulus, elongation at yield, and Izod impact resistance for all the as-prepared roselle fiber-reinforced iPP composites as a function of the fiber loading. Evidently, addition of the fibers did not affect much the tensile strength at yield, but it resulted in a monotonous increase in both the Young's modulus and the impact resistance, at the expense of the elongation at yield, of the composites. Interestingly, a marked increase in the Young's modulus was observed when the fiber content was 40 wt %. Flexural properties for all the composites investigated, shown in Figure 9, exhibited a similar behavior to that of the tensile properties in that the flexural strength was not affected much by the presence of the fibers, while the flexural modulus increased monotonically with increasing fiber loading and a marked increase in the property value was observed at the fiber loading of 40 wt %.

On the basis of the results obtained, the 40 wt % WS (20–40) filled iPP (HP500N) composite was further investigated for the effect of the addition of compatibilizer on the mechanical properties of the resulting composites. The amount of MAPP added ranged between 1 and 10% by weight of the iPP/WS (20–40)

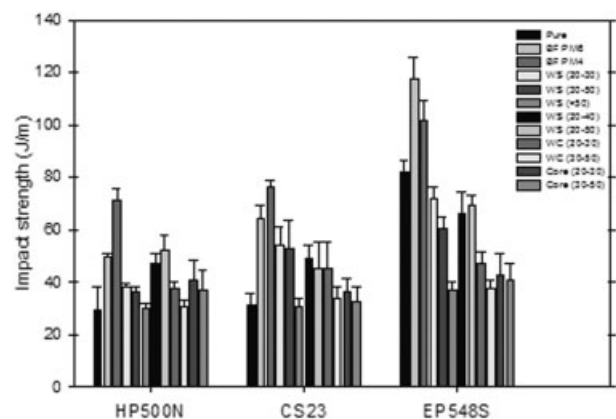
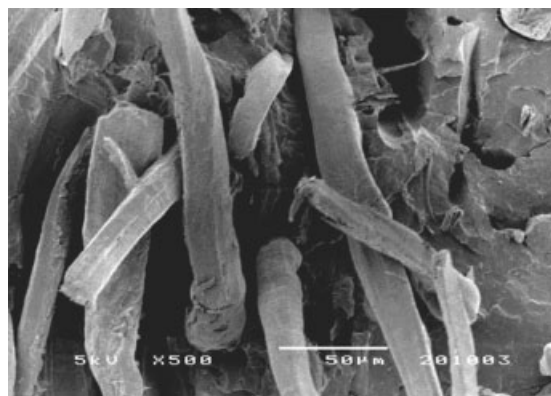
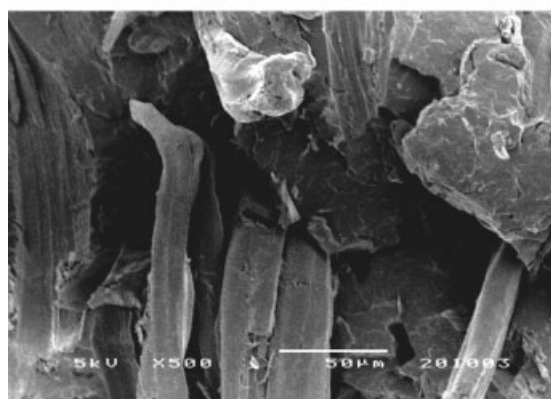


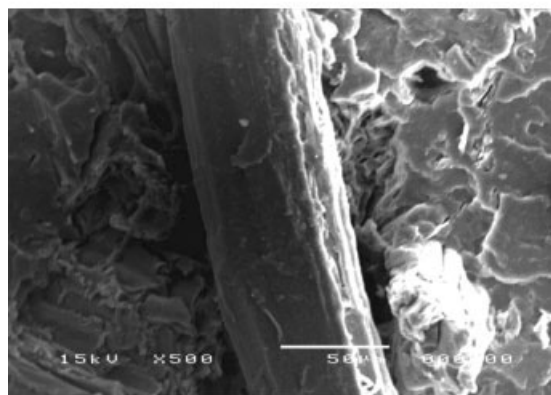
Figure 5 Izod impact resistance for all the as-prepared roselle fiber-reinforced iPP composite materials at a fixed fiber loading of 30 wt %.



(a)



(b)



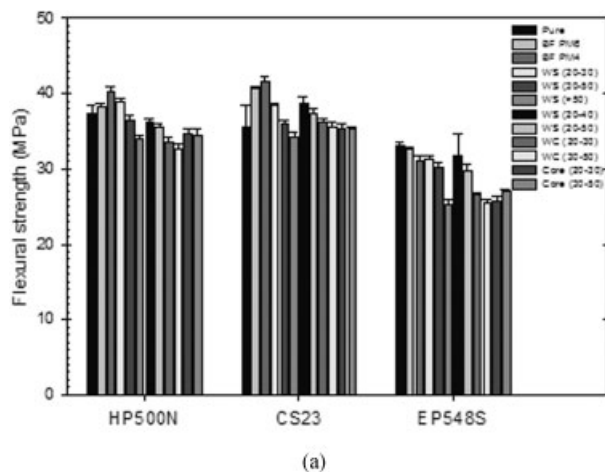
(c)

Figure 6 SEM images of surfaces of some impact specimens of (a) bast fiber-reinforced HP500N, (b) WS (20–50) fiber-reinforced HP500N, and (c) Core (20–30) fiber-reinforced HP500N composites at a fixed fiber loading of 30 wt %.

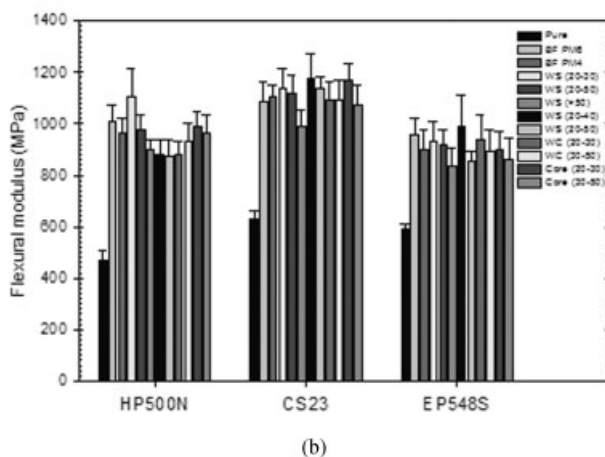
base compound. Figure 10 shows tensile strength at yield, Young’s modulus, elongation at yield, and Izod impact resistance for all the as-prepared roselle fiber-reinforced iPP composites as a function of the MAPP loading. Addition of only 1 wt % of MAPP resulted in a marked increase in the tensile strength at yield (i.e., about 25%), but it decreased both the elongation (i.e.,

about 25%) and the impact resistance (i.e., about 16%) of the resulting composite. Both the tensile strength at yield and the elongation at yield increased with further increase in the MAPP content up to about 5 wt %, after which the property values leveled off, while the impact resistance showed a monotonous decrease in the property value.

Figure 11 shows flexural properties such as flexural strength and flexural modulus of the as-prepared MAPP-compatible iPP/WS (20–40) composites. Apparently, initial addition of MAPP (at 1 wt %) resulted in marked decrease in the property values (i.e., about 21% for flexural strength and about 48% for flexural modulus). With further increasing the MAPP content, flexural strength was found to increase. Interestingly, at the MAPP contents of 7 and 10 wt %, flexural strength of the resulting composites was much greater than that of the uncompatibilized composite (i.e., an increase in the property value of about 31%). With regards to the flexural modulus, it was not found to be affected when the MAPP content was less than about 5 wt %, but, when the MAPP content was

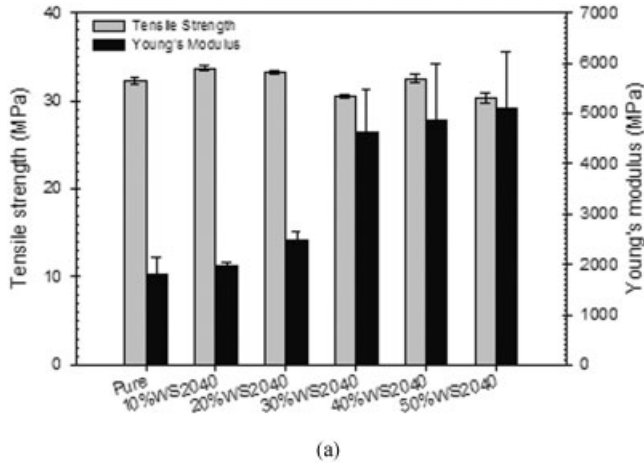


(a)

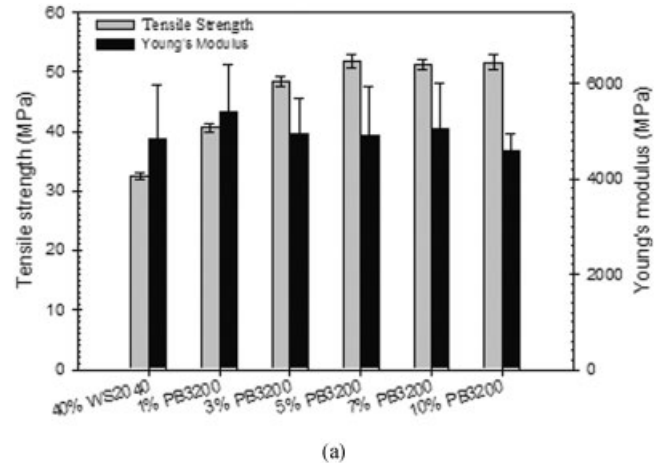


(b)

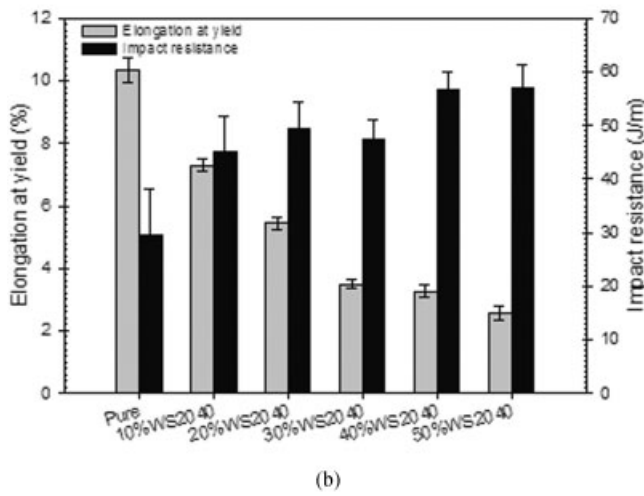
Figure 7 (a) Flexural strength and (b) flexural modulus for all the as-prepared roselle fiber-reinforced iPP composite materials at a fixed fiber loading of 30 wt %.



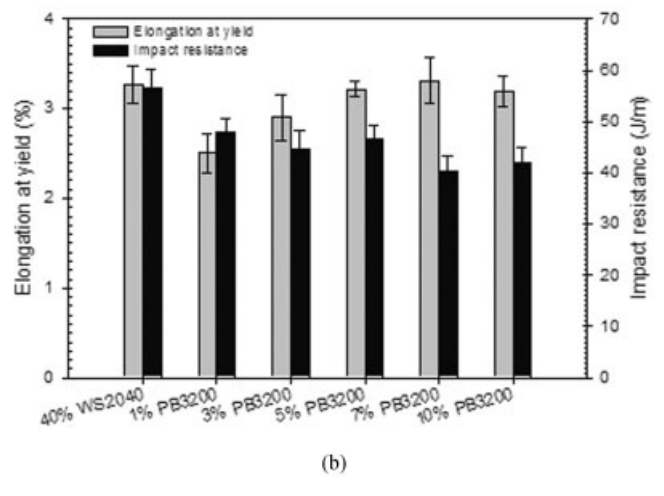
(a)



(a)



(b)



(b)

Figure 8 (a) Tensile strength at yield and Young's modulus and (b) elongation at yield and Izod impact resistance for pure and WS (20–40) fiber-reinforced iPP composites of varying fiber content (ranging from 10 to 50 wt %).

Figure 10 (a) Tensile strength at yield and Young's modulus and (b) elongation at yield and Izod impact resistance for uncompatibilized and compatibilized 40 wt % WS (20–40) fiber-reinforced iPP composites of varying MAPP content (ranging from 1 to 10 wt %).

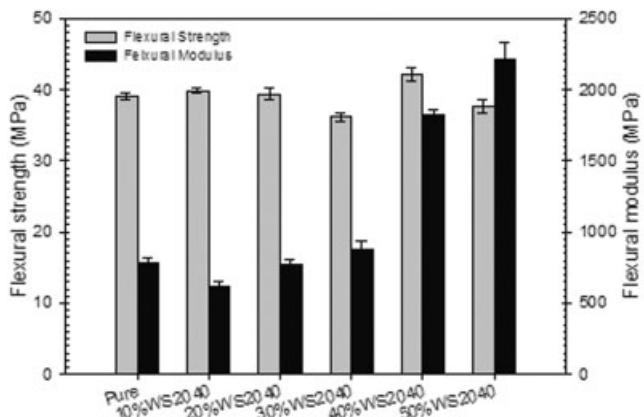


Figure 9 Flexural strength and flexural modulus for pure and WS (20–40) fiber-reinforced iPP composites of varying fiber content (ranging from 10 to 50 wt %).

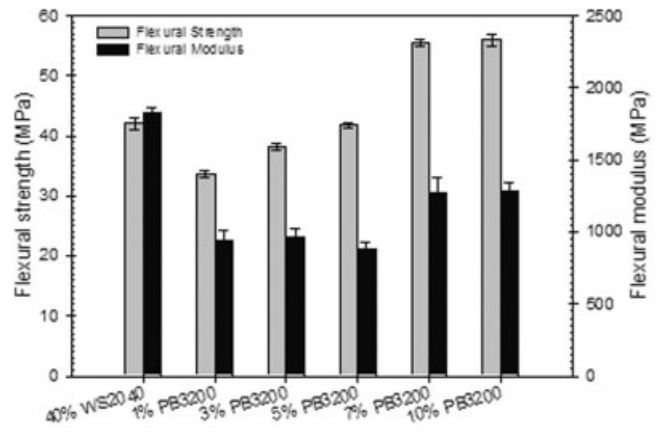


Figure 11 Flexural strength and flexural modulus for uncompatibilized and compatibilized 40 wt % WS (20–40) fiber-reinforced iPP composites of varying MAPP content (ranging from 1 to 10 wt %).

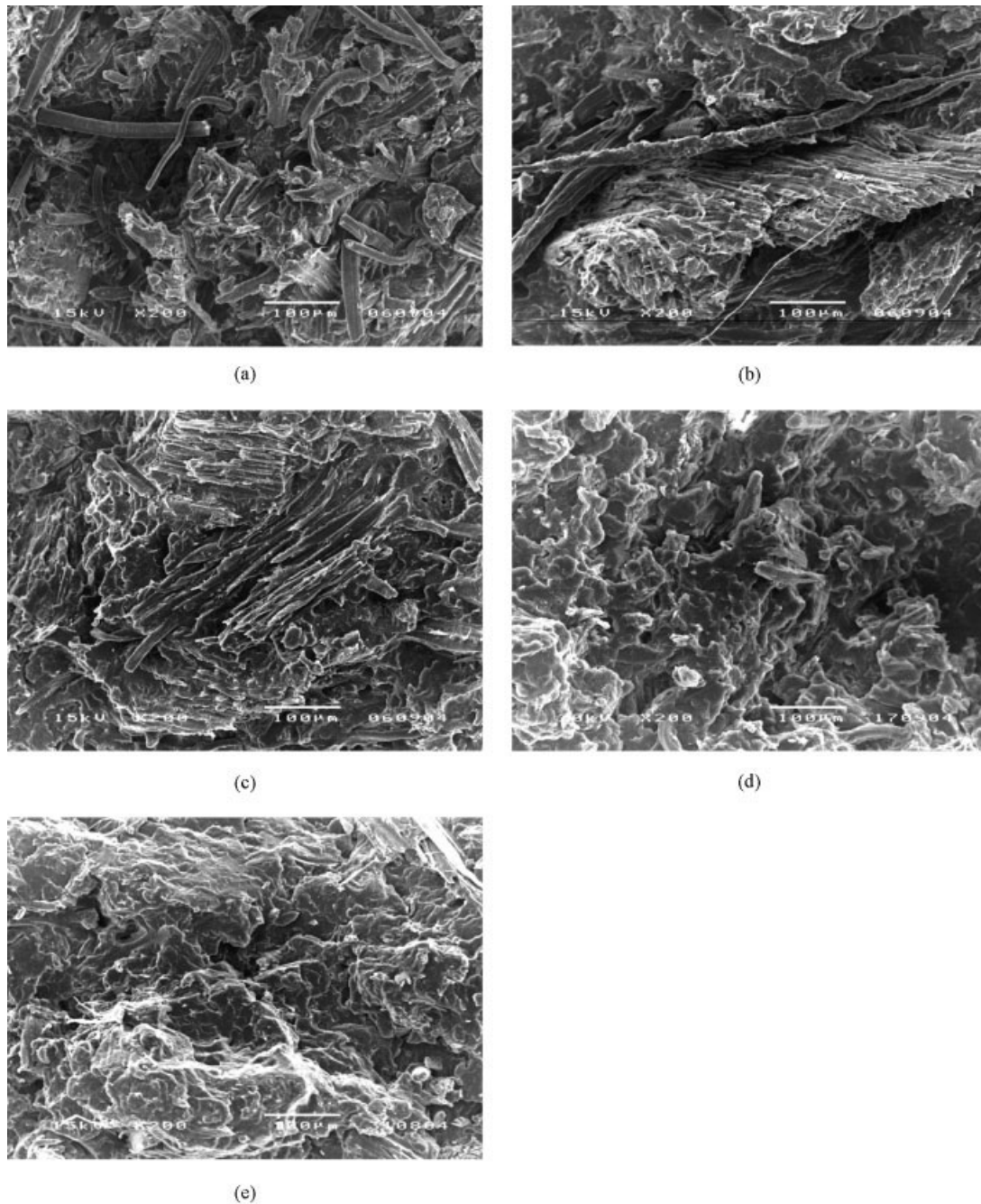


Figure 12 SEM images of surfaces of some impact specimens of 40 wt % WS (20–40) fiber-reinforced iPP composites compatibilized with (a) 1, (b) 3, (c) 5, (d) 7, and (e) 10 wt % MAPP.

greater than about 7 wt %, a sudden increase in the property value was observed (but the value was still lower than that of the uncompatibilized composite).

Figure 12 shows SEM images of fractured surface of some selected impact specimens of the as-prepared MAPP-compatibilized iPP/WS (20–40) composites. It was previously shown that the rather clean surface of the fibers was observed on the fractured surface of uncompatibilized composite [see Fig. 6(b)]. Such a feature was also observed in the SEM image of frac-

tured surface of the composite compatibilized with 1 wt % MAPP [see Fig. 12(a)]. With further increasing the MAPP content to 3 wt %, the surface of the fibers appeared to be rougher. Interestingly, at the MAPP content of greater than about 5 wt %, the presence of the fibers within the iPP matrix was not clearly distinguishable, indicating a much improvement in the compatibility between the two components. The much improvement in the interfacial adhesion between iPP matrix and the roselle fibers at the MAPP content of

greater than about 5 wt % was the most likely explanation for the observed increase in the tensile strength at yield and the flexural strength of the resulting composites.

CONCLUSIONS

Injection-molded roselle (*H. sabdariffa* L.) fiber-reinforced iPP composites were successfully prepared and reported in the present contribution for the first time. On the effects of type and size of roselle fibers on the mechanical properties of the resulting composites at a fixed fiber loading of 30 wt %, It was found that the addition of pure roselle bast fibers resulted in the overall improvement in the tensile, flexural, and impact properties of the as-prepared composites, while incorporation of pure core fibers only improved the Young's and the flexural modulus of the composites. The property values were generally found to increase with increasing bast fiber content or with increasing lengths, hence the aspect ratios, of the incorporated fibers. In addition, the composites became more rigid, as evidenced by the loss in the elongation at yield. Microscopic observation of fractured surfaces of some selected impact specimens revealed that the surface of the fibers was smooth, indicating poor interfacial bonding of the iPP molecules on the fiber surfaces. On the effects of roselle fiber content and the presence and

content of maleic anhydride (MAPP) on mechanical properties of the resulting iPP composites, incorporation of the roselle fibers at about 40 wt % resulted in a much improvement in many categories of the mechanical properties investigated, without sacrificing much the processability of the resulting composites. Lastly, about 7 wt % of MAPP was required for the compatibilizing effect to become effective in improving some categories of the mechanical properties of the compatibilized composites.

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