



Reinforcing potential of recycled carbon fibers in compatibilized polypropylene composites

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Abstract

Materials that are low cost and lightweight are essential for future automotive and aerospace industries. While carbon fibers have been shown to achieve high mechanical properties for high-end applications, their high cost has generated interest to find cost-effective alternatives. The recent advances in recycling carbon fibers has created an opportunity to develop low cost composite materials. Using a maleic anhydride grafted polypropylene (MA-g-PP), the interfacial adhesion between the polypropylene (PP) matrix and the recycled carbon fibers is greatly improved. The integration of 20 wt% recycle carbon fibers result in 2.4 times, 4.9 times, and 5.7 times enhancements in tensile strength, notched Charpy impact strength, and flexural modulus, respectively. This enhancements in mechanical properties are significantly higher than those reported in previous works. The addition of recycled carbon fibers also increases the heat deflection temperature (HDT) of the neat PP sample, indicating the higher resistance of the composite samples at high temperature. This work demonstrates the great potential of the recycled carbon fibers in manufacturing cost-effective and high-performance PP composites.

Keywords Recycled carbon fibers · Polypropylene composites · Mechanical properties · Compatibilizer

Introduction

The light weight of manufactured products has become a key characteristic of advanced materials in modern industries. This is one of the key reasons scientists and engineers strive to develop polymer compounds and composites with low densities to replace metals and ceramics. The development of lightweight products made of short-fiber reinforced polymer (SFRP) composites is growing in various industries, thanks to their ease of production and remarkable mechanical properties.

Polypropylene (PP) is a widely used polymer because of its low density, low cost, chemical resistance, and good processability. However, its application in advanced products has been limited due to inadequate mechanical properties [1–4]. PP composites reinforced with particles and short fibers are commonly produced by mass production methods [5]. Glass fibers are typically used to reinforce commodity and engineering polymers [6]. However, carbon fibers are becoming more popular in advanced composites for high-tech industries, such as aerospace as well as luxury and sport cars, owing to their higher performance compared to glass fibers [1, 7–11].

Advanced composites employed in structural engineering applications are either under high static loads or repeatedly subjected to impact loadings and high deformation rates. The fracture behavior of these composites is primarily evaluated by the Charpy and Izod impact tests. The following mechanisms contribute in dissipating energy during failure of SFRP: (1) plastic deformation, crazing, and microcracking of the matrix that take place in an area in front of the crack tip, that is absent in brittle matrices, (2) fiber breakage if its length is supercritical and the stress level exceeds beyond the local fiber strength, (3) fiber pull out when the fibers are not long enough (i.e., subcritical length), and (4) fiber-matrix debonding if the strength of the fiber-matrix

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interface is less than the applied load that is transferred to the fibers. Notably, when the fiber fractures, the stored strain energy is mainly dissipated as heat and acoustic energy. Mechanical performance of composites is closely related to the interfacial stress transfer between the components and fiber volume fraction. Effective transfer of stresses between matrix and fibers, which promotes fiber breakage versus fiber pull out, requires a strong interfacial adhesion.

The interfacial adhesion between the fibers and the matrix can be improved through fiber surface modification or employing a coupling agent [11–22]. Wong et al. [16] studied the influence of various grades of polypropylene-grafted maleic anhydride (MA-g-PP) on PP composites containing recycled carbon fibers. Improved interfacial adhesion between the composites' components was observed for the compatibilized samples, leading to less polymer-fiber debonding and fewer fiber pull out. It was found that a higher anhydride content was more efficient in improving the tensile and flexural strengths while the maximum strength was governed by the molecular weight of the compatibilizer to achieve sufficient chain entanglements with the host matrix. Liang et al. [23] studied poly(butylene succinate) composites loaded with carbon fibers at various concentrations, from 5 wt% up to 20 wt%. They observed fiber agglomeration at high fiber concentrations, which weakened the level of mechanical properties reinforcement. Incorporation of 20 wt% carbon fiber into the matrix led to an increase in tensile strength from ~26 MPa to ~62 MPa and ~79% enhancement of impact strength while a dramatic reduction in elongation at break from ~509% to ~8% was observed. In another study, polyethylene (PE) composites containing up to 30 wt% recycled carbon fibers were produced via melt blending. No significant reduction in elongation at break was observed at composites containing 1, 3, and 5 wt% carbon fibers [24]. In contrast, the elongation at break reduced significantly at higher concentrations of recycled carbon fibers, resulting in brittle composites. A 180% increase in the Young's modulus of the composites was reported as the result of incorporating 30 wt% recycled carbon fibers within the PE matrix.

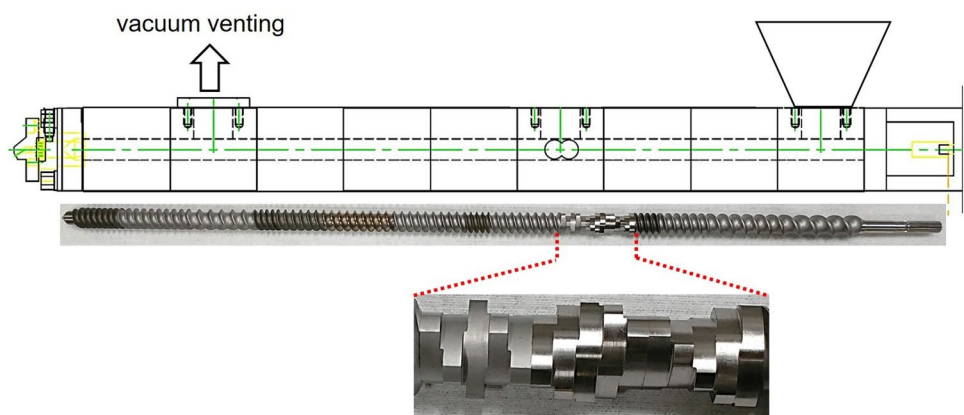
The application of polymer composites reinforced with carbon fibers is rapidly growing. This is because the remarkable gain in the final properties such as strength and stiffness compensates for the relatively high cost of carbon fibers. Since carbon fibers are generally more expensive than commodity and engineering polymers, achieving optimal fiber content that meets the performance requirement of the desired application is crucial. Hence, in this study, recycled carbon fibers, which are around 70% less expensive than the virgin fibers, were used to reinforce PP. This work systematically explores the reinforcement potential of the recycled carbon fibers at various loading in PP composites. A coupling agent is employed to enhance the level of stress transfer between carbon fibers and the PP matrix. A fractographic study using scanning electron microscopy (SEM) is performed to investigate the state of adhesion between the composites' components. To minimize fiber breakage, only forward-conveying screw elements are used after the side feeder, where the carbon fibers are introduced. The potential of this screw profile at a low rotation speed in dispersing the recycled carbon fibers within the PP matrix is investigated. The morphology, stiffness, notched impact resistance, strength, deformability, processability, crystallization, and heat deflection characteristics of the reinforced composites are evaluated at various loadings of the recycled fibers to comprehensively assess the performance of the developed composites. The fiber efficiency factor for various composites is determined according to the modified rule of mixtures and the influence of fiber volume fraction on this parameter is discussed.

Experimental

Materials

A PP homopolymer, Adstif HA801U, produced by Lyondell-Basell, was used in this study. The melt flow index (MFI) of Adstif HA801U was measured as 64 g/10 min, suitable for high-speed injection molding of thin-walled parts. 3 wt% of

Fig. 1 Screw configuration of the twin-screw extruder with one mixing zone, consisting of elliptical kneading elements with staggering angles of 30° and 60°



a maleic anhydride-grafted polypropylene, MA-g-PP, (Polybond 3200, Crompton), with a maleic anhydride grafting level of 2 wt%, was employed as a compatibilizer. 40 mm chopped recycled carbon fibers were supplied by SGL Company and were mixed with the PP matrix at concentrations of 5, 10, and 20 wt%.

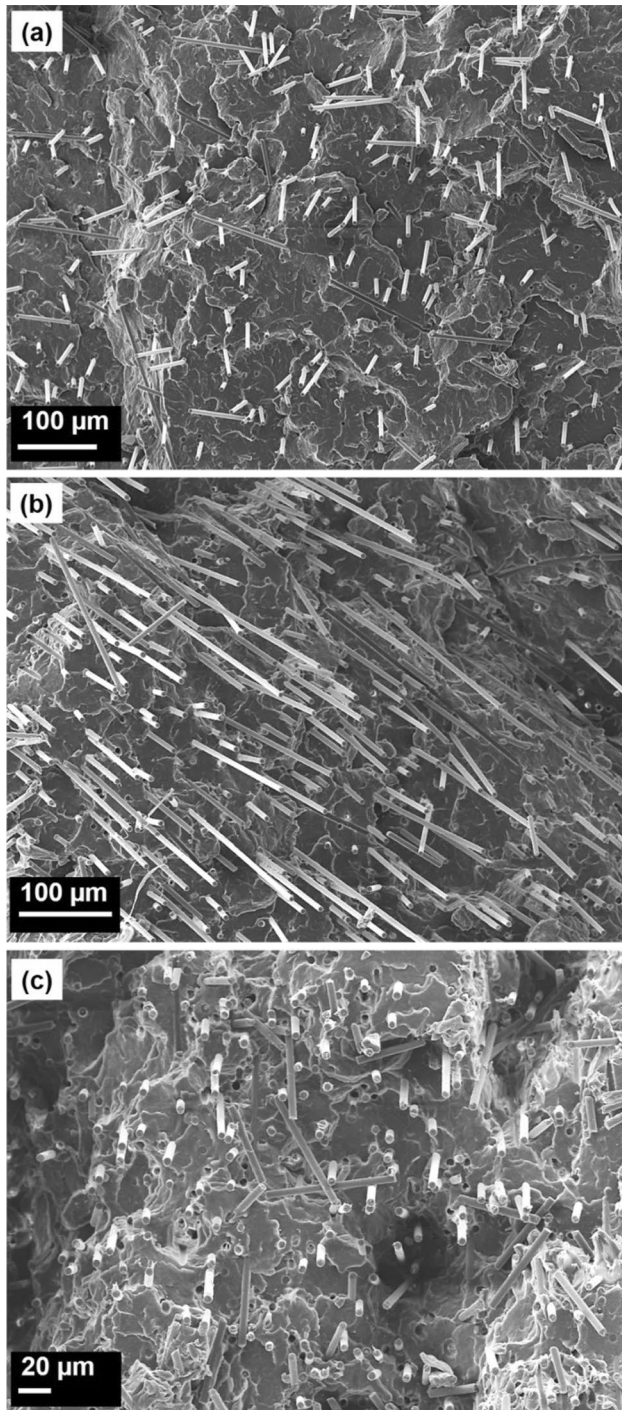


Fig. 2 SEM images of the cross-section morphology of the PP composites containing 5 wt% (a), 10 wt% (b), and 20 wt% (c) recycled carbon fibers

Sample preparation

Carbon fiber reinforced composites used in this work were manufactured by a twin-screw extruder with $L/D = 36$. A side feeder was employed to add the recycled carbon fibers. Melt compounding was performed at 100 rpm and a temperature profile of 180 °C to 230 °C. Figure 1 exhibits the screw geometry of the extruder consisting of kneading elements with staggering angles of 30° and 60°. The extrudate was dried prior to molding ISO 527 Type 1A dumbbell-shaped test bars with a nominal gauge length, width, and thickness of 75 mm, 10 mm, and 4 mm, respectively, using a Battenfeld injection molding machine at ~220 °C.

Characterization

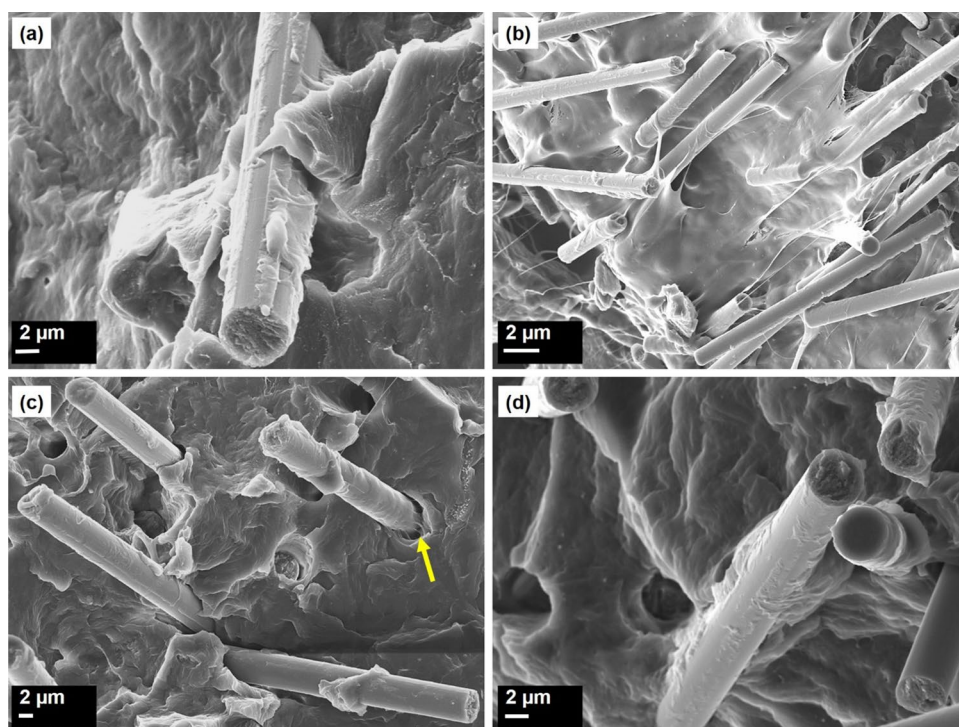
A Tinius Olsen machine H10KS tensometer equipped with a 10 kN load cell was used to evaluate tensile and flexural characteristics of the samples at a rate of 50 mm/min and 2 mm/min, according to ISO 527 [25], determination of tensile properties, and ISO 178 [26], determination of flexural properties, respectively. The notched Charpy impact strength at 23 °C was determined according to ISO 179 [27], determination of Charpy impact properties, using a pendulum impact tester (Testing Machines Inc.). Between five to ten samples were tested for evaluating the mechanical properties. Melt flow rate of the samples were measured using MP600 (Tinius Olsen) according to ISO 1133 [28], determination of the melt mass-flow rate, at 230 °C using a weight of 2.16 kg. The heat deflection temperature (HDT) characterization was conducted under 1.82 MPa bending load according to ISO 75-1 [29], determination of temperature of deflection under load, by employing 603 HDTM instrument (Tinius Olson).

Differential scanning calorimetry (DSC) measurements were performed under a helium atmosphere (DSCQ1000, TA Instruments). The samples were heated from 20 °C to 200 °C and held at that temperature for 3 min, then cooled to 20 °C and heated again to 200 °C at a constant rate of 10 °C min⁻¹. The state of fiber dispersion in various samples was evaluated by scanning electron microscopy (SEM) using a Hitachi S4700 microscope. SEM samples were prepared by cryogenically fracturing the neat polymer or composites in liquid nitrogen and coating with a 3 nm layer of gold before the analysis.

Results and discussion

The morphology of the composites was examined by SEM to determine the state of carbon fiber dispersion in the polymer matrix, the reinforcement mechanisms, and the state of interface between the matrix and the recycled fibers. The

Fig. 3 Cryogenic breakage morphology of the PP composites reinforced with 5 wt% (a), 10 wt% (b and c), and 20 wt% (d) recycled carbon fibers



observation of the surface morphology and cross section of the composites revealed that the carbon fibers were dispersed relatively uniformly in the composites and there were no obvious agglomerations. The surface morphology of the composites (not shown here) exhibited that carbon fibers were preferentially aligned in the direction of flow. This preferential orientation of the carbon fibers within the PP matrix can be better observed in the cryogenically fractured cross section of the composites (Fig. 2). Injection molded fiber reinforced thermoplastics usually exhibit a marked fiber orientation in the flow direction and are consequently anisotropic. This preferential orientation is the result of the shear

stresses that the molten composites experience while filling the mold [1, 8, 30].

Detailed analysis of the cryogenically fractured cross section of the composites (Figs. 2 and 3) revealed that the majority of the carbon fibers at the fracture surface were broken. This observation suggests that there is a strong interaction between PP and carbon fibers emanating from the good compatibility of the composite components. Some carbon fibers were pulled out of the PP matrix leaving holes within the fractured surface. The mechanical characteristics of the composites are governed by the microfailure mechanisms at the fiber/matrix interface. Careful observation of

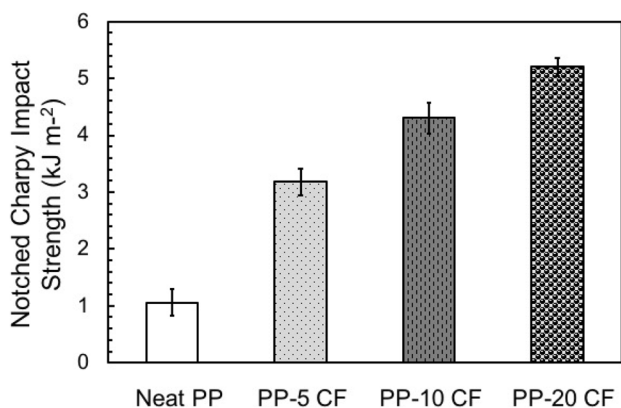


Fig. 4 Impact strength of the neat PP homopolymer and its composites reinforced with various concentrations of the recycled carbon fibers

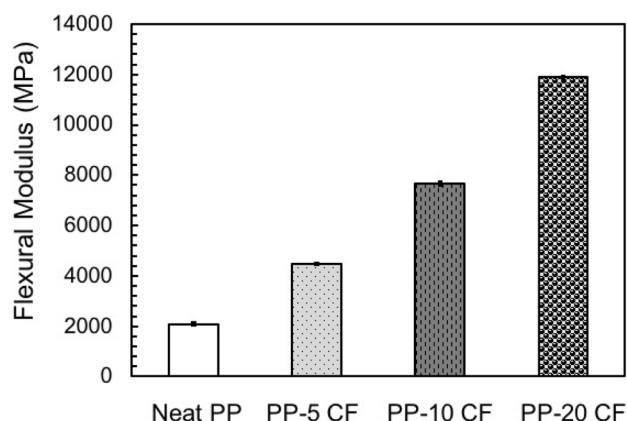
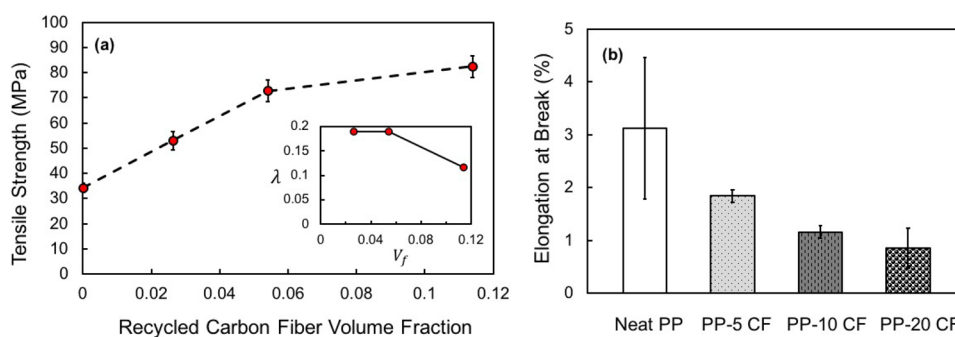


Fig. 5 Flexural modulus of the neat PP homopolymer and its composites reinforced with various concentrations of the recycled carbon fibers

Fig. 6 Tensile strength (a) and elongation at break (b) of the neat PP homopolymer and its composites reinforced with various concentrations of the recycled carbon fibers. The inset in (a) shows the fiber efficiency factor as a function of carbon fiber volume fraction



the SEM micrographs at higher magnifications (Fig. 3) indicated that the cohesive failure of the matrix close to the interface caused the interfacial microfailure, rather than fiber/matrix debonding. The arrow in Fig. 3c indicates the elongated strips of the matrix at the interface that prevents fiber debonding and pullout. Further, carbon fibers were also covered with residual polymer, confirming the strong fiber/matrix interfacial adhesion. These results imply that carbon fibers are well embedded within the compatibilized PP matrix.

Figure 4 presents the impact strength of the PP homopolymer and its carbon-fiber reinforced composites. The impact strength of the matrix increased monotonically with the carbon fiber's concentration whereby at 20 wt% recycled carbon fibers, the notched Charpy impact strength of the homopolymer showed 390% enhancement (from $\sim 1.1 \text{ kJ m}^{-2}$ to $\sim 5.2 \text{ kJ m}^{-2}$). Crack propagation in the notched composites is hindered by fiber pull-out, interfacial debonding, fiber bridging, and matrix plastic deformation [7, 18]. Since brittle matrices like PP homopolymers have low impact strengths, the matrix-dominated energy absorption mechanisms are marginal in these composites. Thus, the improvement in impact strength of the composites is mainly associated to extrinsic toughening mechanisms induced by the fibers [7].

The flexural modulus of the neat matrix and composites was also measured to investigate the bending resistance of the produced composites (Fig. 5). A steady increase in stiffness of the composites was observed with the carbon

Table 1 Summary of the mechanical properties of the neat PP homopolymer and the reinforced composites containing 5, 10, and 20 wt% recycled carbon fibers

Sample Name	Impact Strength (kJ m^{-2})	Flexural Modulus (MPa)	Tensile Strength (MPa)
Neat PP	1.1 ± 0.2	$2,084 \pm 59$	34.2 ± 1.9
PP-5 CF	3.2 ± 0.2	$4,482 \pm 37$	53.0 ± 3.5
PP-10 CF	4.3 ± 0.3	$7,670 \pm 88$	72.8 ± 4.4
PP-20 CF	5.2 ± 0.1	$11,900 \pm 56$	83.0 ± 4.3

fiber's concentration. The composites reinforced with 20 wt% recycled carbon fibers exhibited a flexural modulus of 11,900 MPa, which is around 5.7 times larger than that of the neat polymer. This enhancement in modulus is the result of an effective load transfer from the PP matrix to the rigid carbon fibers. The improvement of flexural modulus achieved in this study is higher than the enhancement value of just below 4 times reported for PP composites reinforced with the same concentration of recycled carbon fibers [19]. This result indicates that the highly reinforced PP composites with recycled carbon fibers produced in this work can withstand high bending deformations and thus have the potential to be used as structural parts.

The analysis of the tensile properties of the neat matrix and composites (Fig. 6) showed that the tensile strength of the matrix increased steadily with the carbon fiber concentration from $\sim 34 \text{ MPa}$ for the neat PP polymer to 83 MPa for the composite with 20 wt% recycled carbon fibers. This is equivalent to a remarkable enhancement of 144% in tensile strength, which is significantly higher than the enhancement values of 77% and 114% reported in the literature at the same carbon fiber concentration [2, 3]. The tensile strength of composite systems can be explained by the rule of mixtures. The extended form of rule of mixtures presented by Cox [31] and Krenchel [32] takes into account the effects of

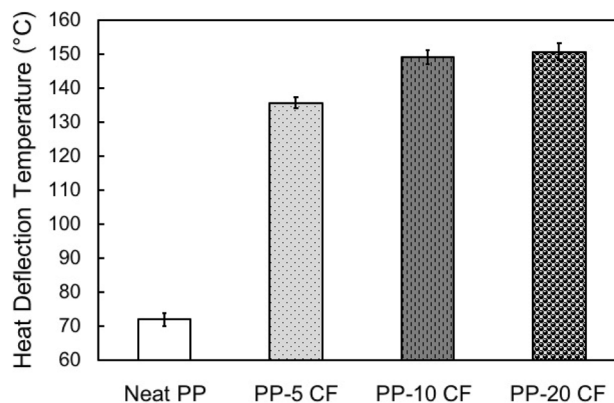


Fig. 7 HDT of the neat PP homopolymer and its composites reinforced with various concentrations of the recycled carbon fibers

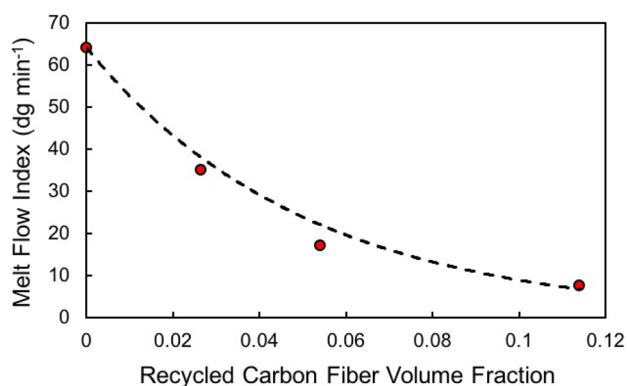


Fig. 8 MFI of the neat PP homopolymer and its composites reinforced with various concentrations of the recycled carbon fibers

fiber length and orientation. The modified rule of mixtures (Eq. 1) with a fiber orientation factor η_0 and a fiber length efficiency factor η_1 was used to model the tensile strength of the composites σ_c and to evaluate the fiber efficiency factor λ at various concentrations:

$$\sigma_c = \lambda \sigma_f V_f + \sigma_m (1 - V_f) \quad (1)$$

$$\lambda = \eta_0 \times \eta_1 \quad (2)$$

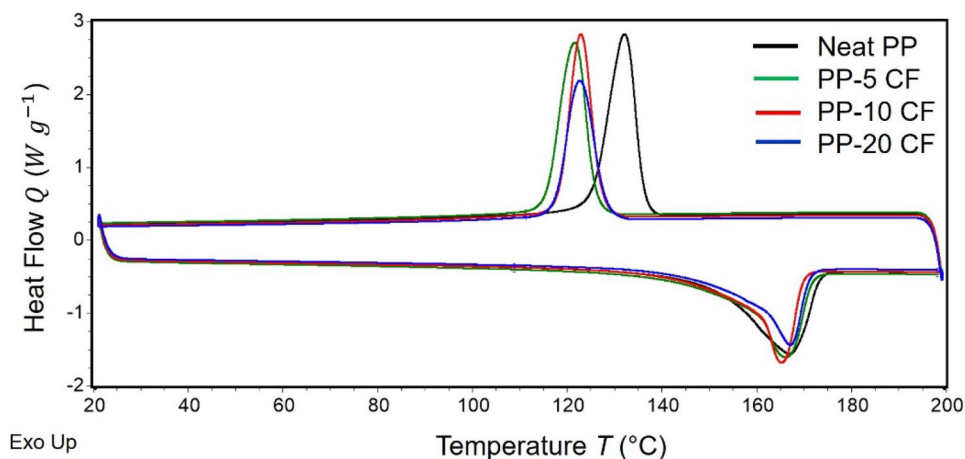
where σ_f and σ_m are fiber and matrix strength, respectively and V_f is the volume fraction of the fibers in the composites.

In this study, the fiber orientation factor η_0 was assumed to be equal to 1, as the fibers showed high alignment along the flow direction in all composites. Hence, the fiber efficiency factor in injection molded composites with oriented fibers is mainly influenced by fiber length [1]. For carbon fibers composites in this work, the fiber efficiency factor decreased with increasing carbon fiber concentration (inset in Fig. 6a). This is because, at high concentrations, the probability of fiber

attrition increases due to fiber–fiber and fiber–equipment interactions during processing. Accordingly, the reduction of fiber length is more pronounced at higher fiber concentrations which is reflected in relatively lower enhancements in tensile strength at high fiber loadings [30]. Figure 6b shows the elongation at break of the neat PP homopolymer and the reinforced composites. The elongation at break of the neat polypropylene homopolymer is fairly low (~3%). The neat samples and the reinforced composites fractured in a brittle manner with very low deformations. The introduction of the fibers reduced the tensile strain of the samples to even lower values due to higher stress concentrations at the fiber ends, which facilitates microcrack initiation. Moreover, carbon fibers are almost non-deformable and hinder polymer chains from sliding one another, during tensile testing, due to the strong interfacial adhesions. Table 1 summarizes the values of tensile, flexural, and impact properties of the neat samples and composites reinforced with the recycled carbon fibers at various loadings.

HDT measurements were performed to assess the maximum temperature at which the reinforced composites can be used as a rigid material (Fig. 7). The comparison of the neat PP homopolymer and the reinforced composites (under a load of 1.82 MPa) showed that the presence of carbon fibers led to a significant enhancement of HDT in the reinforced composites. For instance, by introducing just 5 wt% recycled carbon fibers, the HDT of the samples increased by 64 °C compared to the neat polymer. The HDT of the reinforced composite further increased to ~151 °C upon incorporating 20 wt% recycled carbon fibers (79 °C higher than HDT of the neat polymer). The higher HDT of the composites reinforced with the carbon fibers could be due to the modulus enhancement and the restricted relaxation of polymer chains in the presence of the fibers at high temperatures. This restricted relaxation of polymer chains is caused by the strong interactions between the fibers and the PP matrix due to the presence of the MA-g-PP compatibilizer.

Fig. 9 Thermal properties of the neat PP homopolymer and its composites reinforced with various concentrations of the recycled carbon fibers



The MFI of the polypropylene homopolymer and the composites were measured to evaluate the effect of carbon fibers, at various loadings, on the processability of the composites. Incorporation of fillers and reinforcing particles into thermoplastic polymers usually increases viscoelastic properties of the matrix. Figure 8 exhibits that the flowability of the matrix reduces almost exponentially by fiber concentration. The higher melt viscosity of the composites is attributed to fiber entanglement and network formation, which is more pronounced at higher concentrations, perturbing the flow of molten polymer. Moreover, restriction of macromolecules mobility due to strong interactions between the fibers and polymer chains may hinder flowability of the matrix. The introduction of 20 wt% carbon fiber reduces the MFI value of the matrix to ca. 8 dg min⁻¹, confirming that injection molding, as a common processing technique, can be employed to produce composite parts even from the highly reinforced samples [33–36].

DSC has also been used to explore thermal properties of the neat and composite samples. The DSC thermograms of the neat and composite samples (Fig. 9) showed that the addition of the recycled carbon fibers had no remarkable influence on melting temperature of the reinforced composites. The DSC results were further analyzed and the thermal characteristics of the samples were summarized in Table 2. While the melting temperature (T_m) was constant at ~166°, the hot crystallization temperature (T_{hc}) reduced from ~132° to ~123° with the addition of carbon fibers. Several factors affect nucleation and growth of crystals in polymers and composites. The strong interfacial adhesion between the fibers and polymer chains restricts the migration and diffusion of the macromolecules to the surface of the nuclei, leading to lower T_{hc} [37–40]. It seems that the heterogeneous nucleating effect of carbon fibers on T_{hc} is suppressed by macromolecular mobility hindrance. The degree of crystallinity (χ_c) of the samples was also calculated according to the Eq. 3:

$$\chi_c = \frac{\Delta H_m}{w_m \times \Delta H^\circ} \quad (3)$$

where ΔH_m , w_m , and ΔH° are the melting enthalpy, matrix weight fraction, and the enthalpy of fusion of a pure crystalline

Table 2 DSC analysis results of the neat PP homopolymer and the reinforced composites containing 5, 10, and 20 wt% recycled carbon fibers

Sample Name	T_{hc} (°C)	T_m (°C)	ΔH_m (J g ⁻¹)	χ_c (%)
Neat PP	132.1	166.9	113.2	54.7
PP-5 CF	121.5	166.2	97.2	49.4
PP-10 CF	122.9	165.2	90.1	48.4
PP-20 CF	122.7	167.1	82.1	49.6

PP (~207 J g⁻¹ [41]), respectively. The crystalline content of the composites reduced slightly with the addition of carbon fibers. For instance, χ_c of the composite sample was ~49.6 which is ~9% lower than that of the neat matrix (~54.7). This decrease in χ_c could be due to the immobilization of the matrix chains due to the addition of carbon fibers, which prevents them from obtaining ordered crystal lattice alignment, thus obstructing the crystal growth in the matrix [37–40].

Conclusion

Polypropylene composites containing recycled carbon fibers at various concentrations were prepared by extrusion followed by injection molding. To maximize the reinforcement potential of the carbon fibers, the interfacial shear strength between the matrix and the carbon fibers was enhanced using a polypropylene grafted maleic anhydride compatibilizer. The morphological assessment revealed that the recycled carbon fibers were uniformly distributed and individually dispersed within the matrix even at the high concentration of 20 wt%. The composites exhibited a remarkably higher tensile strength, flexural modulus, impact strength, and heat deflection temperature compared to the neat matrix. The results showed that the fiber efficiency factor for tensile strength decreased with increasing fiber volume fraction. Detailed morphological characterizations indicated that the interfacial microfailure was predominantly induced by the cohesive failure of the matrix at the vicinity of the carbon fiber/matrix interface, rather than debonding between the composite components. The thermal characterization of the samples showed that while melting point was not influenced by the addition of the carbon fibers to PP, the hot crystallization temperature and the crystalline content of PP reduced slightly. This work demonstrated the great potential of recycled carbon fibers in the manufacturing of cost-effective high-performance polypropylene composites.

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