

## Research Article

# Carbon Fiber Composites of Pure Polypropylene and Maleated Polypropylene Blends Obtained from Injection and Compression Moulding

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A comparative study of the mechanical performance of PP and PP/PP-g-MAH blends reinforced with carbon fibre (CF) obtained by two different moulding techniques is presented. Three filler contents were used for fabricating the composites: 1, 3, and 5 pph (parts per hundred). The crystallisation behaviour of the composites was studied by differential scanning calorimetry. Morphological and structural features of these samples were observed by atomic field microscopy and Fourier-transform infrared spectroscopy, respectively. Mechanical properties of the injection and compression moulded composites were evaluated by means of tensile and impact resistance tests. The fracture surface of the impacted samples was observed by scanning electron microscopy. The processing method had a noticeable effect on the results obtained in these tests. Young's modulus was enhanced up to 147% when adding 5 pph CF to a PP matrix when processed by compression moulding. Addition of PP-g-MAH and CF had a favourable effect on the tensile and impact strength properties in most samples; these composites showed improved performance as the filler content was increased.

## 1. Introduction

Polypropylene possesses an attractive combination of mechanical properties which can be further improved by the modification of this polymer in order to obtain polymer blends and also by the incorporation of reinforcing fillers [1–3]. Grafting maleic anhydride (MAH) into the PP molecular segments is a common approach for the modification of this polymer. Maleated PP (PP-g-MAH) can be used as a nucleating agent or compatibiliser for blends and composites [2–4]. Polymer blending is a very important technique in the industrial practice and it is mainly used to achieve properties that are more advantageous than those of the individual components [1, 5].

The miscibility between PP and PP-g-MAH has been studied by several authors. Depending on the molecular weight and MAH content in PP, the blending can either cocrystallise or phase separate; this in turn will influence the mechanical properties of PP/PP-g-MAH blends [6, 7]. Furthermore, isothermal crystallisation of PP/PP-g-MAH blends confirmed that there is a correlation between the crystallisation rate and the resulting morphologies [6]. Other authors have also shown that the thermodynamic properties of PP/PP-g-MAH blends have improved the mechanical responses depending on the content of the compatibiliser [8, 9]. Additionally, the mechanical properties of polymers are found to be strongly dependent on the processing technique [7]. They can impart different morphologies and

crystal structures in semicrystalline polymers [5, 10–12]. Moulding techniques are widely used in the plastic industries where injection and compression moulding are typically used for obtaining a variety of products. Another approach for improving the mechanical properties of polymers is by the addition of reinforcement fillers, such as those based on carbon [9–12]. Carbon fibre (CF) reinforced polymer composites have demonstrated having many applications in diverse fields due to their ability to obtain a synergetic effect when combining toughness of the thermoplastic polymer with the stiffness and strength of carbon fibres [12]. Carbon based composites can be found in the high technology sector, which includes aerospace and the automotive industries, including engineering components such as bearings, gears, fan blades, and chassis structures. Due to the importance of these composites and the high number of applications PP moulded parts have in the industry, this paper presents the influence of CF on the tensile and impact strength of PP and PP-g-MAH/PP blends. Furthermore, to our knowledge, this is the first study that shows the effects of these composites when moulded by two different methods, injection and compression moulding, and the behaviour that the PP-g-MAH has when rigid fillers are added to PP.

## 2. Experimental Methods

**2.1. Materials.** PP homopolymer XH1760 was supplied by Indelpro (Mexico) with a melt flow index of 3 g/10 min at 230°C/2.16 kg. The material was a commercially stabilized polymer without any further additives, especially without nucleating agents. Polybond 3200 with a melt flow of 115 g/10 min at 230°C/2.16 kg obtained from Chemtura was employed as PP-g-MAH compatibiliser. AGM-99 milled CF produced from PAN with a mean length of 150 microns and a diameter of 8 microns were obtained from Asbury Carbons and used as received.

**2.2. Sample Preparation.** A twin-screw corotating Brabender Plasti-Corder was used to prepare the PP composites and composite blends (PP/PP-g-MAH) which contained 5 pph PP-g-MAH. The screw speed was 60 rpm and they were compounded at  $200 \pm 10^\circ\text{C}$  during 10 min. The polymer and polymer blends were reinforced with CF at three different contents: 1 pph, 3 pph, and 5 pph. These composites were moulded using compression and injection compounding. A Beutelspacher single screw extruder at a screw speed of 50 rpm and temperature profile set from 175°C in the feed section to 190°C at the die was used to prepare the extruded samples. The compounds were fabricated by a Sumitomo injection machine at a moulding temperature of 200°C. The compression moulded samples were prepared at 200°C and 5 tons for 5 min in a Carver press which was followed by cold pressing at 5 tons for 10 min.

**2.3. Characterisation Techniques.** Fourier-transform infrared (FTIR) spectroscopy was carried out on Perkin Elmer Spectrum 100 spectrometer with an ATR device. The sample films were analysed by atomic field microscopy (AFM) in tapping

mode using an Innova Veeco microscope. Differential scanning calorimetry (DSC) studies were conducted on a Q2000 TA Instrument. All samples weighed approximately 6 mg and were sealed within aluminium pans. The samples were heated from  $-30$  to  $200^\circ\text{C}$  and maintained at this temperature for 1 min and then they were cooled down to  $-30^\circ\text{C}$  to be equilibrated at 1 min; both heating and cooling rates were conducted at  $10^\circ\text{C}/\text{min}$ . Subsequently, a similar procedure was done at  $50^\circ\text{C}/\text{min}$ .

Compression moulded samples ( $1.5 \times 3 \times 9 \text{ mm}^3$ ) and injection moulded samples ( $3 \times 3 \times 9 \text{ mm}^3$ ) were subjected to tensile test using an Instron 3365 Universal Materials testing machine at room temperature using a crosshead speed of 5 mm/min and a 5 kN full-scale load cell. All results presented are the average values of three measurements. Izod impact tests were done following ASTM D256 using CEAST Izod pendulum impact tester using a nominal hammer energy of 2 J, and measurements were done in ten samples. Morphology analysis of the impacted areas was made by scanning electron microscopy (SEM) using a JEOL JSM-7600TFE microscope at 2 kV accelerating voltage.

## 3. Results and Discussion

Extruded composites obtained from the Brabender Plasti-Corder were analysed by calorimetry in order to see the crystallisation properties of these samples. Table 1 shows the resulting melting and the crystallisation temperatures ( $T_m$  and  $T_c$ , resp.) obtained from the melting and crystallisation curves during the first ( $10^\circ\text{C}/\text{min}$ ) and second ( $50^\circ\text{C}/\text{min}$ ) run. All samples presented a melting temperature  $\sim 160^\circ\text{C}$  corresponding to the presence of  $\alpha$ -crystals. More evident changes were found in the crystallisation thermograms, where at low cooling rate crystallisation started at higher temperatures, because there was more time to overcome the nucleation energy barriers while at high cooling rate the nuclei became active at lower temperatures. Furthermore, addition of CF led to an increase in the crystallisation temperature as the filler content was increased in those samples containing PP-g-MAH as compatibiliser, indicating its addition favoured the formation of nuclei due to a synergistic effect with the CF. This could be related to the incorporation of PP-g-MAH which is well known for increasing the flow properties, therefore favouring the dispersion of the CF in the PP matrix which allowed the composite blends to crystallise in a smaller time. The crystallisation temperature was increased up to  $7^\circ\text{C}$  for the sample obtained from PP/PP-g-MAH and containing 5 pph CF.

According to the fusion enthalpy values included in Table 1, there were noticeable differences between the first and the second run of the samples and also between the composites obtained from the pure PP or the PP/PP-g-MAH blend. Additionally, incorporation of carbon fibres also had an effect in the crystallinity of the samples. The considerable differences between the first and the second value are related to the recrystallisation process during the second run conducted at higher rate. As explained previously, this allowed the crystallisation of the sample at higher temperature and

TABLE 1: Crystallisation properties of the samples.

Sample	Heating and cooling rate					
	10 C/min		50 C/min			
	$T_m$ , C	$T_c$ , C	$\Delta H_m$ , J/g	$T_m$ , C	$T_c$ , C	$\Delta H_m$ , J/g
Pure PP	156.45	116.07	91.21	158.75	107.12	75.97
1 pph CF	161.41	116.12	78.78	160.1	105.47	61.34
3 pph CF	158.62	117.1	74.5	157.84	107.19	60.06
5 pph CF	157.58	118.66	72.77	156.53	109.73	58.94
PP/PP-g-MAH	156.42	113.24	72.99	158.03	103.99	70.83
1 pph CF	157.58	118.66	90.77	156.53	109.73	59.94
3 pph CF	157.09	118.46	83.53	159.43	110.01	62.93
5 pph CF	159.37	119.48	75.38	159.69	111.05	64.12

applying this higher cooling rate after the first melting also restricted the amount of crystals to be nucleated and caused a lower degree of crystallinity. The crystallinity degree can be calculated from the enthalpies of fusion ( $\Delta H_m$ ) using the following equation:  $X_c = 100(\Delta H_m/\Delta H_0)$ , where  $\Delta H_m$  is the enthalpy of fusion of the samples obtained from the integration of DSC melting curves and  $\Delta H_0$  is the enthalpy of fusion of 100% crystalline polypropylene taken as 209 J/g [9]. Samples containing pure PP had a higher percentage of crystallinity (44%) compared to those obtained from the PP/PP-g-MAH blend (35%). It is well known that PP-g-MAH has lower degree of crystallinity; therefore, this could be an explanation to the crystallinity reduction in the samples obtained from the blends [13]. Interestingly, addition of CF in the blends has been shown to improve this property, especially at lower content of this reinforcing material. On the other hand, addition of CF to the pure PP matrix was not favourable for the crystallinity of the samples.

AFM was employed to investigate the surface morphologies of the samples; Figure 1 contains the images of this analysis. Lee et al. found that iPP and PP-g-MAH exhibited different surface morphologies [14]. The features found by them agree well with the relatively smooth surface of PP-g-MAH displayed in Figure 1(a). Carbonyl groups in MAH have been related to promote heterogeneous nucleation; therefore, small sized spherulites may have been developed in this sample which could not be noticeable in our samples [15]. On the other hand, the rest of the samples clearly showed spherulitic morphology. The PP/PP-g-MAH sample displayed large spherulites with dimensions ranging from 20 to 40  $\mu\text{m}$ ; see Figure 1(b). In addition, as displayed in Figure 1(c), incorporation of CF exhibited smaller spherulites as seen in 1 pph CF/PP, their dimensions are within 10–20  $\mu\text{m}$ , and this can be explained by the impingement of neighbouring spherulites by the CF subsequently truncating these structures in their contact regions and stopping their growth. Interestingly, CF/PP/PP-g-MAH sample (Figure 1(d)) also showed a decrease in spherulite size which is not as evident as the previous sample due to the reduction on the surface roughness attributed to the incorporation of MAH. The few parallel trenches and small defects were originated from the film manufacturing process.

TABLE 2: FTIR peak assignments of iPP.

Peak position, $\text{cm}^{-1}$	Vibrational modes
810	$\gamma(\text{CH}_2)$
841	$\nu(\text{CH}_2)$
900	$\gamma(\text{CH}_3)$
970	$\gamma(\text{CH}_3), \nu(\text{C}-\text{C})$
1000	$\gamma(\text{CH}_3), \nu(\text{C}-\text{CH}_3)$
1170	$\nu(\text{C}-\text{C})$
1380	$\delta(\text{CH}_3)$ asymmetrical
1455	$\delta(\text{CH}_3)$ symmetrical
2840	$\nu(\text{CH}_2)$ symmetrical
2920	$\nu(\text{CH}_2)$ asymmetrical
2950	$\nu(\text{CH}_3)$ asymmetrical

Key:  $\gamma$ : rock,  $\nu$ : stretch, and  $\delta$ : bend.

Figure 2 shows the FTIR spectra of PP/PP-g-MAH, 5 pph CF/PP, and 5 pph CF/PP/PP-g-MAH. The peaks located within the 800–1300  $\text{cm}^{-1}$  range are characteristic of the stretching and bending vibrations of the hydrocarbon structure of PP. Table 2 details more information regarding this area [2, 16, 17]. The peak located at  $\sim 1730 \text{ cm}^{-1}$  is due to the C=O stretch of carboxylic acid of hydrolysed anhydride [18]. Samples containing the compatibiliser show this peak which is associated with the maleic anhydride from the PP-g-MAH.

Figures 3 and 4 show Young's modulus values of the CF composites. In the compression moulded samples, addition of 5 pph CF enhanced this property up to 77% in the composites obtained from pure PP, as seen in Figure 3. On the other hand, when PP-g-MAH was blended into the PP matrix, Young's modulus was increased to a lower degree (12% enhancement at the highest CF content). Addition of 1 pph CF to PP/PP-g-MAH decreased slightly the stiffness of the compression moulded composites, accompanied by an increase in tensile strength, as it will be seen in the next section. Nevertheless, at higher CF content, the modulus was improved, indicating that the content of the filler was high enough to favour this property imparting rigidity to the polymer matrix. In a similar trend as described previously, the reinforcement effect provided by CF was superior in

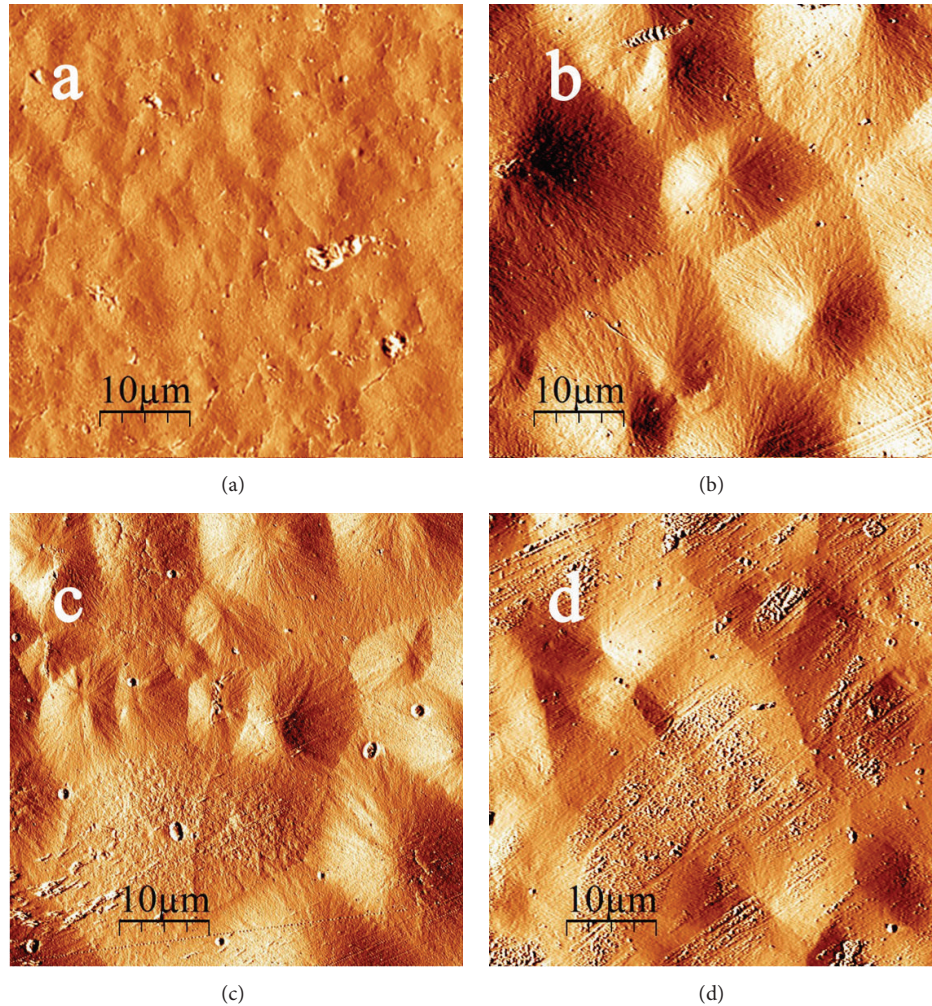


FIGURE 1: AFM images of (a) PP-g-MAH, (b) PP/PP-g-MAH, (c) 1 pph CF/PP, and (d) 1 pph CF PP/PP-g-MAH.

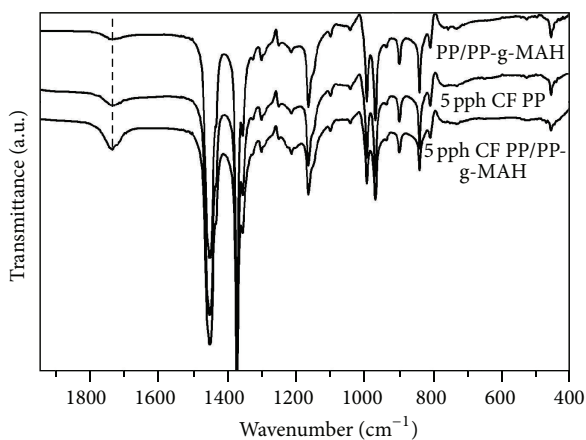


FIGURE 2: FTIR of the extruded samples.

the injection moulded samples containing pure PP. Young's modulus showed an increased response upon incorporation of CF in the PP matrix. This property was enhanced up

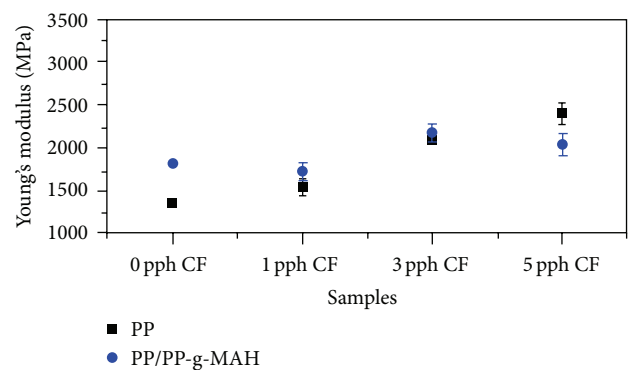


FIGURE 3: Young's modulus of compression moulded samples.

to 147% when adding 5 pph CF, as displayed in Figure 4. Regarding the composites obtained from the PP/PP-g-MAH blend, the incorporation of CF is favoured to the stiffness of all the injection moulded samples. Young's modulus was increased up to 35% for the highest CF content.

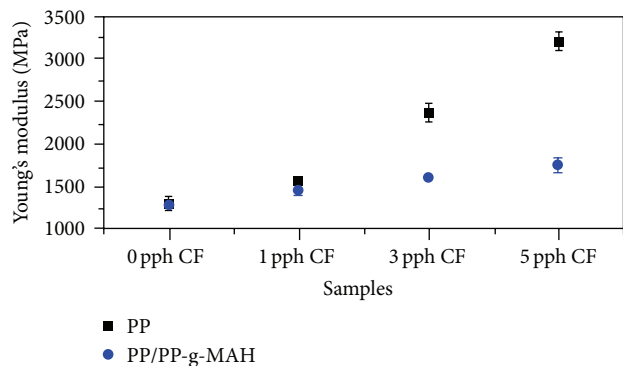


FIGURE 4: Young's modulus of injection moulded samples.

TABLE 3: Tensile strain at break of the compression moulded composites.

Sample	Elongation, %	Std. error
PP	1160.45	53.27
1 pph CF	661.09	87.94
3 pph CF	12.83	1.55
5 pph CF	11.86	2.98
PP/PP-g-MAH	114.88	11.03
1 pph CF	143.28	60.27
3 pph CF	11.64	4.84
5 pph CF	12.06	3.79

The results show the different behaviour that PP-g-MAH can provide when compounded by two different techniques, which is more evident for the injection moulded samples. In the compression moulded samples, the PP/PP-g-MAH blend showed superior modulus at 0 and 1 pph CF content, as compared to pure PP, and this was associated with a brittle behaviour of the samples, resulting in a large reduction of elongation, which is further reduced by a higher content of CF, as displayed in Table 3. A more noticeable difference in the modulus of the samples was found in the injection moulded samples. From Figure 4, it is clearly seen that this property is greatly affected when adding the maleated PP to the PP matrix. Remarkably, this reduction in the modulus is accompanied by a moderate increase of the elongation of the samples and the values are kept even at high loading of the reinforcing filler (see Table 4), indicating the PP-g-MAH had a plasticizer effect in the blend when the composites were obtained by this technique. Järvelä et al. have shown that, at a suitable ratio of PP to PP-g-MAH (similar to that used in this work), the latter had a plasticising function when compounded by injection moulding [19].

Addition of rigid fillers usually restricts the chain mobility of polymer molecules. Since the chains cannot move freely, the strain at break is reduced with increasing CF content, which explains the elongation behaviour found in the compression moulded samples. This behaviour is in agreement with the study conducted by Karsli and Aytac [9]. On the contrary, elongation values were superior in the injection moulded samples. Furthermore, incorporation of the CF

TABLE 4: Tensile strain at break of the injection moulded composites.

Sample	Elongation, %	Std. error
PP	1016.58	54.30
1 pph CF	1195.763333	27.55
3 pph CF	1212.49	30.97
5 pph CF	984.97	2.70
PP/PP-g-MAH	1288.83	64.79
1 pph CF	1236.52	25.17
3 pph CF	871.73	136.53
5 pph CF	994.10	79.73

TABLE 5: Yield strength of the compression moulded composites.

Sample	Strength, MPa	Std. error
PP	38.82	1.21
1 pph CF	38.26	2.14
3 pph CF	44.16	1.13
5 pph CF	43.27	0.80
PP/PP-g-MAH	37.42	0.41
1 pph CF	44.43	1.30
3 pph CF	40.31	0.56
5 pph CF	42.68	1.72

showed only a slight decrease in this property, indicating a synergistic behaviour between the filler and the matrix, and this is further favoured by the addition of PP-g-MAH as shown in Table 4. This synergistic effect will be better seen in the values of impact strength.

From the above mentioned results, it is noticeable that the performance obtained by the samples processed by injection moulding stands out when compared with compression moulded composites. The different behaviour between both processes upon the incorporation of CF can be explained in terms of the orientation of the polymer chains and the filler. Injection moulding allows the polymer chains and CF to orientate parallel to the flow direction, which is not the case for the compression moulding process. The injection moulding samples were tested in the flow direction thus favouring the elongation response; this explains the large difference between the elongation responses of both processes.

Table 5 shows that the tensile strength was slightly improved in the compression moulded composites. The highest enhancement of this property was accomplished by the addition of 1 pph CF in the PP/PP-g-MAH blend, resulting in a 20% increase. Table 6 displays that the injection moulded samples did not show noticeable increases; these results indicate that CF contributed mainly to the stiffness of the composites. However, addition of CF to the PP samples showed a good mechanical performance based on the reinforcing effect seen by improving the modulus and also by maintaining the tensile strain close to that of the pure polymer.

Plastic deformation of the polymer matrix is the main energy absorbing process in impact tests [20]. Since injection

TABLE 6: Yield strength of the injection moulded composites.

Sample	Strength, MPa	Std. error
PP	61.64	1.42
1 pph CF	60.21	1.67
3 pph CF	60.16	1.02
5 pph CF	57.98	0.72
PP/PP-g-MAH	55.59	1.22
1 pph CF	59.46	1.62
3 pph CF	49.34	4.73
5 pph CF	49.73	2.70

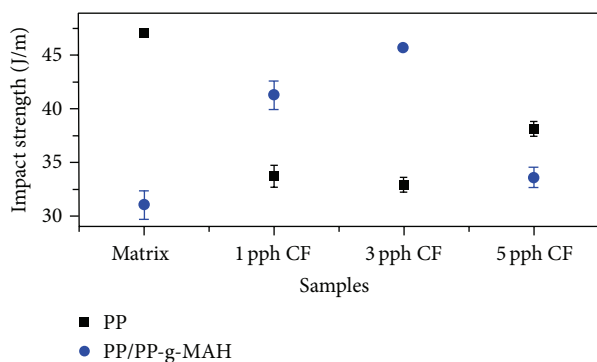


FIGURE 5: Izod impact test values of the injection moulded samples.

moulded samples showed a longer plasticity region, these samples were studied in order to see the effect of the CF and PP-g-MAH on the impact strength of the composites. Figure 5 shows that the composites based on the PP matrix had lower values of impact strength and at higher content of the reinforcing filler this property was improved slightly. The drop of this property with addition of CF could have been by the restriction of chain mobility due to this rigid filler and to the poor interfacial bonding between the filler and the polymer matrix and also due to the creation of stress region concentrations around the carbon fibres that required less energy to initiate the cracking propagation and the resulting microcracks propagated, therefore reducing the resistance to impact [9, 21]. However, when increasing the CF content, some of the energy was absorbed by the filler thus increasing the resistance to impact.

Figure 6 displays a lower value on the impact strength upon addition of PP-g-MAH to the PP matrix which can be explained in the following manner: addition of the compatibiliser caused more boundaries in the PP chains and fracture probably initiated and propagated from these defects when the samples were impacted. This can be related to phase separation of PP/PP-g-MAH blends and it could also explain the lower stiffness of the blend samples when compared to those obtained from only PP. Jiang et al. also found that PP-g-MAH brought about more defects in crystals which might be another reason for the reduced mechanical properties in the blends. Addition of PP-g-MAH was expected to increase the impact strength of the samples, as it has been already found in other studies [22]. This was noticeable at 1 and 3 pph CF

(see Figure 6). At these CF contents, the increased resistance to impact could be attributed to a similar absorbing energy filler behaviour that was explained earlier. This could not be maintained at the higher CF loading and the number of voids around the fibres accounts for the inferior resistance to impact.

SEM micrographs of the polymer and composite fracture surfaces are included in Figure 6. These images show that PP matrix attained poor wetting behaviour on the CF in the absence of compatibiliser. Some of the defective areas attributed to CF/polymer debonding are highlighted in the circled areas of the PP composites. Furthermore, in these composites, the crack propagation resulting from the fillers is shown by arrows, explaining the reduced resistance to impact behaviour achieved in Izod tests. On the other hand, in the PP/PP-g-MAH based composites, the number of debonded CF decreased, especially at lower filler content. Crack propagation of the sample obtained from the blend and reinforced with 5 pph CF is also highlighted by arrows; this feature agrees with the reduced impact strength compared to the rest of the composites.

#### 4. Conclusions

Incorporation of PP-g-MAH has been shown to provide an advantageous effect when used as a compatibiliser in CF composites according to the DSC results which showed higher crystallisation temperatures and also higher degree of crystallinity achieved when compared to those composites based only on a PP matrix. Additionally, composites obtained by compression moulding achieved higher stiffness when the polymer blend was used and with the addition of low content of CF; on the other hand, a reduction in the modulus and a dramatic drop in the elongation of the samples were observed at higher CF content. The mechanical performance in the injection moulded composites differed more noticeably between the samples obtained from pure PP and the PP/PP-g-MAH blend. It was found that addition of CF greatly enhanced the modulus in the composites obtained from the former and the impact strength was reduced compared to the matrix. While the composites obtained from the PP/PP-g-MAH blend had a modest increase in the stiffness by the addition of CF up to the highest loading used in this work and also maintained the values of tensile strain, in these samples, the impact strength was improved with the incorporation of the filler when compared to the unreinforced sample. Impact strength was reduced in the composites obtained from the pure polymer, indicating stress region concentrations around fillers contributed mainly to this response. An absorbing energy filler behaviour was obtained in the samples prepared from the polymer blends as displayed by their enhanced impact strength.

#### Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

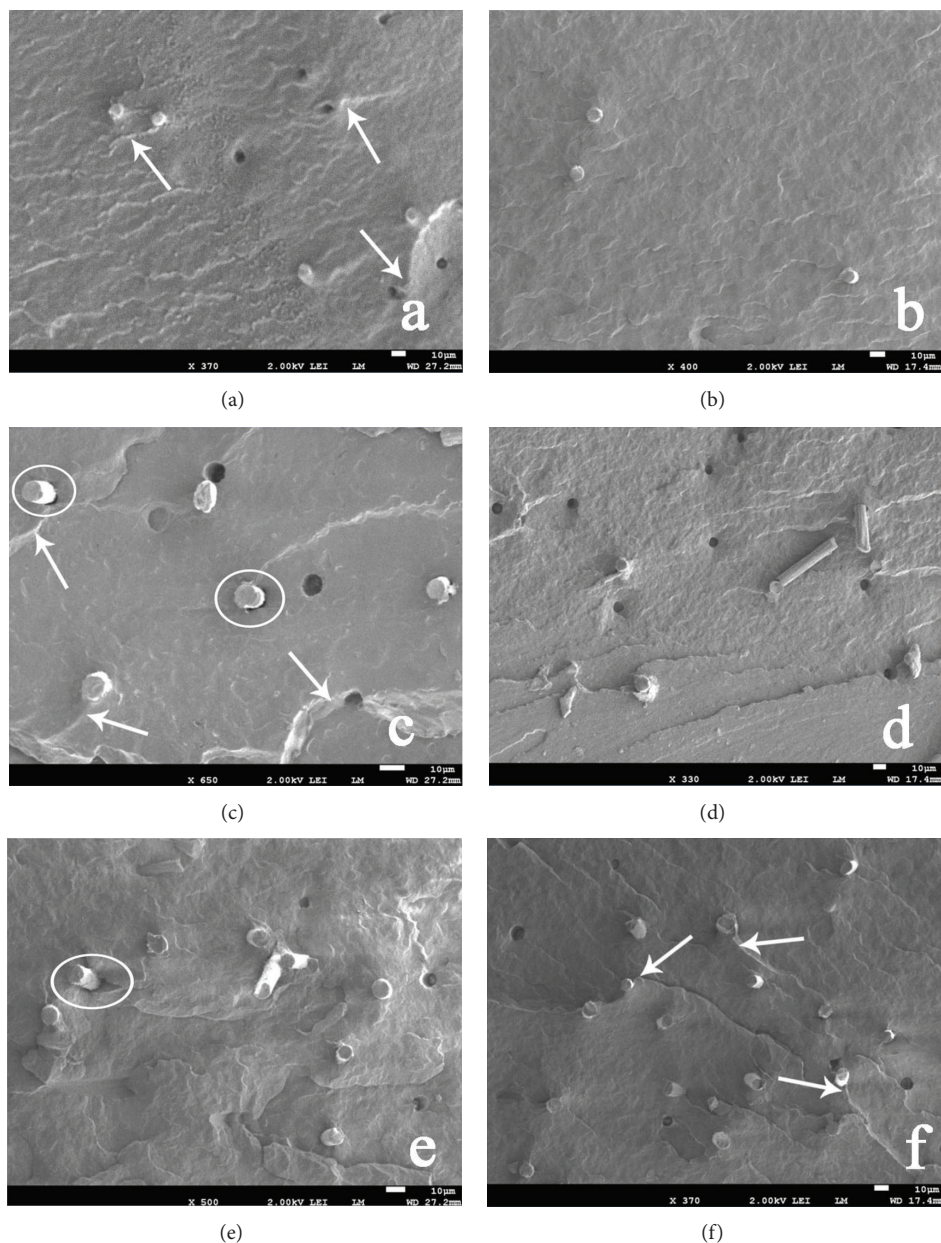


FIGURE 6: SEM images of the impacted samples: (a) 1 pph CF/PP, (b) 1 pph CF/PP/PP-g-MAH, (c) 3 pph CF/PP, (d) 3 pph CF/PP/PP-g-MAH, (e) 5 pph CF/PP, and (f) 5 pph CF/PP/PP-g-MAH.

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