

# Mechanical and Thermal Properties of Polypropylene/Recycled Polyethylene Terephthalate/Chopped Rice Husk Composites

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**ABSTRACT:** The various ratios of recycled polyethylene terephthalate (rPET) into polypropylene (PP) filled with 40 parts chopped rice husk per hundred part of polymer have been studied. Composites were prepared using a corotating twin screw extruder at temperature zones of 165–215, well below 250°C (rPET mp temperature) and characterized by mechanical and thermal properties. To improve the compatibility between different components, PP grafted with maleic anhydride was added as a coupling agent in all the compositions studied. The results showed that the addition of rPET improved the tensile and flexural modulus and impact strength of the composite while reducing its tensile

and flexural strength. The scanning electron microscopy micrographs of samples in the injection direction showed that some particle shaped rPET inside the composites appear as drawn fibrils and some appear as plates. Differential scanning calorimetric studies showed that the addition of rPET particles to the composites decrease the PP crystallization temperatures. © 2008 Wiley Periodicals, Inc. *J Appl Polym Sci* 110: 1979–1985, 2008

**Key words:** recycled polyethylene terephthalate; polypropylene; chopped rice husk; mechanical properties; morphology; DSC

## INTRODUCTION

A major part of factory packaging waste consists of thermoplastic compounds which are produced during forming processes. Polypropylene (PP) and polyethylene terephthalate (PET) are two of the most widely used packaging materials.<sup>1</sup> The necessity of recycling certainly is not only for ecological but also for economical reasons. Thus, various methods for the recycling of plastics waste materials have been developed.<sup>2</sup> Recycling options for PET can be divided into three categories: chemical recycling (i.e., depolymerization,<sup>3</sup> hydrolysis,<sup>4</sup> aminolysis<sup>5</sup>), mechanical recycling,<sup>6</sup> and energy recovery.<sup>7</sup> For the economic reasons, the mechanical recycling in PET has become more important than the chemical recycling process. On the other hand, separation of PET from plastic waste stream is expensive and very time consuming.

Blending polyolefins (e.g., PP) with engineering plastics (e.g., PET) can usually produce materials with improved mechanical properties. There are many studies carried out on the processing, morphology and mechanical properties, and crystalliza-

tion behavior of PET/PP blends.<sup>8–20</sup> Because PET and PP are immiscible,<sup>9</sup> some of these studies have focused on techniques to improve the compatibility between the two polymers.<sup>8,14,17</sup> PP grafted with maleic anhydride (MAPP) can be used as a compatibilizer in PET/PP blends.<sup>15–19</sup> It has been reported that the use of MAPP in PET/PP blends can improve the dispersion and adhesion between the two components.<sup>16</sup> It has also been indicated that using maleated PP as a compatibilizer improves the strength and stiffness of PP/PET blends.<sup>18</sup> However, there are some contradictory reports on the effect of compatibilizer. For example, Bataille et al. reported that when an acrylic acid–PP copolymer is added to PET/PP blends it does not have a significant effect on mechanical properties.<sup>20</sup> However, they found that the modulus increased with the weight percentage of PET. In contrast, Xanthos et al. observed improved compatibility and improved mechanical properties upon the use of acrylic acid grafted PP in PET/PP blends.<sup>19</sup>

Attempt to use recycled polyethylene terephthalate (rPET) as filler was reported by some of the researchers.<sup>21–23</sup> Demir and Tincer reported the use of rPET as filler into high-density polyethylene (HDPE).<sup>21</sup> They reported that silane, maleic anhydride (MA), and chain extender improved the mechanical properties of the resulting composites. There are also some reports on the plasma treat-

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ment of rPET particles for improvement in interaction with polyvinyl chloride (PVC).<sup>22</sup> Santos and Pezzin have reported that rPET can be used in PP and increases the impact strength of the resulting composite.<sup>23</sup>

While the tensile strength of the recycled plastics is usually reasonable, the other mechanical properties such as stiffness and creep resistance are generally fairly low and this limits their use in a number of applications. One possibility for improvement of the stiffness and the creep resistance of the recycled plastics is the addition of natural fibers into the composite. Recently, lignocellulosic-plastic composites are introduced as a new composite group. In this group of composites, the mixture of different natural fibers and plastic materials are produced by using different plastic processing technologies. The usage of natural fibers instead of the synthetic fibers has some advantages, e.g. the natural fibers composites are cheaper and have higher strength and modulus by considering the comparative fiber densities.<sup>24</sup>

The lignocellulosic-plastic composites are usually produced from the convenient semicrystalline polymers like polyethylene (PE), PVC, and PP. Furthermore, the thermoplastics having melting temperatures below 200°C are commonly used in making the composites because of the limited thermal stability of natural fibers.<sup>25</sup> As mentioned earlier, the natural fibers are inexpensive, having low density and high specific properties. The other advantages are their biodegradability and also ready availability.<sup>26</sup> Through a wide range of natural fibers, rice husk (RH) was chosen in this study because of its special properties. RH is produced as a by-product in large quantities in Iran. RH is the outer covering of paddy and accounts for 20% of its weight.<sup>27</sup> RH is a by-product in rice milling process and it contains 35% cellulose, 25% hemicellulose, 20% lignin, and ash of 17% by weight.<sup>28</sup> Chopped rice husk (CRH) can be produced in various grinding machines. CRH consists of typical lignocellulosic components. However, its lignin and hemicellulose amounts are lower than wood flour, and the CRH filler can be processed at higher temperatures than wood flour. CRH has a degradation and decomposition temperature about 250°C whereas wood flour has stability drawbacks at temperatures above 200°C. Therefore, the manufacturing processing of CRH composites can be performed up to 250°C.<sup>29</sup>

Several main factors should be taken into account when designing the CRH-PP composites. Because of the hydrophilic nature of the fibers and strong hydrogen bonding between the fibers, their mixing with hydrophobic polymeric matrix leads to heterogeneous systems whose properties are inferior. The fibers form aggregations, while there is also weak adhesion between the fiber and the matrix.<sup>30,31</sup> A

practical way for improvement of the interfacial adhesion between the nonpolar polymer matrix and the polar natural fiber can be achieved by using some coupling agents, making the polymer more reactive towards the natural fiber surface. MAPP has been evaluated as a coupling agent in CRH composites to promote interfacial bonding and improve the mechanical properties.<sup>29,32</sup> Besides, the hydrophilic nature of the fibers makes them very sensitive towards water absorption which decreases the mechanical properties abruptly. Therefore, drying of the fibers before processing is an important precondition.<sup>33</sup>

Marti-Ferre et al. reported that the addition of MAPP in CRH-PP composites introduce increasing storage modulus and decreasing loss tangent value at various temperatures.<sup>29</sup> This improvement has been attributed to the increase of compatibility between the RH and PP. Rozman et al. have also found that MA has a positive effect on the mechanical properties of RH-polyester composites and can improve the tensile and flexural properties.<sup>34</sup>

In our previous study, the mechanical properties and water absorption behavior of CRH-PP composites were evaluated.<sup>35</sup> The investigation showed that the mechanical properties of the composites remained in an acceptable level by 40 php CRH in the matrix. Relatively good dispersion and adhesion was observed from the examination of SEM micrographs which indicated that the addition of MAPP improved the interphase adhesion, thereby leading to improved properties.

In this study, rPET was ground into flakes and introduced as filler in a PP containing 40 php CRH by melt mixing. The objective of this work is to explore the possibility of using rPET and the effects on mechanical and thermal properties of the PP/CRH composites. The fracture surface of the composite is also examined.

## EXPERIMENTAL

### Materials

CRH was obtained from a local rice milling plant and after drying in air was ground in a Wieser grinding machine (WG-LS 200/200). The PP homopolymer was supplied by Bandar Imam Petrochemical Co., Iran, as the grade Poliran (PI0800) and with the melt flow index of 8 g/10 min. The dried PET bottles were cut into small flakes (approximately 0.5 cm × 0.5 cm) with grinding machine. MAPP with melt flow index of 450 g/10 min (190°C and 2.16 kg) was obtained from DuPont as the grade Fusabond MD353D and used as a coupling agent. Irganox 1010 supplied from Ciba, was also added into the composites as a heat stabilizer.

TABLE I  
Percent Weight of Each Component in  
PP/rPET/CRH Composites

Polymer ingredients in the samples	1	2	3	4	5	6	7	8	9
PP	97	92	87	82	77	72	67	62	57
MAPP	3	3	3	3	3	3	3	3	3
rPET	0	5	10	15	20	25	30	35	40

### Sample preparation

CRH and grinding rPET particles were dried at 120°C for 24 h before extrusion, and used without any treatment.

Composites including different amounts of rPET with 0–40% weight of polymer (wt %) were prepared by using a Brabender Plasticorder model DSE 20 twin-screw extruder. The screws with a 20 mm diameter and a 40 : 1 length/diameter ratio were employed and the screws speed was 130 rpm. The recipe for the preparation of PP/rPET/CRH composites is presented in Table I. In all samples, the content of MAPP, Irganox 1010, and CRH were kept constant. The ratio of MAPP was 3 wt % of the polymer content, while Irganox 1010 and CRH were 0.1 and 40 php, respectively. The temperature of five different barrel zones and the die were set to 165, 165, 215, 210, 195, and 185°C, respectively. The output of extruder was formed as the pellets and dried at 120°C for 24 h prior to injection into the mold. The injection machine used was manufactured by Imen Machine Co., Iran. Samples for different experiments were prepared by injection of the material into the appropriate mold.

### Mechanical experiments

All tensile and flexural tests were performed on an Instron testing machine (model 6025) and each experimental data is the average of five recordings. Tensile samples were prepared as dumbbell-shaped specimens at a cross-head speed of 5 mm/min. The thickness and width of the samples were 4 and 10 mm, respectively.

Flexural tests were examined in a three-point bending mode at the constant deflection rate of 2 mm/min. The samples were in the shape of rectangular bars of 10 mm × 10 mm × 110 mm and the span length was taken to be 50 mm.

The impact strength of the PP/rPET/CRH composites was also investigated by using a Zwick Impact Pendulum Machine (model 5102) in the Izod impact mode according to ASTM D256. The experiment was carried out on the notched samples. The reported data for each point is the average of at least 10 experiments.

### Thermal analysis

A Polymer Laboratories differential scanning calorimeter (DSC) was used for investigating the thermal behavior of composites. Samples were weighed and enclosed in aluminum pans and an empty aluminum pan was used as a reference. Thermal studies were carried out in the temperature range of 40–230°C at a rate of 10°C/min. In the first run, the samples were heated to 230°C and maintained at this temperature for 3 min; they were then cooled until reached ambient temperature. In the second run, the samples were reheated up to 230°C at the same heating rate. The thermograms were recorded as a function of temperature. The degree of crystallinity,  $X_C$ , of the samples were calculated by using the following equation:<sup>36</sup>

$$X_C = \left( \frac{\Delta H_f}{\Delta H_f^0} \right) \left( \frac{100}{w} \right)$$

where  $\Delta H_f$  is heat of crystallization of the sample,  $\Delta H_f^0$  is the heat of crystallization for 100% crystallized PP, taken as 207.1,<sup>36</sup> and  $w$  is the weight fraction of PP in the composites.

### Morphological investigation

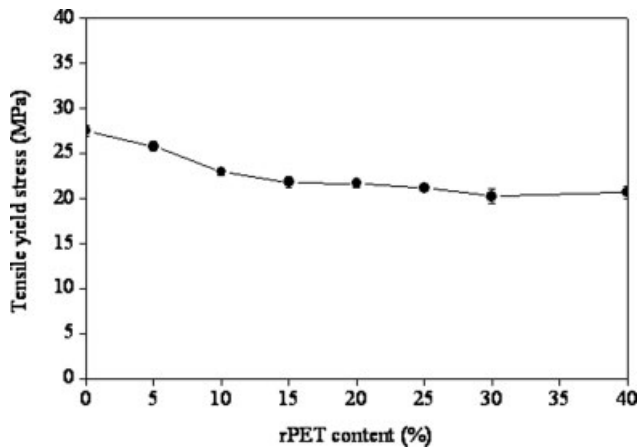
The morphology of the fracture surface of each composite was studied by using a Cambridge scanning electron microscope (model S360). To examine the fracture surface, the composites were snapped to two halves under liquid nitrogen. The samples were mounted on the sample stub and the surface was sputtered with gold.

## RESULTS AND DISCUSSION

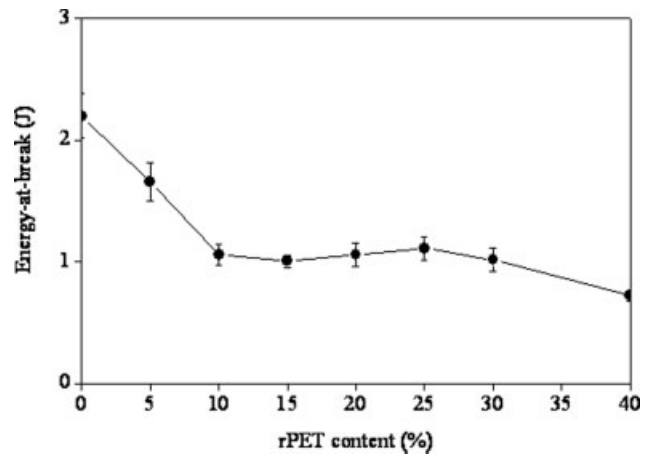
### Mechanical properties of PP/rPET/CRH composites

The variation of tensile yield stress of PP/rPET/CRH composites against rPET content is shown in Figure 1. As it can be seen, the decreasing of yield stress is marked in the range 0–15 wt % rPET and then it levels off. This behavior could be of economical benefit to industries as one could add the greater amount of rPET (ranging between 15–40 wt %) to replace the polymer without significant changes in mechanical properties, and therefore leading to cost savings. In fact, tensile yield stress was more affected by the presence of rPET not its quantity. Santos and Pezzin<sup>23</sup> have reported that increasing rPET in PP matrix does not affect the tensile strength of composite.

Tensile modulus and energy-at-break of PP/rPET/CRH composites are shown in Figures 2 and 3, respectively. As shown in Figure 2, the tensile



**Figure 1** Variation of tensile yield stress against rPET content of the composites.



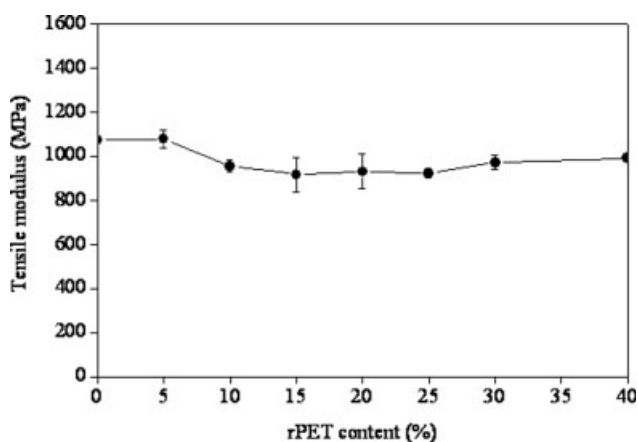
**Figure 3** Variation of energy-at-break against rPET content of the composites.

modulus indicates a decreasing slope till 10–15 wt % rPET and then it is leveled off. Since the injection molded tensile bars do not contain fully oriented rPET, the tensile modulus value is relatively constant. During the compounding process, various shapes of rPET can be formed and these forms (i.e., fibril or plate) can affect the mechanical properties of the composites.<sup>9</sup> It seems that the rPET plates are slipped during tensile test, decreasing the tensile modulus of the composites containing 15% rPET. However, the increasing of rPET weight ratio in the composite can increase the shear stress in the compounding process resulting in the formation of rPET fibrils form. Therefore, simultaneous presence of plate and fibril rPET forms can equilibrate the mechanical properties of the composites.

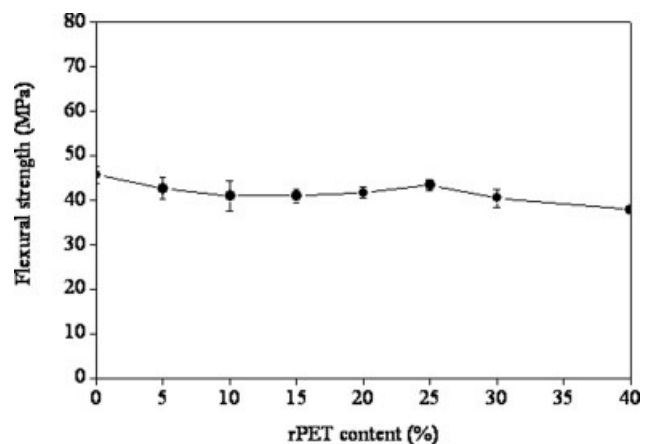
The energy-at-break was significantly affected by the addition of rPET into the composite. Figure 3 shows that it sharply decreases up to 10–15 wt % rPET and then remains relatively constant up to 25

wt % rPET and decreases again. This behavior can be explained by the reduction of ductile PP percentage and also the presence of various rPET forms from plates to oriented fibrils in the composites. This rPET morphological changes result in increasing crystallinity and decreasing rPET ductility which can be the main reason for decreasing energy-at-break of the composites. Demir and Tincer have also shown a decreasing trend in elongation-at-break of the HDPE-rPET composites with rPET addition.<sup>21</sup>

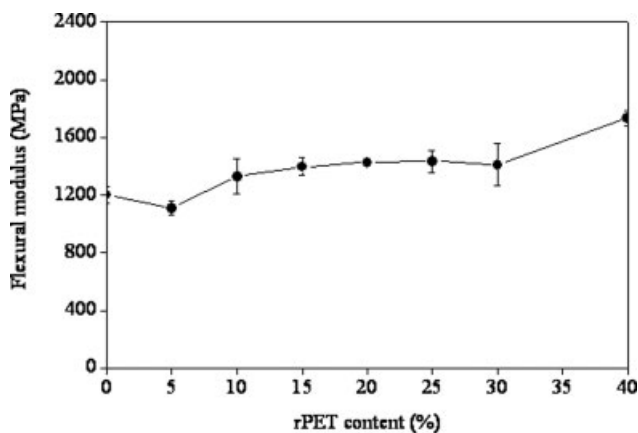
Figures 4 and 5 show the variation of flexural strength and modulus of the composites having various rPET contents, respectively. It was found that although the flexural strength relatively remained constant, the flexural modulus increased by about 44% comparing to the matrix alone. By addition of rPET particles to the matrix, flexural modulus of the composites was increased because of increasing crystallinity of PP in the matrix and the reinforcement effect of rPET fibrils aligned in the bar direction.



**Figure 2** Variation of tensile modulus against rPET content of the composites.



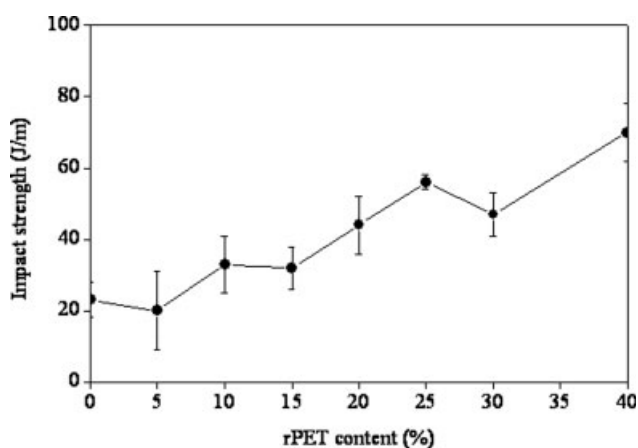
**Figure 4** Variation of flexural strength against rPET content of the composites.



**Figure 5** Variation of flexural modulus against rPET content of the composites.

Furthermore, it can be concluded from Figure 5, that a critical content of rPET (5 wt %) is required before the modulus of composites becomes greater than that of the matrix without rPET.

The impact properties of the composites are shown in Figure 6. The impact strength increases monotonously with increasing of rPET content. It means that the rPET content has a highly positive influence on impact strength of the composites. As it can be seen, with increasing 40 wt % rPET, the impact strength of composites has tripled that of the matrix without rPET. The similar behavior was also seen for the HDPE base composites containing 5 and 15% rPET. As reported, these composites are highly resistant towards the impact force as none of the samples were fractured.<sup>21</sup> The higher impact strength can be attributed to a greater amount of rPET that acts as a shock absorber in the bulk of composites and increases the length of the crack around the rPET particles.<sup>23</sup>



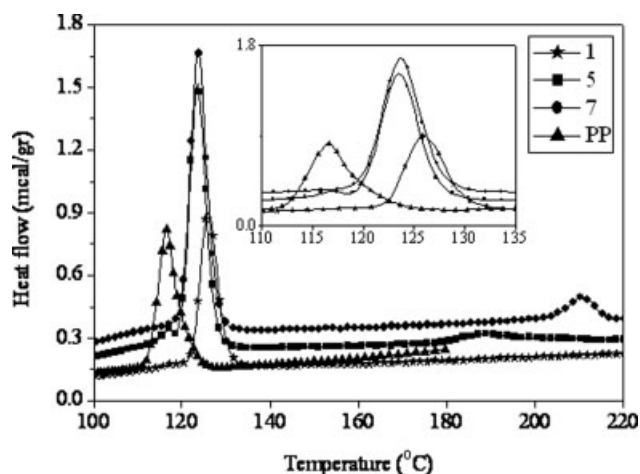
**Figure 6** Variation of impact strength against rPET content of the composites.

**TABLE II**  
Variation of PP Crystallinity in the Composites

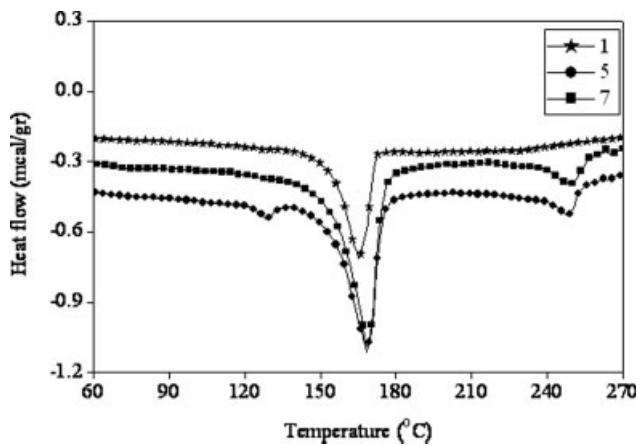
Property	Samples		
	1	5	7
$X_C$	25.6	39.2	40.4
$T_m$ (°C)	165.3	168.5	168.5
$T_c$ (°C)	124.1	123.5	123.5

### Differential scanning calorimetry

It is well known that melting temperature ( $T_m$ ) and the shape of endothermic peak offer valuable information on crystal size, its distribution, and degree of crystallinity of polymers. In the case of binary blends, the shape of thermograms depends on the degree of miscibility of the components. The miscibility of two components at the molecular level results in one melting point or crystallization peak in the thermogram. On the contrary, two peaks related to each component can be observed when the two components are immiscible. Table II presents thermal analytical data of CRH filled PP matrix and the two different contents of rPET composites (the sample codes in Table II are the same as Table I). CRH addition affected the crystallization behavior of PP matrix to a large extent, as shown in Figure 7. Compared with the PP matrix, the crystallization peak of the composites is narrower while shifting to higher temperature. The presence of CRH may be viewed as a nucleation agent for PP. Thus, CRH can promote the crystallization of PP at its surface and hence leading to a composite with higher  $T_c$  values. This kind of behavior has also been reported for RH powder in PP composites.<sup>37</sup> Other studies also showed that the use of MAPP<sup>38</sup> or surface roughness<sup>39</sup> can affect nucleation of the matrix.



**Figure 7** Crystallinity spectrum of the composites in various rPET contents.



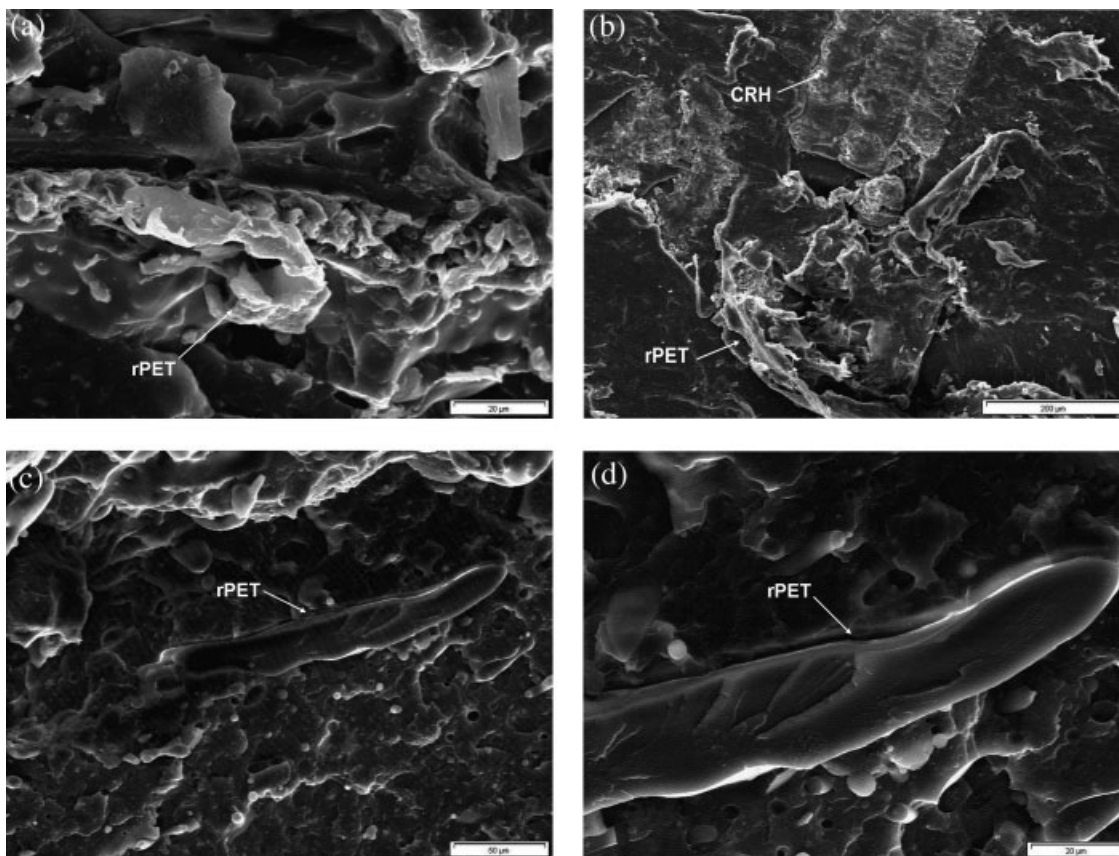
**Figure 8** Variation of melting temperature against rPET content of the composites.

However, the addition of rPET appears to change the crystallization behavior of the PP containing CRH. The addition of rPET increases PP crystallinity degree ( $X_C$ ) in the composites. It seems that rPET can also act as an external entity generating manifold nuclei for PP crystallization and increases degree of crystallinity.<sup>12</sup> Furthermore, the shift of PP exothermic peak to lower temperatures in these com-

posites (i.e., comparing the spectra of samples 1, 5, and 7 in range 110–135°C) is an indicative of good dispersion and compatibility between the PP and rPET components (Fig. 7). The endothermic peaks in Figure 8 confirm the crystallization behavior. As it is seen in this figure, the surface area of melting peak (i.e., in range 150–180°C) in the composites containing rPET are greater than PP matrix. Furthermore, the presence of rPET shows remarkable influence on melting and crystallization temperatures (Figs. 7 and 8), independent of its quantity, as given in Table II. The displacement of melting peaks of PP and rPET in the composites and shifting towards each other can be attributed to the role of MAPP in the miscibility of different samples.

### Morphology

Figure 9(a–d) shows the SEM micrographs of fracture surfaces of 30 wt % of rPET filled composites. As it can be seen from Figure 9(a,b), no smooth surface is noticeable between the rPET particles and CRH in the matrix. It seems that undrawn rPET forms have good physical interaction in the matrix. In addition, increasing the rPET amount which results in the increasing of mechanical properties can



**Figure 9** SEM micrographs of fracture surfaces. (a,b) rPET plate in the composite, (c,d) rPET fibril in the composite in two magnifications.

be attributed to the change of its forms from plate to the fibril rPET particles as seen in the Figure 9(c,d). The fibrillation of the rPET particles is due to shear force of extrusion and injection processes. However, the use of MAPP in the composite has a profound effect on creating a reasonably good chemical interaction between the components, which are confirmed with the mechanical studies.<sup>12,15-19</sup> As the SEM micrographs [Fig. 9(c,d)] show rPET surface is adhered to the PP matrix. Although a small gap observed around rPET particle might be directly to the result of impact energy in which the particle was forced to move. Furthermore, MAPP content is considered as 3% in all the composites under study, which may not be sufficient in high rPET content. However, the mechanical properties, particularly impact strength can also confirm the relatively good interaction and fibril morphology of rPET in the PP matrix.

### CONCLUSION

This work would be an indication that grinded and modified rPET can be used in the PP matrix as a reinforcing agent. The results have shown that the addition of rPET improved tensile modulus and impact strength while it reduced the tensile strength. The results also revealed that although flexural strength remained relatively constant, the flexural modulus increased by 44%. Impact strength of the composites has tripled compared to PP-CRH matrix. The improvement in the impact strength and flexural properties of the composites can be attributed to fibril morphology of rPET in the composite and its good adhesion to PP matrix as revealed by SEM micrographs.

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