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## Effects of electron beam radiation dose on the compatibilization behaviour in recycled polypropylene/microcrystalline cellulose composites

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# Effects of electron beam radiation dose on the compatibilization behaviour in recycled polypropylene/microcrystalline cellulose composites

N Samat<sup>1</sup>, S N R Motsidi<sup>1</sup> and N H M Lazim<sup>1</sup>

<sup>1</sup>Department of Manufacturing and Materials Engineering, International Islamic University Malaysia (IIUM), Jalan Gombak, 53100 Kuala Lumpur, Malaysia

Email: noorasikin@iium.edu.my

**Abstract.** The purpose of this research was to evaluate the influence of dose level of electron beam on the compatibilization behavior of recycled polypropylene (rPP) in rPP/microcrystalline cellulose (MCC) composites. Initially, the rPP was irradiated with various dose of electron beam (5 kGy up to 250 kGy) which then mixed with unirradiated rPP (u-rPP) at a ratio of 30:70 respectively. The composites were prepared by incorporating a series wt% of MCC fibers into rPP (u-rPP : i-rPP) using extruder and finally moulded with an injection moulding machine. The compatibility behavior of irradiated rPP (i-rPP) were analysed with mechanical tensile and thermal methods. The results of mechanical analysis showed great improvement in tensile modulus but an increase in radiation dosage gradually decreased this property. Nevertheless, the tensile strength exhibited a minor effect. The thermal stability of composites is lowered with increase in the absorbed dose, more significantly at higher content of MCC. Fracture surface observations reveal adhesion between the cellulose and rPP matrix.

## 1. Introduction

Radiation processing is a technique that can be used to modify the chemical and physical structure of polymeric materials. Therefore, the mechanical and thermal properties of polymers will change. In this technique, the polymers will be exposed to the irradiation and the common sources of irradiation are electron beams, gamma rays, X-ray etc. The application of irradiation to polymers results in several reactions such as crosslinking and chain scissions [1]. Both reactions can occur concurrently. However, the occurrence of crosslinking is preferred as it generally promotes the improvement of polymer's property. Moreover, this technique offers several advantages such as simpler, cheaper and easier to use.

According to Luguó et al. [2], radiation dosage is an important parameter to change the properties of polymers. Khalid et. al [3] reported that the high irradiation dose up to 200kGy increased the tensile strength and hardness of natural rubber nanocomposites. A study by Li et. al [4] in silk fibroin (SF) fiber-reinforced poly( $\epsilon$ -caprolactone) (PCL) composite also found that sample added with 45% fiber and irradiated with 150 kGy resulted in the optimum tensile strength and modulus. On the contrary an improvement at low dose (<50 kGy) was reported by Ndiaye et. al [5] for polypropylene/wood composites. But in this work the sample was irradiated with gamma beam.

In polymer composites, the compatibility between matrix and filler is necessary to the enhancement of their properties. Incompatibility between these constituents will hinder efficient stress transfer from





the matrix to filler. Most previous studies reported that polymer composites added with compatibilizer or coupling agents exhibited higher property [6-8]. As mentioned earlier, besides the chemical-based compatibilizer or chemical reagents, the ionizing radiation process also is able to modify the properties of a material. Studies by Samat et. al [9] on the compatibility effects of irradiated recycled polypropylene (i-rPP) in rPP/microcrystalline cellulose (MCC) composites, found that the tensile modulus of rPP/MCC was comparable to the chemical-based coupling agent. The improvement was associated with the existence of i-rPP in the rPP matrix with a ratio of 50:50 and the radiation dose was below 50 kGy.

Thus, the main objective of this study was to evaluate the influence of variation in radiation dose on the compatibility effect i-rPP in the rPP/MCC composites. The irradiation dose was varied from low to high dose and the ratio of u-rPP: i-rPP was 70:30. The compatibility behaviour associated with the changes in irradiation dose was determined from the mechanical and thermal stability properties. Field emission scanning microscopy analysis was also carried out.

## 2. Experimental procedures

Recycled polypropylene (rPP) was purchased from Top Flow Sdn. Bhd., Shah Alam, Selangor. The reinforcement material is microcrystalline cellulose (MCC). It was obtained from Sigma-Aldrich Co. (M) Sdn. Bhd. and has a density of 0.6 g/cm<sup>3</sup>. The polymer and the MCC were dried in an oven at 40-60°C for at least 7 hours before use.

The rPP pellets were irradiated using a 3MeV electron beam (EB) using the EB model EPS-3000 with an accelerator voltage of 0.5-3.0 MeV at room temperature. The radiation dosages were varied from 5, 10, 50, 100, 150, 200 and 250 kGy. The unirradiated (u-rPP) and irradiated rPP (i-rPP) pellets were blended at a ratio of 70/30 wt% by weight prior to the addition of various MCC loadings (5, 20, and 40 wt%). Several compositions, radiation dose and designation of composite in present study is given in table 1. These materials were compounded in a Brabender twin-screw extruder machine at temperatures ranging from 180-190°C with a screw speed of 70-90 rpm. The compounded materials were then crushed into small pellets and then injected moulded into standard specimens (ASTM D638) at a temperature 180°C.

**Table 1.** Composition, radiation dose and designation of blends

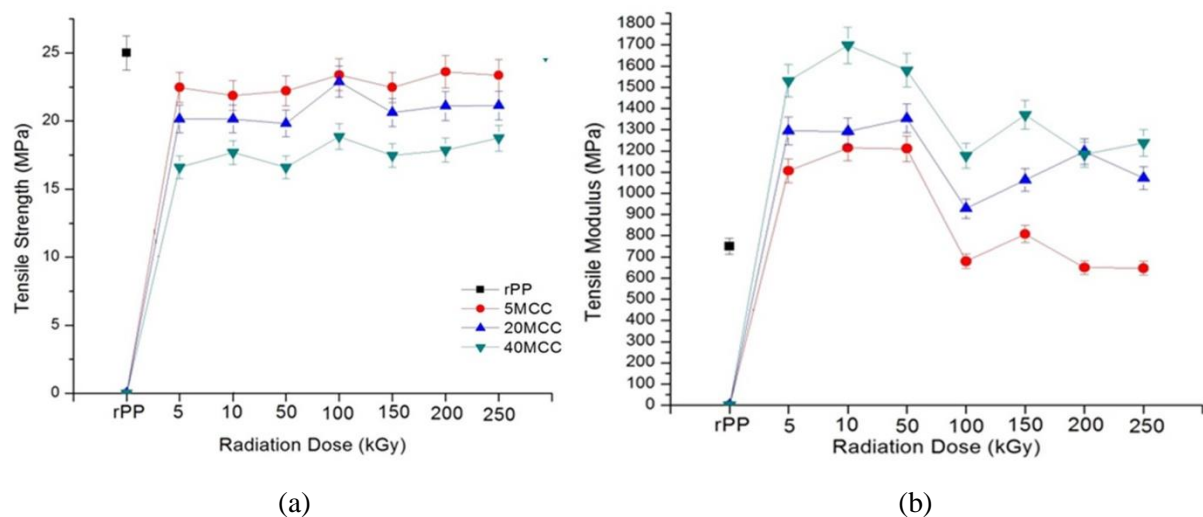
Sample designation	u-rPP (%)	i-rPP (%)	MCC (wt%)	Irradiation dose (kGy)
rPP	100	-	-	-
rPP/5D/5M	70	30	5	5
rPP/5D/20M	70	30	20	5
rPP/5D/40M	70	30	40	5
rPP/50D/5M	70	30	5	50
rPP/50D/20M	70	30	20	50
rPP/50D/40M	70	30	40	50
rPP/100D/5M	70	30	5	100
rPP/100D/20M	70	30	20	100
rPP/250D/5M	70	30	5	250
rPP/250D/20M	70	30	20	250
rPP/250D/40M	70	30	40	250

The tensile were determined according to ASTM D638 standards by using a Universal Machine (LLYOD Instruments LR10K Plus) at a cross speed of 50 mm/min. Thermogravimetric analysis was conducted on a STA7300. The samples were scanned from 50 to 700°C with a heating rate of 10°C/min in the presence of nitrogen gas. The weight loss and its derivative were measured and recorded as a function of temperature. The fracture surface morphology's of the composites were

observed using field emission scanning microscopy (JEOL-JSM 5600) at 10 kV. The sample surfaces were sputter coated with gold coating before analysis to eliminate any electron charging effect

### 3. Results and Discussion

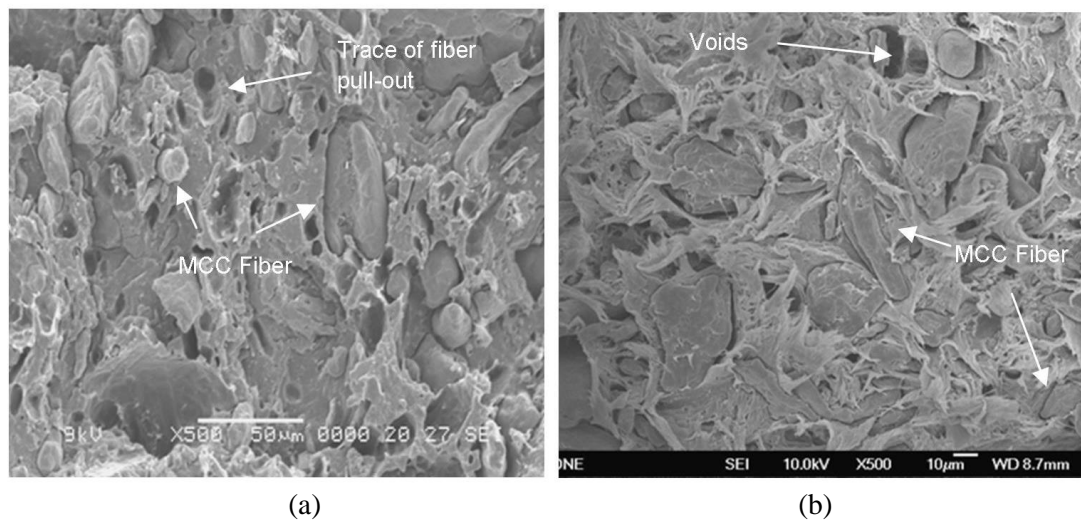
The effect of radiation dose and MCC loading on the tensile properties of rPP composites are shown in figure 1(a). In comparison with the neat rPP, the tensile strength of composites decreased slightly as the content of MCC was increased from 5 to 40wt%. The lower tensile strength at 40wt% of MCC is presumed due to agglomeration that resulted from the poor interfacial adhesion between the MCC and the matrix. A study by Zulkifli et. al [10] found that the presence of agglomerated MCC or filler with weak bonding strength would induced the formation of voids and led to fast crack propagation rates. Nevertheless, irrespective of the MCC content, no noticeable differences in the tensile strengths were observed for all irradiation dose. It seems that the tensile strength of rPP/MCC composites is insensitive to varying irradiation dose. Similar findings were reported by Chaudhari et al. [11] for PP-PE blends. These blends were irradiated with 100-500kGy.



**Figure 1.** Tensile properties (a) tensile strength and (b) tensile modulus of rPP/MCC composites with various MCC loadings and radiation dose.

From figure 1(b), it is evident that the Young's modulus of composites is affected by variation in the radiation dose and MCC content. Clearly, the presence of MCC fibers has caused an increase in the tensile modulus, including in samples added with 5wt% of MCC. In other word, though the amount of MCC was low but the improvement in Young's modulus is still significant. Therefore, this observation signifies that the enhancement in the Young's modulus is not merely caused by the stiffness property of MCC fibers but also by the existence of 30% of i-rPP, which acts as a compatibilizer. The i-rPP possesses higher degree of crosslinking that resulted from irradiation process with electron beam. Thus, the presence of high crosslink level also assisted to improve the modulus or stiffness of fabricated composites [12].

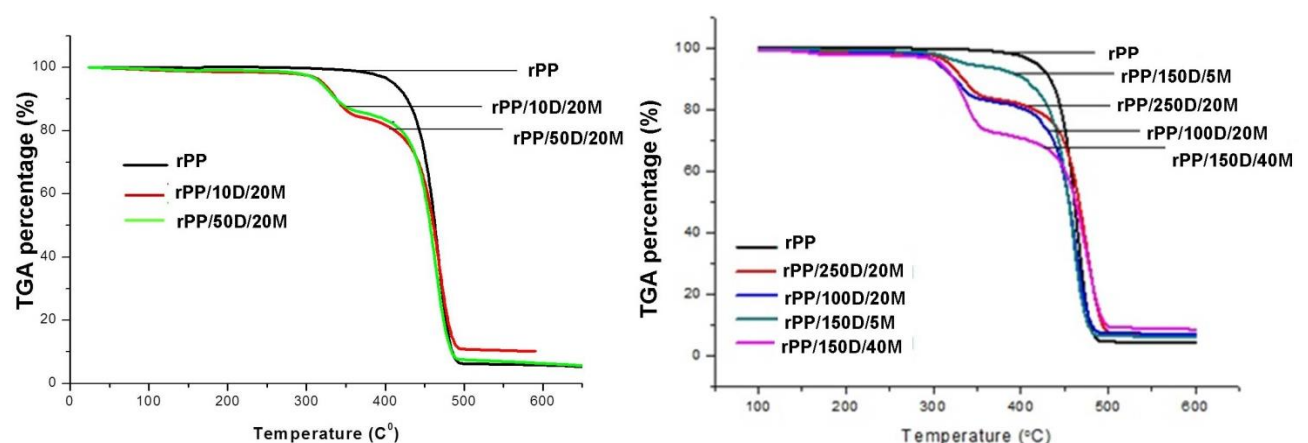
Moreover, changes in the modulus values were noted along with the increase in the radiation dose level beyond 50 kGy. Results from the electron spin resonance analysis by Lazim et. al [13] demonstrated that higher radiation dose produced more oxidation products (carbonyl and C-O compounds) and free radicals which associated with the chain scissions. Hence, it is suggested that irradiation of rPP at high dose diminishes the compatibility effects which resulted from the occurrence of chain scission phenomenon and formation of oxidation compounds.



**Figure 2.** FESEM micrograph of rPP/MCC composites for (a)rPP/10D/5MCC and (b)rPP/150D/5MCC

FESEM investigations on the tensile fracture surface of rPP composites which contained i-rPP at low and high dose is depicted in figures 2a and b, respectively. The fracture surface exhibits several features: traces of fiber pull-out, dissimilar size of MCC and voids. Interestingly, the cellulose-rPP matrix adhesion is also evident as observed from figure 2. This indicates that the presence of i-rPP improves the compatibility between the fiber and matrix. In this study although the ratio of i-rPP to u-rPP was only 30%, which is lower than in a work by [9](i-rPP = 50%), but the compatibility effects of i-rPP is still present.

The thermogravimetric analysis (TGA) was carried out to investigate the thermal properties of the produced composites. The result is presented as residual weight vs. temperature (Figure 3). Dissimilar to neat rPP, the composite samples exhibit the two-mass loss step feature. The first degradation temperature occurs at a range of 250°C to 350°; which attributed to the presence of MCC fibers [9] or natural fiber [14]. Meanwhile, the second degradation corresponds to the neat rPP.



**Figure 3.** Thermogravimetric mass loss curves of rPP/MCC composites with various MCC loadings at (a) low radiation dose and (b) high radiation dose

Table 2 shows the analysis of thermal degradation temperature for weight loss at 5% ( $T_5$ ) and 50% ( $T_{50}$ ). From this table, it is noticed that independent of the MCC content, the presence of i-rPP

compatibilizer with different radiation dose level affects the thermal stability of composites. For instance, the irradiation at 250kGy has caused the degradation to occur at 10°C faster than the neat rPP. The degradation at higher irradiation dose is in accordance with previous observation in Young's modulus and a work by [5]. The highest char residue implies greater thermal stability in composites with lower radiation dose. Thus, it can be emphasized that for polymer based-cellulose/natural fibers; the high irradiation dose would lead to chain scission mechanism [1,13] and resulted in lower thermal stability.

Besides the radiation dose, the thermal stability is also dominated by the presence of MCC fibers. Comparison of samples rPP/150/40M and rPP/150/5M shows that the thermal stability deteriorated with the increasing loadings of MCC fibers (Table 2). The incorporation of MCC fibers from 5 to 40wt% lower increased the decomposition rate, which is attributed to their lower thermal stability. Similar findings were reported by Kiziltas et. al [15] for PET-PTT blends/MCC composites.

**Table 2:** The degradation temperature of rPP/MCC composites

MCC(wt%)	Dose (kGy)	Degradation Temperature (°C)		Char residue at 590°C.
		$T_5$	$T_{50}$	
0	0	411.2	461.3	4.18
20	10	321.1	461.1	10.17
20	50	318.7	457.0	7.91
20	100	313.4	460.9	6.74
20	250	311.5	458.8	6.85
5	150	341.6	458.5	5.98
40	150	304.3	458.7	8.45

#### 4. Conclusion

The following conclusions can be drawn from the results obtained:

- (1) The presence of i-rPP at 30% enhanced the adhesion between the cellulose fiber and rPP matrix.
- (2) The compatibilizing effect of i-rPP in polymer based-cellulose is efficient at irradiation below 50 kGy.
- (3) The high radiation dose along with high concentration of MCC fibers reduced the thermal stability of rPP/MCC composites

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