Effects of Electron Beam Irradiation on Mechanical Properties of PP/EPDM Nanocomposites

H. ANUAR^{*}, N.A. JAMAL^{*#} AND A.R. SHAMSUL BAHRI^{**}

Nanocomposites are materials created by introducing nanoparticulates commonly referred to as a filler into the matrix. Blends of polypropylene (PP)/ethylene propylene diene monomer (EPDM)/montmorillonite (MMT) were treated by compatibiliser maleic anhydride polypropylene (MAPP) and irradiated using an electron beam. The effects on mechanical properties for both samples were compared with the untreated nanocomposites. The effects of montmorillonite clay loading on mechanical properties of PP/EPDM/MMT were investigated. The d-spacings of the clay in nanocomposites were examined using x-ray diffraction (XRD) and the extent of delamination was observed by a transmission electron microscope (TEM).

Keywords: electron beam irradiation; nanocomposite; maleic anhydride polypropylene; polymer blend; montomorillonite clay; mechanical properties; nanoparticulates

In recent decades, polymer-layered inorganic nanocomposites have experienced an important development over the past ten years across the world. Nanocomposites are particle-filled polymers for which at least one dimension of the dispersed particles is in the nanometer range and can have superior physical properties such as thermal, mechanical and barrier properties or some new properties when compared to the original polymer/filler composites¹.

An electron beam is a stream of electrons (as from a betatron) generated by heat (thermionic emission), bombardment of charged atoms or particles (secondary electron emission), or strong electric fields (field emission). Electron beam crosslinking is a good alternative to crosslinking by chemical agents especially for blends or multimaterials². When polymers are crosslinked, the molecular movement is severely impeded, making the polymer stable against heat. The main effects of electron beam irradiation are chain scission, oxidation and increased unsaturation, depending on the dose rate and the oxygen content in the exposed environment³.

The purpose of this paper is to study comparatively the effects of high energy radiations on the mechanical properties of PP/EPDM nanocomposites. The effects of adding compatibiliser in PP/EPDM nanocomposites in comparison to irradiated

^{*}Department of Manufacturing and Materials Engineering, Faculty of Engineering, International Islamic University Malaysia, PO BOX 10, Kuala Lumpur, 50728, Malaysia

^{**}Crop Improvement and Protection Unit, Production Development Division, Rubber Research Institute Malaysia (RRIM), 47000, Sungai Buloh, Selangor Darul Ehsan, Malaysia

[#] Corresponding author (e-mail: ayuni_jamal@yahoo.com)

samples and untreated samples were studied. The effects of clay loadings from 0 to 8% by weight on mechanical properties of samples were also investigated.

EXPERIMENTAL

A polypropylene with a density of $0.92 \text{ g/} \text{cm}^3$ was supplied by Polypropylene Malaysia Sdn Bhd and ethylene propylene diene monomer (EPDM) with a density of $0.87 \text{ g/} \text{cm}^3$ was obtained from Centre West Industrial Supplies Sdn Bhd. The portion used was 70:30 of PP and EPDM. The clay (with density of 1.00 g/cm^3) used was 1.31 PS grades, containing silane modification supplied by Nanocor Inc. The clay content was 0-8% by weight as shown in *Table 1*. The compatibiliser used was maleic anhydride polypropylene, MAPP with a density of 0.95 g/cm^3 from Aldrich Chemical Co. USA. The weight of MAPP used was 3%.

TABLE 1. THE COMPOSITION OF CLAY USED

Matrix (wt %)	MMT (wt %)
100	AND .
98	2
96	4
94	6
92	8

In this study, three types of nanocomposites samples were prepared. First are the untreated samples of PP/EPDM, with different clay contents. Second are compatibilised samples (PP/EPDM/MAPP with different clay contents). Last are the irradiated samples (PP/EPDM with different clay loadings). All samples were prepared in an internal mixer with Banburry blade rotors at a temperature of 180°C and a rotor speed of 40 r.p.m. for 10 minutes of direct mixing. Compression moulding was used to compress the mixed samples into sheets of 1 and 3 mm thickness. The temperature used was 180°C. It took 12 minutes to complete a cycle; pre-heating time was 5 minutes, venting was 1 minute, full pressing cycle was 3 minutes and the cooling cycle was 3 minutes. The samples were cut according to *ASTM Standard D412*.

Radiation was conducted after the compression moulding process. Samples were exposed to an irradiation dose of 100 kGy. For the samples with compatibiliser, the MAPP was added together during the mixing. The samples were characterised using tensile test, impact test, XRD and TEM.

RESULTS AND DISCUSSION

Tensile Test

Tensile test was carried out to observe the effects of clay content, compatibiliser and radiation on the strength of the nanocomposites. These samples were compared with the untreated samples as well as samples at different clay loadings. The compatibiliser added was MAPP and the irradiation dose was fixed at 100 kGy. The results are summarised in *Table 2* and *Figure 1*.

From Figure 1, the tensile strength of the studied nanocomposite with addition of compatibiliser is higher than the untreated and irradiated nanocomposite. The tensile results for the untreated and irradiated samples both showed a decrease in tensile strength which slightly differs with radiation to give slightly better tensile strength than the untreated samples. It is stated that the effects of the applied treatment on the properties of the polymer blends depend both on the exposure type and on the composition of the blends³. Thus, improvement in radiation is also associated with the composition of the blends. The introduction of compatibiliser showed an increase in tensile strength. The improvement

Clay composition (wt %)	Without MAPP (MPa)	Standard deviation values	With MAPP (MPa)	Standard deviation values	Radiation (MPa)	Standard deviation values
0	16.743	0.686	16.993	0.283	17.313	0.141
2	14.890	0.257	17.248	0.754	15.115	0.590
4	14.060	0.287	18.261	0.446	13.895	0.349
6	13.120	0.447	16.880	0.553	14.121	0.280
8	12.959	0.077	15.397	0.202	12.896	0.663

TABLE 2. THE DATA OBTAINED FROM TENSILE TEST



Figure 1. Effects of clay composition on the tensile strength of nanocomposites.

in strength by MAPP is larger compared to the irradiated samples. The increase in tensile strength can be attributed to the improved dispersion of clay by the addition of compatibiliser⁴.

Clay composition in the samples also plays an important role as it has a limit in improving the tensile strength. From the graph of untreated and irradiated samples, both showed that the tensile strength decreased as the clay content increased. However, the irradiated samples of 6 wt % showed an increase in strength before it decreased again. This pattern may occur due to agglomeration of the clay particles and poor dispersion of clay in the mixture. The graph of samples treated by compatibiliser showed an increase in tensile strength with maximum clay composition of 4 wt % and decreases as the clay increased. Thus, the 4 wt % of clay content is the optimum value. This also attributed to better dispersion. The enhancement in tensile strength is directly attributed to the dispersion of nanosilicate layers in the matrix and strong interaction between matrix and clay, and the lower tensile strength can be attributed to inevitable aggregation of the silicate layers with high clay content⁵.

Impact Test

Table 3 and *Figure 2* show the summarised results obtained from the study of nanocomposites with addition of MAPP. The impact strength increased as the clay content increased, but reached an optimum value at 4 wt % of clay loading. The impact strength at 4 wt % clay loading with the addition of MAPP is 9.726 kJ/m^2 while the impact strength for untreated nanocomposites at the same clay loading is 4.428 kJ/m^2 . The graph showed a decrease in value before it starts to increase. The minimum value is at 4 wt % of clay loading.

X-ray Diffraction (XRD)

X-ray diffraction is an effective way to characterise the formation of a nanocomposite. In an immiscible mixture, the gallery height of clay, in terms of its d-spacing should be virtually identical to that of the pristine clay; if a nanocomposite is formed, the d-spacing must increase⁵. By using XRD, the clay dispersion by two possible cases could be evaluated. If a peak is seen at larger d-spacing than in the pristine clay, that indicates an intercalated structure. If no peak is seen, that may indicate either an exfoliated structure or disordering of the clay layers. *Figure 3* below shows the

Clay composition (wt %)	Without MAPP (kJ/m ²)	Standard deviation values	With MAPP (kJ/m ²)	Standard deviation values
0	4.803	0.362	4.825	0.297
2	4.645	0.245	5.608	0.159
4	4.428	0.479	9.726	0.337
6	5.072	0.291	5.803	0.125
8	5.107	0.273	4.300	0.358

TABLE 3. THE DATA OBTAINED FROM IMPACT TEST



Figure 2. Effects of clay composition on impact strength of nanocomposites.



Figure 3. XRD patterns of samples (a) without MAPP (b) with MAPP (c) irradiated (d) with clay powder.

XRD pattern of nanocomposites from samples untreated, with MAPP and irradiated. All samples of nanocomposites showed a slight shift in peak towards the left with increase in d-spacing, indicating the structure of nanoclays are intercalated.

Transmission Electron Microscope (TEM)

X-ray diffraction only provides a partial picture about the distribution of nanoclays in disordered-intercalated polymer nano-composites⁶. Thus, to confirm the complete characterisation of nanocomposites morphology requires microscopic investigation that can be done using TEM. The images of TEM

in *Figure 4* show that clay is intercalated in all samples. This may occur due to agglomeration of the clay. Other than that, the processing parameters also play an important role in clay distribution. The intercalated structures might occur due to the low rotor speed during mixing of materials.

CONCLUSION

The mechanical properties of nanocomposites were studied by comparing the tensile strength and impact strength. The use of MAPP as compatibiliser gave better improvement in mechanical properties than the irradiated samples. The clay loading also affects the



Figure 4. TEM images of nanocomposites (a) without MAPP (b) with MAPP (c) irradiated.

mechanical properties of nanocomposites. The optimum value for clay loading was found to be 4 wt %. XRD and TEM micrographs showed that nanoclay is intercalated, thus reduces the mechanical properties of the composite.

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