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Effects of MAPE and EB Irradiation Techniques on the Mechanical Properties of Crosslinked Rubber Toughened Nanocomposites

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

Improving the mechanical properties of high density polyethylene (HDPE)/ethylene propylene diene monomer (EPDM) matrix is the main target of developing HDPE/EPDM filled organophilic montmorillonite (OMMT). Both HDPE/EPDM matrix and HDPE/EPDM filled OMMT were first prepared via *intercalation technique at different OMMT loadings. Two types of crosslinking techniques were applied namely, maleic anhydride polyethylene (MAPE) and electron beam (EB) irradiated systems. The effectiveness of MAPE and EB irradiation crosslinkings were then compared with control and analysed based on the mechanical tests and morphological examination. The mechanical tests revealed that control, MAPE and EB irradiated systems had attained the optimum mechanical properties at 4 vol% OMMT content. EB irradiation absorbed dose of 100 kGy showed excellent mechanical properties with higher crosslinking degree is proved by gel content analysis. X-ray diffraction (XRD) analysis confirmed the existence of delamination of OMMT by MAPE and EB irradiation techniques based on disappearance of the characteristic peak. The degree of delamination was further investigated by transmission electron microscope (TEM).*

Keywords: mechanical properties; organophilic montmorillonite; electron beam radiation; maleic anhydride polyethylene; gel content

Compatibiliser or crosslinking agent is commonly introduced in a nanocomposite purposely to reduce the surface tension between immiscible polymers and filler as well as to improve the surface adhesion of filler. This is to obtain the optimum filler dispersion which will improve the properties of nanocomposite. Moreoever, compatibiliser is proved to enhance the rigidity of the polymer[1,2]. Compatibilising agent is typically a molecule having one hydrophilic and one organophilic functional group. Compatibilisers containing maleic anhydride functionalities are commonly used and are effective in improving the physical properties of composites. Studies claim that the miscibility of polymer matrix

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with clay can be enhanced by introducing compatibilisers containing polar groups such as maleated polyethylene (MAPE) and carboxylated PE[3,4]. In this study, MAPE has been applied as a compatibiliser agent for both the HDPE/EPDM blend and HDPE/EPDM filled OMMT.

Irradiation processing has been used for many years to modify the properties of formed polymer parts. The property enhancements achieved include increased operating temperature, improved mechanical properties, increased chemical and solvent resistance. It should be noted that polymer structure influences the crosslinking ability of a polymer. Chain crosslinking and scission are the two reactions that occur during electron beam (EB) processing of polymers. Polymers typically undergo simultaneous scission and crosslinking, but in most cases with one or the other clearly predominating[5,6]. Crosslinking is the intermolecular bond formation between polymer chains. The degree of crosslinking is proportional to the radiation dose[7]. Much work has been done on radiation crosslinking of uncrosslinked polymers and crosslinking of various rubbers and plastics by electron beam irradiation[7–9].

In contrast, scission is the opposite process of crosslinking in which the rupturing of C-C bond occurs. Scission reduces crosslinking efficiency and degrades the properties of polymers (chemical resistance, mechanical and thermal properties). The current study initiates to vary and highlight the interest of EB irradiation technique for HDPE/EPDM, where pristine composites and nanocomposites were prepared with different irradiation doses rate and OMMT loading. The results obtained for the EB irradiated system were then compared with control and MAPE systems for the determination of the optimum system.

EXPERIMENTAL

Materials

Homopolymer high density polyethylene (HDPE) (Melt flow index 3–6 g/10 min, density 900 kg/cm^3) supplied by Cementhai Chemicals Group, Thailand and Ethylene Propylene Diene Monomer rubber (EPDM) supplied by Centre West Sdn Bhd, Malaysia were used as the base polymer matrix. MAPE-Polybond@ 3009 obtained from Uniroyal Chemical Company was used as a coupling agent to improve surface adhesion. Commercially available organophilic montmorillonite (OMMT) surface modified with 15–35 wt% octadecylamine and 0.5 wt% aminopropyltriethoxysilane obtained from Sigma-Aldrich Group, Malaysia was used as a reinforcing agent to prepare nanocomposites.

Compounding

Melt blendings of HDPE (70 vol%), EPDM rubber (30 vol%), MAPE agent (optimum content of 3 vol%) and OMMT at different loadings (2, 4, 6 and 8 vol%) were carried out in an internal mixer (Thermo Haake Rheomix 600P). The processing parameters were set to 135ºC of mixing temperature, 100 r.p.m. of rotor speed and 13 min of direct mixing. Prior to mixing, the matrix polymer and the nanoclays were dehumidified in a dry oven at 110ºC for a period of 1 h.

Specimen Preparation

Subsequently, the blended samples were compression moulded as per *ASTM-F-412* using a compression moulding machine at a temperature range of 135–155ºC, 8 tonne pressure for 14 min.

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TABLE 1. COMPOSITION, PARAMETERS AND MODIFICATION OF NANOCOMPOSITES

Systems	Polymer matrix	Polymer matrix content (vol%)	OMMT content (vol%)	Surface modification	Total Composition
Control	HDPE/EPDM	100	-	-	100
MAPE	HDPE/EPDM	97	-	3 vol% MAPE	100
50 kGy	HDPE/EPDM	100	-	Electron beam irradiation at 50 kGy	100
100 kGy	HDPE/EPDM	100	-	Electron beam irradiation at 50 kGy	100
150 kGY	HDPE/EPDM	100	-	Electron beam irradiation at 50 kGy	100
200 kGy	HDPE/EPDM	100	-	Electron beam irradiation at 50 kGy	100
Control/OMMT	HDPE/EPDM	98, 96, 94, 92	2, 4, 6 and 8	-	100
MAPE/OMMT	HDPE/EPDM	95, 93, 91, 88	2, 4, 6 and 8	3 vol% MAPE	100
50 kGy/OMMT	HDPE/EPDM	98, 96, 94, 92	2, 4, 6 and 8	Electron beam irradiation at 50 kGy	100
100 kGy/OMMT	HDPE/EPDM	98, 96, 94, 92	2, 4, 6 and 8	Electron beam irradiation at 50 kGy	100
150 kGy/OMMT	HDPE/EPDM	98, 96, 94, 92	2, 4, 6 and 8	Electron beam irradiation at 50 kGy	100
200 kGy/OMMT	HDPE/EPDM	98, 96, 94, 92	2, 4, 6 and 8	Electron beam irradiation at 50 kGy	100

Control system = untreated or non-irradiated system; OMMT = organophilic montmorillonite; HDPE = high density polyethylene; EPDM = ethylene propylene diene monomer; MAPE = maleic anhydride polyethylene (optimum content of 3 vol%)

High Energy EB Irradiation

The melt compounding samples were exposed under high energy EB irradiation at different absorbed doses of 50, 100, 150 and 200 kGy at room temperature using a 3 MeV electron beam accelerator. The acceleration energy, beam current and dose rate were set to 2 MeV, 2 mA and 50 kGy/pass respectively.

CHARACTERISATION TECHNIQUES

Mechanical Properties

Test specimens for analysing mechanical properties were initially conditioned at 23 ± 1ºC and 55 ± 2% RH for 24 h prior to testing. These conditioned specimens were subjected to mechanical testing and an average from the five testing measurements was reported. The corresponding standard deviation along with the measurement uncertainty value for the experimental data showing maximum deviation was also included.

Tensile Test

Specimens with dimensions of 125 × 1 × 1 mm were subjected to a tensile test as per *ASTM F412*, using Instron 5567 machine with 5 kN load. Crosshead speed and gauge lengths were set to 50 mm/min and 60 mm.

Flexural Test

Specimens with 125 × 15 × 3 mm dimension were tested in accordance to *ASTM D790* by using the Instron 5567 machine. A span of 100 mm was used in a 5 kN load cell with a crosshead speed of 50 mm/min.

Impact Test

Notched Izod impact test as specified by *ASTM D256* standard test method was applied by using Ceast 6545/000 model. Specimens with 62 × 15 × 3 mm dimension were subjected to an impact test with 2.54 mm depth of notch. Before the test, each sample was immersed into liquid nitrogen for 30 s. The energy absorbed by the specimen in the breaking process is known as the breaking energy (J/m).

Gel Content Analysis

The gel content of the samples was determined by boiling the samples with xylene for 24 h in accordance to *ASTM D2765* procedure. The extracted samples were vacuum dried to constant weight for 16 h at 75ºC. The gel content was calculated as the ratio of weight of dried sample after extraction to the initial weight of the sample before extraction. The results reported were the average of three specimens. The gel content percentage of crosslinked samples was calculated using the formula below:

$$(\%)\ \text{Gel content} = \frac{\text{weight after extraction}}{\text{weight before extraction}} \times 100 \qquad \ldots 1$$

X-Ray Diffraction Analysis (XRD)

X-ray diffractograms of OMMT and the nanocomposites were recorded using Shimadzu 6000 (Japan), X-ray crystallographic unit equipped with nickel filtered Cu Kα radiation source operated at 40 kV and 40 mA. The basal spacing or d_{001} reflection of the samples was calculated from Bragg's equation by monitoring the diffraction angle 2θ from 2 to 10º.

Morphological Examination using Transmission Electron Microscope (TEM)

The morphology of the nanocomposites was observed using a JEOL JEM electron microscope with an accelerating voltage of 100 kV. Ultrathin specimens of 100 nm thickness were cut from the middle section of the compression moulded bar using a Leica ultra microtome. The specimens were collected on a trough filled with water and placed on a 200 mesh grid.

RESULTS AND DISCUSSION

Tensile Strength and Modulus

The effects of different clay loadings on control, MAPE and EB irradiated systems are demonstrated in *Figures 1* and *2*. It is observed that the tensile strength and modulus for HDPE/EPDM filled OMMT began to increase up to 4 vol% of OMMT. As clay loading exceeded 4 vol%, the tensile strength and modulus of all systems were found to decrease. Similar improvements in tensile strength and modulus were also reported by previous researchers in their work on any polymer/organoclay nanocomposites[10,11]. The primary cause for such improvement was attributed to the presence of immobilised or partially mobilised polymer phases. This is due to the better interaction of polymer chains with clays and large number of interacting molecules due to the dispersed phase volume ratio characteristic of delamination clay platelets as evidenced by TEM micrographs. In contrast, deagglomeration of clay particles at higher clay loading could be attributed to the decrease of strength and modulus.

Tensile strength and modulus of both HDPE/EPDM matrix and HDPE/EPDM filled OMMT were further improved with MAPE agent, as shown in *Figures 1* and *2*. An increase of 34% (stdev = 0.098) and 22% (stdev = 0.608) in tensile strength and modulus was observed for MAPE system as compared to control where only 31% (stdev = 0.24) and 20% (stdev = 0.31) increments at 4 vol% OMMT were elucidated. Such slight increase was due to the enhanced adhesion of OMMT sheets to the HDPE/EPDM matrix.

For EB irradiated system, the tensile strength and modulus are maximised at 19 MPa (stdev = 0.18) and 701 MPa (stdev = 0.27) at 4 vol% of OMMT and absorbed dose of 100 kGy. This indicates the formation of radiation induced crosslinking in the rubber and plastic phases as confirmed by the gel content analysis. At higher doses rate, the tensile strength decreased due to the breakdown of the network structure.

Elongation at Break

A moderate increment of the elongation at break with initial incorporation of 2 vol% organoclay loading and followed by a sudden dropping of elongation at break upon 6 vol% loading of organoclay is obtained as in *Figure 3*. Similar results were reported before[12,13]. The improvement in elasticity may be attributed to the plasticising effect of the OMMT gallery and their contribution to the formation of dangling chains but also probably due to the improvement of surface interaction between the clay and HDPE/EPDM matrix interface[14].

Furthermore, almost 10% (stdev = 0.77) reduction in elongation at break at optimum clay loading of 4 vol% was observed for the MAPE system. This is due to the MAPE reduced chain slippage on the surface of the fillers by the reaction of both filler and matrix. As a result, the elongation at break of the MAPE nanocomposite decreased.

EB irradiated system experienced a continuous decrease in elongation at break with increased dose rate. Reductions of 10% (stdev = 0.72), 11% (stdev = 0.12), 16% (stdev = 0.71) and 21% (stdev = 0.65) at optimum clay loading of 4 vol% were obtained at absorbed doses of 100, 50, 150 and 200 kGy. Generally, increasing the radiation dose resulted in the reduction of elongation at break of nanocomposite. Therefore, the reduction in elongation at break at higher filler loading could be attributed to the reduced segmental mobility of polymer chains which resulted from chain degradation. Also, increasing irradiation dose led to increased crosslinked

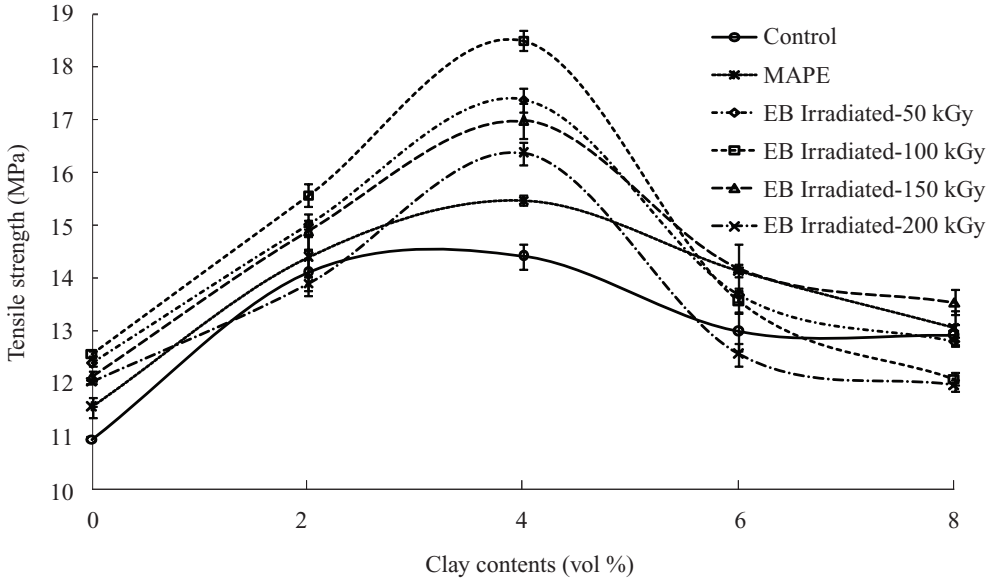

Figure 1. Effect of OMMT content and surface modification on tensile strength by control, MAPE and EB irradiated systems.

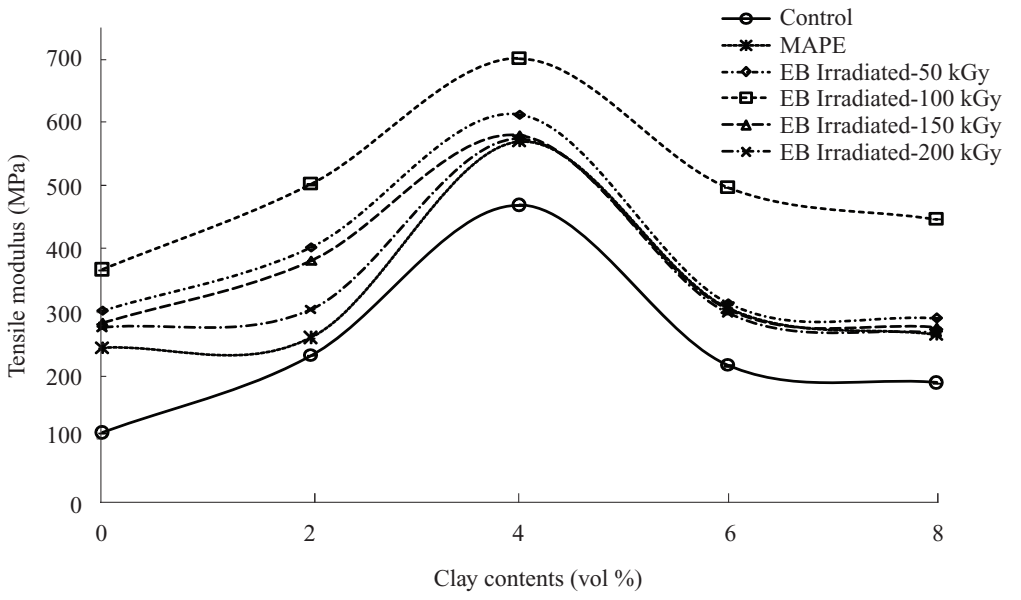

Figure 2. Effect of OMMT content and surface modification on tensile modulus by control, MAPE and EB irradiated systems.

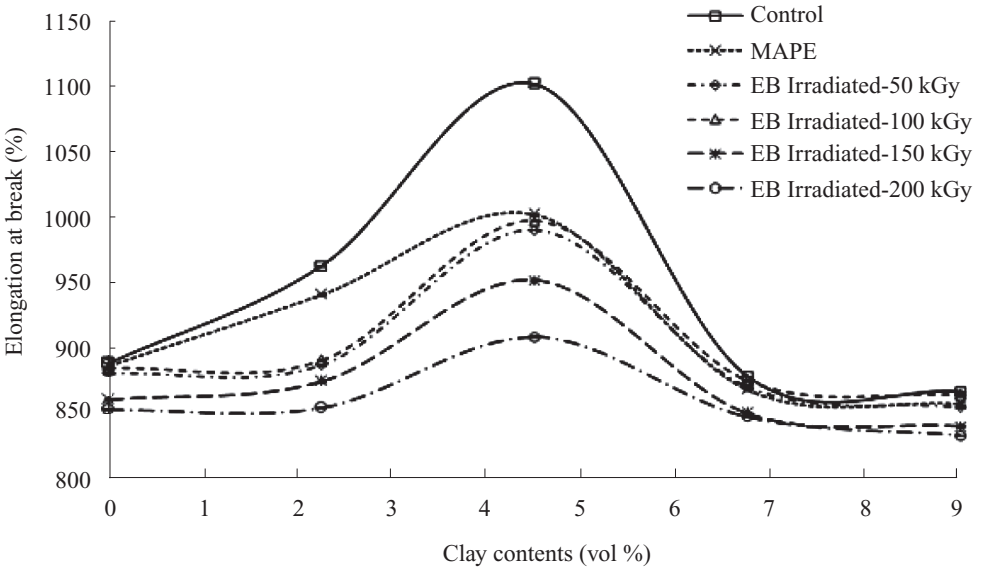

Figure 3. Effect of OMMT content and surface modification on elongation at break by control, MAPE and EB irradiated systems.

density and hence hindered the extension of chains which resulted in lower values of elongation at break. The enhanced crosslinking density may not necessarily increase the tensile properties of the polymer matrix, as in the case of elongation at break due to the EB irradiation induced scission of polymer chain as well as brittleness.

Flexural Strength and Modulus

The effects of organoclay loadings on the flexural strength and modulus for control, MAPE and EB irradiated systems are illustrated in *Figures 4* and *5*. The same trends in flexural strength and modulus were obtained as in tensile properties. Moderate increase in flexural strength and modulus were observed for the MAPE system. This indicates that functionalisation lowered the hydrophobicity of polymer matrix, imparting polarity which makes it more compatible with hydrophilic clay.

Significant increases in flexural strength and modulus were observed with EB irradiated system. An improvement of 39% (stdev = 0.48) and 40% (stdev = 0.33) were achieved at 100 kGy absorbed dose. As expected, flexural strength and modulus increased with radiation dose and continue to decrease at absorbed dose of 150 kGy and above. Such increase may be attributed to the transfer of electron energy to the polymer. According to Park *et al.*[15], during irradiation, the electron loses kinetic energy and momentum due to interactions with the polymer matrix. The authors added that two possible types of interactions involved during EB irradition are known as elastic and inelastic scattering. Increase in flexural strength of HDPE/EPDM filled OMMT with irradiation dose is believed to be due to the process of inelastic scattering as it involves transfer of electron energy to the polymer. This process will lead to bond crosslinking rather than scission depending on the structure of the polymer. In contrast, the decrease in strength and modulus at higher absorbed doses

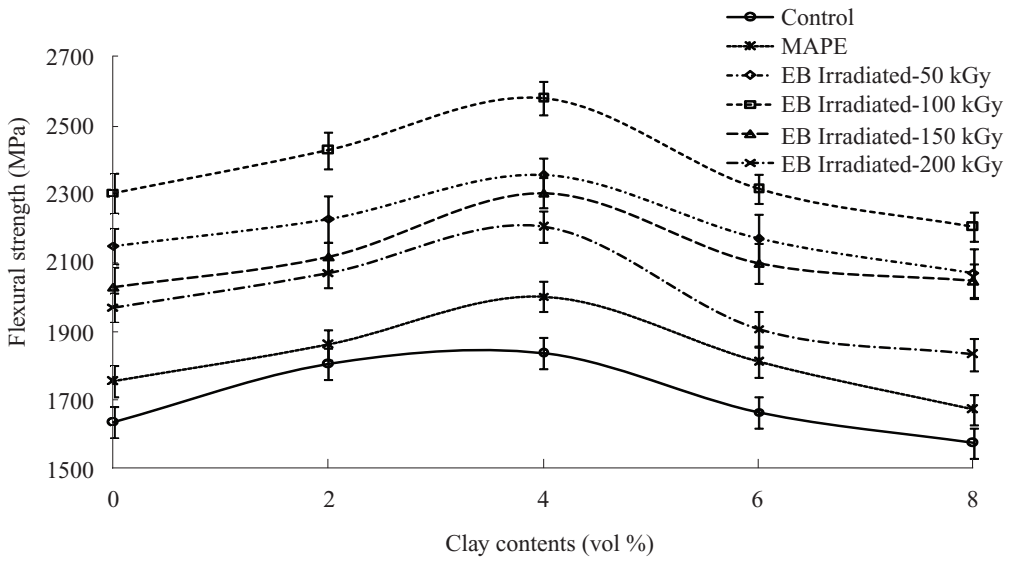

Figure 4. Effect of OMMT content and surface modification on flexural strength by control, MAPE and EB irradiated systems.

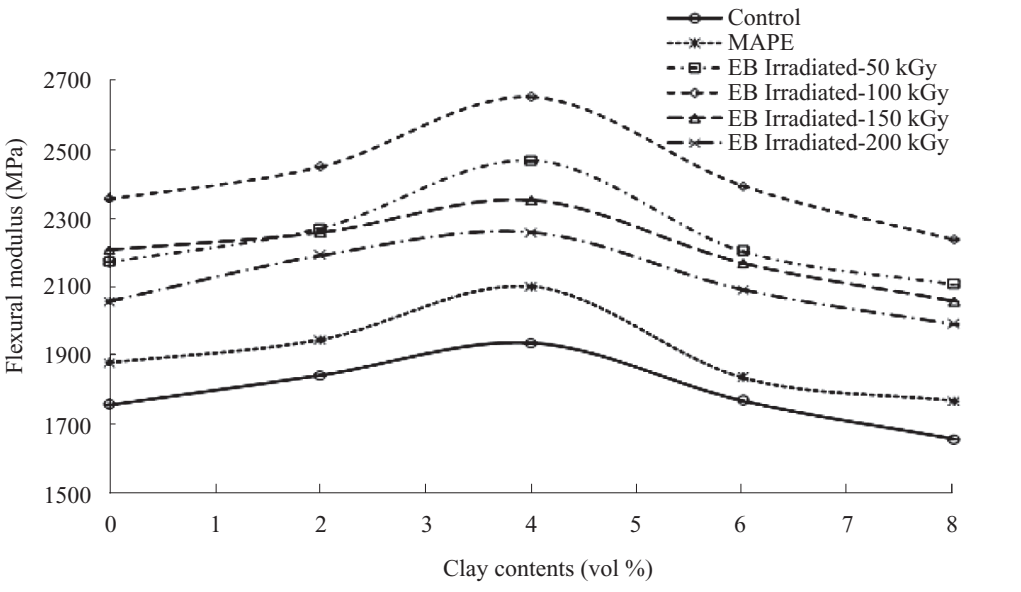

Figure 5. Effect of OMMT content and surface modification on flexural modulus by control, MAPE and EB irradiated systems.

have been attributed to the already formed crosslinked network between HDPE/EPDM matrix and clay particles interface had become smaller due to the scission process.

Impact Strength

The effects of clay loadings on control, MAPE and EB irradiated system of notched Izod impact strength are depicted in *Figure 6*. It can be seen that a moderate increment on the impact strength for both HDPE/EPDM matrix and HDPE/EPDM filled OMMT with initial incorporation of 2 vol% OMMT followed by a sudden dropping at 6 vol% of OMMT loading. This is possibly due to the presence of acceptable amounts of rubber compound as impact modifier along with the aids of OMMT dispersed as spherical domains in polymer matrix and contributes to improved toughness. Similar pattern of behaviour was also reported in previous studies[15–19]. Similar explanation as in tensile strength and modulus section could be attributed to the decrease in impact strength.

The presence of MAPE significantly modified the impact strength value by 19% (stdev = 0.52) as compared to the control system at an optimum level of 4 vol% OMMT loading. This is believed to be due to the higher diffusion of the lower molecular weight MAPE chains through the polymer matrix and its easier penetration into the octadecylamine group formed at the OMMT surface.

EB irradiated system showed the highest value of impact strength as compared to control and MAPE systems. The impact strength of EB irradiated increased with clay loading but decreased as clay loading reached 6 vol% and above. For HDPE/EPDM matrix, an increase of 30% (stdev = 0.38) is achieved at optimum absorbed dose of 100 kGy followed by 23% (stdev = 0.52), 22% (stdev = 0.61) and 20% (stdev = 0.17) improvement at 50, 150 and 200 kGy. Moreover, the introduction of 4 vol% OMMT loading enhanced the impact strength by 25% (stdev = 0.9), 20% (stdev = 0.62), 18% (stdev = 0.73) and 15% (stdev = 0.46) at absorbed doses of 100, 50, 150 and 200 kGy. This might be due to the crosslinking effect in HDPE/EPDM matrix and HDPE/EPDM filled OMMT which resulted in three dimensional and gel-like structures. In contrast, the reduction in impact strength contributed to the radiation induced scission which caused the break of C-C bond.

Gel Content Analysis

The susceptibility of HDPE/EPDM matrix as well as HDPE/EPDM filled OMMT to crosslinking process was estimated from the gel fraction determination. The gel content of control, MAPE treated and irradiated samples increased with the presence of OMMT at optimum loading of 4 vol%. As illustrated in *Figure 7*, there was no gel formation observed for control system without the addition of OMMT. Only 20% of gel formation was obtained with 4 vol% OMMT loading. The gel percentage of HDPE/EPDM matrix increased with OMMT loading for both untreated and treated nanocomposites. This may be attributed to a better dispersion of clay particles in HDPE/EPDM matrix.

In this study, the tensile strength property was identified to reflect the extent of crosslinking *via* gel formation. A linear relationship is obtained between the tensile strength and gel formation percentage. An improvement of 27% and 31% are achieved for both HDPE/EPDM matrix and HDPE/EPDM filled OMMT with MAPE. This shows that the MAPE can act as a compatibiliser and thus decrease the interfacial energy between the two phases. Thus, the wetting of the OMMT with polymer matrix material is improved. As a result, the percentage of gel content increased.

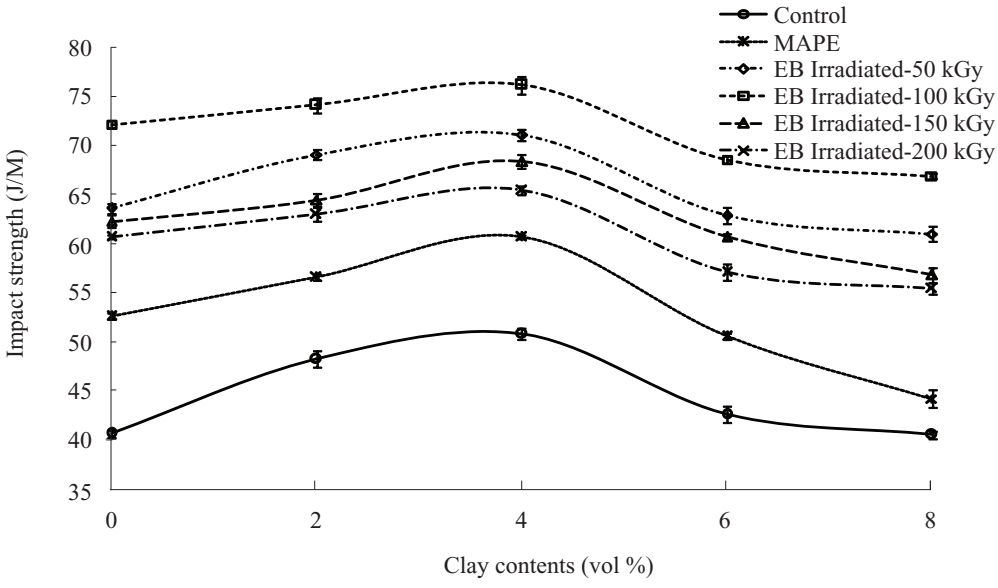

Figure 6. Effect of OMMT content and surface modification on impact strength by control, MAPE and EB irradiated systems.

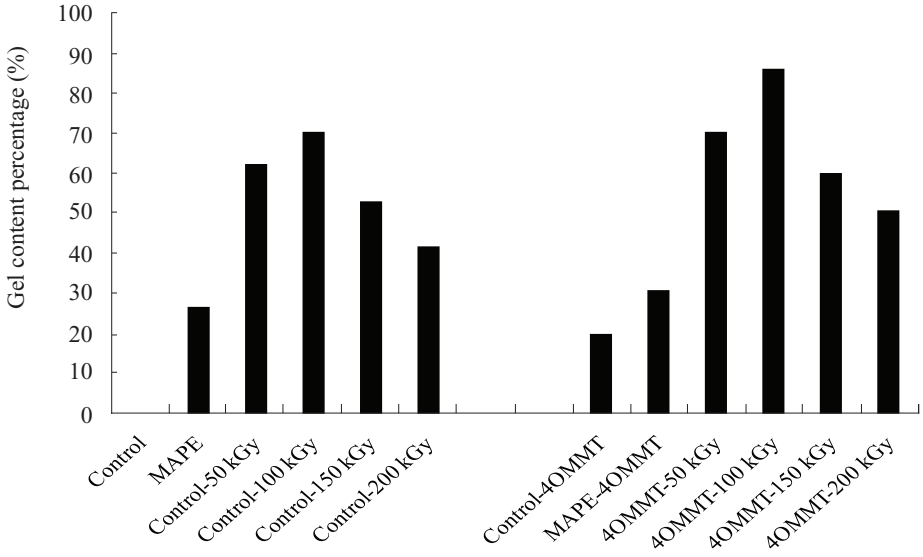

Figure 7. Effect of OMMT content and surface modification on gel content.

For EB irradiated system, the gel content increased rapidly by increasing the radiation dosage up to 100 kGy, beyond which it slowly decreased. An enhancement of 63%, 70%, 53% and 42% was observed for HDPE/EPDM matrix. Moreover, for HDPE/EPDM filled OMMT, an improvement of 70%, 86%, 60% and 51% was obtained in gel formation. As EPDM and HDPE are organic polymers which are categorised as radiation crosslinkable materials, then such formation of crosslinks upon irradiation are to be expected in them. Kim[21] mentioned that the degree of crosslinking increases with irradiation dose rate due to the increase in the concentration of the free radicals. As the crosslinking and the chain scission during irradiation occur simultaneously, the decrease in gel formation beyond 100 kGy might be attributed to the 'loosening' of the network upon irradiation.

X-Ray Diffraction Analysis (XRD)

Judging from the above results, the optimum OMMT content and EB irradiation absorbed dose were taken as 4 vol% and 100 kGy. Therefore, from this section onwards, the discussions were based on these optimum parameters. The changes in the interlayer distance of clay can generally be elucidated using XRD. The peak for control system (untreated) was dulled, indicating that most of the clay is still in the original stacked condition. The diffraction angle and interplanar spacing values are summarised in *Table 2*.

As MAPE was added, the (001) peak still appeared, but its intensity was obviously lowered. This suggests that the diffraction peak of MAPE system has shifted towards a lower angle and higher diffraction peak which were about 0.4º and 4 Å. This shows intercalation of OMMT layers resulting in broadening of XRD peak. As a result, the interlayer spacing of the clay increased, and the interaction of the layers should be weakened. However, in the current study, some clay still kept the original stacking condition with MAPE as revealed by the TEM micrographs.

Nanocomposites with exposure to EB irradiation revealed intercalated structure based on the absence of any basal reflections in the XRD patterns as evidenced by TEM image. It can be seen from *Figure 8* and *Table 2*, that the diffraction peak was significantly shifted towards a lower angle and higher diffraction peak which were about 0.8º and 6.30 Å. This suggested that the surface interaction between the OMMT particles layers was decreased for the benefit of improving the surface interaction between OMMT particles and polymer matrix. Moreover, this might imply that the interlayer distance of OMMT particles have reduced due to the formation of crosslinking structure[20]. However, the shift in the diffraction peak to lower 2θ values may not necessarily offer evidence for complete exfoliation or intercalated structure, which has been indicated by TEM examination.

Transmission Electron Microscope (TEM)

The nanometer scale dispersion of the treated clays OMMT within the polymer matrix is further corroborated with TEM images as depicted in *Figures 9, 10* and *11*, respectively. The lighter region represents HDPE part, the dark region represents EPDM part whereas the dark lines correspond to silicate layers (OMMT).

Silicate layers with bulky stacks were found to be intercalated in the polymer matrix for control and MAPE systems as shown in *Figure 9* and *Figure 10*, respectively. In the case of control system, the non-uniformity of OMMT agglomerates was easily detected.

TABLE 2. INTERPLANAR SPACING AND DIFFRACTION ANGLE VALUES FOR CONTROL, MAPE AND EB IRRADIATED SYSTEMS

Systems	2θ (º)	Interplanar spacing, d (Å)
Control/4 vol% OMMT	3.6	24
MAPE/4 vol% OMMT	3.2	29
EB irradiated-100 kGy/4 vol% OMMT	2.8	31

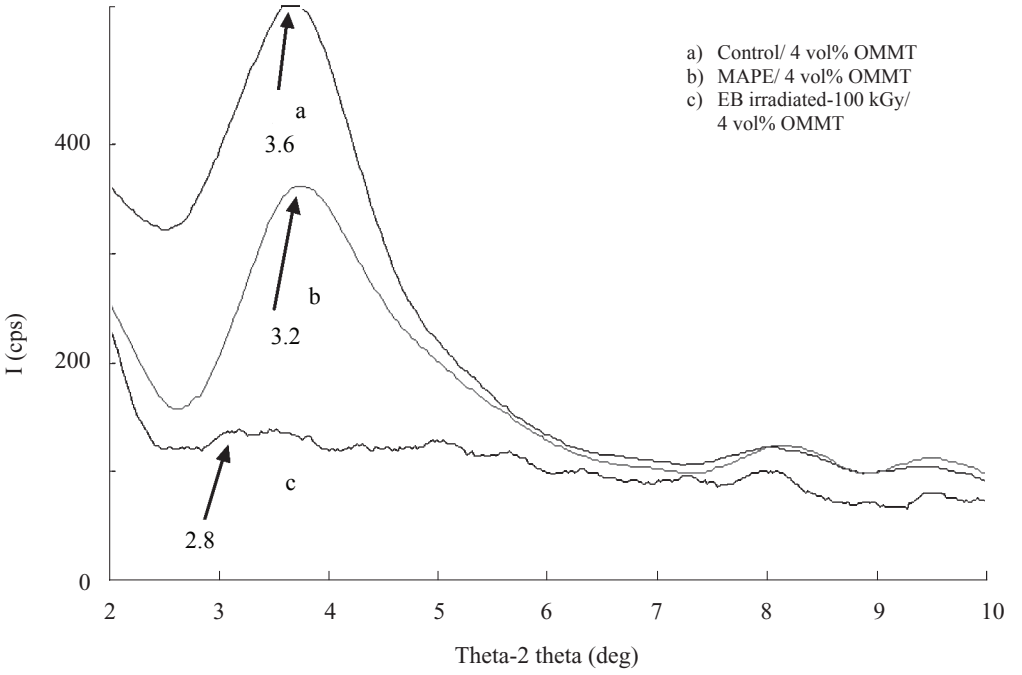

Figure 8. XRD analysis of control, MAPE and EB irradiated systems.

Moreover, it can be seen that the dispersion of the clay particles was poor with large aggregates.

However, in the case of EB irradiated system as shown in *Figure 11*, the large aggregates of clay layers had broken down between three and four aggregates corresponding to the intercalation structure which resulted in slightly smoother and finer surface as compared to control and MAPE systems. This indicates that the surface interaction between OMMT particles and HDPE/EPDM matrix had slightly improved. On the other hand, modification of clay with high energy EB irradiation lowered the electrostatic interactions between the clay layers enlarged their intra-gallery spacing thus facilitating intercalation and exfoliation with efficient dispersion of the clay[20–22]. It also demonstrated superior nanocomposites performance as discussed in the mechanical properties section.

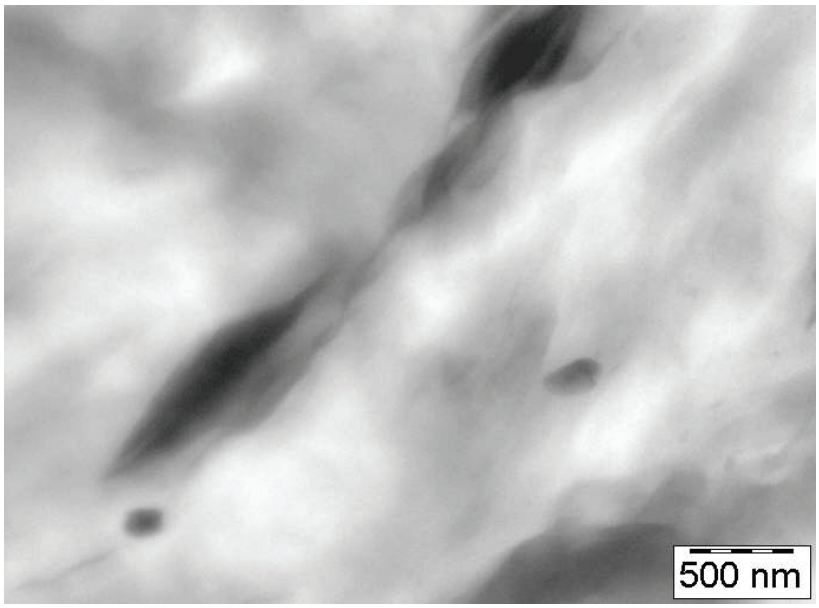

Figure 9. TEM micrographs of control system (untreated).

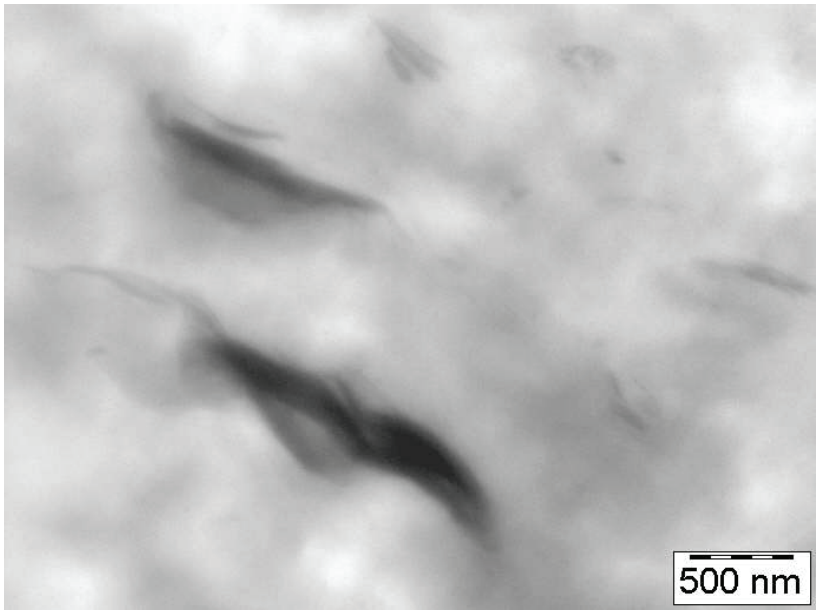

Figure 10. TEM micrographs of MAPE system.

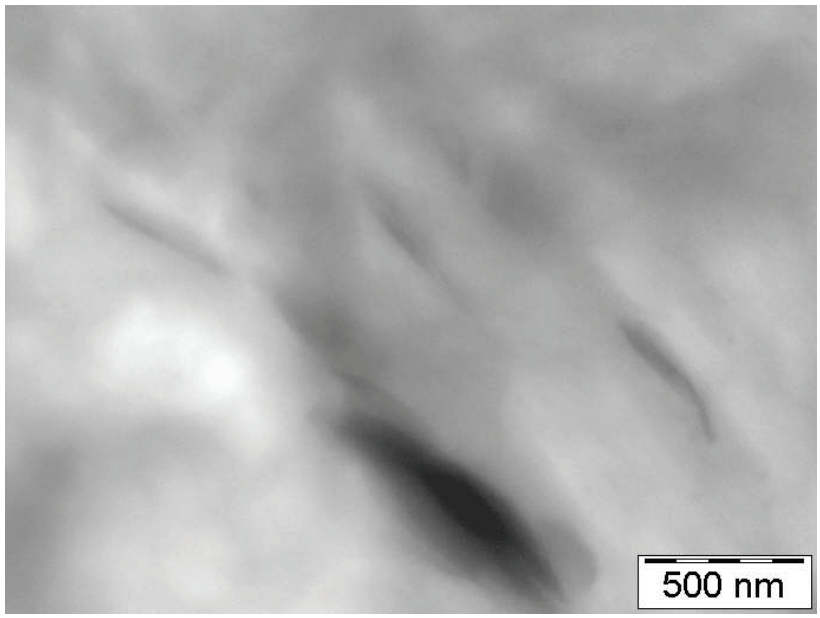

Figure 11. TEM micrographs of EB irradiated system

CONCLUSIONS

The effects of MAPE and EB irradiation crosslinking on the mechanical properties and gel content formation of both HDPE/EPDM matrix and HDPE/EPDM filled OMMT were investigated in the current study. The findings are summarised as below:

- A good balance of properties in terms of stiffness and strength was achieved at 4 vol% OMMT content.
- Electron beam (EB) radiation has induced high crosslinking as evidenced by gel content analysis thus enhancing the mechanical properties of both HDPE/EPDM matrix and HDPE/EPDM filled OMMT.
- A linear relationship between mechanical properties and gel content has been achieved where the higher the mechanical properties, the greater the gel formation.
- High energy EB irradiation can be an alternative as better impact and surface modification of nanocomposites system in which it can replace the role of chemical crosslinking which has been applied in many studies for decades.

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