

## Research Article

# The Influence of EB-Irradiated Treatment on Enhancing Barrier Property and Crystallization Behavior of Rubber-Toughened Nanocomposites

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Electron beam (EB) irradiation technique was introduced to modify the crystallization and oxygen (O<sub>2</sub>) barrier properties of high density-polyethylene (HDPE)/ethylene propylene diene monomer (EPDM) matrix and HDPE/EPDM filled with organophilic montmorillonite (OMMT). The absorbed dose for EB-irradiation was fixed at 100 kGy. HDPE/EPDM matrix and HDPE/EPDM filled with OMMT at 4 vol% loading were prepared via melt intercalation method. It was found that the barrier resistance of HDPE/EPDM filled with OMMT against oxygen (O<sub>2</sub>) transmission was significantly enhanced by EB-irradiation absorbed dose of 100 kGy as compared to the control system. The crystallization temperature,  $T_c$ , and melting temperature,  $T_m$ , were also improved with the addition of OMMT along with the aids of EB-irradiation technique. Field emission scanning electron microscope (FESEM) revealed that the stacking condition of OMMT particles was greatly reduced by EB-irradiation treatment as evidenced by finer surface and less formation of voids.

## 1. Introduction

For many years, increasing interest has been devoted to polymeric alloys because blending is an important route for the preparation of materials with synergistic and modified properties [1, 2]. Polymer blends play an important role in achieving the required and desired properties that cannot be gained from a polymer alone. However, such a generalized concept should be more clearly defined to include the scale of the reinforcements since systems can be reinforced chemically at both microscopic and nanoscopic levels. Because of this reason, nanosized filler is introduced in order to develop nanocomposite with desired properties. The nanosized filler is based on the use of a low concentration of expandable smectite clays, such as organomontmorillonite (OMMT), in the matrix. Multifunctional benefits such as mechanical performance, oxygen permeability resistance and flame-retardant characteristics are obtained simultaneously.

Different types of filler or reinforcement can be incorporated into polymer blending system, but the selection of the silicate layer is more preferable. This is due to the fact that use of traditional filler such as talc and mica increases the mechanical properties and yet additionally increases weight which is typically considered a negative factor in designing a material. Because of this reason, clay silicate as an emerging class of filler which requires less content to achieve similar or improved mechanical and thermal properties as well as barrier property has been used in this research work [3, 4]. Small amounts of well-dispersed natural clay can lead to environmentally friendly and inexpensive plastic composites with improved specialized properties and thus produce a new class of lightweight materials. However, immiscibility or incompatibility of most polymers is a serious barrier to processing blends of polymer matrix and nanofiller. A common approach to alleviate this problem involves the addition (or the in situ formation) of an interfacially active

agent, or the so-called compatibilizer, to the blend [5]. Crosslinking agents or the so-called compatibilizer agents have been studied and applied for years due to their ability to improve both physical and chemical interactions between polymer matrix and filler and thus leads to better mechanical and thermal properties.

On the other hand, EB irradiation as one type of high-energy radiation technique has been a popular method of crosslinking instead of chemical crosslinking as it is a fast, pollution free, and simple process. Although there are other high-energy methods like gamma irradiation claimed by Croonenborghs et al. [6] to give similar effects in the investigated polymer properties, gamma irradiation is time consuming as compared to EB irradiation [7, 8]. The importance of doing research work is to make use of time and at the same time reduce the processing cost. Dubey et al. [7] stated that EB irradiation is an excellent irradiation processing compared to other electromagnetic irradiations as it induces the chemical changes in irradiated system radiation by the use of high-energy ionizing radiation. Understanding how and to what extent the characteristics of polymer blend can be altered as a function of the level of radiation exposure is crucial to predicting the performance and utility of irradiated plastics.

Although a lot of work has been done on elastomer blends, studies on the blends of EPDM and HDPE with the addition of OMMT as filler as well as its ability in gas barrier resistance are meager. Therefore, this research initiates to explore the possibilities for making engineered thermoplastic and rubber compound nanocomposite by the treatment of EB irradiation technique. The development of this nanocomposite system is aimed to vary the existing research done in this area as well as being a future and important reference particularly in enhancing the barrier property of nanocomposite.

**1.1. Overview of the Current Stage of Packaging Industry in Malaysia.** For decades, the flexible packaging consumption has grown rapidly within the global community in the United States (USA) in addition to Europe and Japan. In contrast to Malaysia, the fabrication of individual or composite material with superior barrier property is still at a critical stage. This is due to the little effort and interest implemented in the area of study and research in enhancing barrier property for commercial packaging applications as compared to other countries such as USA, Europe, and Japan. An innovation to enhance barrier property of material produced is by developing polymer nanocomposite technology which holds the key to future advances in flexible packaging.

A majority of consumer products use polymeric material or composite material in packaging areas which are commonly applied in the plastic industry. Many different types of commercial plastics, flexible and rigid, are commonly used including polypropylene (PP), nylon, polyethylene terephthalate (PET), and polyethylene (PE). Polymer matrix (HDPE/EPDM) used in this study is known to

possess good water vapor barrier properties. However, it is easily permeated by oxygen, carbon dioxide, and hydrocarbons. Thus the necessity of developing more effective barrier polymers has given rise to different strategies to incorporate and optimize the features from several components. Most schemes to improve gas barrier properties involve either the addition of higher barrier plastics via a multilayer structure (processing techniques) or by introducing filler with high aspect ratio in the polymer matrix.

Polymer nanocomposites which are constructed by dispersing a filler material into nanoparticles that form flat platelets are an ideal system for barrier property construction. These platelets are then distributed into a polymer matrix creating multiple parallel layers which force gases to flow through the polymer in a torturous path, forming complex barriers to gases and water vapor [9]. As more tortuosity is present in a polymer structure, higher barrier properties can be obtained. Once these tiny flat platelets are dispersed into the plastic, they create a path that gases must follow to move through the material, thus greatly slowing their transmission [10]. Therefore, nanocomposites would ease the transition from current packaging with metal layers and glass containers to flexible pouches or rigid plastic structures. The advantages that nanocomposites offer far outweigh the costs and concerns, and with time the technology will be further refined and processes more developed.

## 2. Experimental

**2.1. Materials.** Homopolymer high-density polyethylene (HDPE) (melt index 3–6 g/10 min, density 900 kg/cm<sup>3</sup>) supplied by Cementhai Chemicals Group, Thailand and ethylene propylene diene monomer (EPDM) supplied by Centre West Sdn Bhd, Malaysia were used as the base polymer matrix. 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 reinforcing agent to prepare HDPE/EPDM filled with OMMT.

**2.2. Compounding.** Melt blending of HDPE (70 vol%) and EPDM rubber (30 vol%) as polymer matrix was carried out first in an internal mixer (Thermo Haake Rheomix 600P). Then, the HDPE/EPDM blend was mixed with OMMT at HDPE/EPDM : OMMT ratio of 96 : 4 vol%. The melt mixing conditions were 150°C and at a rotor speed of 100 rpm. Prior to mixing, the polymer matrix and the nanoclays were dehumidified in a dry oven at 110°C for a period of 1 hr. Details on the preparation of HDPE/EPDM and HDPE/EPDM filled with OMMT are summarized in Tables 1 and 2.

**2.3. Specimen Preparation.** Subsequently, the blended samples were compression-molded as per ASTM-F-412 using a compression molding machine at a temperature range of 135–155°C and 8 tone metric pressures for 14 min.

TABLE 1: Preparation of HDPE/EPDM blend (polymer matrix) via melt blending method.

Polymers	Polymer content (vol%)	Ratio of HDPE to EPDM blend	Total composition
High-density polyethylene (HDPE)	70	70 : 30	100
Ethylene propylene diene monomer (EPDM)	30	70 : 30	100

TABLE 2: Preparation of HDPE/EPDM and HDPE/EPDM filled with OMMT via melt blending method.

Systems	Polymer matrix	Polymer matrix content (vol%)	Filler content, OMMT (vol%)	Total composition	Surface modification
Control	HDPE/EPDM	96	—	100	—
100 kGy	HDPE/EPDM	96	—	100	Electron beam irradiation at 100 kGy
Control	HDPE/EPDM	96	4	100	—
100 kGy/OMMT	HDPE/EPDM	96	4	100	Electron beam irradiation at 100 kGy

OMMT = organophilic montmorillonite.

TABLE 3: DSC Results for unfilled composites and nanocomposite systems.

Systems	Composite	$T_m$ (°C)	$T_c$ (°C)	$\Delta H$ (J/g)	$X_c$ (%)
Control	HDPE/EPDM	122.5	113.8	110.9	40.04
EB irradiated-100 kGy	HDPE/EPDM	130.8	118.9	132.7	47.91
Control	HDPE/EPDM/OMMT	131.8	115.7	120.5	43.5
EB irradiated-100 kGy	HDPE/EPDM/OMMT	134.1	120.7	146.2	52.78

OMMT = organophilic montmorillonite;  $T_m$  = melting temperature;  $T_c$  = crystallization temperature;  $\Delta H$  = melting enthalpy;  $X_c$  = crystallization percentage.

**2.4. High-Energy EB Irradiation.** The melt compounding samples were exposed to high-energy EB irradiation absorbed dose of 100 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.

### 3. Characterization

**3.1. DSC Analysis.** DSC of each sample was performed using Perkin Elmer DSC 7. Samples with weight from 7 to 9 mg were used for the analysis. Measurements of the glass transition temperature,  $T_g$  and crystallization temperature,  $T_c$  were recorded as a function of temperature in the range of  $-80^\circ\text{C}$  to  $200^\circ\text{C}$  with heating rate of  $10^\circ\text{C}/\text{min}$ . The crystallinity % of nanocomposites was determined using the following relationship:

$$(\% \text{ crystallinity}) \quad X_c = \frac{\Delta H_f}{\Delta H_f^0} \times 100, \quad (1)$$

where  $\Delta H_f$  and  $\Delta H_f^0$  are the enthalpy of fusion of the system and the enthalpy of fusion of perfectly (100%) crystalline HDPE, respectively. For  $\Delta H_f^0$  (HDPE), a value of 277 J/g was used for 100% crystalline HDPE homopolymer [11].

### 3.2. Morphology

**3.3. Field Emission Scanning Electron Microscope (FESEM).** Examination of cryogenic fractures surface etched with nitric acid has been performed using JEOL JSM 6700F scanning electron microscope. All samples have been measured after sputter coating with carbon to avoid electrostatic charging and poor image resolution.

**3.4. Gas Barrier Test: Oxygen Transmission Rate ( $O_2TR$ ).**  $O_2TR$  of nanocomposites was analyzed in MOCON OX-TRAN 2/20 devices in accordance to the ASTM D3985 standard method. Specimens having circular shape with thickness of 1 mm and diameter of 130 mm were conditioned in a desiccator at room temperature for a minimum of 48 hours. The test conditions used in determining the  $O_2TR$  of nanocomposites were  $23^\circ\text{C}$  and 0% RH with nitrogen flow rate of 20 mL/min. The resulting permeability was recorded as  $\text{cm}^3/\text{m}^2/\text{day}$ .

## 4. Results and Discussion

**4.1. Differential Scanning Calorimeter Analysis (DSC).** The effect of OMMT loading and EB-irradiated system on the crystallization temperature,  $T_c$ , melting temperature,  $T_m$ , and crystallinity content,  $X_c$  of nanocomposite systems was analyzed by DSC thermograms. The  $T_c$ ,  $X_c$ ,  $T_m$ , and heat

of fusion,  $\Delta H$ , values for both systems are outlined in Table 3. The DSC thermograms of heating scan ( $T_m$ ,  $X_c$ , and  $\Delta H$ ) for unfilled polymer matrix and nanocomposite at 4 vol% clay loading are presented in Figures 1 and 2, respectively. Moreover, the DSC thermograms of cooling scan ( $T_c$ ) for unfilled polymer matrix and nanocomposites at 4 vol% clay loading are demonstrated in Figures 3 and 4. All the heating and cooling scans show only one endothermic as well as exothermic peak, but some differences in the peak temperatures can be revealed. Durmuş et al. [12] mentioned that a single crystallization peak observed for the nanocomposite system could be attributed to the compatibility of the polymer matrix and nanoclay. This indicates that the presence of multiple peaks (possibly attributed to  $\beta$  and  $\gamma$  phases) in the nanocomposite system may be due to the presence of either imperfect or small-sized crystallites [13].

According to Figures 2 and 4, the values of  $T_m$  and  $T_c$  for control and EB-irradiated systems at 4 vol% OMMT were shifted toward the higher temperature range. The  $\Delta H$  values were also increased as compared to the unfilled polymer matrix (refer to Table 3). Similar observations were also reported by previous researchers [12–14]. The addition of OMMT at optimum level of 4 vol% increased the  $T_m$  values for the whole nanocomposite system. The increasing in  $T_m$  values signifies that the crystal thickness in nanocomposites is more perfect than in polymer matrix [14]. Similar improvement pattern was observed for  $T_c$  and  $X_c$  values suggesting that the crystallization mechanism of neat polymer matrix was enhanced. The nucleation mechanism is responsible for the growth of crystals in nanocomposites as OMMT silicates acting as a heterogeneous nucleating agent have increased the overall crystallization rate and the crystalline fraction [15].

The presence of OMMT is identified to narrow the width of the crystalline peak which also indicates an increase in the crystallization rate of the polymer matrix chains. However, such observation is not significant for the current study. Mingliang and Demin [16] stated that the strong interaction existing between polymer matrix molecules and the layers of organoclay has resulted in immobilizing of some polymer matrix as organoclay easily absorbs the polymer molecule segments. These immobilized molecules of polymer matrix contribute to the crystallization process of nanocomposite; therefore, the crystallization of polymer matrix molecules has occurred at higher temperature thus increasing the  $T_c$  values of nanocomposite systems.

The improvement in the values of  $T_m$ ,  $T_c$ ,  $\Delta H$ , and  $X_c$  was obtained with the presence of 4 vol% OMMT by EB irradiation 100 kGy absorbed dose. Such increase can be referred to in Table 3. The increase in crystallinity percentage is believed to be due to the rearrangement of more mobile polymer chains associated with chain scission. Oxidation inhibits crosslinking of nanocomposites and leads to chain scission in the amorphous regions. As a result, the shortened segments can arrange to form new crystals and enhance the degree of crystallinity in irradiated polymer. Moreover, the increase in crystallinity percentage is also caused by fine rearrangements by radiation-induced backbone chain

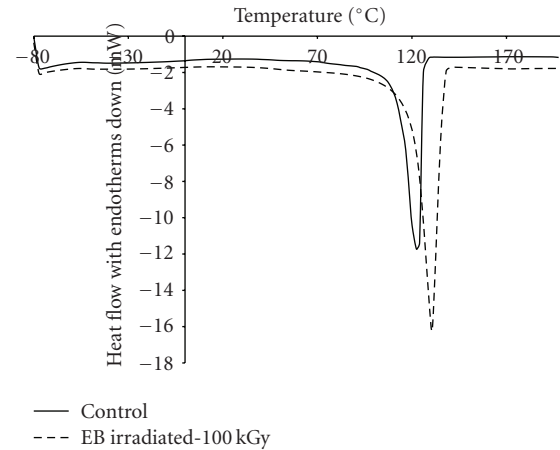


FIGURE 1: Melting endotherms of unfilled polymer matrix for control and EB-irradiated systems.

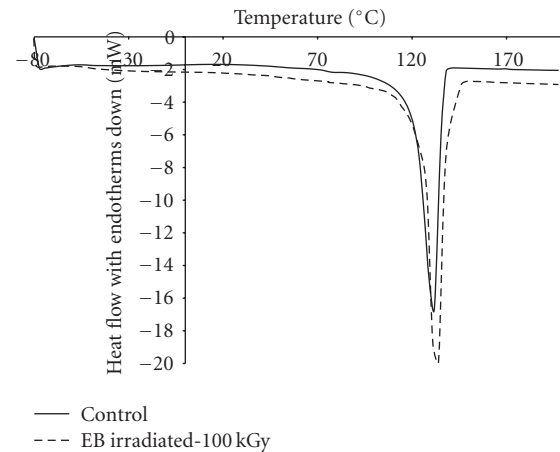


FIGURE 2: Melting endotherms of nanocomposites at 4 vol% clay loading for control and EB-irradiated systems.

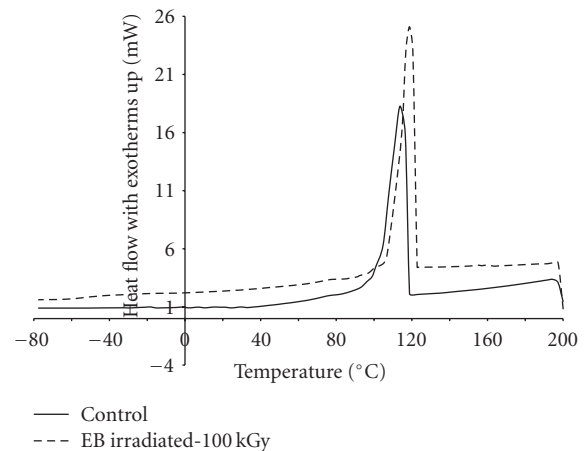


FIGURE 3: Crystallization exotherms of unfilled polymer matrix for control and EB-irradiated systems.



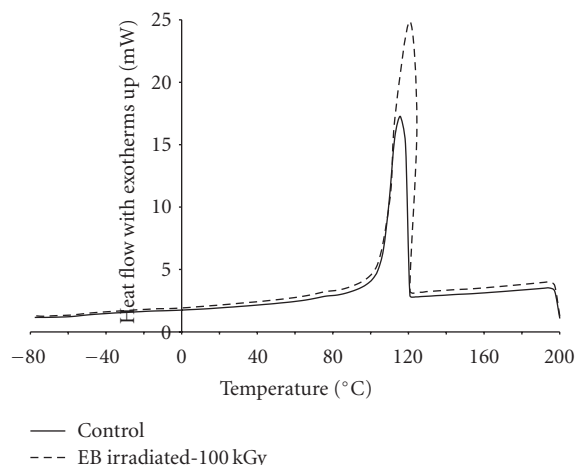


FIGURE 4: Crystallization exotherms of nanocomposites at 4 vol% clay loading for control and EB-irradiated systems.

scission in the lamellae or at the crystal-amorphous interface. Furthermore, the chain freed by scission can move easily and recrystallize [17, 18].

The melting temperatures of the irradiated samples at 100 kGy for both unfilled and 4 vol% filled composites are plotted in Figures 1 and 2. The initial high increase can be attributed to the increase of crystal domains as discussed earlier. The high peak of melting temperatures also suggest that the melting process is being kinetically arrested by the crosslinking, thus producing higher melting temperatures at the relatively fast heating rate of 10°C/min [17, 18]. It can be suggested that crosslinks in the crystal domains may play some role in enhancing the melting temperature of irradiated HDPE/EPDM filled with OMMT. Nho et al. [19] added that the penetration of electron beam shall be more effective in the loosely packed amorphous area because of the amount of oxygen required for the chain scission is higher in the amorphous region as well.

The chain scission and crosslink, therefore, take place predominantly in the amorphous area first. As the polymer chains undergo chain scission in the amorphous region, the increased chain ends provide the mobilities of the entangled chains, leading to the recrystallization of the less-ordered crystals [16, 19]. As the irradiation dose increases and reaches a certain energy level, the electron beam is now able to penetrate into the crystalline domains. The crosslinking points in the crystal domains would hold the chains, reducing the mobility gain from the thermal energy, thus enhancing the thermal stability and requiring higher thermal energy to be melted [19]. Consequently,  $T_m$  values of EB-irradiated system for both unfilled and 4 vol% OMMT-filled composites increased as depicted in Table 3 as well as in Figures 1 and 2.

**4.2. Gas Barrier Testing: Oxygen Transmission Rate ( $O_2TR$ ).** A protection of samples produced against the gas permeation is measured by using the oxygen transmission rate technique ( $O_2TR$ ). The effect of clay loading at 4 vol% as well as

crosslinking techniques on the oxygen ( $O_2$ ) permeability is summarized in Figure 7. As evidenced from Figure 7, the  $O_2$  permeability decreased with the addition of clay loading whereas the introduction of EB irradiation techniques further reduced the value of  $O_2$  permeability. It should be noted that the lower the permeation, the better the barrier. For the control system without any crosslinking treatment, no enhancement in  $O_2$  permeability was observed. Such result is in parallel with the result obtained in Section 4.1 where the higher the crystallinity percentage, the lower the value of  $O_2$  transmission. This is because the crystal regions are impervious to water and oxygen transmission while in the amorphous regions, the water and oxygen molecules are diffused easily. This concept readily accounts for the general observations that gas permeability is reduced by increasing the crystallinity and by decreasing the amorphous phase density.

In the context of gas transport, the nanocomposite is considered to consist of a permeable phase (polymer matrix) in which nonpermeable nanoplatelets are dispersed [20]. In general, there are three main factors that influence the permeability of a nanocomposite which are the volume fraction of the nanoplatelets, the orientation of nanoplatelets relative to the diffusion direction and the aspect ratio [20]. Low fractions of clays which affected in the reduction of the polymer matrix volume are required to achieve equivalent properties in comparison with the traditional composites. Therefore, a decrease of the solubility is expected in the nanocomposite due to the reduced polymer matrix volume, as well as a decrease in diffusion due to a more tortuous path for the diffusing molecules. In addition, the nanoclay layers are believed to be able to act as a trap to preserve the active oxygen foragers in the polymer while reducing the rate of oxygen transmission.

A little enhancement of 11.4% was obtained by EB irradiation absorbed dose of 100 kGy for unfilled composite whereas a significant improvement of 32% was observed with the incorporation of 4 vol% OMMT loading. This indicates that the introduction of EB-irradiation technique significantly modified the barrier property of the system with the aid of OMMT filler. The radiation-induced crosslinking could serve as oxygen scavenger by binding oxygen molecules and thus contribute to the reduction in the oxygen permeability. Moreover, the improvement in barrier property could be related to the enhanced interfacial region between the polymer matrix and OMMT particles which resulted in more homogenous structure (intercalated and exfoliated) as the hydrophobic nature of OMMT was reduced and thus providing more tortuous path. In other words, the reduction of the permeability in the platelet-reinforced nanocomposite was attributed to the lowering of the diffusion coefficient due to a more tortuous path for diffusing molecules as a result of crosslinking formation. Upon irradiation, dense network is always desired as at this stage crosslinking is more prominent than chain scission and thus resulted in improving the thermal properties. This is believed due to the decrease of the free volume that facilitates the diffusion process as the easier pathways has been created for the  $O_2$  transmission.

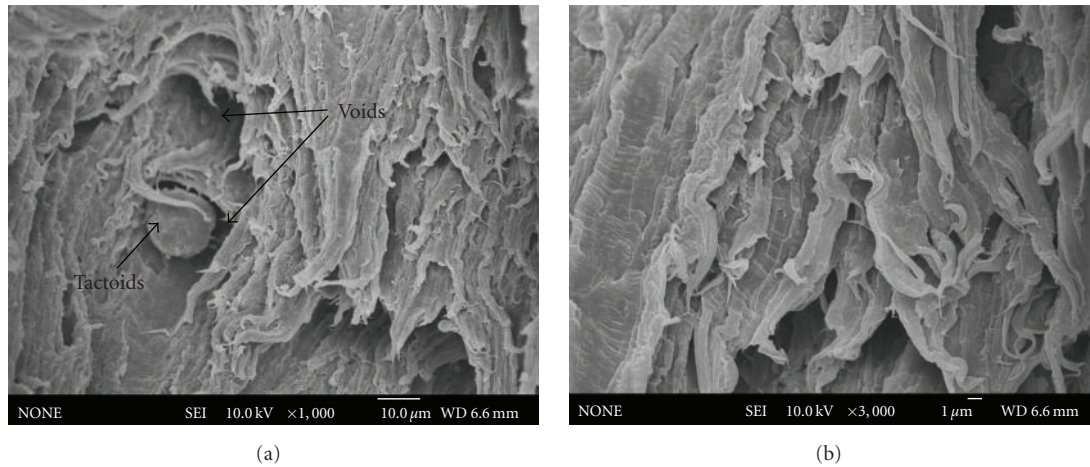


FIGURE 5: FESEM micrographs of control sample.

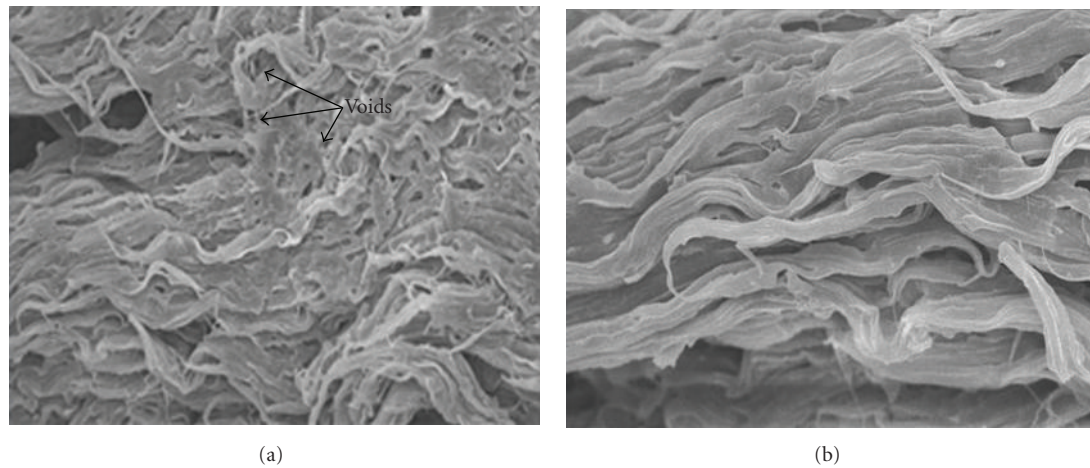


FIGURE 6: FESEM micrographs of EB-irradiated sample.

**4.3. Field Emission Scanning Electron Microscope (FESEM).** FESEM micrographs of HDPE/EPDM and HDPE/EPDM filled with OMMT for control and EB irradiated samples are presented in Figures 5 and 6, respectively. It can be seen that the formation of clay agglomerates known as tactoids could be identified in Figure 5. This is expected as polymer matrix and OMMT particles are immiscible; thus, the clay particles tend to agglomerate as a result of strong interaction existing among these particles rather than interaction with polymer matrix. This resulted in the formation of void which is bigger when compared to EB-irradiated sample as evidenced in Figure 6. The voids being observed earlier are almost covered up after treatment with EB-irradiation absorbed dose of 100 kGy. As a result, there is no presence of tactoids was observed and the clay particles seemed to move freely apart from each other. The formation of fibrils was also visible as a result of the EB irradiated treatment indicating the morphological properties of the strongly enhanced interactions [21]. This resulted in enhancement in

both crystallization as well as barrier properties as discussed in Sections 4.1 and 4.2.

## 5. Conclusion

The findings of the current study demonstrated that EB irradiation is an effective technique to enhance the crystallization and gas barrier properties of HDPE/EPDM and HDPE/EPDM filled with OMMT. Such increase indicated that crosslinking network was successfully created in this system. The addition of 4 vol% OMMT was found to further enhance the thermal and gas barrier properties of HDPE/EPDM as clay particles were able to create more tortuous path due to its impermeable nature towards gases. FESEM micrographs revealed that the formation of bigger voids and tactoids for control system was greatly improved by EB irradiation treatment as voids and tactoids became less visible which resulted in finer and smoother structure.

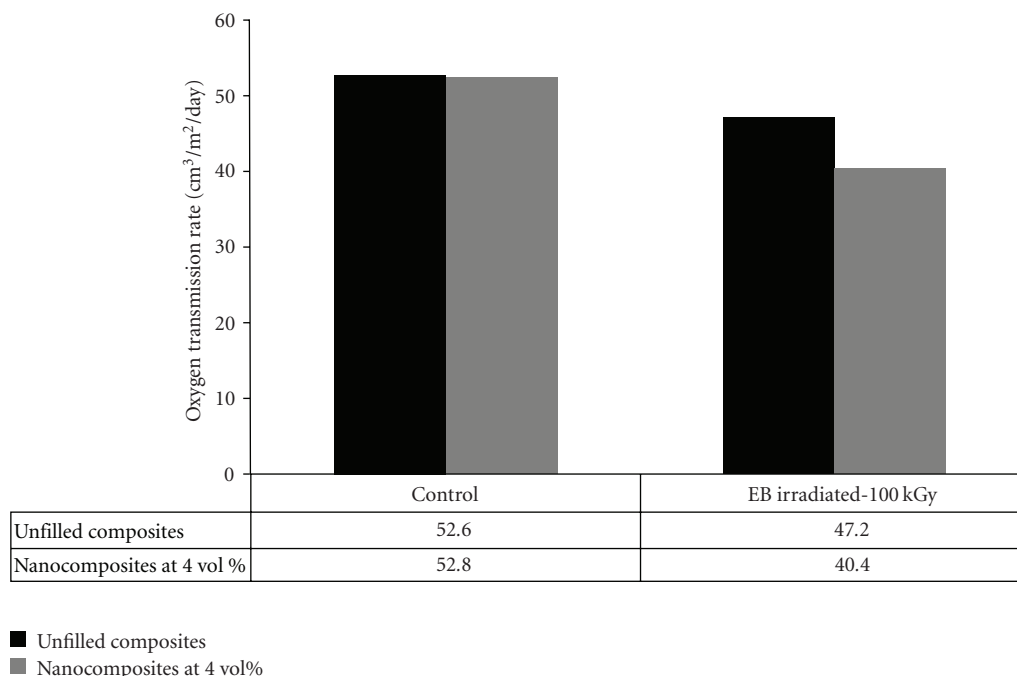


FIGURE 7: Oxygen transmission rate ( $O_2TR$ ) of unfilled composites and nanocomposites for control and EB-irradiated systems.

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