# Characterization of Irradiation-Induced Crosslink Ethylene Vinyl Acetate/Waste Tire Dust (EVA/WTD) Blend

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#### ABSTRACT

The effect of irradiation on 90/10 Ethylene Vinyl Acetate/Waste Tire Dust (EVA/WTD) blend was studied. Blends of EVA/WTD were prepared by using a Haake mixer at 140 °C. The blend was then irradiated by using a 3.0MeV electron beam machine at doses ranging from 50 to 200 kGy in air and room temperature. The dynamic mechanical properties, tensile properties, and gel fraction of the blends were measured. It was found that the tensile strength and modulus increased with irradiation with a concomitant decline in elongation at break. Results on gel fraction revealed that under the irradiation condition employed, the EVA/WTD blend crosslinked by electron beam irradiation. The addition of TMPTA and TPGDA was found to be effective in increasing the degree of crosslinking.

Keywords: Additive, crosslinking, EVA/WTD blend, irradiation

# Introduction

Tires are among the largest and most problematic sources of waste, due to the large volume produced and their durability. Those same characteristics which make waste tires such a problem also make them one of the most re-used waste materials, as the rubber is very resilient and can be reused in other product. Whole scraped tires can be used in a variety of non-transportation applications, such as erosion control, playgrounds, and artificial reef. Recycling of tires includes use of retreads and grinding into crumb for use in pads, mats, carpet backing, moisture barriers, rubber-modified asphalt, and sport tracks. One of the methods that can be use to recycle tires is by blending it with other polymer. Blending of polymer is a common technology. The aim of polymer blends, mixing of two or more polymers, is to obtain materials, which as far as possible combine their advantages, but not their disadvantages (Zurina et al., 2006).

One of the promising alternatives to utilize waste tire rubber is the preparation of thermoplastics elastomers. Besides having physical properties of both thermoplastics and elastomer as well as processability similar to that of thermoplastics, thermoplastics elastomers provides better material utilization, as scrap and rejects can be recycled (Ismail, et al.,2006).

There has been growing interest in blending natural rubber with ethylene-vinyl acetate copolymers (EVA) because it has excellent ozone resistance, weather resistance, and excellent mechanical properties. In this work, the influences of electron beam irradiation (EB irradiation) on the properties of EVA/WTD blend are reported.

## Materials and Methods

#### Materials

Ethylene-vinyl acetate (EVA) (Grade H2020) having 15% vinyl acetate content with a MFI value of 1.5 g/10 min and density of 0.93 g/cm<sup>3</sup> was purchased from The Polyolefin Company, Singapore. Rubber dust from recycle tires (WTD) (40 mesh) used in this study was obtained from Sin Rubtech Consultancy Sdn.Bhd. The Trimethy-lolpropane triacrylate (TMPTA) was a product of UCB Asia Pacific Company and were used as received. The 90/10, EVA/WTD blends were pre-

pared by mixing 90 parts EVA with 10 parts WTD. The recipes used to prepare the blends are given in Table 1.

# **Blend Preparation**

Melt blending was carried out at 120°C and 50 rpm rotor speed in a Haake Rheomix Polydrive R600/610. The blending was done as follows: When the desired temperature was reached, EVA was charged into the mixing chamber and mixed for 2 min. The additives and WTD were then added, and the blending was continued for a further 8 min. The samples were then compression molded at 123°C for 5 min. After that the samples were cooled for 2 min and produce into 1 mm thick sheets.

Table 1:Recipes of EVA/WTD Blends

Material	EWC	EWT	EWP
EVA	90	90	90
WTD	10	10	10
TMPTA	-	4	-
TPGDA			4

Notes; EVA- ethylene-vinyl acetate, WTD- waste tire dust, TMPTA- trimethyl-lolpropane triacrylate, TPGDA-, EWC-, EWT-, EWP-

#### Irradiation

The molded sheets were irradiated using a 3 MeV electron beam accelerator NHV EPS-3000 at dose range of 0-200 kGy. The acceleration energy, beam current, and dose rate were 2 MeV, 2 mA, and 50 kGy per pass, respectively.

### **Gel Content**

The gel content of the crosslinked samples were determined by the extraction of samples in boiling xylene for 24 h using Soxhlet apparatus. The extraction samples were dried in oven at 50 °C till constant weight. The gel fraction was calculated as:

$$= \frac{W_0 - W_1}{W_0} \times 100$$
% Gel content

Where  $W_1$  and  $W_0$  are the weights of the dried samples after extraction and before extraction, respectively.

## **Tensile Properties**

The tensile properties were measured with Instron Universal Testing Machine 4301 H119 at 50 mm/min crosshead speed. The molded samples of 1mm thick were cut into dumb bell shaped test pieces using BS6746 cutter. Five samples were used for tensile test and average results were taken as the resultant value.

## **Dynamic Mechanical Analysis**

Dynamic mechanical properties were measured with Perkin Elmer, dynamic mechanical analyzer DMA 7e. The experiment was conducted in a 3 point bending mode at a frequency of 1 Hz. The temperature was increased at 5°C/min over the range of -80°C to 40°C. The dimensions of the samples were approximately 1 mm thick, 10 mm length and 10 mm width.

# **Results and Discussion**

## **Tensile Properties**

Figure 1 depicts the effect of irradiation on the Tensile strength (Ts) of the 90/10 EVA/WTD blends. The increase in Ts with irradiation dose for EWC is attributed to the formation of irradiation-induced crosslinking in the blend, as well reported elsewhere for PVC/ENR blends (Ratnam and Zaman, 1999 and Ratnam et al., 2001), ENR (Ratnam et al., 2000) and EVA (Sujit et al., 1996). Apparently from Figure 1, EWP and EWT at 50 kGy results in the best enhanced in Ts, but for EWC, it reach the highest value for Ts at 200 kGy. This is again attributed to the enhanced radiation-induced crosslinking by TMPTA and TPGDA as observed from gel content. As the crosslink density increase, higher applied stress is required to cause rupture (Ratnam and Zahid, 2006). Figure 1 also indicates that EWT and EWM blends show gradual drop in Ts after 100 kGy irradiation dose. This decline in Ts could be associated with excessive crosslink in the polymer molecule that make the polymer brittle.

Elongation at break of the samples depends upon the nature of the polymer as well as on the degree of crosslink, which restrict the movement of the polymer chain against the applied force (Zurina et al., 2006). Generally, increasing the irradiation dose cause a reduction in elongation at break of the blend, because the blends become increasingly brittle as a consequence of the increase in crosslink density with irradiation. From Figure 2, it is clear that EWT shows lowest Eb because with the presence of TMPTA, it will result in higher crosslink density, thus asserting the acceleration of crosslinking by the additive (Chantara, 2006).not in references

From Figure 3, modulus at 100% strain (M100) shows no significant change in stiffness at 100% elongation of irradiated and non-irradiated for EWP and EWC blends. The same result was reported for irradiated metallocene elastomer/PA 6 blend and PVC/ENR blend, but with the addition of TMPTA, M100 increased. It indicate that the acceleration of crosslinking by tri-functional monomer additives occur. The modulus depends directly on crosslink density or a perfect network. Crosslink density for EWC and EWP quite low compared to EWT. These agree with the gel fraction test in later section.

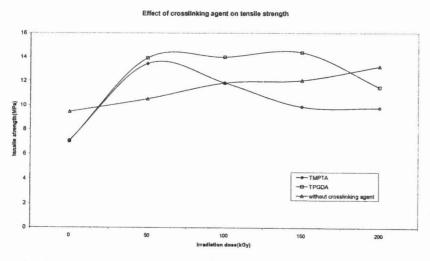


Figure 1:Effect of Crosslinking Agent on Tensile Strength of 90/10 EVA/WTD Blend.

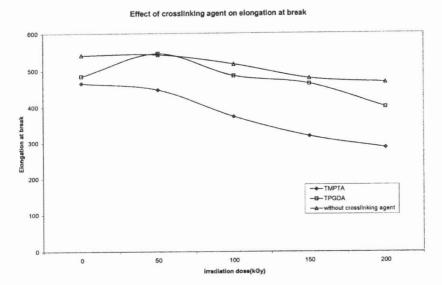


Figure 2: Effect of Crosslinking Agent on Elongation at Break of 90/10 EVA/WTD Blend.

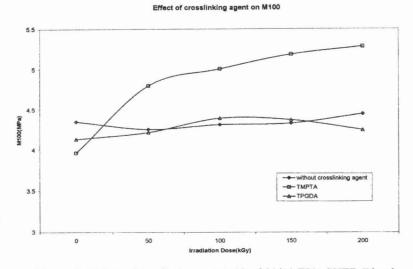


Figure 3: Effect of Irradiation on M100 of 90/10 EVA/WTD Blend.

#### **Gel Fraction**

In general, the yield of irradiation-induced crosslinking can be estimated from gel fraction determination. The variation of gel content with irradiation dose for 10/90 WTD/EVA blend and the blend containing TMPTA and TPGDA are shown in Fig.4. A similar increase in gel fraction was reported by (Ratnam et al., 2000), and (Sujit et al., 1996) in their studies on ENR and EVA. A sharp increase in gel fraction was observed as the irradiation dose was increase from 50 to 100kGy. It can be concluded that an increase in irradiation doses will increase the percentage of gel fraction in all of the blends. The enhancement in the gel fraction with the addition of TMPTA and TPGDA ionomer indicates that those additives accelerate the radiation-induced crosslinking in blend. However, TPGDA ionomer affect crosslinking at relatively lower extent compared to TMPTA because TMPTA is a tri-functional monomer while TPGDA is di-functional monomer that make TMPTA have higher degree of crosslink.

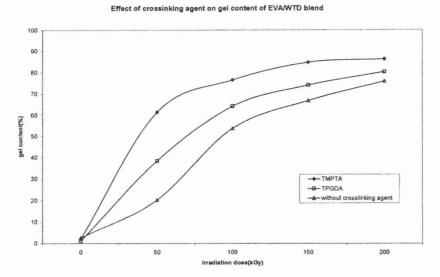


Figure 4: Effect of Crosslinking Agent on Gel Content of EVA/WTD Blend.

#### **Dynamic Mechanical Analysis**

Miscibility or compatibility in a polymer system is the key to the physical and mechanical properties of blends. Hence, dynamic mechanical analysis was done to determine the transition behavior and compatibility of the EVA/WTD blend. The change in storage modulus and tan delta ( $\tan\delta$ ) of EWC, EWT and EWP blend is presented in Fig. 5 and 6. The temperature at maximum  $\tan\delta$  peaks is taken as the glass transition temperature ( $T_g$ ). Fig. 5 shows that  $T_g$  shift slightly to lower temperature when additive was added due to the lubricating effect of the monomer. This can be supported by the tensile test that shows at 0kGy, EWT and EWP gives lower value compared to EWC. Fig. 5 also shows two peaks of  $\tan\delta$ . It indicates that WTD is immiscible in EVA. In fig. 6, storage modulus for EWT and EWP shows almost the same trend as EWC because TMPTA and TPGDA is a low molecular monomer.

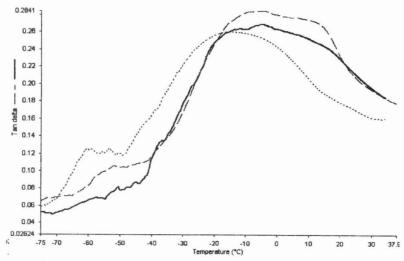


Figure 5: Temperature Dependence of Tano of 90/10 EVA/WTD Blend at 0 kGy.

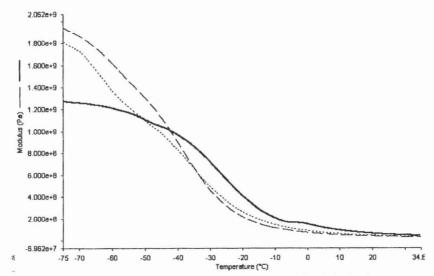


Figure 6: Temperature Dependence of Storage Modulus of 90/10 EVA/WTD Blend at 0 kGy.

#### Conclusion

It was observed that irradiation-induced crosslinking occurs in the EVA/WTD blend and it improved the tensile strength of EVA/WTD blend. Addition of TMPTA and TPGDA has significantly enhanced the irradiaton-induced crosslinking in the blend.

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