

Mechanical Characteristics of Dynamically Vulcanized and Compatibilized Polyoxymethylene/Ethylene Vinylacetate Blends

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Abstract— Dynamically vulcanized blends of polyoxymethylene (POM) and ethylene vinyl acetate (EVA) with and without compatibilizer were prepared by melt mixing in a twin screw extruder. Maleic anhydride (MAH) grafted EVA (EVA-g-MAH) has been used as a compatibilizer. Dicumyl peroxide was used for vulcanizing the elastomer phase in the blends. Mechanical and dynamical mechanical properties of the blend systems have been investigated as a function of blend composition and compatibilizer content. The impact strength of both dynamically vulcanized blends and compatibilized/dynamically vulcanized blends increases with increase in elastomer content with decrease in tensile strength. Dynamic mechanical analysis shows decrease in $\tan\delta$ values as the elastomer and compatibilizer content increased.

Index Terms—Dynamic vulcanization, compatibilization POM / EVA blends.

I. INTRODUCTION

Polyoxymethylene (POM) is used for a variety of industrial applications because of its high strength, stiffness and excellent chemical resistance. However its low impact strength limits its application. Generally toughening of such resins can be carried out by blending with low modulus elastomers. Dynamic vulcanization may be carried out in the case of plastics/rubber blend system to enhance their processing characteristics.[1,2]. Dynamically vulcanized blends have a judicial control of morphology accomplished by choice of mixing methods, rheological properties, control of surface energy and chemical reaction during mixing have resulted in production of high quality materials.[3] Francesco Pilati studied the characteristics of poly (ethyleneterephthalate) with 15 wt % EVA.[4] Thomas reported the tensile impact strength of blends of polypropylene and ethylene vinyl acetate copolymer with special reference to the effect of blend ratios. It was observed that the impact strength reached a maximum at 70 wt % EVA content.[5] Gupta et al [6] investigated the potential of ethylene vinyl acetate copolymer (EVA) as an impact modifier for isotactic polypropylene (PP). They reported the properties of PP / EVA blend in the range 0-40 wt % EVA.

Three grades of EVA containing 9, 12, and 19 wt % vinyl acetate content were used. Ray et al [7] deduced a correlation between morphology, dynamic mechanical, thermal, physiomechanical properties and electrical conductivities of EVA / LDPE blends. They showed that an interpenetrating polymer network (IPN) was formed with a minimum of 50 wt % EVA in the blend. Maciel et al [8] prepared polypropylene / ethylene vinyl acetate (PP / EVA) copolymers with compositions ranging from 90 / 10 to 10 / 90. The results showed a wide range of spatial structures which correlated well with the corresponding measurements of elastic modulus of the blends. Soares et al [9] investigated the influence of poly [(ethylene-co-vinyl acetate)-g-polystyrene] (EVA-g-PS) on the mechanical and morphological properties of polystyrene and PS / EVA blends. The addition of the graft copolymer enhanced the mechanical properties and impact resistance of the PS matrix and PS / EVA (90: 10 wt %) blends. Legros et al [10] studied the insitu compatibilization of poly(ethyleneterephthalate) (PET) copolymer with ethylene vinyl acetate (EVA) copolymer via catalysed transesterification reactions and reported the rheological, morphological and mechanical properties. Jafari et al [11] correlated the impact strength and dynamic mechanical properties of elastomer modified polypropylene. EPDM and EVA were used as an impact modifier for PP. EPDM was better than EVA as an impact modifier for PP. Ramirez Vargas et al [12] studied the morphological and mechanical properties of polypropylene [PP] / poly (ethylene vinyl acetate) [EVA] blends. They reported enhancement of both elongation at break and impact strength. Seon et al [13] studied the reactive compatibilization of poly (butylene terephthalate) (PBT) and ethylene vinyl acetate copolymer (EVA) blends with maleic anhydride (MAH). The impact strength of PBT / EVA-g-MAH (80/20) blend showed about three fold increase in comparison with PBT / EVA (80/20) blend due to the enhanced interfacial adhesion by the formation of insitu compatibilizer. Peon et al [14] studied the rheological behavior of LDPE / EVA blends with respect to the vinyl acetate content of EVA.

Xiaodong Wang et al [15] investigated nylon 6 and ethylene vinyl acetate copolymer (EVA) blends compatibilized with an ethylene acrylic acid copolymer (EAA). They reported that neither EVA nor EAA are compatible with nylon 6. However, the combination of the two resulted in toughened nylon 6. The compatibilization was revealed by the dramatic increase in impact strength, the smaller particle size and finer dispersion of EVA in the nylon 6 matrixes in the presence of EAA. Bhattacharyya et al [16] reported the mechanical properties of nylon 6 and ethylene vinyl acetate (EVA) blends with composition from 0 to 50 wt % EVA. The notched izod impact strength of nylon 6 increased with the incorporation of EVA. The increase was more than 100 % in blends which contained 10 % EVA. The tensile strength and tensile modulus of the blends decreased steadily as the weight percent of EVA increased. Tang et al [17] reported the influence of polarity of EVA copolymers on the morphology and mechanical properties of their uncompatibilized blends with polystyrene. Moly et al [18] reported the melt rheological properties of linear low density polyethylene (LLDPE) / ethylene vinyl acetate (EVA) blends. The blends were investigated with special reference to the blend ratio, temperature, shear rate, compatibilization and dynamic vulcanisation. Bhattacharyya et al [19-20] studied the compatibilization of PA6 / EVA blends with the addition of an ionomer. The effect of compatibilization on the mechanical properties and morphology were reported as a function of ionomer concentration. Ana Cristina et al [21] reported the influence of the processing temperature on the co-continuous morphologies of polystyrene / ethylene vinyl acetate blends. Ramirez Vargas et al [22] studied the degradation effects on rheological and mechanical properties of multi-extruded blends of impact modified polypropylene and poly(ethylene-co-vinyl acetate).

Bhattacharyya et al [23] studied the mechanical properties and morphological characteristics of compatibilized blends of PA6 / EVA-g-MA and PA6 / EVA / EVA-g-MA as functions of maleic anhydride content (MA) and dispersed phase (EVA-g-MA) concentrations respectively at blending composition of 20 wt % dispersed phase (EVA-g-MA or combination of EVA and EVA-g-MA). It was observed from the phase morphological analysis that the average domain size of the PA6 / EVA-g-MA blends decreased gradually as the MA content of EVA-g-MA increased. A similar decrease was also observed in PA6 / EVA / EVA-g-MA blends with increase in EVA-g-MA content which suggested the coalescence process was slower in presence of EVA-g-MA.

Moly et al [24] studied the nonisothermal crystallisation, melting behavior and wide angle X-ray scattering of linear low density polyethylene (LLDPE)/ethylene vinyl acetate (EVA) blends and reported the effects of compatibilization and dynamic crosslinking.

In the present work an attempt has been made to improve the miscibility and properties of POM / EVA blends systems by dynamic vulcanization and compatibilization. By this method it may be possible to develop POM / EVA blends with the rubber phase finely dispersed in the polyacetal matrix. The mechanical and dynamic mechanical, properties of the blend systems are proposed to be investigated as a function of blend composition and compatibilizer content.

II. EXPERIMENTAL

Materials

Polyoxymethylene with trade name Celcon M90, a medium viscosity copolymer providing optimum performance in general purpose injection molding and extrusion with melt flow rate 8.6 g/10 min was supplied by Polyplastics, USA. Ethylene vinyl acetate (EVA) with Vinyl acetate % by wt. 18.0 and Melt index 2.5g/10min is a copolymer of ethylene and vinyl acetate with trade name Elvax 460 supplied by Dow Elastomers. Dicumyl peroxide(DCP) 98% active was obtained from Himedia Laboratories Ltd.,India. A commercial grade maleic anhydride (MAH) 99 % pure was used for the preparation of maleic anhydride grafted ethylene propylene diene terpolymer.

Methods

Grafting of MAH on EVA

The EVA was grafted with maleic anhydride by melt mixing using dicumyl peroxide as initiator in a laboratory model co-rotating twin-screw extruder (Berstorff ZE 25). The EVA granules were mixed with 2 % maleic anhydride containing 0.02 % dicumyl peroxide. Then the treated EVA was introduced into the extruder .The grafting reaction was carried out at 180 °C for 4 minutes with a screw rotational speed of 60 rpm. The extrudate was cooled and passed through a pelletizer and cut into granules. The EVA-g-MAH granules were dried before the preparation of blends [25].

Preparation of Blends

Dynamically vulcanized blends of POM and EVA were prepared with various weight ratios of the constituents in the presence of 0.1 % DCP using a co-rotating twin screw extruder (Berstorff ZE 25) with temperature range 140 °C to 190 °C and screw rotational speed of 60 rpm.

The samples were passed through a cooling bath and pelletizing was subsequently carried out. The blends were further dried at 80 °C before injection moulding the test specimens for the measurements of mechanical properties. Based on the optimization of mechanical properties for the above dynamically vulcanized blends, the ratio of plastic / rubber was kept constant (90/10) and the compatibilized /dynamically vulcanized blends were prepared by similar method using various weight ratios of EVA-g-MAH as compatibilizer. The composition of the various blends prepared is presented in Table 1.

Fourier transform infrared spectroscopy (FTIR) measurements were performed using Nicolet AZ006 FTIR spectrometer. Thin films of the EVA and EVA-g-MAH were prepared by electrically heated compression moulding machine.

Table :1
Composition of POM /EVA blends

Composition*	POM (wt %)	EVA (wt%)	EVA-g-MAH (phr)
PEV ₀	100	0	-
PEV ₅ D	95	5	-
PEV ₁₀ D	90	10	-
PEV ₁₅ D	85	15	-
PEV ₂₀ D	80	20	-
PEV ₁₀ C _{2.5} D	90	10	2.5
PEV ₁₀ C _{5.0} D	90	10	5.0
PEV ₁₀ C _{7.5} D	90	10	7.5
PEV ₁₀ C _{10.0} D	90	10	10.0

*P-POM; EV-EVA; D-dynamically vulcanized; C-compatibilizer EVA-g-MAH; DCP 0.1% of the total elastomer content.

The tensile test specimens were prepared by injection moulding using a reciprocating screw injection machine (L & T 65 ton). The tensile properties were determined with an Instron Universal Testing Machine (Instron 4467) according to ASTM D638. All the tests were done at room temperature, using a cross head speed of 50 mm / min and average values of five measurements are reported. Impact specimens conforming to ASTM D256 were used to measure the notched izod impact strength using a Ceast impact testing machine at room temperature. The average values of five measurements are reported.

DMA measurements were carried out using NETSCH DMA 242 analyzer at a heating rate of 2 °C / min at 10 Hz from -140 °C to 180 °C. The dynamic mechanical loss tangent were determined for all the blend systems.

III. RESULTS AND DISCUSSION

FTIR spectroscopy

The characteristic infrared bands of EVA and EVA-g-MAH are shown in Fig. 1. The absorption bands at 2923 cm⁻¹ and 2852 cm⁻¹ are due to the presence of C-H stretching of the ethylene groups. The C=O stretching around 1738 cm⁻¹ is seen in both EVA and EVA-g-MAH. It is not easy to identify the characteristic peaks of MAH which are grafted onto EVA, because MAH and EVA possess the same C=O group [26]. The C-O stretching is observed at 1241cm⁻¹. The only assumption for the grafting reaction is that disappearance of the C=C absorptions of maleic anhydride which may be involved in the grafting reaction.

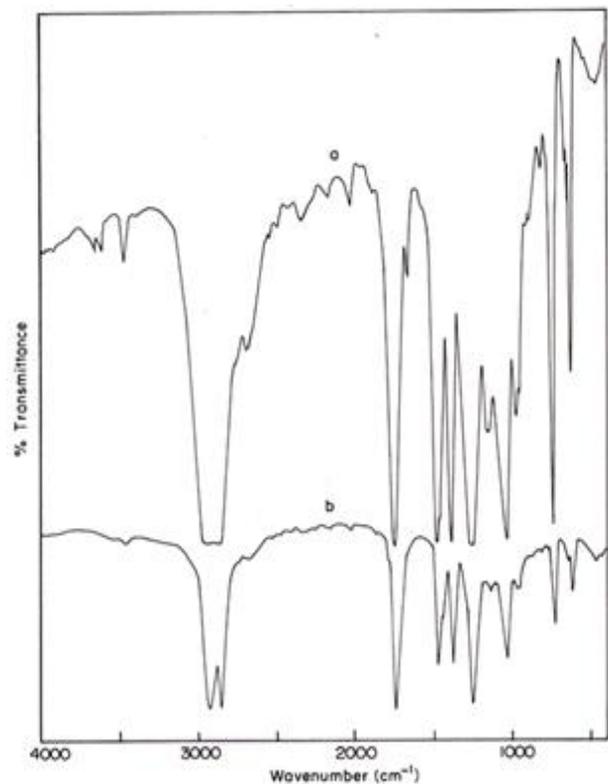


Fig.1 FTIR spectra of (a) EVA and (b) EVA-g-MAH

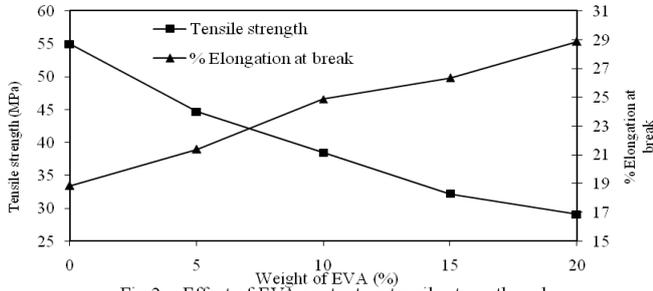


Fig. 2. Effect of EVA content on tensile strength and % elongation at break of dynamically vulcanized POM / EVA blends

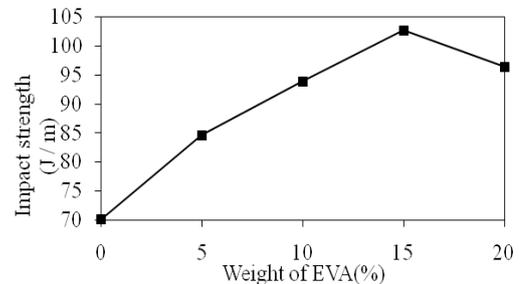


Fig. 4. Effect of EVA content on impact strength of dynamically vulcanized POM / EVA blends

Mechanical Properties

The mechanical properties like tensile strength, modulus, % elongation at break and notched izod impact strength values of dynamically vulcanized blend systems are shown in fig.2-4.

As shown in fig.2, the tensile strength of dynamically vulcanized blends decreases with increasing elastomer content. This decrease in tensile strength is due to the presence of elastomer phase in the POM matrix. However, the % elongation at break increases when the elastomer content in increased in the blend which may be due to improved interaction between the blend constituents. The variation in tensile modulus as function of EVA content for dynamically vulcanized blends is shown in fig.3. It is observed that increasing elastomer content reduces the values of tensile modulus. Elastomers possess low modulus when compared to plastics hence it decreases the modulus of the blends. This shows that dynamic vulcanization reduces interfacial tension between POM and EVA .

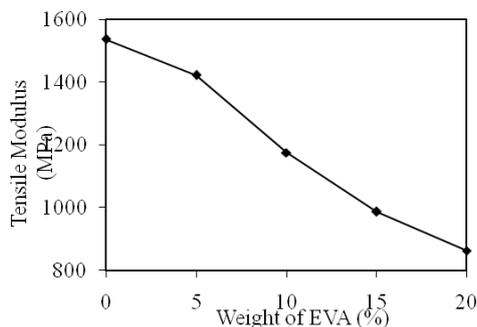


Fig. 3. Effect of EVA content on tensile modulus of dynamically vulcanized POM / EVA blends

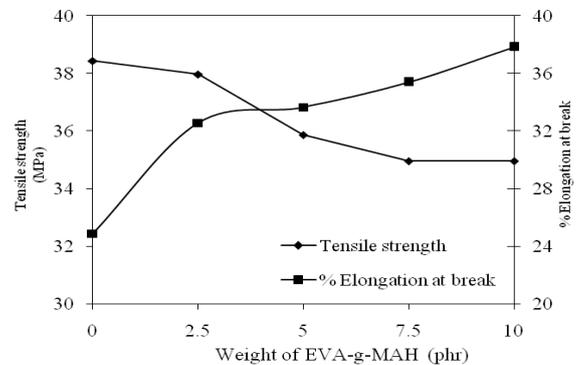


Fig. 5 Effect of EVA-g-MAH on tensile strength and % elongation at break of compatibilized / dynamically vulcanized POM / EVA blends

The variation of impact strength with the elastomer content is shown in fig.4. The impact strength depends on the dispersed phase particle size, the rubbery material being used and the adhesion between the particles and the matrix. The notched izod impact strength of dynamically vulcanized blends increases gradually up to 15 % EVA content. However increased elastomer content beyond 15 % causes a marked decrease in impact strength due to the increase in particle size of the elastomer phase and coalescence of the rubber particles. Dynamic vulcanization enhances the compatibility between the component polymers up to 15 %.

The effect of EVA-g-MAH compatibilizer on the tensile strength, % elongation at break, tensile modulus and izod impact strength of dynamically vulcanized POM / EVA (90 / 10) blend are presented in figs.5-7. From fig.5, the value of tensile strength decreased with the increasing addition of compatibilizer.

This is due to the elastomeric nature of the compatibilizer. The increasing content of EVA-g-MAH causes marked increase in % elongation because it imparts flexibility. This increase in percentage elongation is attributed to the improved adhesion of the component polymers in the presence of EVA-g-MAH.

As shown in fig 6, compatibilized blends possess low values of tensile modulus compared to uncompatibilized blends. The values of tensile modulus decreases as the content of EVA-g-MAH increased. This implies that the presence of compatibilizer enhances the elastomeric characteristics in POM / EVA blends.

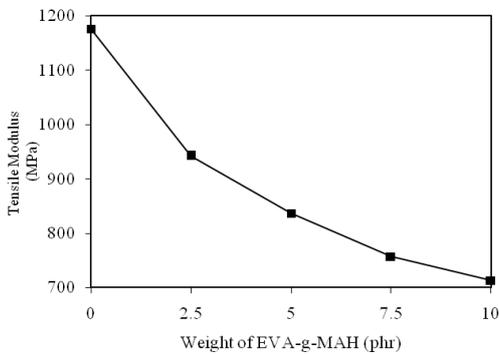


Fig. 6 Effect of EVA-g-MAH on the tensile modulus of compatibilized dynamically vulcanized POM / EVA blends

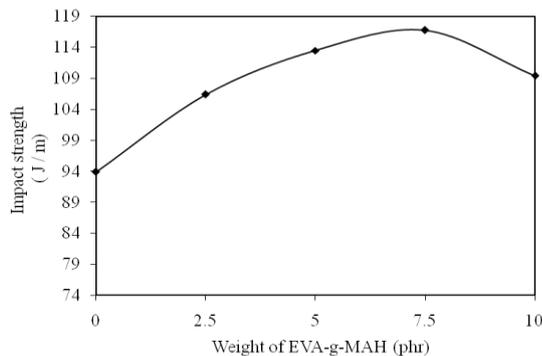


Fig. 7 Effect of EVA-g-MAH on impact strength of compatibilized / dynamically vulcanized POM / EVA blends

As shown in fig.7, the addition of EVA-g-MAH as compatibilizer to a 90 / 10 POM / EVA system significantly increases the notched izod impact strength when compared to dynamic vulcanization. This may be explained by the fact that the compatibilizer forms an interface between the two components, enhancing the miscibility of POM and EVA and thus significantly improving the impact strength.

Dynamic Mechanical Analysis (DMA)

DMA is often used to study polymer / polymer miscibility in polymer blends. The glass transition region can be studied using loss factor curves. The results of dynamic mechanical analysis add information about the behavior of the blends and phase morphology. For an incompatible blend the $\tan \delta$ vs. temperature curve shows the presence of two $\tan \delta$ peaks corresponding to the glass transition temperature of the individual polymers whereas in a miscible blend only a single peak that is located in between the transition temperature of the components of the blend is observed. In the case of partially compatibilized polymer blends two separate peaks corresponding to the individual polymer components are also observed but with their position shifted to a higher or lower temperature depending on the blend composition and the influence of their microstructure [27-30]. The DMA scans illustrating the temperature dependence of $\tan \delta$ of the dynamically vulcanized blends and compatibilized dynamically vulcanized blends are shown in fig.8 and 9.

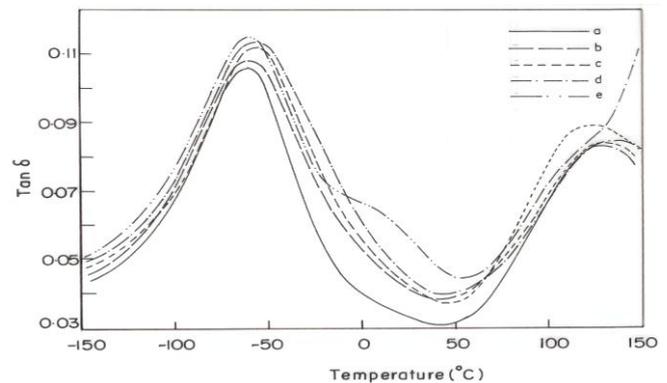


Fig.8 Variation of $\tan \delta$ as a function of temperature for dynamically vulcanized POM / EVA blends (a) PEV₀ (b) PEV₅ D (c) PEV₁₀ D (d) PEV₁₅D (e) PEV₂₀D

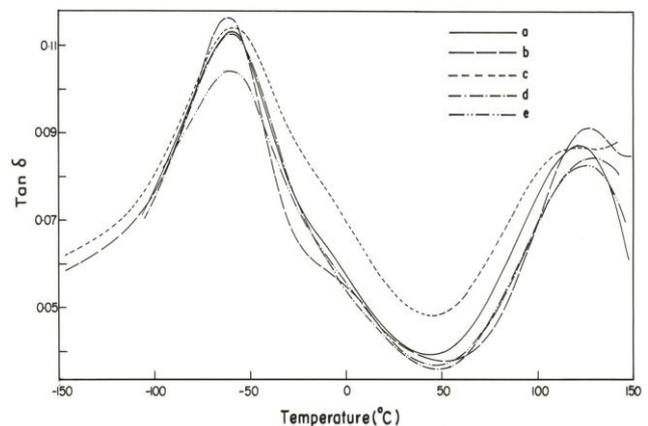


Fig.9 Variation of $\tan \delta$ as a function of temperature for compatibilised / dynamically vulcanised POM / EVA blends (a) PEV₁₀ D (b) PEV₁₀C_{2.5} D (c) PEV₁₀C_{5.0} D (d) PEV₁₀C_{7.5} D (e) PEV₁₀C₁₀ D

As shown in fig 8, the $\tan \delta$ curve of POM shows two prominent peaks similar to the relaxations of POM which have already been reported [31-33]. The α transition at 125 °C is associated with the molecular motion within the crystalline phase. The peak maximum temperature of $\tan \delta$ at -61.3 °C corresponds to the T_g of POM. In case of dynamically vulcanized blends, the peak maximum temperature of $\tan \delta$ increases upward when the elastomer content is increased. This indicates that the EVA is crosslinked and the interaction between EVA and POM has increased. Byrne and Hourston investigated the dynamic mechanical behavior of EVA / PP and reported that $\tan \delta$ is increased by crosslinking which is ascribed to the decrease in the degree of crystallinity of the blends [34, 35]. Furthermore the T_g of POM is shifted towards T_g of EVA which is around -53 °C according to the supplier specification. The shifting of T_g to higher value is pronounced with increasing content of EVA. This indicates that dynamic vulcanization improves the adhesion between the constituents of blends.

The $\tan \delta$ curves obtained for the blends with varying content of EVA-g-MAH are compiled in fig 9. The inclusion of EVA-g-MAH as a compatibilizer increases the height of $\tan \delta$ curves. Further, a marked shift in the T_g to higher value is observed when the addition of EVA-g-MAH is increased. The blend incorporating 7.5 phr of EVA-g-MAH (PEV₁₀C_{7.5}D) shows maximum shift in T_g value owing to the better interaction between POM and EVA. However, the addition of EVA-g-MAH beyond 7.5 phr does not increase the $\tan \delta$ value. This indicates that the optimum concentration of EVA-g-MAH required to achieve maximum compatibility is 7.5 phr. The EVA-g-MAH content above 7.5 phr does not contribute towards the interaction of the blend components. The $\tan \delta$ curves for the blends in all composition ranges are located above the $\tan \delta$ curve for uncompatibilized blend and peaks are broader when compared to peak for uncompatibilized blend. This rise in height and broadening of $\tan \delta$ peaks clearly indicates that EVA-g-MAH significantly improves the compatibility of the polymers by reducing interfacial tension between POM and EVA. This shifting of T_g to higher value confirms that EVA-g-MAH played a role in bridging the matrix and dispersed phase. The improved interaction between the component polymers leads to a shift in T_g to higher value.

IV. CONCLUSION

The grafting of EVA was carried out using maleic anhydride in the presence of dicumyl peroxide in a twin screw extruder.

In the case of dynamically vulcanized POM / EVA blends increasing content of elastomer up to 15-wt % results in significant improvement in izod impact strength and morphology of the blend system. In the case of compatibilized dynamically vulcanized blends of POM / EVA a marked increase in impact strength and improvement in thermal properties is obtained. The increase of impact strength of the blends indicates improved interaction between the components in the presence of EVA-g-MAH, which resides at the interface and reduces the interfacial tension in the melt and results in finer phase domains. Therefore this enhanced interfacial adhesion would lead to better mechanical properties. The compatibilization of POM and EVA blends has been achieved by means of dynamic vulcanization and incorporation of EVA-g-MAH as compatibilizer.

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