

Melt free-radical grafting of glycidyl methacrylate (GMA) onto EPDM backbone and effect of EPDM-g-GMA on the morphology and mechanical properties of PS/EPDM/PA6 ternary blends

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ABSTRACT

Melt free-radical grafting reactions between ethylene-propylene-dieneterpolymer (EPDM) and glycidyl methacrylate (GMA) were investigated in a batch mixer (170°C, 60rpm). Effect of dicumylperoxide (DCP) initiator and GMA functionalizing monomer concentrations was studied on the grafted EPDM characteristics. Titration results indicated an increase in the graft degree (GD) and gel content (GC) values with increasing DCP concentration as a result of increasing primary free radical concentration and strengthening cross-linking side reaction. FTIR spectrums confirmed that GMA functionalities have been grafted onto EPDM with appearing carbonyl (C=O) peak. After that, the resultant EPDM-g-GMA was used as compatibilizer in PS(polystyrene)/EPDM/PA6 (polyamide6) ternary blends. The effect of rubbery compatibilizer on the blend morphology and mechanical properties was studied. The ATR-FTIR spectra of ternary blends, etched to remove unreacted PA6, demonstrated that the compatibilizing reactions occurred during melt blending. By investigating the SEM micrographs it was revealed that the EPDM-g-GMA compatibilizer at the concentration range of 5 wt.% to 15 wt.% changed the size and type of the blend morphology from separated dispersed to multicore-shell morphology. The finest morphology was achieved by using 7.5 wt.% EPDM-g-GMA. Also, the presence of compatibilizer up to 7.5 wt.% could improve the tensile modulus, yield stress and impact strength, but a decreasing trend was observed at higher concentration of the compatibilizer. **Polyolefins J (2021) 8: 1-9**

Keywords: Functionalizing; glycidyl methacrylate; polystyrene/ethylene-propylene-dieneterpolymer/polyamide6; microstructure; impact strength.

INTRODUCTION

The inert nature of polymers is a defect for applications in which interaction with polar components is required such as polymer blends [1]. Functionalizing is a common method to obviate the mentioned problem. The functionalization of polymers is often done through free-radical grafting of functionalizing monomers onto the polymer backbone in the presence of an initiator as a free-radical generator. In the free-radical grafting method, the usual used functionalizing monomers are maleic anhydride (MAH) [2-4] and

glycidyl methacrylate (GMA) [5-8]. Dicumyl peroxide (DCP) [4, 9, 10] and benzoyl peroxide (BPO) [5, 11] commonly are used as an initiator in the functionalizing systems.

Addition of suitable block copolymer as a compatibilizer [12] using ultrasonic extrusion [13], cross-linking between phases [14] using a graft copolymer (modified polyolefins) as a reactive compatibilizer [15] are suggested for this purpose in literature. As a compatibilizer in polymer blends, modified polyolefins

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containing epoxy functions e.g. GMA have the potential tendency to react with many functional groups such as hydroxyl, amine and carboxyl [16, 17]. There are several studies assaying the factors affecting the free-radical functionalization reaction of some polyolefins such as high-density polyethylene [7, 18], low-density polyethylene [19], polypropylene [20], linear low-density polyethylene [21] and ethylene-propylene rubber [6, 22]; whereas free radical grafting onto EPDM has rarely been studied.

Polystyrene (PS) is a thermoplastic which is easily synthesized and processed, but it performs so brittle at ambient temperature. Synthesis of high-impact PSs (HIPS) has been recommended as a primary solution, but HIPS has a low resistance against natural light radiation due to the probable photodegradation of polybutadiene (PB) segments [23]. In some researches, PB is replaced by EPDM and the PS/EPDM binary blends have been studied [12, 13]. Despite the improvements achieved in the impact strength, undesirable falls would be observed in Young's modulus and stiffness of the material. PA6 was innovatively added to PS/EPDM binary blend as the third stiff phase to improve Young's modulus and stiffness in a previous paper [17]. Also, the prepared ternary blend (PS/EPDM/PA6) was compatibilized by GMA functionalizing monomer and DCP direct injection during blending. In this study, free-radical grafting onto EPDM backbone at different DCP initiator and GMA functionalizing monomer concentrations was studied. Then the functionalized EPDM was used as the compatibilizer of PS/EPDM/PA6 ternary blends and the blend morphology alterations and mechanical properties against compatibilizer content were investigated.

EXPERIMENTAL SECTION

Materials used in this research were: ethylene-propylene-diene terpolymer (EPDM, Kep270) supplied by Kumho Polychem (Korea), Mooney viscosity ML(1+4) at 125°C 71 M, ethylene content 57%, termonomer content 4.5 ENB%; general purpose polystyrene (GPPS, Solarene® G-144) supplied by Hyundai Engineering Plastics (Korea), MFI 8.5 g/10min at 200°C and 5kg; polyamide 6 (PA6, Ultramid® B3S) supplied by BASF (Germany), MFI 197.75 g/10min at 275°C and 5kg; glycidyl methacrylate monomer (GMA, 97%, Aldrich

Chemical Co., Japan); dicumyl peroxide (DCP, 98%, Merck Millipore, Germany). Xylene, formic acid, acetone, ethanol, trichloroacetic acid (TCA), phenolphthalein and potassium hydroxide used for titration and purification were all of analytical grade from Merck Millipore.

Grafting GMA Functionalizing Monomer onto EPDM

Melt grafting reactions were conducted using a 60 mL internal mixer (Brabender W50, Duisburg, Germany) preheated to 170°C. Regarding the volatility of GMA functionalizing monomer at the processing temperature, twenty minutes prior to the mixing, GMA functionalizing monomer and DCP initiator were physically poured on EPDM in a closed container. Impregnated EPDM was then melt mixed for 8 min at 60 rpm. Finally, every sample was immersed in the cold water to stop reactions. All EPDM-g-GMA samples were prepared according to Table 1.

Blend Preparation

All ternary blend samples were prepared at 220°C in an internal batch-mixer (Brabender GmbH, Germany) with rotor speed of 120 rpm for overall mixing time. Before mixing, all components were dried for 24 h at 80°C to remove moisture. All components were blended together using the internal mixer for 10 min then immersed in the cold water in order to freeze microstructure. All prepared samples are listed in Table 2.

Purification and Titration of EPDM-g-GMA

Gel content (GC) and graft degree (GD) were measured according to a published research by Saeb et al. [7]. EPDM-g-GMA sample was put in boiling xylene for 1 h. The remaining solid filtrate was dried in a vacuum oven at 80°C. Gel content was then calculated according to Equation (1):

$$GC = \frac{w_1}{w_0} \times 100 \quad (1)$$

Table 1. Melt free-radical grafting components.

Sample Code	DCP, phr	GMA, phr
Blank1	0	5
C1	0.5	5
C2	0.3	5
Blank2	0	3
C3	0.3	3
C4	0.15	3

Table 2. Ternary blends components.

Sample Code	PS (%wt.)	EPDM (%wt.)	EPDM-g-GMA (%wt.)	PA6 (%wt.)
B0	70	15	0	15
B5	70	10	5	15
B7.5	70	7.5	7.5	15
B10	70	5	10	15
B15	70	0	15	15

Where, w_0 and w_f are the sample weights before and after xylene solution, respectively.

The filtrate was stirred with acetone when the solution became opaque. To eliminate un-reacted monomers, the opaque solution was washed with acetone after filtration. The residue is, in fact, purified EPDM-g-GMA.

Pure EPDM-g-GMA was weighed and solved in boiling xylene, and TCA (0.3M) was added to the solution before putting in 90°C oven where the reaction between GMA epoxy rings and TCA would progress to complete. Reacted EPDM-g-GMA was then settled by introducing acetone/ethanol (1:1). The filtrate was titrated using 0.1M KOH. Graft degree was calculated according to Equation (2):

$$GD = \frac{142.15 \times (C_0 V_0 - C_2 V_2)}{w} \times 100 \quad (2)$$

Where, C_0 and C_2 are the TCA and KOH standard solution concentrations, respectively. V_0 and V_2 are the TCA standard solution volume and KOH standard solution volume used through titration, respectively. w indicates the initial sample weight.

FTIR and ATR-FTIR Analysis

Purified EPDM-g-GMA samples were solved in boiling xylene. Then a Fourier transform infrared (FTIR) spectroscope (Bomem-102, Canada) was used for prepared solutions to track the expected reactions and estimate a grafting degree criterion.

Samples B5, B7.5 and B15 were typically characterized with an attenuated total reflection-Fourier transform infrared (ATR-FTIR) spectroscope (Bruker Vertex 70, United states). Moreover, each sample was also analyzed after keeping immersed in formic acid for one week to remove un-reacted PA6. ATR-FTIR analysis was used to confirm the compatibilizing reaction during melt blending.

Morphological Study

A Philips (XL30, Netherlands) scanning electron

microscope (SEM) was used to observe the phase morphology of ternary blends. Fracture surface of impact tests was etched by n-heptane to remove EPDM phase. Etched surfaces were then coated with a thin layer of gold before SEM imaging.

Mechanical Properties

Standard ternary blend specimens for tensile and impact strength tests were machined from sheets compression-molded at 240°C. Tensile tests were conducted by a Galdabini testing machine (Italy) with a crosshead speed of 1 mm/min at room temperature according to ASTM D638. Un-notched Izod impact tests were carried out using a Ueshima impact tester machine (Japan) according to ASTM D4812.

RESULTS AND DISCUSSION

EPDM-g-GMA

EPDM rubber was modified with free-radical grafting GMA functionalizing monomers onto EPDM backbone. In the following, tracking of grafting reaction and grafting degree variation have been investigated by FTIR analysis and titration, respectively.

FTIR Analysis

Figure 1 shows the FTIR spectroscopy of pure EPDM and samples C1-C4. It can be seen in Figure 1, for each sample, some clear peaks have appeared along the related FTIR spectrum.

In fact, the DCP initiator undergoes a homolytic decomposition upon thermo-mechanical heating to yield a pair of primary free radicals [21] that can abstract a tertiary hydrogen from the EPDM chain to form a macromolecular radical [24]. These macroradicals could initiate the GMA functionalizing monomer grafting onto polymer chains with breaking C=C double bonds of GMA functionalizing molecules and emerging C=O carbonyl groups (of GMA functionalizing molecules) onto EPDM backbone or undergo undesirable competing reactions depending on the polymer backbone nature [25]. As seen in Figure 1, a clear and sharp peak has appeared at 1730cm⁻¹ for samples C1, C2, C3 and C4. This main characteristic peak confirms the formation of C=O carbonyl groups during the free-radical grafting reaction of GMA functionalizing monomers onto EPDM chains [26]. The epoxy group, responsible for the compatibilizing fea-

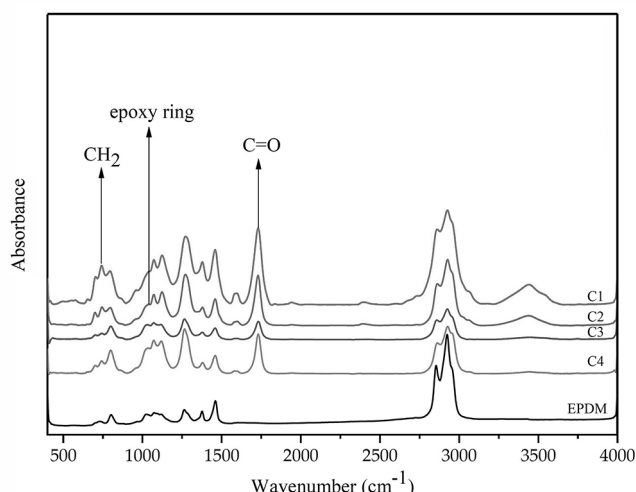


Figure 1. FTIR spectra of pure EPDM and the EPDM-g-GMA samples.

tures of EPDM-g-GMA, was detected as a weak peak (or a shoulder) at 990cm^{-1} . The newly appeared peak at 740cm^{-1} for all samples and pure EPDM has been attributed to stretching CH_2 available in EPDM backbone. In some published researches, the carbonyl to stretching CH_2 peak area (A_{1730}/A_{740}) was used as the graft degree criterion [19, 21, 22]. The calculated amounts of A_{1730}/A_{740} for samples C1, C2, C3, C4 and Blank2 have been reported in Table 3.

The data reported in Table 3 show a drop in grafting degree criterion (A_{1730}/A_{740}) versus decrease in DCP concentration (from C1 to C2 or C3 to C4 and Blank2). It is also clear that as GMA functionalizing monomer concentration is decreased (from C2 to C3) the grafting degree criterion has fallen.

Gel Content and Graft Degree

Figure 2 compares the variations of GMA graft degree and gel content with increasing DCP concentration at fixed GMA content. At both GMA contents studied here (i.e. 3 and 5 phr), a similar increasing trend is observed for GD values versus DCP content. These results are supported by the variations of grafting degree criterion (A_{1730}/A_{740}) represented in Table 3 and calculated based on the FTIR spectrums.

This growth in GD is imputed to the accelerated formation of free-radicals and subsequently macro-radicals along the polymer chain at the presence of

DCP. In fact, the DCP initiator withstands a homolytic decomposition upon thermo-mechanical heating to yield a pair of primary free radicals [21] which can abstract a tertiary hydrogen from the EPDM chain to form a macromolecular radical [24]. These macro-radicals could begin the grafting GMA functionalizing monomers onto polymer chains or undergo unpleasant competing reactions depending on the polymer backbone nature [25]. However, crosslinking might be more contingent to happen as a competing pathway at the presence of higher amounts of free-radical and this would weaken the mentioned increasing trend as clearly observed in Figures 2(a) and 2(b).

Gel content has also increased with DCP content at the same time with the graft degree but the trend is somehow different. At GMA content of 5phr, the increasing rate is almost uniform along all DCP contents studied here; where for C3 and C4 containing 3phr GMA, a greater slope is observed at higher DCP contents when DCP amount changes from 0.15 phr to 0.3 phr since crosslinking competing reactions would be more likely at the presence of higher concentrations of macro-radicals. This would produce more gel-like structures in the sample bulk. At GMA contents of 5 phr, the increasing rate was observed to be unaffected by DCP concentration which might be due to the homo-polymerization of GMA functionalizing molecules besides the crosslinking reactions. In such conditions, a part of the free-radicals present in the reaction media is involved in the GMA homo-polymerization. This would repress the expected crosslinking reactions. Limited solubility of the monomer in the polymer, yielding high local concentration, has also known to be responsible for oligo-GMA grafts [27].

It can be concluded that increasing initiator concentration beneficially affects the improvements observed in gel content values where the grafting degree rate is slightly suppressed. Similar behavior has also been reported by Hu et al. [25] studying the effect of DCP on the reactions grafting GMA functionalizing monomer onto EPR. Analyzing the graft degree and gel content results showed that sample C4 with grafting degree of 0.72 wt.% and gel content of 2.12 wt.% could be selected as the best sample to use as a compatibilizer in polymer blends.

Table 3. Grafting degree criterion calculated based on the FTIR spectrum.

Sample Code	C1	C2	C3	C4	Blank2
A_{1730}/A_{740}	12.01	9.95	7.33	6.91	0

PS/EPDM/PA6 Ternary Blends

In the following, C4 was used as a compatibilizer for PS/EPDM/PA6 ternary blend in the different weight percent (from 0 %wt. to 15 %wt.). Morphology and mechanical properties (impact strength and tensile properties) of prepared ternary compatibilized blends were investigated. Also all SEM micrographs were analyzed by ImageJ analyzer to study the variations of number and particle size.

ATR-FTIR Analysis

Figure 3 shows the ATR-FTIR spectroscopy of typical prepared ternary blends. As seen in Figure 3, for selected ternary blends, some clear peaks have appeared in the related ATR-FTIR spectrum.

As seen in Figure 3, primary and secondary amine groups of PA6 have revealed peaks at $3000\text{-}3100\text{cm}^{-1}$ (a double bond) and 3303cm^{-1} (a single bond), respectively. Unexpectedly, these peaks are not thoroughly disappeared for formic acid etched samples. This would be attributed to the occurrence of a reaction between some amine end groups of PA6 and the epoxy groups of GMAs grafted on the EPDM backbones. It is evident that these reacted PA6 chains are not soluble

in the etching liquid. These observations confirm the compatibilizing reactions during melt blending.

Morphological Study

Figure 4 shows SEM micrographs of etched fracture surface of PS/EPDM/EPDM-g-GMA/PA6 ternary blends prepared according to Table 2. As shown in the corner of Figure 4(a), PA6 and EPDM phases are visible as white cores and black holes in all SEM images, respectively. It is clear that by adding EPDM-g-GMA (C4) as a compatibilizer to PS/EPDM/PA6 ternary blend, morphology has changed dramatically.

The comparison between Figures 4(a) and 4(b) shows an obvious morphological change from separation dispersion (including large rubber droplets) to partial core-shell and a little separated rubber droplets. In fact, epoxy-amine reaction has led to large decrease in the interfacial tension between EPDM and PA6 phases. As a result, PA6 phase is completely removed from PS bulk and is partially surrounded by EPDM shell. It is clear in Figure 4(c) that when the EPDM-g-GMA content has reached 7.5 wt. %, and followed by an increase in the amount of epoxy-amine reaction, the rubber shell has penetrated into the PA6 core and

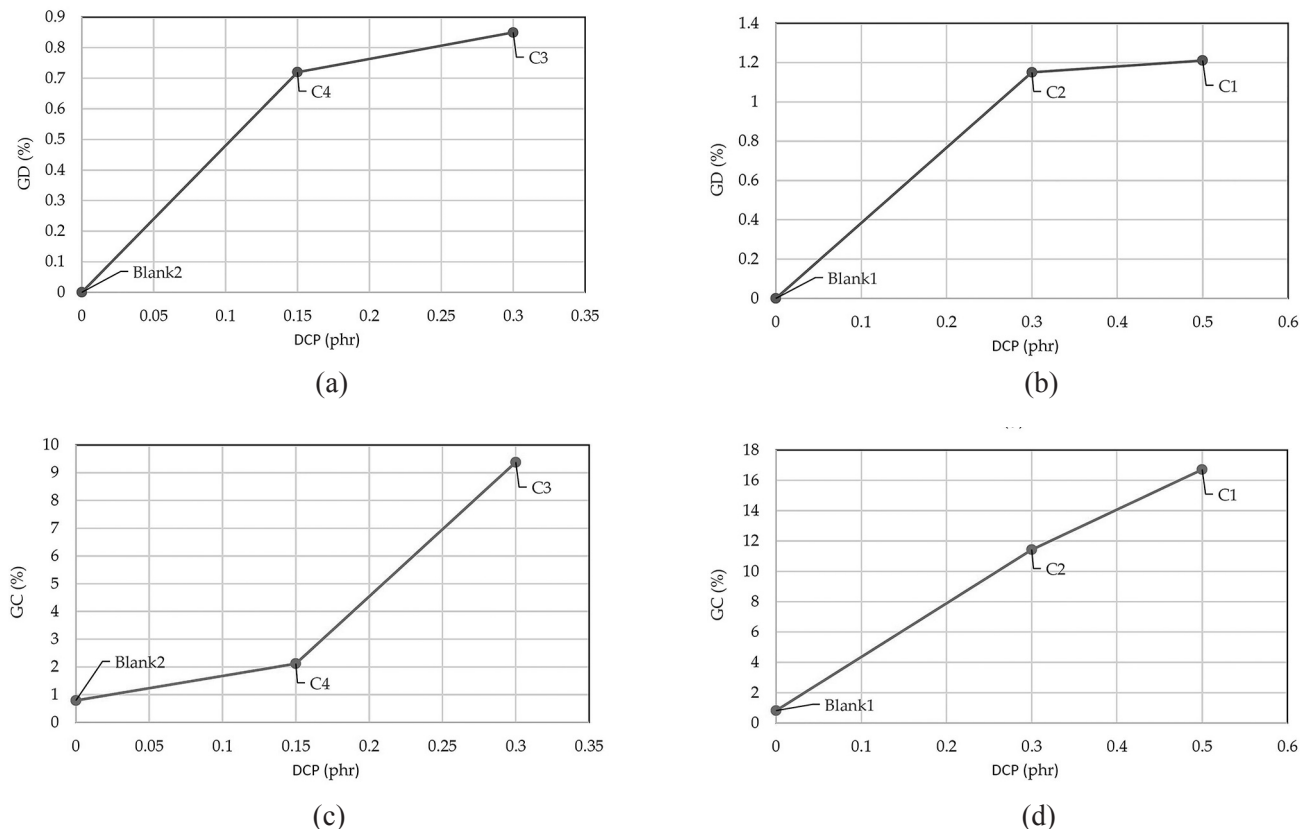


Figure 2. Graft degree and gel content versus DCP concentration at different GMA concentrations: (a) 3phr, (b) 5phr, (c) 3phr and (d) 5phr.

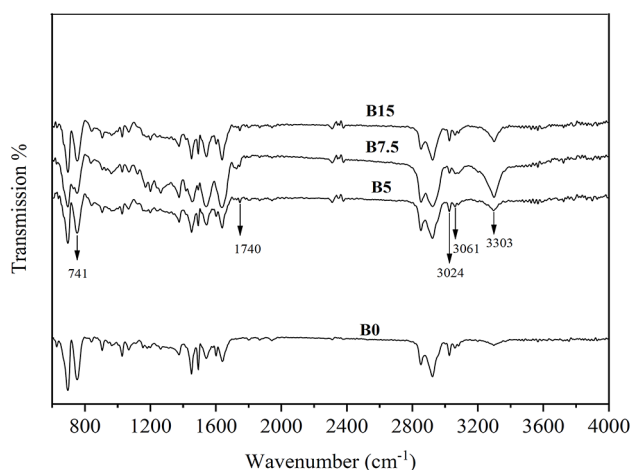


Figure 3. ATR-FTIR spectra of all prepared ternary blends (B0, B5, B7.5 and B15).

created multi-core. This sample (B7.5) has the finest ($0.582 \mu\text{m}$) and the most PA6 cores among all the samples according to ImageJ analyzer. The size of PA6 cores has changed to $0.755 \mu\text{m}$ without any change in the type of morphology with the arrival of EPDM-g-GMA content to 10 wt. % (Figure 4(d)). In fact, by increasing compatibilizer content, rubber shells have tended to coalesce. As a result, PA6 cores and rubber phase interface have dropped and PA6 multi-cores have become larger in size. Finally, it is clear in Figure

4(e) that the heavy coalescence of rubber phase has led to a core-shell morphology including coarse PA6 single cores in 15 wt. % EPDM-g-GMA. So it is worth to note adding compatibilizer has led to change the size and the type of microstructure.

Mechanical Properties

Interactions and reactions done during the melt blending are one of the most effective factors on the blend microstructure and following by mechanical properties. Therefore, in order to continuously investigation this process, the mechanical properties of prepared ternary blends have measured and then reported in Table 4. Also, their stress-strain curves are shown in Figure 5.

As seen in Figure 5, under loading, all specimens have shown the variable yielding behavior and tensile modulus. Among the studied ternary blends, B0 has disclosed the lowest tensile modulus ($1674 \pm 15 \text{ MPa}$) according to the reported data in Table 4. It can be claimed that the coarse, soft and stretched EPDM droplets within the continuous phase have led to low modulus for uncompatibilized ternary blend [28]. According to Table 4, it is clear that B7.5 sample has presented the lowest modulus (1958 MPa) among compatibilized ternary blends (B5, B7.5, B10 and

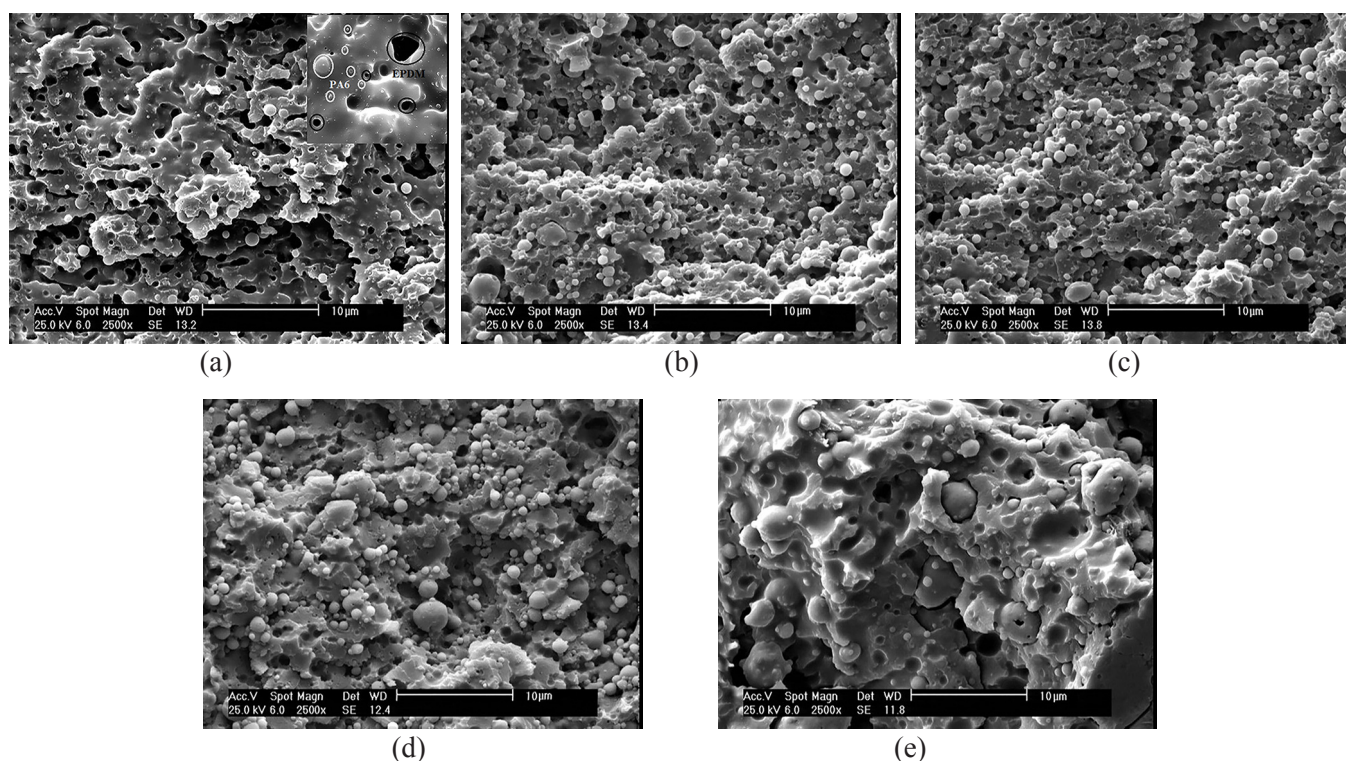


Figure 4. SEM micrographs of etched fracture surface of PS/EPDM/EPDM-g-GMA/PA6 ternary blends: (a) B0, (b) B5, (c) B7.5, (d) B10 and (e) B15.

Table 4. Mechanical properties of the prepared PS/ EPDM/ EPDM-g-GMA/PA6 samples.

Sample Code	Tensile modulus (MPa)	Yield stress (MPa)	Impact strength (J/m)
B0	1674±15	19.4±0.0	7.0±0.6
B5	1986±22	20.1±0.2	14.03±1.0
B7.5	1958±21	23.7±0.4	16.7±0.9
B10	2018±12	23.0±0.7	11.98±1.7
B15	2203±19	16.6±0.5	12.25±1.8

B15). Analyzing Figure 4(c), by ImageJ analyzer, has revealed that B7.5 sample has the most and the finest (0.582 μm) hard PA6 cores. It can be the main reason for a minimize tensile modulus [29].

Uncompatibilized sample B0 has showed the low yield stress. It can be confidently said, the reason of low yield stress for B0 ternary blend (19.4±0.0 MPa) is the weak interfacial adhesion between the components especially between the EPDM phase and both the PS and PA6 phases [28, 30]. The variation of yield stress, with increasing the amount of compatibilizer, is hill like with the peak at B7.5 sample (23.7±0.4 MPa). According to Figure 4(c), B7.5 microstructure does not contain any dispersed rubber particles but has partial core-shells with the finest hard PA6 cores. This microstructure represents the most interfacial adhesion. So the maximum yield stress for B7.5 sample is expected.

According to data reported in Table 4, uncompatibilized ternary blend has represented the minimum impact strength (7.0±0.6 J/m). It is because of the weak interfacial interaction between the blend phases [28]. On the other hand, regarding the microstructure including dispersed hard PA6 particles, with the nega-

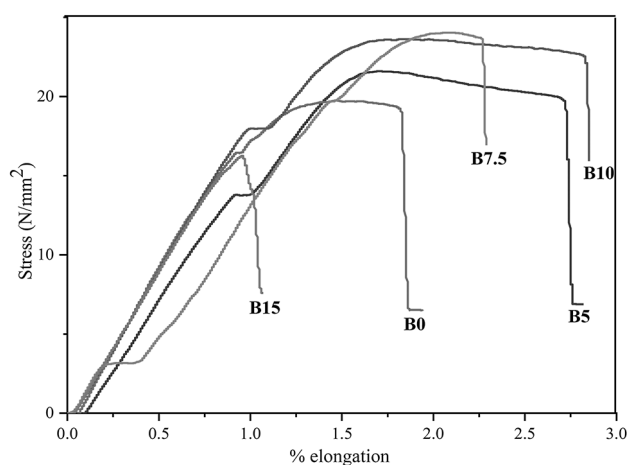


Figure 5. Stress-strain curves of all prepared ternary blends (B0, B5, B7.5, B10 and B15).

tive effect on the impact strength, minimizing impact strength for B0 is expected [29]. The variation of impact strength has been a hill like that shown a peak at B7.5 (16.7±0.9 J/m). Figure 4(c) shows that B7.5 sample does not have any dispersed hard PA6 particles but includes the fine composite droplets containing PA6 multicores with negative and positive effect on the impact strength, respectively. So the maximum impact strength is expected by the morphology mentioned for B7.5 sample. It is clear in the Figure 4 that the composite droplets are too large at high content of compatibilizer especially at 15 wt. %. On the other hand, it is obvious that the larger composite droplets more likely can concentrate stress and certainly decrease the impact strength [29, 31]. So the impact strength drop observed at high content of EPDM-g-GMA is expected.

CONCLUSION

In this study, melt free-radical grafting reactions between ethylene-propylene-dieneter polymer (EPDM) and glycidyl methacrylate (GMA) were investigated in a batch mixer (170°C, 60rpm). Effect of dicumylperoxide (DCP) initiator and GMA functionalizing monomer concentrations was studied on the grafted EPDM characteristics. After that, the EPDM-g-GMA obtained was used as a compatibilizer in the PS (polystyrene)/EPDM/PA6 (polyamide6) ternary blend. The effect of rubbery compatibilizer on the blend morphology and mechanical properties was studied. It is concluded that:

- Titration results indicated an increase in the graft degree (GD) and gel content (GC) values with increasing DCP concentration.
- FTIR spectrums confirmed that the GMA functionalizing monomer has been grafted onto EPDM.
- ATR-FTIR spectrums of etched ternary blends confirmed the compatibilizing reaction during melt blending.
- By investigating SEM micrographs, it can be claimed that the EPDM-g-GMA compatibilizer in the concentration range of 5 wt. % to 15 wt. % changes the size and the type of the blend morphology.
- The finest morphology was achieved by using 7.5 wt. % EPDM-g-GMA.
- Also, the presence of the compatibilizer up to 7.5 wt. % can improve tensile modulus, yield stress

and impact strength, but at higher concentration of compatibilizer these mechanical properties are fallen.

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