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Polyolefins Journal, Vol. 12, No. 1 (2025)
DOI: 10.22063/poj.2025.35539.1323
ORIGINAL PAPER

Photo-aging changes in mechanical properties of high impact polystyrene, polystyrene-based binary and compatibilized ternary blends

Marzieh Alidadi-Shamsabadi[1,*], Shirin Shokoohi[2], Mahnaz Shahzamani[1], Homa Abbasian-Peykani[3]

[1]Chemistry & Chemical Engineering Technical Centre, Academic Centre for Education, Culture and Research (ACECR), Isfahan University of Technology branch, Isfahan, Iran
[2]Chemical, Polymeric and Petrochemical Technology Development Research Division, Research Institute of Petroleum Industry, Tehran, Iran
[3]Chemical Engineering Department, Isfahan University of Technology, Isfahan, Iran

Received: 22 September 2024, Accepted: 11 January 2025

ABSTRACT

In this work, the photo-aging behavior of high impact polystyrene (HIPS), polystyrene/ethylene propylene diene monomer (PS/EPDM) binary blends, and compatibilized polystyrene/ethylene propylene diene monomer/ polyamide 6 (PS/EPDM/PA6) ternary blends was studied and compared together. Photo-degradation of polymer blends faces considerable challenges, because a polymer blend is a compound of multiple components with particular interactions and its components may function as degrading or stabilizing agents. Photo-aging generally can cause changes in the color and mechanical properties of polymer compounds. Attenuated total reflection Fourier transform spectroscopy (ATR-FTIR) was conducted to study the chemical interactions between components in the prepared samples. The morphological structure of blends was studied by using scanning electron microscopy (SEM). The impact and tensile strength of the samples were measured and compared after exposure to UV radiation. To study the changes in the appearance, the yellowness index values of the samples were followed at different periods of exposure to UV irradiation. The post-radiation results showed similar mechanical performance of ternary and binary blends with the retention of mechanical properties close to each other. The impact strength and elongation-at-break for the HIPS sample were greatly reduced compared to the blends, showing their retention by 8.46 and 7.86%, respectively. The ultimate tensile strength retention in each sample is between 70 and 82% and there is no significant difference between them. The final yellowness index of HIPS was measured to be 1.6 and 1.2 times higher than that of the binary and ternary blends, respectively. **Polyolefins J (2024) 12: 45-52**

Keywords: Ternary blend; photo-degradation; photo-aging; high impact polystyrene; UV radiation.

INTRODUCTION

Polystyrene (PS) is a thermoplastic polymer that can be simply synthesized and processed, but its brittle behavior at ambient temperature districts its application in many fields[1]. Developing high-impact polystyrene (HIPS) through dispersion of rubber particles in rigid PS matrix to improve the toughness has far been recommended as a primary solution for this problem [2,3].

HIPS, as a toughened polymer, is usually synthesized through radical polymerization of styrene in the presence of polybutadiene allowing the formation of PS-g-PB copolymers as in situ compatibilizers [4-6]. Despite toughness improvement, HIPS presents limited resistance against natural light radiation due to the probable photo-degradation of polybutadiene (PB) segments [4,7]. Thus, PB could be replaced by saturated or partially-saturated rubbers such as ethylene propylene diene monomer (EPDM). EPDM as a rubber with high resistance to ozone and oxidation has been used to enhance the impact strength of PS. Polyethylene, polypropylene, and other similar materials have also

*Corresponding Author - E-mail: alidadi@acecr.ac.ir

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been suggested for this purpose [2,3]. HIPS would be produced through bulk polymerization of styrene in the presence of EPDM or by a simple blending method. Polymer blends can be readily prepared and are preferred in many industries. However, PS and EPDM are chemically different and hence show thermodynamically-incompatible behavior, resulting in poor mechanical properties raised by the lack of physical/chemical interactions [4,7,8]. Thus, different methods have been studied to increase PS/EPDM binary blends compatibility [1,4,6-10].

Among different possible methods, production of ternary blends is more interestedly focused in the last decade [2,11]. Introduction of a third stiff component like polyamide 6 (PA6) along with a proper compatibilization is expected to balance the mechanical properties of toughened PS binary blends [2, 12]. Improvement of Young's modulus and stiffness with the addition of PA6 was observed in previous studies [6,13].

PB involves different isomers with different degradation stability behaviors, which also affect the PS matrix. Exposure to sunlight significantly decreases impact resistance through the UV-initiated photo-oxidation of the unsaturated rubber phase. This would limit the polymers lifespan when used in outdoor applications. Polymers absorb solar UV radiation, leading to photolysis and photo -oxidation, which suppress the physicomechanical, optical and other properties of the material[14, 15]. On the other hand, it is important to consider the photo-aging properties of plastic products [16], and exposure to sunlight is considered the main cause of plastic aging, as it triggers polymer chains reactions as well as chain scission. Also, color change in polymers is typically caused by photo-aging[17,18]. In the outdoor environment, the most important reason for the degradation is UV radiation providing dissociation energy of the chemical bonds [19].

Functional changes undergone by polymer blends when exposed to UV radiation has rarely been investigated. Polymer blends photo-degradation faces considerable challenges, as a polymer blend is a compound of two or three discrete components with particular interactions, and its components may function as degrading or stabilizing agents. Comprehending the photo-degradation mechanism of a developed polymeric blend with a main matrix, two dispersed phases, and a compatibilizing component could be complex [20]. Due to HIPS's poor resistance to UV radiation, it is essential to replace PB, the main cause of this weakness, with a saturated or semi-saturated rubber like EPDM. This replacement can be studied using the blending method for both binary and ternary blends. Thus, in this work, photo-degradation behavior of HIPS, PS/EPDM and compatibilized PS/EPDM/PA6 blends is studied at different UV radiation exposure periods.

EXPERIMENTAL

Materials

Substances used in this research are: KEP270 EPDM by Kumho from Korea (Mooney viscosity 71 M, ethylene content 57%, termonomer content 4.5% ENB (2-ethylidene-5-norbornene)), general purpose polystyrene (GPPS, Solarene® G-144) supplied by Hyundai from Korea (MFI 8.5 g/10min @200˚C & 5kg), polyamide 6 (PA6, Ultramid® B3S) supplied by BASF (MFI 197.75 g/10min @275˚C & 5kg), glycidyl methacrylate monomer (GMA, 97%, Aldrich , Japan), dicumyl peroxide (DCP, Merck Millipore, Germany) and formic acid prepared from Merck Millipore.

Blend preparation

A ternary blend of PS/(EPDM+GMA+DCP)/PA6 and a binary blend of PS/EPDM was prepared at 220°C using an internal batch-mixer (Brabender GmbH, Germany) with a rotor speed of 120 rpm for the overall mixing time of 10 min [2,21]. Prior to mixing, all components were dried for 24 hours at 80°C to eliminate moisture. Half of the weighed EPDM was cut into small, flaky pieces and then physically impregnated with DCP and GMA. The mixture was placed in a closed container for 20 minutes at ambient temperature to allow the

Table 1. Prepared samples: compositions and nomenclature.

Components (Sample Code)	PS (wt. %)	EPDM (wt. %)	PA6 (wt. %)	EPDM (wt. %)	GMA* (phr)	DCP* (phr)
	Blend components			compatibilizer		
PS/EPDM (B)	70	30	0	0	0	0
PS/(EPDM+GMA+DCP)/PA6 (T)	70	7.5	15	7.5	3	0.15
PS/EPDM/PA6 (T_0)	70	15	15	0	0	0

* Based on half of EPDM weight.

physical absorption of the active components onto the surface of the EPDM. Then all components were simultaneously blended using the internal mixer for 10 minutes. The composition containing EPDM, DCP and GMA acts as an in situ compatibilizer during the final melt blending of the ternary blend [2]. B and T_0, as a blank sample for analyzing the reactions during T preparation, were prepared by simultaneously blending of all components under the same processing conditions for T. Composition and nomenclature of prepared samples are listed in Table 1.

ATR-FTIR analysis

The ternary blend sample (T) and a blank sample (T_0) were characterized using an ATR-FTIR spectrometer (Bruker Vertex 70, United States). Additionally, T was analyzed before and after one week immersion in formic acid to extract any unreacted PA6. In fact, the reacted portion of PA6 phase is bonded with epoxide rings grafted onto the EPDM backbone, making it resistant to removal by formic acid as a solvent. The sample, consisting of fine particles smaller than 1 mm, was mixed with KBr and pressed to form a 1.2 cm diameter tablet for analyzing. ATR-FTIR analysis was utilized to monitor the expected compatibilizing reactions during the melt blending process.

Morphological analysis

The fracture surfaces of the impact tests for T and B samples were etched with n-heptane to remove the EPDM phase. Following this, the etched surfaces were coated with a thin layer of gold using a BAL-TEC 005 sputter coater. The morphological structures of the samples were then observed using a Philips XL30 scanning electron microscope.

Photo-aging

The standard binary blend (B), ternary blend (T) and HIPS specimens for tensile and impact strength tests were machined from sheets compression-molded at 240˚C. The specimens were submitted to accelerated photo-aging, with only one side of the specimens being exposed, according to the procedures described in ASTM G53 using a particular set consisting of UVA PHILIPS model CLEO performance 80 W mercury Lamps from Eindhoven, the Netherlands, which emits light in the range of 315 to 400 nm. The aging program consisted of 22 h cycles of UV irradiation at a temperature of 25-30°C, followed by 2 h of cold steam introduction into the test chamber at 50°C [3]. Samples were exposed for 0, 168, 336, 504, and 720 hours.

Mechanical tests

After each time period, mechanical tests were performed on the three exposed samples. Tensile tests and un-notched Izod impact tests were conducted by a Galdabini testing machine (Italy) with crosshead speed of 1 mm/min according to ASTM D638 and a Ueshima impact tester machine (Japan) according to ASTM D4812, respectively.

Yellowness index

Yellowness index was measured after each time period for each sample by a spectrophotometer model 64-SP X (China), containing Color I QC software, according to ASTM E313 to evaluate photo-aging changes. All samples were placed on the same gray background and yellowness index was measured at three surface points at the ambient temperature.

RESULTS AND DISCUSSION

ATR-FTIR analysis

Figure 1 shows the ATR-FTIR spectrum of the studied (T) and blank (T_0) ternary blend. The spectrum of sample T, after etching, shows a peak at 1740 cm^{-1} associated with C=O groups. This peak, indicating the presence of C=O due to GMA grafting onto the EPDM backbone, is not present in sample T_0. This observation supports the conclusion that a grafting reaction occurred during the in situ compatibilization of sample T [2,22,23]. As seen in Figure 1, some specific peaks representing primary and secondary amine groups of PA6 have revealed at 3000-3100 cm^{-1} (a double band) and 3303 cm^{-1} (a single band), respectively. Surprisingly, etched samples still reveal these peaks. This can be ascribed to the compatibilizing reactions between some amine end groups of PA6 and the epoxy groups of GMAs grafted on the EPDM backbone. It is obvious that these reacted PA6 chains are not soluble in formic acid as the etching liquid. Thus, expected compatibilizing reactions progress during the melt blending process is confirmed.

Morphological analysis

The SEM micrographs for all studied samples are shown in Figure 2. Figure 2b clearly shows the morphological structure of the binary blend (B) where PS and EPDM act as the matrix and dispersed phase respectively. The structure consists of large, stretched, non-spherical EPDM particles dispersed in the PS matrix. In contrast, Figure 2d illustrates the morphology

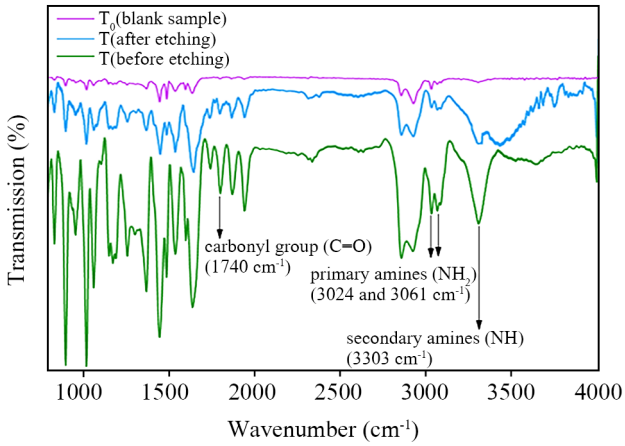


Figure 1. ATR-FTIR spectrum of PS/EPDM/PA6 ternary blends.

of compatibilized ternary blends (T) with a core-shell structure. In this arrangement, PA6 serves as the core (white cores), surrounded by EPDM shell, along with a spherical separate dispersed phase of EPDM (black holes). This type of morphological structure in T sample has been observed in similar blends in previous research [2]. This morphological structure results from changes in interfacial tensions due to the effect of compatibilization [24]. The comparison between Figures 2c and 2d illustrates a significant morphological change from a separated dispersion morphology, which includes large rubber droplets, to a core-shell structure with separated rubber droplets. The epoxy-amine reaction has led to a substantial reduction in the interfacial tension between the EPDM and PA6 phases [6]. Consequently, the PA6 phase is completely removed from the PS bulk and is partially surrounded by an EPDM shell. This phenomenon confirms the occurrence of a compatibilization reaction. The morphological structure can directly influence the mechanical properties.

Mechanical properties

Mechanical properties of the studied samples, including tensile and impact properties, at different radiation periods, are reported in Table 2. The comparison of the tensile properties between T and T_0 indicates that T_0 exhibits a lower tensile modulus of 1674±15 MPa, as shown in Table 2. This reduction in modulus can be

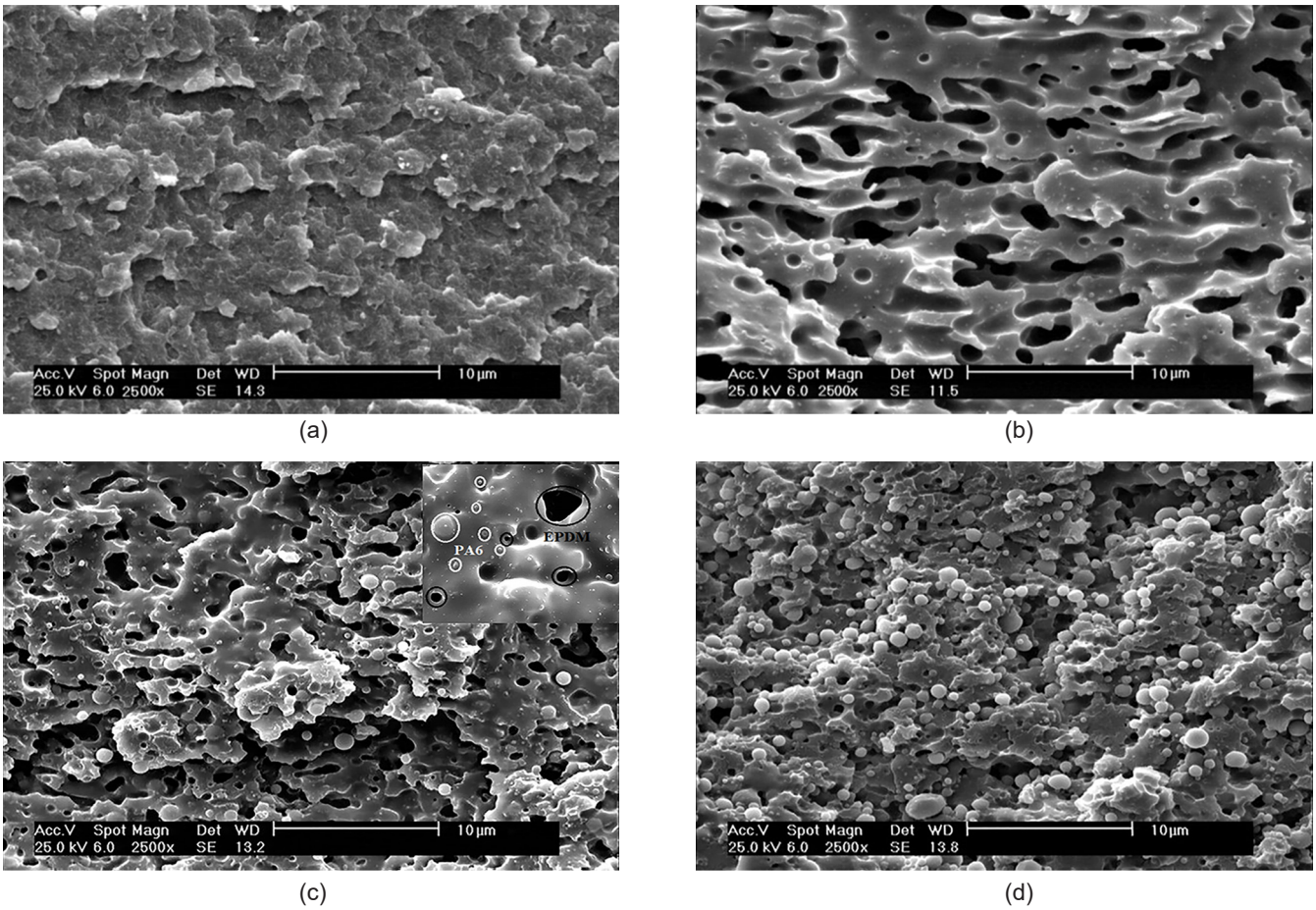


Figure 2. SEM micrographs of fracture surface of samples: (a) HIPS, (b) PS/EPDM(B), (c) PS/EPDM/PA6(T_0) and (d) PS/(EPDM+GMA+DCP)/PA6(T).

attributed to the presence of coarse, soft, and elongated EPDM droplets in the continuous phase [25], illustrated in Figure 2c, which leads to a low modulus in the uncompatibilized ternary blend. According to the data presented in Table 2, T_0 also shows a lower impact strength of 7.0±0.6 J/m, ultimate tensile strength of 22.38±0.4 MPa, and elongation-at-break of 2.2±0.19%. These figures result from weak interfacial interactions between the blend phases. On the other hand, the microstructure depicted in Figure 2c includes dispersed hard PA6 particles, which negatively impact the impact strength and contribute to the expected reduction in impact strength for B0 [26].

The modulus (Table 2) reveals an extreme increase in the tensile modulus of HIPS and T specimens in the early time of UV irradiation. However, the increase for B is gradual. After some time, a greater increase in the modulus was observed for B and T specimens than for HIPS, which could be attributed to the crosslinking reactions caused by non-conjugated double bonds of EPDM unsaturated monomers (2-ethylidene-5-norbornene, ENB) [3]. The increase in modulus of T is greater than sample B which might be attributed to DCP decomposition residues in sample T intensifying the crosslinking reactions.

As seen in Table 2, the elongation-at-break of all specimens decreased with increasing UV irradiation time. The decrease in elongation-at-break has been attributed to the crosslinking reactions of non-conjugated double bonds of the rubber unsaturated monomers [1]. Despite the fact that the ternary blend does not contain stabilizer, unlike HIPS, and even contains a small amount of unreacted DCP, it was found that the elongation-at-break of the ternary blend is greater than that for HIPS. It can be attributed to the larger separated rubber droplets in T morphology than the rubbery districts in HIPS morphology, as shown in Figure 2a and Figure 2d. [27]. Emerson et al. [1] observed a similar trend in tensile properties of PS/EPDM binary blend and HIPS exposed to UV radiation at different periods of time as 0, 168, and 720 hours. The impact strength of HIPS is almost the same as the binary and ternary blends after 720 h of UV irradiation confirming that high impact strength is a distinctive feature of HIPS.

The amount of increase in tensile modulus and mechanical properties retention for all three samples under ultraviolet exposure are presented in Table 3. The properties retention values are obtained from the ratio of their values after UV irradiation to the values before. Comparing the results in Table 3, it was found that the ultimate tensile strength retention values ranged from 70 to 82% and there is no significant difference between them. This is while the impact strength and elongation-at-break of HIPS greatly decreased compared to the blends and showed the retention about 8.46 and 7.86%, respectively. The reason for this sharp decrease in the properties of HIPS might be the lower stability of this sample against optical degradation compared to the binary and ternary blend samples [4]. Emerson et al.[3] also observed a similar phenomenon in the photo-degradation of HIPS and PS/EPDM blend. Thus, the large decrease in elongation-at-break and impact strength of HIPS compared to the polymer blend could be due to the rubber phase cross-linking as well as degradation of

Table 2. Mechanical properties of samples at different UV radiation time intervals.

Components	Sample Code	UV Irradiation Time (hr)	Tensile Modulus (MPa)	Ultimate Tensile Strength (MPa)	Elongation-at-break (%)	Impact Strength (J/m)
HIPS	H	0	1964±17	27.3±2.4	24.6±1.9	130±2
		168	2167±95	26.6±1.4	2.2±0.1	6.5±0.4
		336	2223±45	25.3±0.5	2.0±0.4	6±0.4
		504	2351±362	23.7±0.0	2.0±0.0	10.1±0.4
		720	2397±192	22.4±0.5	1.9±0.3	11±3.1
PS/EPDM (70/30 wt.%)	B	0	1288±168	21.3±1.3	3.1±0.3	8.84±0.4
		168	1311±98	17.5±0.7	3.3±1.0	10.8±1.0
		336	1336±10	16.4±0.9	3.4±0.8	9.0±2.3
		504	1543±21	16.1±0.5	2.7±0.5	8.5±0.6
		720	1793±31	15.9±0.6	2.2±0.3	8.2±0.9
PS/EPDM/PA6	T_0	0	1674±15	22.38±0.4	2.2±0.19	7.0±0.6
PS/EPDM+(EPDM+DCP +GMA)/PA6	T	0	2015±24	25.7±2.5	2.9±0.2	25.7±0.9
		168	2302±35	20.8±2.2	2.8±0.1	12.7±0.6
		336	2454±269	19.4±0.5	2.7±0.5	10.4±0.6
		504	2458±18	19.2±0.7	2.4±0.0	10.1±0.3
		720	2976±139	18.1±0.4	2.3±0.3	9.9±0.2

Table 3. Added and residual mechanical properties of samples after UV irradiation for 720 hours.

Components	Sample code	Impact strength retention (%)	Tensile modulus increase (%)	Ultimate tensile strength retention (%)	Retention of elongation-at-break (%)
HIPS	H	8.46	22.05	81.82	7.86
PS/EPDM (70/30 wt.%)	B	92.87	39.21	74.95	70.59
PS/EPDM+(EPDM+DCP+GMA)/A6	T	38.62	47.69	70.40	79.73

the main PS phase in HIPS. In addition, the higher stability of EPDM compared to the PB phase present in HIPS plays an important role in the deactivation the PS phase macro-radicals [1, 28].

It was also found that at the first stage, HIPS, ternary blend (T) and binary blend (B) presented the best mechanical performance, respectively (Table 2 and Table 3); but after 720 h, the blends presented higher elongation-at-break compared to HIPS due to the higher resistance of blends against photo-degradation [3]. Although the ternary blend demonstrates the better mechanical performance than the binary blend in most areas, except for elongation-at-break during the initial stage, the mechanical performance of both blends is similar after 720 h. Figure 2a clearly shows that the presence of large, stretched rubber particles during the initial stages of irradiation may explain the superior elongation-at-break observed in B compared T. The superior mechanical performance of T, particularly in terms of impact strength compared to B, can be attributed to its compatibilized core-shell morphology (Figure 2b). This morphological structure enhances the transfer of impact energy from PS matrix to EPDM cores through PA6 shells, while also allowing the rubbery cores to effectively dampen energy [2, 21].

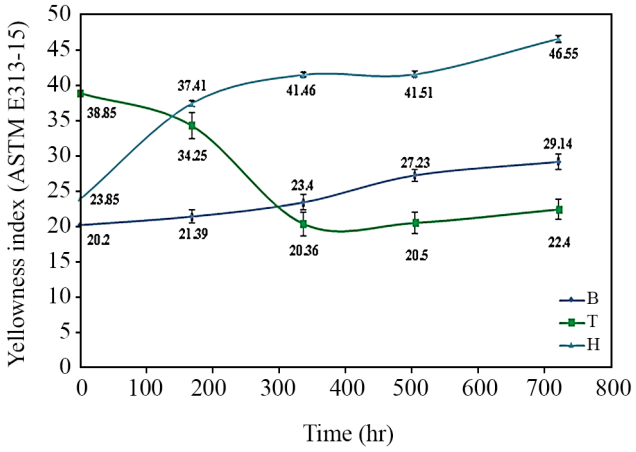


Figure 3. Changes in the yellowness index of samples with UV irradiation time.

Yellowness index

In order to investigate the degradation process through the changes in the appearance of the samples, the yellowness index values of the samples were monitored at different periods of UV irradiation (see Figure 3). It should be noted that the yellow color appeared through degradation is due to the formation of chromophores and their interactions with the aromatic rings of the PS phase [3].

As can be seen in Figure 3, before UV irradiation, the yellowness index and the standard error of the ternary blend (T) were strongly higher than the other two samples (B, H). The high standard error indicates color non-uniformity (yellowing) attributed to the presence of chromophores and their interactions with the aromatic rings of the PS phase which have led to non-uniformity of yellowing in the samples [3]. It can be seen that with the increase of UV irradiation time, the yellowness index of the ternary blend showed a dramatically decrease and then gradually increase. It can be said that the decrease in yellowness is due to using by-products in the cross-linking reactions [29], which is followed with the simultaneous occurrence of optical and chemical aging in this sample leading to an increase in the yellowness index.

It can also be observed that the binary blend and HIPS presented an increase in yellowness index with increasing UV irradiation time. Analyzing yellowness index variations showed that the final yellowness index of HIPS is 1.6 and 1.2 times of the binary and ternary blends, respectively. This phenomenon confirmed the lower stability of HIPS against photo-degradation compared to binary and ternary blends [3].

CONCLUSION

The FTIR spectra confirmed the compatibilizing interactions in PS/EPDM/ PA ternary blends since some amine end groups of PA6 react with the epoxy groups of GMAs grafted on the EPDM backbones. Also, the grafting of GMA onto the EPDM backbone

was confirmed by comparing the ATR-FTIR spectra of compatibilized and non-compatibilized blends.

Mechanical strength values presented an extreme increase in the tensile modulus of three specimens with increasing UV irradiation time. On the other hand, the elongation-at-break of all samples decreased with increasing in UV irradiation duration.

Results showed that:

- The carbonyl (C=O) peak's appearance in the ATR-FTIR spectrum, along with a significant morphological shift from separate dispersion to a core-shell structure, and an improvement in impact strength for T compared to T_0, confirmed the compatibilization reaction in the compatibilized ternary blend (T).
- The ultimate tensile strength retention values are between 70 and 82% and there is no significant difference between them.
- The impact strength and elongation-at-break for the HIPS sample have greatly decreased compared to the blends with a retention rate of 8.46 and 7.86%, respectively.
- The ternary blend (T) showed higher tensile properties, except elongation-at-break in the initial period, than the binary blend (B). However, both samples exhibited similar mechanical performance after 720 h of UV irradiation.
- Following the yellowness index results, the lower stability of HIPS against photo-degradation compared to the binary and ternary blends is confirmed.

ACKNOWLEDGEMENTS

Thanks to the Research Institute of Petroleum Industry (RIPI) for providing its laboratory and equipment facilities.

CONFLICTS OF INTEREST

The authors declared that there is no conflict of interest.

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