Synthesis and Properties of Styrene–EPDM–Vinyl Acetate Graft Polymer

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ABSTRACT: The styrene–EPDM–vinylacetate (SEV) graft polymer, which linked respectively the styrene (St) unit and vinylacetate the (VAc) unit to the ethylene–propylene–diene terpolymer (EPDM) backbone was synthesized by two-step graft polymerizations: First the graft polymerization of VAc onto EPDM was carried out, and then St was added successively in the prepolymerized solution and further polymerized for a given period to obtain SEV. The effects of concentration of EPDM and an initiator, mole ratio of VAc to St, polymerization time, temperature, and solvent were examined on the graft polymerizations. The synthesized graft polymers (SEVs) that have different contents of St or VAc were identified by Fourier transform IR spectrum. The highest graft ratio has been obtained by 10 wt % of EPDM, 1.0 mole ratio of VAc to St, and 1.0 wt % of BPO in toluene for 48 h at 70°C. The glass transition temperature of SEV is lower than that of poly(vinyl acetate) (PVAc) and polystyrene (PS). The thermal stability of SEV is higher than that of PVAc, PS, and the acrylonitrile–butadiene–styrene (ABS) resin. The tensile strength of SEV was improved as compared with that of EPDM. The light resistance and weatherability of SEV were better than those of ABS. © 2000 John Wiley & Sons, Inc. J Appl Polym Sci 77: 2296–2304, 2000

Key words: graft polymerization; styrene–EPDM–vinyl acetate graft polymer (SEV); ethylene–propylene–diene terpolymer (EPDM); acrylonitrile–butadiene–styrene (ABS) resin; glass transition temperature; thermal stability; light resistance, weatherability; tensile property

INTRODUCTION

Ethylene–propylene–diene terpolymer (EPDM) is a synthetic rubber having superior heat and ozone resistance, weatherability, and good electrical insulation property. Therefore, EPDM is used as materials for automobile parts such as hoses, gaskets, belts, and electrical cables, and for footwear. However, it has poor oil resistance and weak adhesion property, and it is difficult to make it compatible with polar thermoplastic polymers to prepare products having desired properties such as good oil resistance, adhesion, and tensile strength. The products based on sulfur vulcanization of EPDM have such demerits as low tensile strength and tear strength.[1] The weak point of materials can be improved by the applying molecular design. As an example, acrylonitrile–EPDM–

styrene, with excellent light and weather resistance, was prepared using EPDM in place of butadiene in acrylonitrile–butadiene–styrene (ABS) which has a low resistance of light and weather. We have also synthesized the high-performance graft polymers containing EPDM to improve demerits of ABS such as poor heat and light resistance.[2–13] In order to synthesize the impact-resistant thermoplastics containing polystyrene, poly(methyl methacrylate), poly(vinyl acetate), etc., the free radical grafting polymerizations of the vinyl monomers onto polybutadiene, EPDM, etc., have been reported by many researchers.[14–19]

Polystyrene (PS) and poly(vinyl acetate) (PVAc) have been used on a large scale in plastic industry because PS has rigid, highly transparent, and good electrical insulation property, and PVAc has adhesive property and it is easy to form film. However, PS and PVAc have poor thermal stability and high brittle properties and they are difficult to use at low temperature.[20]

However, it has been reported[21] that styrene (St) and vinyl acetate (VAc) are very difficult to copolymerize with conventional radical initiators because of a large different reactivity between St and VAc. Thus, there has been no report in the open literature on the systematic study for the graft polymerization of St and VAc onto EPDM by a radical initiator.

The aim of this study was to synthesize the graft polymer of St and VAc onto EPDM by use of conventional radical initiators. For this purpose, first the graft polymerization of VAc onto EPDM was carried out, and then St was added successively in the prepolymerized solution and further polymerized. The structure of synthesized graft polymer, styrene–EPDM–vinyl acetate (SEV), was identified by Fourier transform IR (FTIR) spectroscopy. The effects of concentration of EPDM and initiator, mole ratio of VAc to St, polymerization time, temperature, and solvent on the graft polymerizations were examined. The solubility, glass transition temperature, thermal stability, light resistance, weatherability, and tensile property of SEV were also determined.

EXPERIMENTAL

Materials

EPDM, having 5-methylene-2-norbonene as a termonomer (Aldrich Co., USA, 50 wt % ethylene, 8 wt % 5-methylene-2-norbornene, Mooney viscos-

ity 50), and ABS (Junsei Chemical Co.) were used as received. St (Junsei Chemical Co.) and Vac (Junsei Chemical Co.) were purified by standard procedures. Benzoyl peroxide (BPO, Aldrich Co., USA) was purified by recrystallization from methanol. Toluene, tetrahydrofuran (THF), acetone, methanol (MeOH), chloroform, n-hexane, etc., were distilled prior to use.[22] Sulfur (Junsei Chemical Co.), zinc oxide (ZnO, Junsei Chemical Co.), stearic acid (St-Ac, Junsei Chemical Co.), tetramethylthiuramdisulfide (TMTD, Junsei Chemical Co.), and 2-mercaptobenzothiazone (Junsei Chemical Co.) were used as received.

Synthesis of SEV Graft Polymer

SEV has been synthesized from St, VAc, and EPDM by two step graft polymerizations as follows: First step, the 15 wt % of EPDM, 3.44 g (0.04 mole) of VAc dissolved in 20 mL toluene, and 0.09 g (1 wt %) of BPO dissolved in 10 mL toluene were introduced into the three- neck round-bottomed flask and stirred at 70°C for 3 h. Second step, the solution of 4.16 g (0.04 mole) of St dissolved in 10 mL toluene was dropped slowly into the flask used in the first step with stirring at 70°C, then the graft polymerization was continued for 4 h with stirring at 70°C. After finishing graft polymerization, the reaction mixture was precipitated in methanol. In order to remove PS, PVAc, and poly(VAc-co-St) formed during graft polymerization, the precipitate was extracted by Soxhlet apparatus using acetone for 16 h. Ungrafted EPDM was separated by extraction of the mixture with n-hexane for 12 h. SEV was separated from poly(EPDM-g-St) and poly(EPDM-g-VAc) by Soxhlet apparatus using toluene/n-hexane mixture (50/50 by vol.) for 24 h. The block diagram of SEV separation showed in Figure 1. SEV was dried in a vacuum drying oven at room temperature until constant weight. The SEVs, which have different contents of St and Vac, were synthesized by the various graft polymerization conditions, as shown in Table I.

The total conversion (%), grafting ratio (%), and grafting efficiency (%) of graft polymerization were estimated from the following equations[12]:

Total conversion (%)

$$= \frac{\text{total weight of polymer formed}}{\text{weight of monomer charged}} \times 100 \quad (1)$$
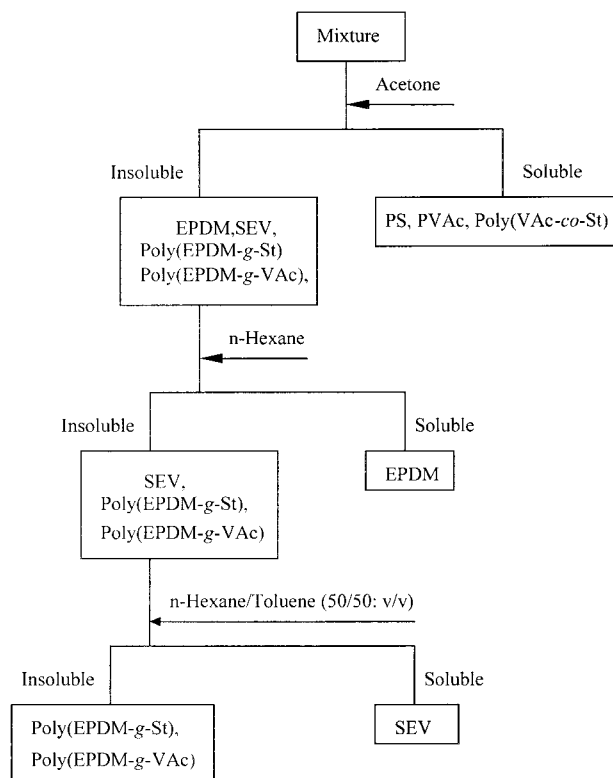
Figure 1 The block diagram of separation of SEV from the mixtures.

Grafting ratio (%)

$$= \frac{\text{weight of polymer in grafts}}{\text{weight of substrate}} \times 100 \quad (2)$$

Grafting efficiency (%)

$$= \frac{\text{weight of polymers in grafts}}{\text{total weight of polymer formed}} \times 100 \quad (3)$$

Measurement

Infrared Spectroscopy

The FTIR spectra of SEVs films were obtained with a Jasco FT/IR-5300 spectrophotometer. These films were cast from 0.5 wt % solution of SEVs in chloroform.

Differential Scanning Calorimetry

Differential scanning calorimetry (DSC) of EPDM and SEV was carried out on a Perkin-Elmer DSC7 (Shimadzu Co.). At the first scan, the temperatures of the samples were raised from -100 to 100°C at a rate of 20°C/min in order to erase their thermal history. Then the second scanning

was heated from -100 to 200°C at a rate of 10°C/ min to determine the glass transition temperature (T_g) of the samples.

Thermogravimetric Analysis

The thermal stabilities of EPDM, ABS, and SEV were examined with a thermogravimetric analysis instrument (Shimadzu DT 30A TGA) at a scanning rate 10°C/min in nitrogen gas.

Light Resistance

The color differences of the EPDM, PS, PVAc, SEV, and ABS were determined by a color difference meter (Minolta Co., Japan). First, the film specimens were cast from 2 wt % of THF solution on a nonyellowing urethane-coated hiding paper. Then they were dried slowly at room temperature and kept under vacuum to constant weight. The cross-sectional area of the film was 3×5 cm^2 and thickness was 35 μm. The color difference (ΔE) of the samples was calculated using the Hunter–Schöfield equation.[23]

Weatherability

The change of color difference (ΔE) of the samples after exposing to UV light (120 h) is used for evaluation of the weather resistance of the samples. The weather resistance (ASTM G53) of the samples was measured by using a Q panel B313 Weathering Tester coupled with a UV lamp of 313–280 nm in wavelength. The film specimens were prepared by the similar method as described for the determination of light resistance.

Tensile Property

The samples for determination of tensile properties were prepared by the following way. EPDM

Table I   Reaction Conditions for Graft Polymerization in the Study

Condition	Description
EPDM concentration (wt %)	5, 10, 15, 20, 25
Mole ratio ([VAc]/[St])	0.5, 1.0, 1.5, 2.0, 2.5
Reaction time (h)	12, 24, 48, 60, 96
Initiator concentration (wt %)[a]	0.5, 1.0, 1.5, 2.0, 2.5
Reaction temperature (°C)	50, 60, 70, 80, 90
Solvent	Toluene, THF, chloroform

[a] The concentration was based on the total weights of monomers and EPDM.

or SEV was mixed with curing agent (sulfur, 3 wt %), activators (ZnO, 5 wt %, stearic acid, 1 wt %), and accelerators (TMTD, 1 wt %, mercaptobenzothiazone, 1 wt %] on a laboratory open two–roll mill at room temperature for 30 min. Then the mixtures were cured by compression molding for 15 min at 170°C. Before determination, all samples were remained at standard conditions at least 24 h. The tensile tests were carried out using an Universal Testing Machine (Model HT Houns Field 500 L). The samples were prepared according to the ASTM D638-84. The crosshead speed was 5 mm/min, and the initial gauge length was adjusted at 25 mm.

RESULTS AND DISCUSSION

Polymerization Procedure

The graft polymerization of St and VAc onto EPDM were carried out by three different procedures as follows: First, EPDM, St, VAc, and BPO were polymerized in a toluene with stirring at 70°C for 48 h. The obtained results showed that the graft polymerization of St and VAc onto EPDM did not occur because of large reactivity difference between St and VAc.[15] The products of this polymerization were EPDM-*g*-St, PS and PVAc. Second, EPDM, St, and BPO were polymerized in a toluene with stirring at 70°C for 3h. Then the solution of VAc was dropped slowly to the prepolymerized solution. The products of this polymerization are also EPDM-*g*-St, PS and PVAc. Third, EPDM, VAc, and BPO were polymerized in a toluene with stirring at 70°C for 3h. Then the solution of St was dropped slowly to the prepolymerized solution. From this polymerization, SEV has been obtained. Therefore, the third is the best suitable procedure for the synthesis of the graft polymer respectively grafted St and VAc onto the EPDM backbone.

Characterization

The FTIR spectrum of SEV exhibited characteristic absorption bands at 3025 cm^{-1} (stretching vibration of aromatic C—H bond due to styrene unit), 2920 cm^{-1} (stretching vibration of aliphatic C—H bond), 1738 cm^{-1} (stretching vibration of C$=$O bond in vinyl acetate unit), 1465 cm^{-1} (scissoring of CH$_2$), 1375 cm^{-1} (bending of CH$_3$), 1240 cm^{-1} (stretching vibration of aliphatic C—O), and
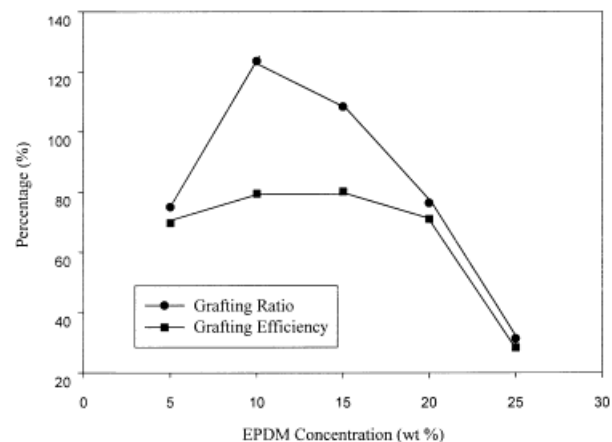

Figure 2 The effect of EPDM concentration on the graft polymerization. [Vac]/[St]: 1.0; polymerization time: 48 h; BPO: 1 wt %; polymerization temperature: 70°C; solvent: toluene.

760 and 700 cm^{-1} (out-of-plane bending vibration of aromatic C—H bond in styrene unit).[24,25]

Effect of Polymerization Condition

Effect of EPDM Concentration

The effect of EPDM concentration on the graft polymerization is shown in Figure 2. The polymerization was carried out with several concentrations of EPDM, 1.0 wt % of BPO, and 1.0 mole ratio of VAc to St in toluene at 70°C for 48 h. The highest grafting ratio and grafting efficiency were 130 and 79% at 10 wt % of EPDM, respectively. This can be explained by the fact that the grafting ratio and grafting efficiency increased with increasing of the graft site as a double bond in EPDM up to 10 wt % of EPDM, Because it has been known that, in general, the graft polymerization depends on the number of active sites available. However, EPDM concentration was higher, the viscosity of reaction medium was increased, as a result, the diffusion and mobility of St and VAc radicals onto EPDM molecule were difficult; thus grafting ratio and grafting efficiency has a tendency to decrease. On the other hand, in order to synthesize SEV as having as large as possible different physical and mechanical properties as compared with EPDM, the graft terpolymerizations were carried out using 15% of EPDM in place of 10% of EPDM in spite of representing the higher grafting ratio at 10% EPDM.

Effect of Mole Ratio of Monomer

Figure 3 showed the effect of mole ratio of VAc to St on the respective graft polymerization. The
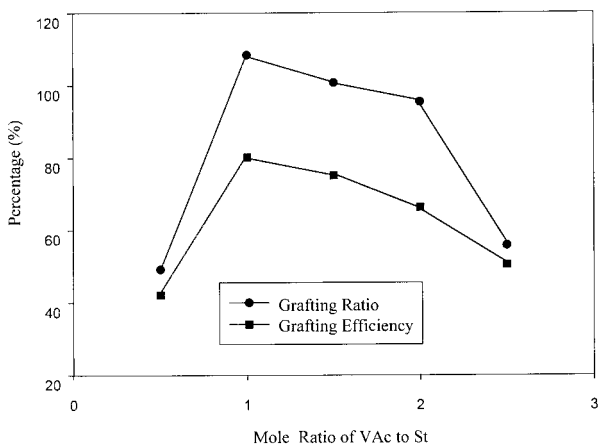
Figure 3 The effect of mol ration of Vac to St on the graft polymerization. EPDM: 15%; polymerization time: 48 h; BPO: 1 wt %; polymerization temperature: 70°C; solvent: toluene.

graft polymerization was performed with several mole ratios of VAc to St, 15 wt % of EPDM and 1.0 wt % of BPO in toluene at 70°C for 48 h. The grafting ratio rapidly increased with an increasing mole ratio of VAc to St up to 1.0, then it slowly decreased to 2.0 mole ratio, and thereafter it rapidly decreased; grafting efficiency increased up to 1.0 mole ratio of VAc to St and thereafter it slowly decreased. These results can be explained by the fact that the homopolymerization of styrene and the graft copolymerization of St onto EPDM preferentially occur as compared with graft terpolymerization of St onto EPDM-*g*-VAc, because the homopolymerization rate of styrene increases with increasing styrene concentration within a certain concentration range—that is, the concentration of styrene monomer to react to the EPDM-*g*-VAc decreases. As a result, the values for the grafting ratio and grafting efficiency are decreased, when the mole ratio of VAc to St is above 1.0. These results can be estimated by eqs. (2) and (3). From the results, in order to obtain the highest grafting ratio and grafting efficiency, the mole ratio of VAc to St of 1.0 is the most suitable for the respective graft polymerization.

Effect of Polymerization Time

The effect of polymerization time on the graft polymerization is shown in Figure 4. The graft polymerization was carried out under several polymerization times and with 15 wt % of EPDM, 1.0 wt % of BPO, and 1.0 mol ratio of VAc to St in toluene at 70°C. The grafting ratio and grafting

efficiency rapidly increased with an increasing polymerization time up to 24 h and thereafter they slowly increased to 48 h, then slowly decreased with further increasing polymerization time. They can be explained by the following: with an increase in polymerization time up to 48 h, the radicals of EPDM, St, and VAc, and high concentration of monomers, St and VAc have more time for the graft terpolymerization and as a result increases in grafting ratio and grafting efficiency were observed. After that time, the grafting ratio and grafting efficiency is decreased by the decrease of the unreacted monomer concentration as well as the increase of viscosity of the reaction medium by graft polymerization.[26]

Effect of Initiator Concentration

Figure 5 demonstrates the effect of initiator concentration on the graft polymerization. The polymerization was performed with several initiator concentrations, 15 wt % of EPDM, 1.0 mole ratio of VAc to St in toluene at 70°C for 48 h. The grafting ratio and grafting efficiency increased with an increasing initiator concentration up to 1.0 wt %. However, thereafter they rapidly decreased. The increasing phenomena of the grafting ratio and grafting efficiency can be elucidated by the fact that the graft polymer was readily formed than each homopolymer.[27] The decrease in grafting ratio and grafting efficiency at higher initiator concentration can be explained by the opposition to explanation for the increasing phenomena. As a result, the grafting ratio and graft-
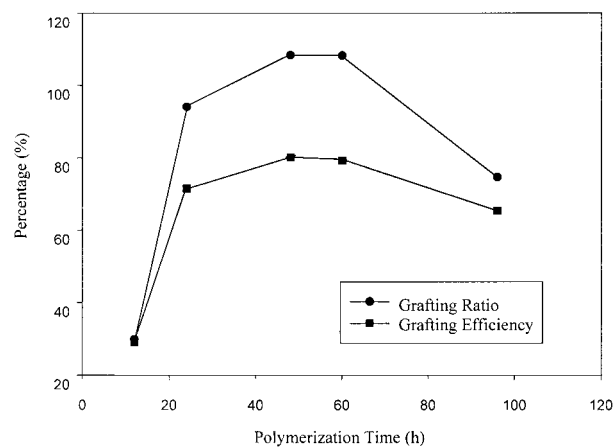

Figure 4 The effect of polymerization time on the graft polymerization. EPDM: 15%; [Vac]/[St]: 1.0; polymerization time: 48 h; polymerization: 70°C; solvent: toluene.
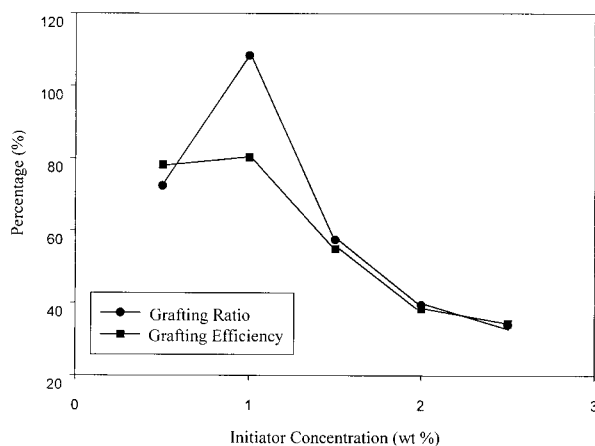
Figure 5 The effect of initiator concentration on the graft polymerization. EPDM: 15%; [Vac]/[St]: 1.0; polymerization time: 48 h; polymerization temperature: 70°C; solvent: toluene.

ing efficiency depend on the formation of the desired graft polymer to that of other polymers, as expected from eqs. (2) and (3).

Effect of Polymerization Temperature

The effect of polymerization temperature on the graft polymerization is presented in Figure 6. The graft polymerization was carried under several polymerization temperatures with 15 wt % of EPDM, 1.0 wt % of BPO, 1.0 mole ratio of VAc to St in toluene for 48 h. The grafting ratio and grafting efficiency passes through a maximum at 70°C and then they decreased with further rising
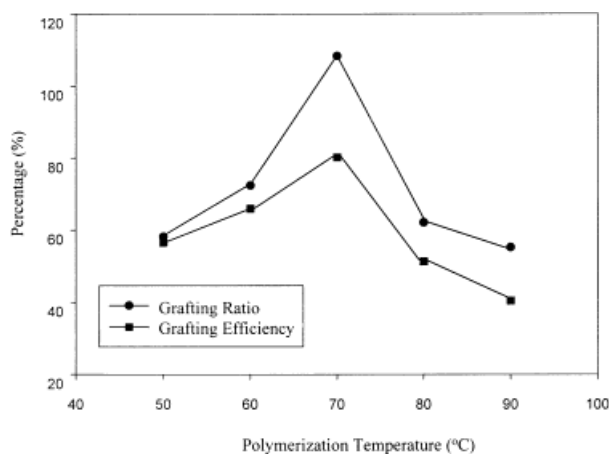

Figure 6 The effect of polymerization temperature on the graft polymerization. EPDM: 15%; [Vac]/[St]: 1.0; polymerization time: 48 h; BPO: 1 wt %; solvent: toluene.

Table II The Effect of Solvent on the Graft Polymerization[a]

Solvent	Solubility Parameter $(cal/cm^3)^{1/2}$	Grafting Ratio (%)	Grafting Efficiency (%)
Toluene	8.9	72	66
THF	9.1	60	56
Chloroform	9.3	54	51

[a] EPDM: 15 wt %; [VAc]/[St]: 1.0; polymerization time: 48 h; BPO: 1.0 wt %; polymerization temperature: 70°C.

polymerization temperature. It has been reported[28,29] that both the graft polymerization rate and the homopolymerization rates of St and VAc increase with a rising polymerization temperature at a certain range. The propagation rates (L/mol s) of styrene[30] were 55 at 30°C and 176 at 60°C, and those of vinyl acetate[31,32] were 1240 at 30°C and 3700 at 60°C, respectively. By the above facts, the grafting ratio and grafting efficiency for this system increased up to 70°C, but on further rising of temperature they decreased. Thus the optimum graft polymerization temperature for this system found to be 70°C.

Effect of Solvent

The graft polymerization was carried out in a solution; therefore, the selection of solvent for this graft polymerization is very important. The demand for such solvent is to dissolve both monomers, EPDM, and graft products. There were only three solvents such as toluene, THF, and chloroform adapted to that demand. The graft polymerization was performed with 15 wt % of EPDM, 1.0 mole ratio of VAc to St, at 60°C for 48 h in toluene, THF, and chloroform, respectively. From the obtained results, it is clear that the grafting ratio and grafting efficiency were highest when toluene was used as a solvent (see Table II). Because the solubility parameter of toluene is lower than other solvents as shown in Table II, toluene can better dissolve EPDM and the formed graft terpolymer (SEV) than THF and chloroform. Thus it can be considered that toluene is the most suitable solvent for the graft polymerization of St and VAc onto EPDM.

Solubility of Monomers, EPDM, and SEV

The solubilities of monomers, EPDM, and SEV are listed in Table III. The monomers (St and

Table III   The Solubility of Monomers, EPDM, and SEV

Solvent	St	VAc	EPDM	SEV
MeOH	S[a]	S	IS[b]	IS
Chloroform	S	S	S	S
Toluene	S	S	S	S
THF	S	S	S	S
Benzene	S	S	S	PS[c]
Acetone	S	S	IS	IS
Ethyl acetate	S	S	IS	IS
n-Hexane	PS	S	S	IS
Cyclohexane	S	PS	S	IS
DMF	S	S	IS	IS

[a] S: soluble.
[b] IS: insoluble.
[c] PS: poorly soluble.

VAc) were soluble in almost organic solvents. EPDM was soluble in n-hexane, cyclohexane, and benzene, but SEV was not soluble or was poorly soluble in the same solvents. It can be explained by the presence of PVAc, which has a polar group in SEV. However, SEV was soluble in chloroform, toluene, and THF. Thus it can be confirmed that SEV has better oil resistance than that of EPDM.

Glass Transition Temperature

As shown in Table IV, the T_g values of EPDM, PS, PVAc, and SEV were −36, 100, 30, and −21°C, respectively. The glass transition temperature (T_g) obtained from DSC diagram of SEV is higher than that of EPDM, but lower that that of PVAc and PS.[33] The T_g value of SEV was lower as

Table IV   The Glass Transition Temperature (T_g) and Thermal Stability of EPDM, PS, PVAc, SEV, and ABS

Sample	T_g (°C)	Decomposition Temperature (°C) T_I[b]	T_M[c]	Residue Weight at 450°C (%)
EPDM	−36	468	480	95
PS[c]	100	320	—	0
PVAc[c]	30	160	—	0
SEV	−21	445	475	72
ABS	—	390	—	25

[a] Reference 34.
[b] T_I: initial decomposition temperature.
[c] T_M: maximum decomposition temperature.
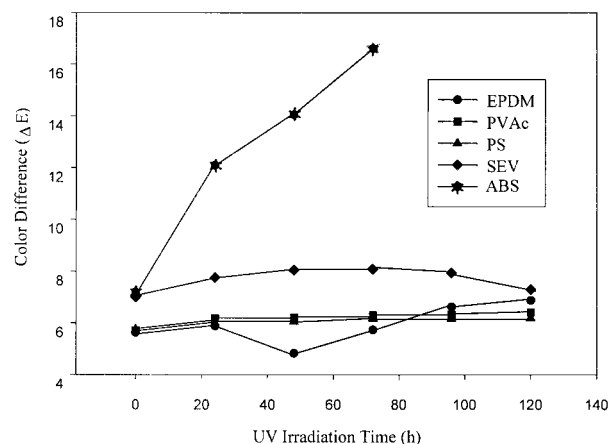
Figure 7 The color difference of EPDM, PS, PVAc, SEV, and ABS as a function of UV light irradiation time.

compared with that of PS and PVAc because of the existence of EPDM backbone having a low T_g value in the graft polymer (see Table IV).

Thermal Properties

The thermal properties of EPDM, PVAc, PS, ABS, and SEV were compared with their decomposition temperature and weight residue at 450°C (Table IV). SEV was synthesized with 15 wt % of EPDM, 1.0 wt % of BPO, and 1.0 mole ratio of VAc to St in toluene, at 70°C for 48 h. As shown in Table IV, the initial decomposition temperatures of EPDM, PVAc, PS, ABS, and SEV were 468, 160, 320, 390, and 445°C, respectively. The weight residue at 450°C of EPDM, PS, PVAc, ABS, and SEV were 95, 0, 0, 25, and 72%, respectively. It is clear that SEV shows better thermal properties than that of PS, PVAc, and ABS. SEV was improved thermal properties as compared with PS, PVAc, and ABS, because of the presence of EPDM having excellent stability to heat.[1,35]

Light Resistance and Weatherability

The light resistance and weatherability were semiquantitatively expressed in terms of ΔE with a National Bureau of Standards Unit. The smaller ΔE value means better light resistance and weatherability.[36] The samples were tested in a Weather-o-Meter for the measurements of light resistance and weatherability according to the time of UV irradiation. Figure 7 demonstrates the ΔE data of EPDM, PS, PVAc, VES, and ABS for the different times of testing. SEV was synthesized with 15 wt % of EPDM, 1.0 wt % of BPO as

Table V The Tensile Property of EPDM, EPDM-*g*-VAc, and SEV

Sample	Tensile Strength (MPa)	Elongation at Break (%)
EPDM	1.9	153.3
EPDM-*g*-VAc[a]	1.8	62.7
SEV	2.5	66.0

[a] [EPDM]/[VAc]: 1/2; BPO: 1 wt %; polymerization time: 48 h; polymerization temperature: 70°C.

an initiator, and 1.0 mole ratio of VAc to St in toluene at 70°C for 48 h. The dependence of light resistance and weatherability of SEV on the variation of UV radiation time was not significant except ABS. It should be noted that SEV obtained in this work shows better light resistance and weatherability than those of ABS resin because SEV contains EPDM, which has excellent outdoor properties in place of butadiene in ABS resin.

Mechanical Properties

The tensile strength and elongation at break of EPDM, EPDM-*g*-VAc, and SEV were listed in Table V. SEV shown in this table was prepared with 15 wt % of EPDM, 1.0 wt % of BPO, and 1.0 mole ratio of VAc to St in toluene at 70°C for 48 h. EPDM-*g*-VAc was synthesized 0.5 wt % ratio of EPDM to VAc and 1.0 wt % of BPO in toluene for 48 h at 70°C. It is clear that SEV has higher tensile strength than that of EPDM and EPDM-*g*-VAc, but its elongation at break is lower than that of EPDM because of the presence of PS having high tensile strength and low elongation at break.[37]

CONCLUSIONS

The SEV graft polymer was synthesized by two-step graft polymerizations: VAc was grafted onto EPDM and followed by St grafted to EPDM-g-VAc to obtain SEV. The dependence of the various conditions on the graft polymerization of St and VAc onto EPDM as well as properties of SEV were studied. The main results are summarized as follows:

1. The highest grafting ratio was 130% in the graft polymerization of St and VAc onto EPDM when 10 wt % of EPDM, 1.0 mole ratio of VAc to St, and toluene were used. However, SEV prepared using 10% of EPDM did not change much in physical

and mechanical properties than EPDM. In order to synthesize SEV having as large as possible different physical and mechanical properties as compared with EPDM, the graft polymer, SEV, was synthesized by using 15% of EPDM.

2. The grafting ratio and grafting efficiency were increased with increasing polymerization time up to 48 h, initiator concentration to 1.0 wt %, reaction temperature up to 70°C. Toluene is the best suitable solvent for the graft polymerization of St and VAc onto EPDM.

3. The glass transition temperature of SEV is lower than that of PS and PVAc but higher than that of EPDM. The thermal properties of SEV were improved as compared with PS, PVAc, and ABS. The light resistance and weatherability of SEV is better than those of ABS are. The tensile strength of SEV is higher than that of EPDM.

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