Original Paper

Styrenic thermoplastic vulcanizates based SEBS/PP/LLDPE with improved compression set for high-performance application via reactive extrusion

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Received: 15 July 2025, Accepted: 14 August 2025

DOI: 10.22063/poj.2025.35737.1366

ABSTRACT

Styrenic thermoplastic vulcanizates (STPV) consist of polypropylene (PP) and linear low density polyethylene (LLDPE) as the continuous phases and a crosslinked hydrogenated styrene block copolymer (SEBS) as the dispersed phase were prepared in an industrial scale using a corotating twin-screw extruder. Morphological study of the TPVs was carried out using a scanning electron microscope (SEM). Rheological study of the SEBS compounds carried with different content of 2,5-Dimethyl-2,5-di(tert-butylperoxy) hexane peroxide (0.5, 1, 2 and 4 wt. %). T_{90} appeared with 4 w% peroxide shows that curing has been completed. Effect of peroxide content (0.5 to 1.2 wt.%) on properties of the TPVs was led to an increase in tensile strength (from 5.6 to 7.4 MPa) and hardness to a certain extent (74 shore A). On the other hand, mechanical properties such as the elongation at break (from 592% to 284%), the melt flow index (MFI) (from 29.6 to 3.14 g/10 min at 190 °C, 10 kg) and tear strength (from 35 to 30 kg f/cm) are decreased. The compression set at low temperature (70 °C) is not changed so much, at high temperature (120 °C), while, is reduced due to higher curing (from 69% to 46%). Heat aging was carried out (at 85 °C for 168 h) on the TPVs which were prepared via the different content of peroxide (0.5 wt. % to 1.2 wt. %). It was found that the results of elongation and the hardness after aging have the lowest amount using 0.9 wt.% peroxide. However, the tensile strength after aging decreased with increasing the content of peroxide. As a result of increasing curing with increasing content of peroxide, the melting point of the prepared TPVs was disappears. The results of using triallylcyanurate (TAC) and bismaleimide (BMI) as a co-curing agent in peroxide curing indicate that all of the tensile strength, hardness and elongation at break (from 897 to 1082%) of the STPV were increased with BMI. Heat ageing study and compression set on the STPV with TAC was slightly raised compared to BMI. Also, the results of dynamic vulcanization on the properties of TPV based on SEBS/LLDPE/PP indicate that Crosslink density and gel content of TPV are close to that of EPDM rubber (fully crosslinked) and higher than that of TPE (without crosslinking). The results show that the tensile strength and density of the dynamically crosslinked blend have not changed significantly under peroxide curing.

Keywords: STPV, rheology, peroxide curing, bismaleimide, crosslink density.

INTRODUCTION

Thermoplastic elastomers (TPEs) are a class of polymers that combine the excellent processability and recyclability of thermoplastics with the high elasticity of vulcanized rubber. For elastomer reversibility, physical cross-links are essential; these cross-links must be reversible and can diminish when the temperature decreases [1-3]. TPEs can be broadly categorized into two groups: blends and multi-block copolymers. The first group includes copolymers such as styrenic block copolymers (SBCs), polyamide/elastomer block copolymers (COPAs), polyether ester/elastomer block copolymers (COPEs), and polyurethane/elastomer block copolymers (TPUs). TPE blends are further divided into two categories: dynamically vulcanized blends (TPVs) and polyolefin blends (TPOs) [1,4,5].

Thermoplastic vulcanizates (TPVs) are a unique type of thermoplastic elastomer in which the rubber phase undergoes dynamic vulcanization during melt mixing at high temperatures, combined with a semicrystalline thermoplastic matrix. To achieve a uniformly distributed vulcanized rubber phase with micron-sized particles, dynamic vulcanization begins with the melting of the rubber and thermoplastic polymer at elevated temperatures, followed by the vulcanization of the rubber using a cross-linking agent. This process significantly enhances the performance of blended thermoplastic elastomers, reduces equipment costs, and increases efficiency compared to traditional rubber vulcanization techniques [6-9]. The characteristics of TPVs are largely determined by their microstructure. Based on this, several factors influence the microstructure of TPVs, including the rubber-plastic composition ratio (R/P), the presence of compatibilizers, the crosslink density (CD) of the rubber phase, as well as the physical and chemical properties of both the plastic and rubber phases. Additionally, the preparation method, processing conditions, and the use of additives also play significant roles [6,9].

The crosslink density (CD) of the rubber phase and the rate of the crosslinking reaction are critical factors influencing the coalescence and disintegration of the rubber phase during dynamic vulcanization (DV). A higher CD typically leads to increased shear stress on the rubber phase and a higher viscosity ratio of the rubber to plastic (R/P), which facilitates phase inversion and morphological development of TPVs during DV [9-12]. Improved elasticity in TPVs is often achieved by increasing the CD of the rubber phase, primarily due to a higher concentration of crosslinking agents [9,13,14]. However, beyond a certain concentration, further increases in the curing agent do not enhance CD and can lead to an optimal elasticity for TPVs cured at this optimum concentration [14]. Additionally, an increase in CD may result in the formation of smaller rubber particles within the TPVs [16], which can further enhance the elasticity of the TPVs [6].

Co-curing agents are reactive additives that enhance the effectiveness of crosslinking, resulting in improved curing rates and conditions. They help reduce unwanted side reactions, mitigate steric hindrance effects, and react rapidly with polymer radicals. Co-curing agents can be broadly classified into two categories: (I) those that quickly react with free radicals, influencing their rate and extent of cure; and (II) those that engage in specific hydrogen-exchange interactions with the radicals. Among the co-curing agent that react quickly and generate stable radicals, which can readily link or polymerize with other co-curing agent molecules, are acrylates, methacrylates, and bismaleimide (BMI). Triallyl cyanurate (TAC), a polyallylaromatic compound, also produces stable radicals but has a significantly lower capacity to extract hydrogen from the polymer, placing it in the second category [17,18].

In this work, styrenic thermoplastic vulcanizates (STPVs) were prepared on an industrial scale using a corotating twin-screw extruder, consisting of polypropylene (PP) and linear low-density polyethylene (LLDPE) as the continuous phases, with crosslinked SEBS as the dispersed phase. The effect of peroxide content (ranging from 0.5% to 1.2% wt.%) on the properties of the TPVs was investigated. Additionally, TAC and BMI were used as co-curing agents in the peroxide curing system during the preparation of the STPVs. The dynamic vulcanization of the SEBS/PP-based TPVs was also examined.

EXPERIMENTAL

Materials

PP (density of 0.9 g/cm^3, MFI of 10.5 g/10 min at 230 °C, 2.16 kg and heat deflection temperature (HDT) of 75 °C) and LLDPE (MFI of 0.9 g/10 min at 190 °C, Vicat softening point of 100 °C, 2.16 kg and density of 0.921 g/cm^3) were purchased as pellets. SEBS was supplied as powder from LCY GRIT CORP, Taiwan (density of 0.91 g/cm^3, SM content of 33% and viscosity of 2000 cP, 10 wt.% in toluene at 25 °C). 2,5-dimethyl-2,5 di(tert-butylperoxy) hexane peroxide as the curing agent was prepared from Coin AKZO Nobel Co. (Netherlands). Tris (2,4-di-tert. -butylphenyl) phosphite, Irgafos 168 antioxidant and pentaerythritol tetrakis (3-(3,5-di-tert-butyl-4-hydroxyphenyl) propionate), Irganox 1010 were supplied by BASF Co., Germany. Triallyl cyanurate (TAC) and bismaleimide (BMI) as a co-curing agent in peroxide curing, was purchased from KETTLITZ (Germany). Aliphatic oil was prepared from Sepahan Co., Iran. Talc (Persitalc® 25 – SW) was purchased by Omya (Switzerland). Ethylene-Propylene-Diene Monomer (EPDM) rubber was supplied from PartLastic Group, Iran.

Sample preparation

Dynamic cross-linking process of the TPE based on SEBS/ LLDPE /PP was carried out in a co-rotating twin-screw extruder. The extruder (Model CTE 65, COPERION Co.) [4] has twelve heating/cooling zones. A feed rate (8 kg/h) by using temperature profiles (140 °C to 160 °C) from feed zone to die zone were used.

Test specimens were produced via injection molding. The formula of the TPEs, except peroxide, was constant and shown in Table 1. Concentration of the peroxide was varied in the range of 0.5–1.2 wt.%. TAC & BMI as co-agent was used in all compounds with twice the ratio of peroxide.

Table 1. Recipe of TPV for peroxide curing systems.

Materials	Wt.%
SEBS	15-40
LLDPE	0-10
PP	10-30
Oil 150	15-40
Irganox 1010	0.1-1
Irgafos 168	0.1-1
Peroxide	0.5-1.5
TAC or BMI	1-3
Talc	15-35

Analysis and testing method

Properties of the samples were determined according to the standard methods shown in Table 2.

Table 2. Standards used for test method.

Characterization	Standard	Test method (standard conditions used)
Heat aging test at 85 °C	ISO 188: 2011	168 h, 85 °C, %
Hardness, Shore A	ISO 7619: 2008	1 s, 15 s
Density	ISO 1183: 2019	---------
Tear strength	D415149: 2008	kg f/cm
Tensile strength-at-break Elongation-at-break Tensile strength (MPa) 20% Elongation Tensile strength (MPa) 20% Extension	ISO 37: 2017	500 mm min^{-1}, 2-kN load cell, MPa
Melt flow index	ASTM 1238: 2013	190 °C /21.6 kg, g/10 min
Compression set	ISO 815 C: 2014	22 h at 70 °C, 120 °C 25% initial thickness, %

Rheological study

Rheology of the SEBS compound with different content of peroxide (0.5, 1, 2 and 4 wt.%) was taken (at a temperature of 180 °C for 3 minutes). For this purpose, a rheometer (Rubber Process Analyzer 2000 (RPA), ALPHA; American), was equipped. Compounds include SEBS and oil, peroxide and co-curing agent with 2 mm thickness were prepared by the roller at 50 °C.

Morphological study

For morphological study, TPV extruded samples were frozen for three minutes in liquid nitrogen. The test strips were removed and split in the middle, then gold spraying was applied to the sample cross sections.

Then, morphological investigation of the cross-sections was carried out using a SEM (ZEISS (LEO) 1450 VP, Germany).

Tensile strength @ 20% extension

Tensile strength at 20% extension refers to the stress a material can withstand when stretched to 20% of its original length. This value is typically determined through a tensile test, where a sample is pulled until it fractures, and the force and elongation are recorded at various points. The tensile strength at 20% extension is a specific point on the stress-strain curve, indicating the material's ability to resist deformation under load.

Crosslink density measurements

Test pieces with weight 0.5 g were used for testing. After immersion in 100 ml cyclohexane the samples were stored at 23 °C in the liquid. After another 72 h the swollen samples were weighed, dried and weighed again. Drying of the samples was performed at 23 °C for 48 h in order to remove the solvent. The relative gel content with respect to the initial mass m_0 was calculated from the difference between m_0 and m_d, where m_d is the mass after drying of the sample. Considering the density of the polymer ρ_p and the density of the solvent ρ_s, the mass m of the swollen sample was used to calculate the polymer volume fraction $Ø_P$ according to Equation (1) [19].

$$Ø_p = \frac{V_p}{\mathrm{V}} = \frac{1}{\left(1 + \frac{m - m_d}{m_d}\right) \cdot \frac{\rho_p}{\rho_s}} \tag{1}$$

The ratio of total volume V of the swollen sample and the volume V_P of the dry polymer sample is defined as the swell ratio Q (Equation 2).

$$Q = \frac{V}{V_P} \tag{2}$$

The reciprocal swell ratio 1/Q can be taken as a measure of the crosslink density. From the comparison of Equations (1) and (2) it becomes obvious that 1/Q is also identical with $Ø_p$.

Calculate the solvent extraction in percent as follows according to Equation (3) and gel content Equation (4) [19]:

Extract, %= weight lost during extraction/ weight of original specimen-weight of filler

$$\text{Extract, }\% = \frac{m_0 - m_d}{m_0} \times 100 \qquad (3)$$

$$\text{Gel content} = 100 - \text{percent extract.} \qquad (4)$$

RESULTS AND DISCUSSION

Rheological properties

To determine the appropriate peroxide concentration for the production of TPVs, we investigated the rheology of SEBS compounds with varying peroxide contents (0.5%, 1%, 2%, and 4% wt.%) as shown in Figure 1. A TAC co-curing agent was included in all formulations at twice the peroxide ratio. The rheological measurements were conducted at a temperature of 180 °C for 3 minutes, during which the half-life of the consumed peroxide was 1 minute. The results indicated that T90 was achieved with 4% peroxide, confirming that curing had occurred. However, this concentration is atypical for TPV production, as noted in previous studies [20,21]. The twin-screw extruder used in the compound preparation is capable of generating higher temperatures and shear rates, which are essential for producing fine rubber domains within the TPVs [22-24]. The increased shear rate elevates the melt temperature, thereby enhancing the curing process and allowing for the dispersion of rubber domains into smaller droplets within the plastic matrix [25-27]. It is important to note that during the rheological study, there was no applied shear force, resulting in a lack of correlation between the extruder data and the rheometer measurements.

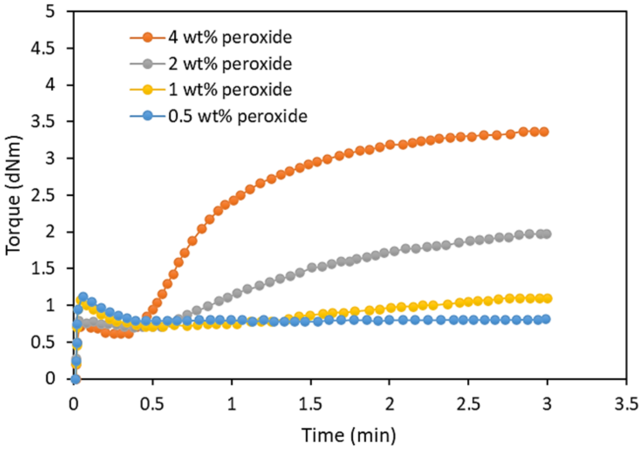

Figure 1. The rheology graph of the SEBS compounds prepared with different percentages of peroxide (0.5, 1, 2 and 4 wt.%).

Effect of peroxide

The curing of the TPV increased with the peroxide concentration within the studied range (0.5% to 1.2% wt.%). This led to improvements in tensile strength (Figure 2) and hardness (Figure 3), although elongation at break (Figure 2), MFI (Figure 4), and tear strength (Figure 3) decreased. The rise in CD, resulting from the higher concentration of crosslinking agents, contributed to improved elasticity in the TPV systems [14,28]. However, optimal elasticity was observed at specific curing agent concentrations; further increases in curing agent could not enhance CD due to saturation of the rubber phase [29,30]. Additionally, an increase in CD may facilitate the formation of smaller rubber particles within the TPVs [6,12], which can also contribute to improved elasticity [6,29].

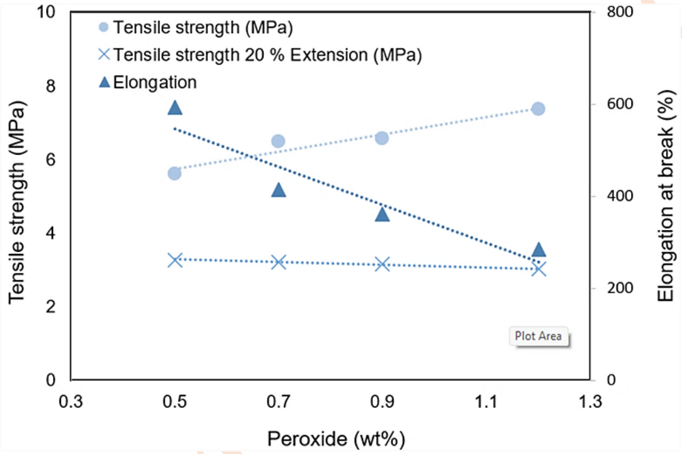

Figure 2. Effect of peroxide concentration on tensile strength and elongation at break of the prepared TPV.

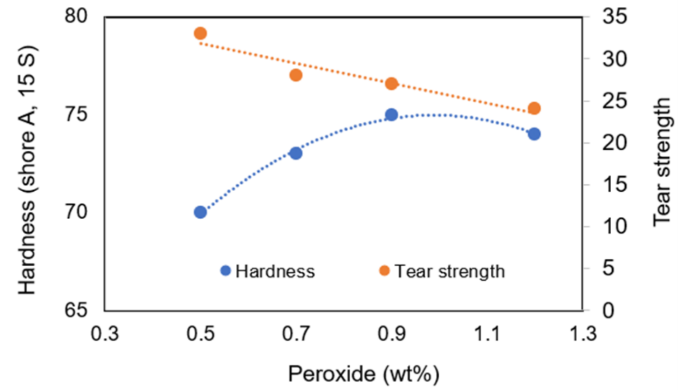

Figure 3. Effect of peroxide concentration on hardness (shore A) and tear strength of the prepared TPV.

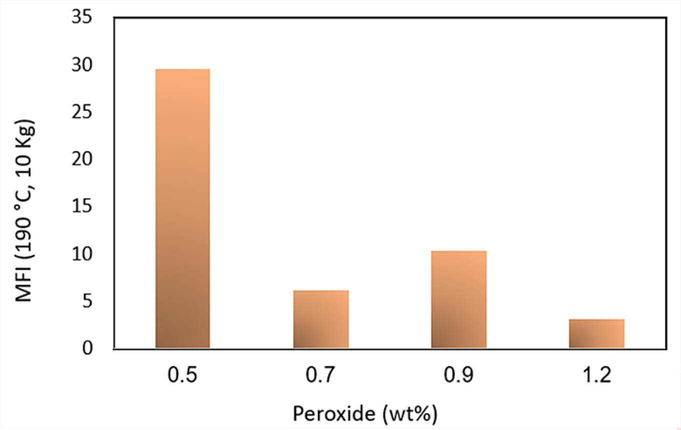

Figure 4. Effect of peroxide concentration on MFI of the prepared TPV.

The morphology of the prepared TPVs was examined using different peroxide concentrations (0.5% to 1.2% wt.%) as shown in Figure 5. Notably, the fracture surfaces displayed a rough texture, particularly in Figure 5 (D), where irregular structures suggest the presence of relatively large rubber domains within the TPVs. In contrast, the fracture surfaces observed in Figures 5 (A-C) exhibited a smoother appearance as the peroxide concentration decreased from 1.2% to 0.5%. This smoother surface morphology indicates the formation of finer rubber phase domains in the resultant TPVs with lower peroxide concentrations [4].

The compression set at low temperature (70 °C) showed little variation with increased crosslinking content. This can be attributed to both chemical and physical cross-links, which contribute to restoring elastic properties at lower temperatures. In contrast, at high temperatures, the plastic phase can aid in elastic recovery; however, this effect diminishes as the temperature rises [6]. Consequently, the compression set at high temperature (120 °C) decreased due to enhanced curing (Figure 6). A heat aging study was conducted on the prepared TPVs (Figure 7). The results revealed that both elongation and hardness exhibited their lowest values at 0.9% peroxide. Conversely, tensile strength decreased with increasing peroxide content. The density remained relatively constant at approximately 1.1 g/cm³ across varying peroxide concentrations, which was expected given the consistent formulation of the TPVs. The reduction and eventual disappearance of the melting point with higher curing agent concentrations is likely due to the disruption of the polymer's structural order resulting from cross-linking (Figure 8).

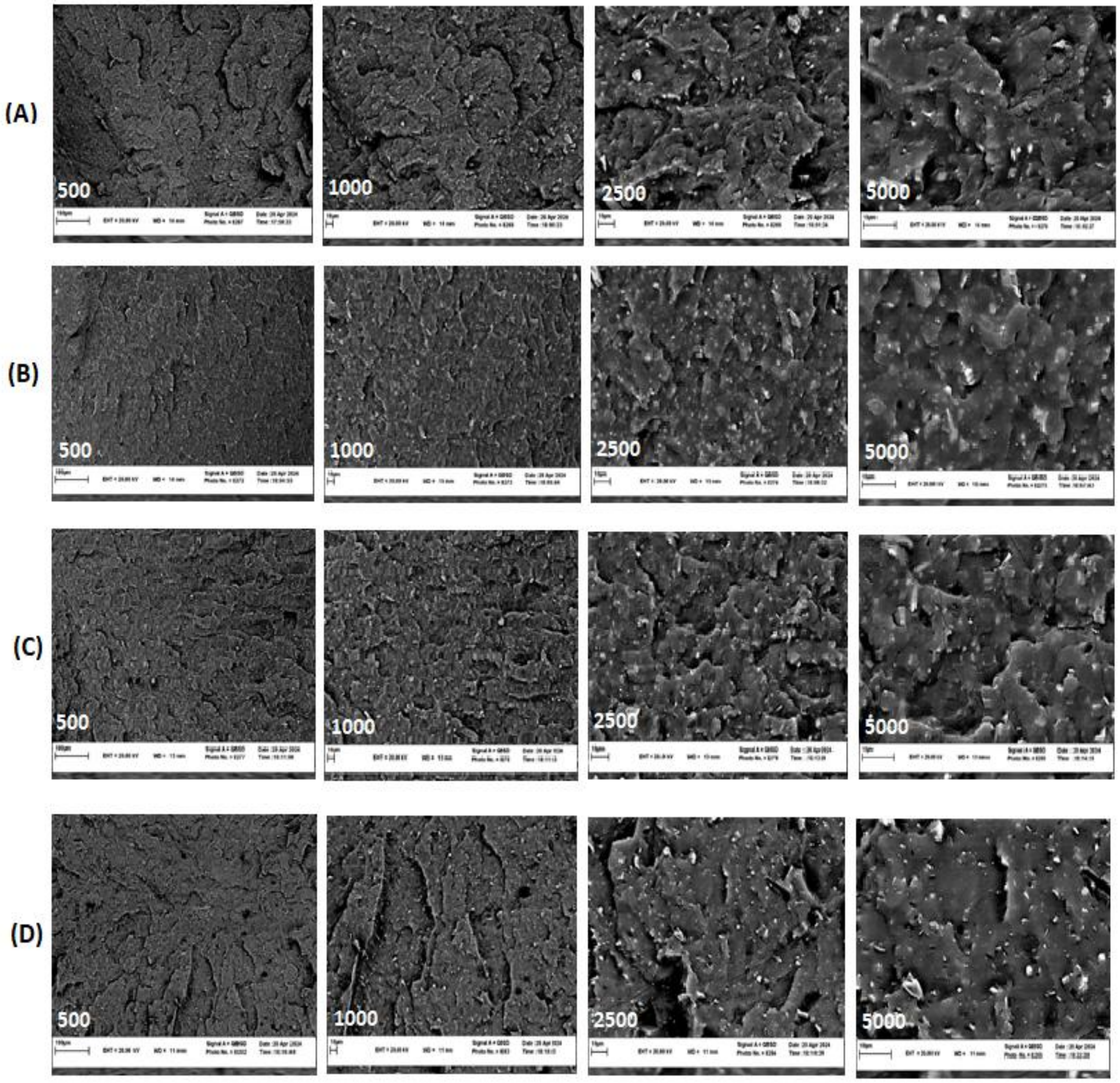

Figure 5. Fracture surfaces of dynamically vulcanized SEBS/LLDPE/PP TPVs: (A) 0.5 wt.%; (B) 0.7 wt.%; (C) 0.9 wt.% and (D) 1.2 wt.%.

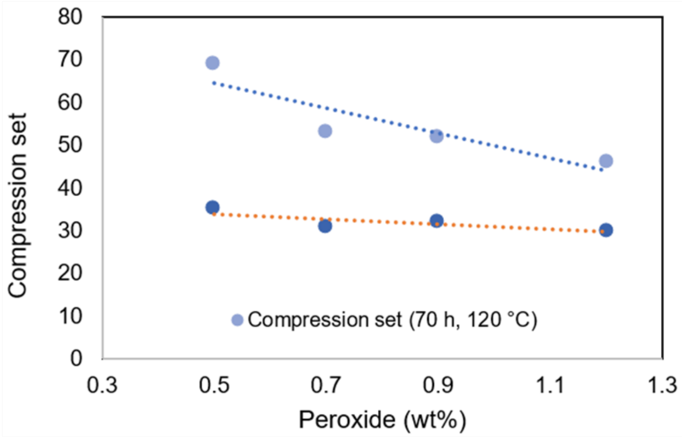

Figure 6. Effect of the peroxide concentration on compression set of the prepared TPV.

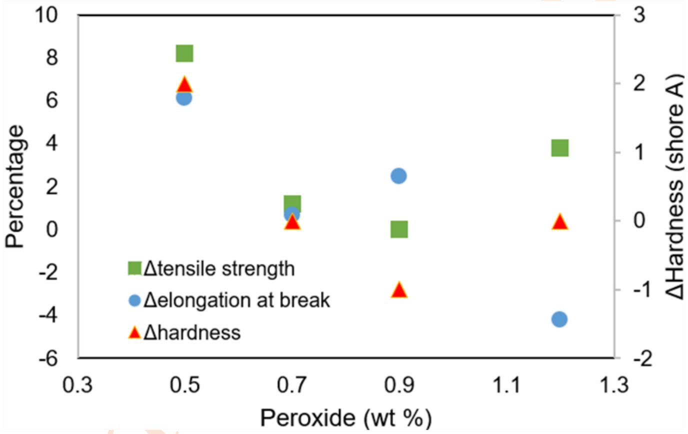

Figure 7. Effect of the peroxide concentration on heat aging of the TPV.

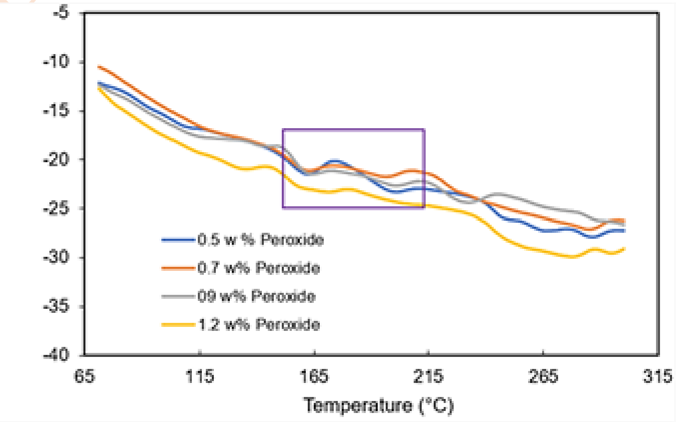

Figure 8. DSC thermograms; effect of the peroxide concentration on melting temperature of TPV samples.

Effect of co-curing agents

This study compared the effects of TAC and BMI as co-curing agents in the peroxide curing system of STPV. The results showed that using BMI as a co-agent significantly improved tensile strength, hardness, and elongation at break (increasing from 897% to 1082%) in the STPV (Table 3). Additionally, the heat aging study indicated that the compression set for STPV with TAC was slightly higher compared to that with BMI. The use of BMI also resulted in an increase in tensile stress at 20% extension, rising from 2.7 to 3.16 MPa. Notably, the co-agents did not significantly affect MFI.

Table 3. Comparison of TAC and BMI as a co-curing agent in the peroxide-cured of thermoplastic vulcanized (TPV).

Test	**TPV produced with TAC**	**TPV produced with BMI**
Tensile strength (MPa)	5.9	6.9
Elongation at break %	897	1082
Tensile strength (MPa) 20% Elongation	2.7	3.2
Tensile strength (MPa) 20% Extension	2.7	3.2
Tensile strength with aging (%, 85 °C, 7 day)	19.9	8.6
Elongation with aging (%, 85 °C, 7 day)	42.1	11.3
Hardness change (Shore A, 85 °C, 7 day)	-5	1
MFI (190 ˚C/ 5 kg, g/10 min)	6.8	6.8
MFI (190 ˚C/ 10 kg, g/10 min)	72.6	76.3
Hardness, shore A	50	57
Density (gr/ml)	1.1	1.1
Compression set (25%, 70 h, 120 °C, rest 120 °C)	50	49
Compression set (25%, 22 h, 70 °C, rest 70 °C)	38	38

Effect of dynamic vulcanization

Hydrogenated SEBS and PP-based uncrosslinked and dynamically crosslinked blends were prepared on an industrial scale using a co-rotating twin-screw extruder. The effect of peroxide curing on the properties of four different types of thermoplastic elastomer-based SEBS/PP was investigated (Table 4). SEM images revealed that the SEBS/PP/LLDPE TPE exhibited a smoother surface compared to the TPV of SEBS/PP/LLDPE (Figures 9 (A) and 9 (B)). This relatively smooth surface suggests the presence of fine rubber phase domains in the resultant TPE. In contrast, the TPV showed rough fracture surfaces (Figure 9 (B)), with irregular structures indicating the crosslinking of the rubber phase. The crosslink density and gel content measurement were performed for TPV, TPE & EPDM rubber. Crosslink density and gel content of TPV are close to that of EPDM rubber (fully crosslinked) and higher than that of TPE (without crosslinking) (Table 5).

Table 5. Crosslink density & gel content of TPV, TPE & EPDM rubber.

Sample	**Crosslink density (mol/cm^3) or Swell ratio**	**Gel content (%)**
Rubber EPDM, 70 shore A	0.233	79
TPV	0.265	70
TPE	0.204	43

Table 4. Effect of dynamic vulcanization on properties of four different types of thermoplastic elastomer-based SEBS/PP.

Test	**TPE$_1$→TPV$_1$**	**TPE$_2$→TPV$_2$**	**TPE$_3$→TPV$_3$**	**TPE$_4$→TPV$_4$**	**TPE→TPV**
Δ*Tensile strength (MPa)	- 0.1	- 1.2	- 0.2	- 2.2	↓
ΔTensile strength 20% Extension (MPa)	- 0.1	- 0.7	- 1.1	-1.2	↓
ΔElongation at break %	-235	-530	-597	-789	↓
ΔTensile strength with aging (MPa, 85 °C, 7 day)	13.1	9.8	5.4	1.6	↑
ΔElongation at break with aging (%, 85 °C, 7 day)	31.2	15.2	-8.2	-4.7	--
ΔHardness with aging (Shore A, 85 °C, 7 day)	-1	0	1	0	--
ΔMFI (190 ˚C/ 10 kg)	22.5	10.2	-7.88	- 6.02	---
ΔHardness (shore A)	5	5	3	2	↑
ΔDensity (gr/ml)	- 0.002	0	- 0.001	0	---
ΔCompression set (70 h, 120 °C) rest 120 °C	2	-9	-34	-27	↓
ΔCompression set (22 h, 70 °C) rest 70 °C	5	5	0	2	↑

* Δ (Property)= Property of TPE- Property of TPV

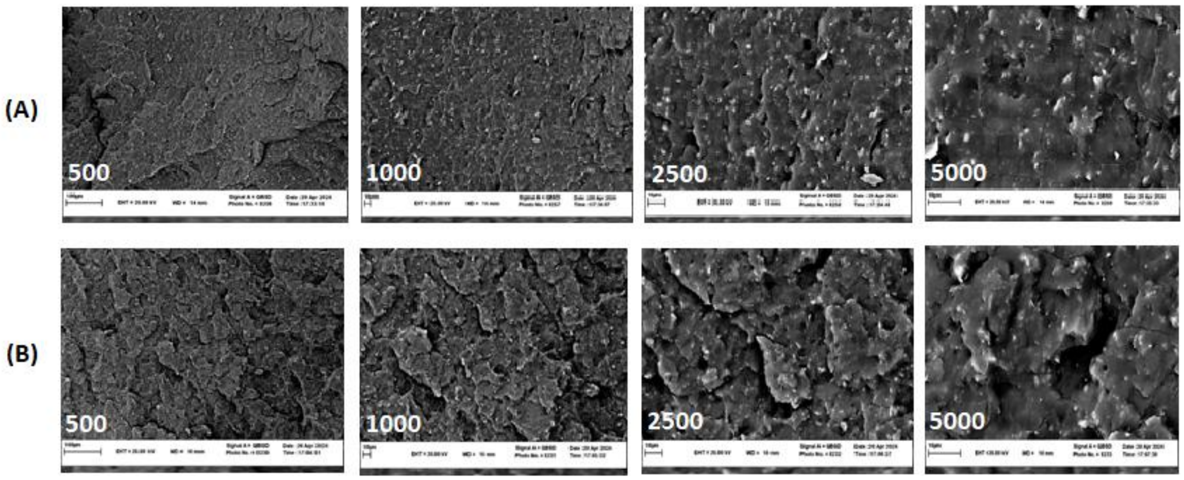

Figure 9. Fracture surfaces of the TPEs and TPVs based SEBS/LLDPE/PP.

The results indicate that both the tensile strength and tensile strength at 20% extension of the TPVs did not change significantly under peroxide curing. When tensile stress is applied to the polymers, the entanglements of the polymer chains are opened, stretched, and aligned. The cross-links formed between the polymer chains partially impede this process, resulting in an increased force requirement for stretching [29]. However, the increase in CD due to peroxide curing was likely not substantial enough to produce noticeable changes. The slight decrease observed may be attributed to disruptions in the structural order and morphology of TPE caused by the curing process. Additionally, the higher tensile strength observed after aging supports this behavior, potentially reflecting enhanced curing. A heat aging study was conducted on the SEBS/PP/LLDPE blend both before and after peroxide curing. The cross-links generated through dynamic curing were sufficient to reduce elongation, MFI, and compression set at high temperature (120 °C). At low temperature (70 °C), the compression set increased due to curing, as both chemical and physical cross-links contribute to restoring elastic properties. However, as previously mentioned, the structural order of the thermoplastic elastomer may have been compromised due to curing, leading to a reduction in physical cross-links, which in turn contributed to the increased compression set at low temperature. At high temperature, the role of chemical cross-links in restoring elastic properties outweighs that of physical cross-links, resulting in a reduced compression set due to curing (Figure 10). The hardness of the compound was affected by the cross-links at 85 °C, with increases likely due to curing. In contrast, the density of the material remained unchanged.

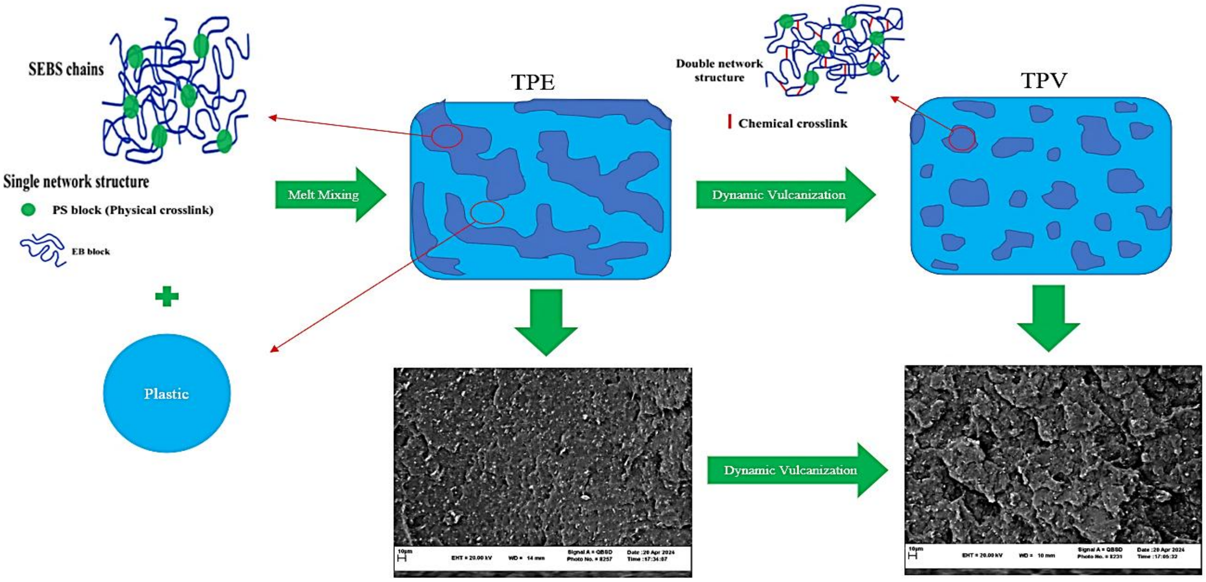

Figure 10. schematic illustration of the chemical/physical interactions between materials.

CONCLUSION

The STPV was prepared on an industrial scale using a co-rotating twin-screw extruder, incorporating PP and LLDPE as the continuous phases, and crosslinked SEBS as the dispersed phase. A rheological study of the SEBS compounds was conducted with varying peroxide concentrations (0.5%, 1%, 2%, and 4% wt.%)

at a temperature of 180 °C for 3 minutes. The presence of T90 at 4% peroxide indicates successful curing. However, the twin-screw extruders operated at higher temperatures and shear rates than the rheometer, leading to discrepancies between the extruder and rheometer data, as anticipated. Increasing the peroxide content from 0.5% to 1.2% wt.% resulted in an increase in tensile strength from 5.6 to 7.4 MPa and hardness up to 74 Shore A. Conversely, elongation at break decreased from 592% to 284%, while MFI dropped from 29.6 to 3.14 g/10 min at 190 °C under 10 kg load, and tear strength decreased from 35 to 30 kg f/cm. The compression set at low temperature (70 °C) remained relatively stable, but at high temperature (120 °C), it decreased from 69% to 46%, likely due to the curing process. Heat aging studies on the TPVs, prepared with varying peroxide content (0.5% to 1.2%), revealed that both elongation at break and hardness exhibited minimum values at 0.9% peroxide, while tensile strength declined with increasing peroxide content. The elevated curing associated with higher peroxide concentrations resulted in the disappearance of the melting point for the prepared TPVs. Additionally, the effects of thermoplastic elastomer co-curing agents, specifically TAC and BMI, were compared in the preparation of the STPV. Results showed that using BMI as a co-agent increased tensile strength from 5.9 to 6.9 MPa, hardness from 50 to 57 Shore A, and elongation at break from 897% to 1082%. In contrast, the STPV with TAC exhibited a slight increase in heat aging and compression set compared to those with BMI. Dynamic vulcanization studies of the SEBS/LLDPE/PP TPV blends indicated that the tensile strength and density of the dynamically crosslinked blends remained largely unchanged under peroxide curing. Furthermore, heat aging improved the performance of the SEBS/LLDPE/PP blend both before and after peroxide curing.

FUNDING DECLARATION

This work was supported by Shahid Beheshti University, Tehran, Iran (Grant numbers [9010923]

and the technical equipment and chemical support of Pouya Gostar Khorasan Co. of PartLastic industrial Group. Co. Iran).

CONFLICTS OF INTERESTS

The authors have no conflicts of interest to declare that are relevant to the content of this article.

AUTHOR CONTRIBUTION DECLARATION

Mehri Dana wrote the manuscript and performed the experimental work. Professor Mohammad Reza Nabid and Professor Gholam Hossein Zohuri supervised and edited the manuscript. Saeid Asadi Shahidi, Sohail Yazdanbakhsh reviewed the manuscript.

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