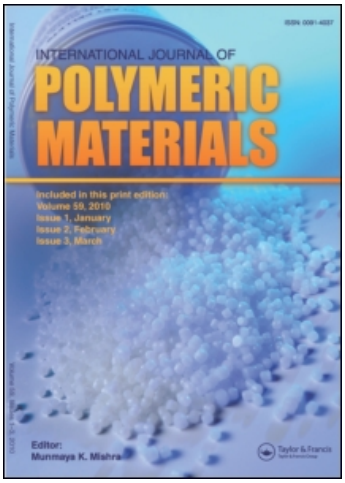

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Dynamically vulcanized thermoplastic elastomer (TPE) blends were first described by Fisher. In the 1960s, the usage of TPE was low. However, the consumption of TPE is increasing today. Due to hard and soft polymer phases in its structure, TPE replaced a lot of materials. TPE materials are preferred today due to their good thermal properties, oxidation resistance, transparency, adhesion, compatibility with other polymers etc. As a result of the studies that were done in 1975, TPV—vulcanized thermoplastic elastomers were developed. In this study, TPV elastomers were produced by forming crosslinks with peroxide from different ratios, of EPDM and PP. Mixing was done with twin screw extruder. After that yield and tensile strength, the modulus of elasticity, % elongation, Izod impact strength, hardness, Melt Flow Index (MFI), Vicat Softening Point, Heat Deflection Temperature (HDT), and density of crosslinks were determined. Thermal transition temperatures and microstructure were determined with DSC and SEM, respectively.

Keywords: dynamic vulcanization, ethylene-propylene-diene terpolymer, peroxide, polypropylene, thermoplastic elastomer

INTRODUCTION

Dynamically cured TPE blends were first described by Fischer [1,2] and developed by Coran and Patel [3–5] and since then have been widely used in the rubber and plastic industries. The outstanding properties of these materials are mainly attributed to their specific microstructure, which consists of a continuous plastic matrix with

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tiny rubber particles dispersed throughout the matrix. This enables the blend to be melt processed even though the particles are crosslinked. Both the mechanical and rheological properties of these materials are, therefore, critically dependent on their morphology [6–8]. Much work has been done that deals with the mechanical and rheological properties of dynamically cured TPEs [7–11]. Also, a few studies have been carried out, mainly to characterize the microstructure of dynamically cured TPEs based on different rubber plastic blend systems [6,9,12–15]. Xia et al. added some materials to form crosslinks in different ratios into EPDM/PP mixtures and investigated the changes in mechanical properties, morphology, and rehology [12]. Naskar et al. investigated the effect of different type of peroxides on the dynamic vulcanized EPDM/PP mixture [13]. Jacob et al. investigated the effect of adding recycled EPDM into EPDM/PP thermoplastic composition [14]. Out of them, Ghosh et al. searched the effect of vulcanization technique on the morphology of polymers and on the mechanical properties [7]. Similarly, Alagar et al. studied the morphology and mechanical properties of EPDM–g-vinyloxiaminosilan (VOS)/LLDPE mixture [15] and Kim et al. investigated the properties of dynamic vulcanize EPDM/LLDPE mixtures [16].

As seen from the studies on EPDM/PP mixtures mechanical and thermal properties as well as morphology of vulcanized thermoplastic elastomers could be changed by changing vulcanization technique, amount of crosslinkers, and type of crosslinkers.

In this study, crosslinks were formed between peroxide and EPDM/PP. Based on the PP and peroxide ratio in dynamic vulcanized thermoplastic elastomers, changes in mechanical and thermal properties and morphology were identified. By changing PP ratio, five groups of EPDM/PP thermoplastic elastomers were prepared. From these five groups, four minor groups were formed by changing peroxide ratio. The ratios of EPDM/PP dynamically vulcanized thermoplastic elastomers that were formed are given in

TABLE 1 Composition of Dynamically Vulcanized EPDM/PP Blends

Groups	EPDM (phr)	PP (phr)	PEROXİDE (phr)	CaSt (phr)
1	39.90	59.90	0.0	0.2
2	39.62	59.43	0.75	0.2
3	39.50	59.30	1.00	0.2
4	39.41	59.14	1.25	0.2

TABLE 2 Characteristics of the Materials Used

Properties	EPDM[17]	PP[18]	PEROXİDE[19]	CaSt[20]
Commercial name	Nordel	Isplen	Peroxan DC	Calcium Stearate
Type	IP 4770 R	PB 150 G2M	Dicumyl Peroxide	
Peroxide ratio (wt%)			98	
Density (g/cm^3)	0.87	0.91		
MFI (g/10 min)		11.30 (230°C–2.16 kg)		
Ash content (%)				9.2–10.2
Bending strength (MPa)		735–784		
Yield strength (MPa)		29–34		
Hardness (Shore A)	88			
Elongation at break (%)		600–700		
Tm (°C)	+42			
Tg (°C)	−45			
Ethylene ratio (wt%)	70			
Propylene ratio (wt%)	25			
ENB ratio (wt%)	5			
Appearance	Granule	Granule	Powder	Powder
Interaction temperature (°C)			+75	
Crystallization (%)	13			

ENB: Ethylene Norbornene; CaSt: Calcium Stearate.

Table 1 and the properties of materials used in this study are given in Table 2.

EXPERIMENTAL

EPDM, also called Norden, was used in this study. This EPDM was produced with 4770 R IP number by DuPont (Germany). Its density is 0.87 g/cm^3. Its composition is 70 wt% ethylene, 25 wt% propylene

TABLE 3 Extrusion Conditions Used in the Preparation of Dynamically Vulcanized EPDM/PP Blends

Parameter	Group 1	Group 2	Group 3	Group 4
Extrusion temperature (°C)	205–160	180–150	180–150	180–150
Torque (%A)	86	63	65	65
Screw speed (Rpm)	350	300	300	300
Capacity (Kg/h)	85	65	65	65

TABLE 4 Injection Conditions Used in the Preparation of Dynamically Vulcanized EPDM/PP Blends

Parameter	Value
Injection temperature (°C)	50–190
Injection pressure (Bar)	70–80
Dwell time in mold (s)	15
Injection speed (mm/s)	70
Mold temperature (°C)	50

and 5 wt% ENB. PP used in this study was produced by Repsol Company (Spain) with Isplen trade name and PB 150 G2M code number. Its density is 0.91 g/cm^3 and MFI value is 11.3 g/10 min. (230°C – 2.16 kg). Elongation at fracture is between 600–700% and yield strength is about 300–350 kg/cm^2. In order to form a crosslink, Dicumyl Peroxide was used. Dicumyl Peroxide was produced by Pergan Company (Germany) with Peroxan DC trade name. Wt% of peroxide is about 98. Interaction temperature is about 75°C. As slip agent, calcium stearat produced by Baerluer Company (Germany) was used. EPDM, PP, and CaSt were mixed by Saray Mixer (Saray Machine Co., Istanbul, Turkey), having two vertical blades, for 5 min at 1400 rpm. After that, samples were mixed with a twin screw extruder (Maris America Corporation, Baltimore, USA). Extrusion conditions are given in Table 3. In order to determine mechanical properties of dynamically vulcanized polymer mixture, tensile samples were obtained by injection molding. Injection conditions are given in Table 4.

Test samples of the granulated polymeric blends were made in Arburg Injection Machine (Arburg GmbH Co., Lossburg, Germany).

TABLE 5 Mechanical Properties of Dynamically Vulcanized EPDM/PP Blends

Test	Unit	Standard	Group 1	Group 2	Group 3	Group 4
Strength at break	MPa	ASTM D412	13	12	11	13
Elongation	%	ASTM D412	400	500	480	530
Permanent Deformation	% (70°C – 24 h)	ASTM D395	77	74	62	81
Hardness	Shore D	ASTM D2240	51	51	48	53
İzod Impact Strength	kJ/m^2(–30°C)	ASTM D256	Not broken	Not broken	Not broken	Not broken

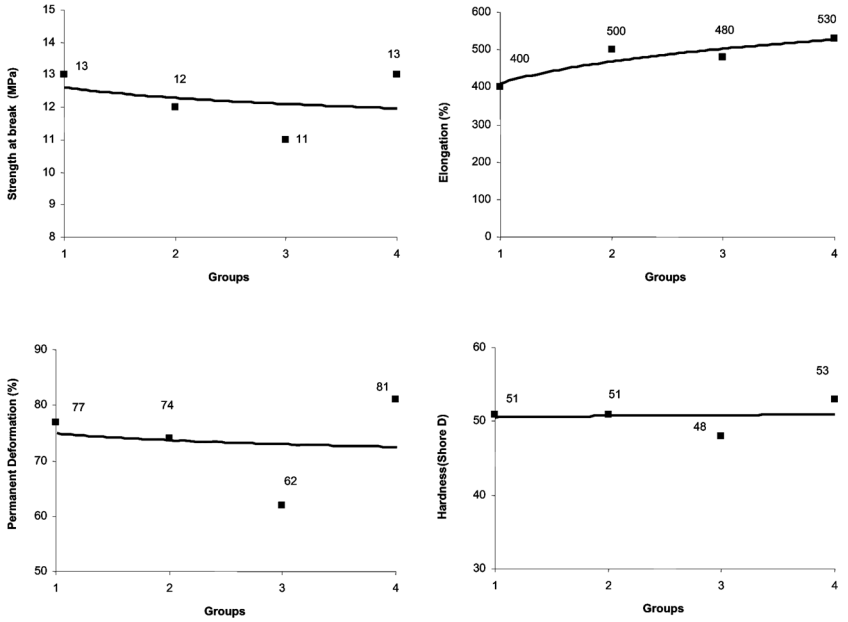


FIGURE 1 Mechanical properties of dynamically vulcanized EPDM/PP blends.

To investigate fracture behavior, Izod impact test was done at −30°C according to ASTM D256 standard with Ceast impact test device (Ceast Spa, Pianezza, Italy). Flow test of all the mixtures was done according to ISO 1133 standard with Ceast MFI device. Thermal transition temperatures were determined by Universal V2.6D DSC (TA Instruments, New Castle, USA). Tensile test was done according to ASTM D 412 standard by Zwick Z010 (Zwick GmbH, Ulm, Germany) testing machine. In tensile tests, 5 mm/min pulling speed

TABLE 6 Thermal Properties of Dynamically Vulcanized EPDM/PP Blends

Test	Group 1	Group 2	Group 3	Group 4
MFI (230°C 2.16 kg) g/10 min.	1.3	0.8	2.8	2.2
EPDM Tg (°C)	−36.88	—	—	−56.74
EPDM Tm (°C)	44.92	—	—	43.48
PP Tg (°C)	—	—	—	—
PP Tm(°C)	168.22	—	—	166.68

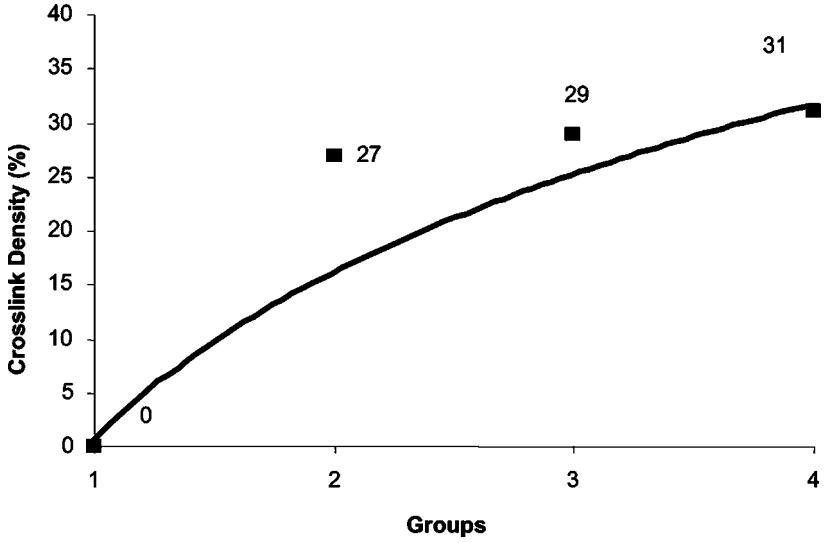


FIGURE 2 Crosslink densities of the dynamically vulcanized EPDM/PP blends.

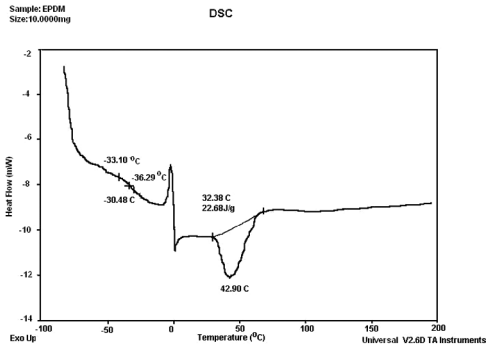
was applied. Microstructure was determined by using JSM-T33O JOEL (Joel, Peabody, MA) SEM at 10 kV. Samples were coated with gold about 40 Å in thickness. Hardness test were done according to ASTM D2240 standard with Zwick hardness measurement device. To determine the degree of Vulcanization, permanent deformation test was conducted according to ASTM D 395 by Dizayn Group's labs (Istanbul, Turkey) at 70°C for 24 h of idle time. Crosslink density test was done according to ASTM D 6338–03 standard.

RESULTS

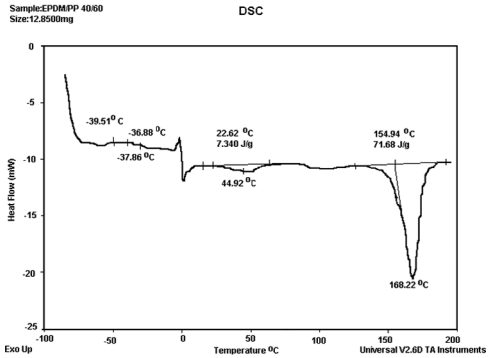
Fracture strength, elongation at fracture, amount of plastic deformation, hardness, and Izod impact strength of EPDM/PP dynamically vulcanized polymer mixtures are given in Table 5 and related graphs are given in Figure 1.

Thermal values obtained experimentally of EPDM/PP dynamically vulcanized polymer mixtures are given in Table 6. Density of crosslinks is shown in Figure 2 and DSC curves are given in Figure 3.

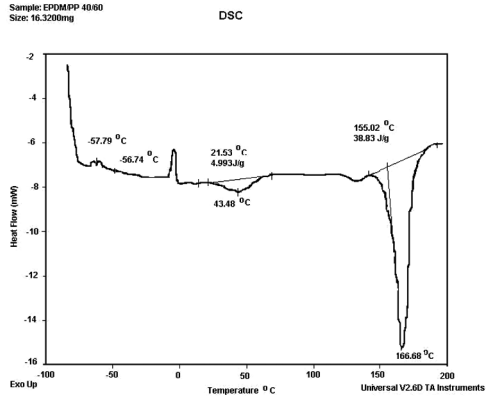
SEM micrographs of fracture surfaces of the Dynamically Vulcanized EPDM/PP Blends are given in Figure 4.



(a) EPDM



(b) Group 1



(c) Group 4

FIGURE 3 DSC Curves for dynamically vulcanized EPDM/PP blends.

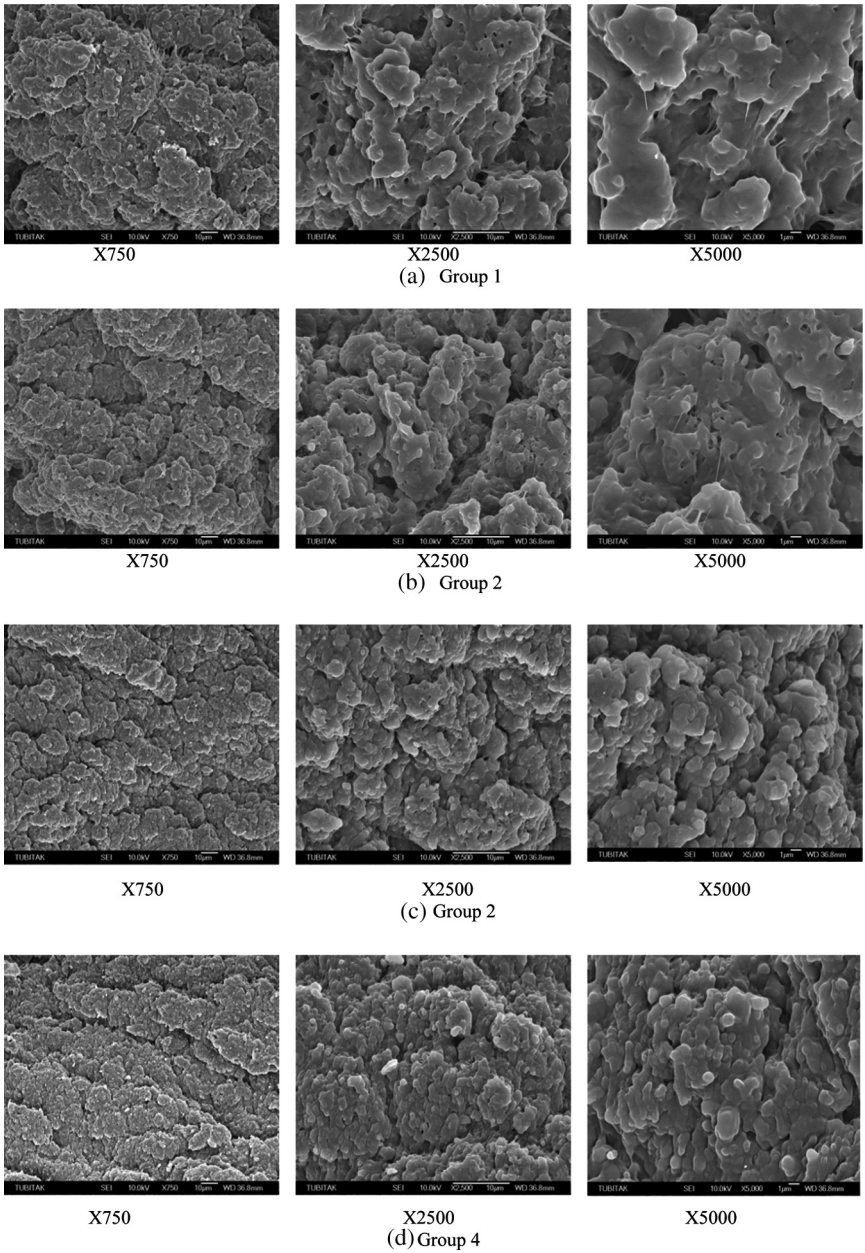


FIGURE 4 SEM micrographs revealing the appearance of fracture surfaces of the dynamically vulcanized EPDM/PP (60/40) blends.

CONCLUSIONS

When mechanical properties of EPDM/PP dynamically vulcanized blends were investigated, it was seen that fracture strength and amount of permanent deformation were decreased by increasing the percent of peroxide. Percent elongation at fracture was increased. Samples tested with Izod impact strength did not show fracture behavior at $-30^{\circ}C$. A slight decrease was determined on the EPDM/PP dynamically vulcanized blends at the melt flow index experiment of Group 2 at $230^{\circ}C$ and 2.16 kg. Increasing the amount of peroxide in EPDM/PP dynamic vulcanized blends, increased the density of crosslinks. Based on the DSC results, increasing the amount of peroxide in EPDM/PP dynamically vulcanized blends decreased, the melting and glass transition temperature of EPDM and the melting temperature of PP was decreased. From the SEM analysis, increasing the peroxide resulted in increasing interphase adhesion.

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