Recycling of EPDM Waste. II. Replacement of Virgin Rubber by Ground EPDM Vulcanizate in EPDM/PP Thermoplastic Elastomeric Composition

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ABSTRACT: Virgin ethylene propylene diene monomer (EPDM) rubber in a thermoplastic elastomeric blend of polypropylene (PP) and EPDM rubber was substituted by ground EPDM vulcanizate of known composition, after which the mechanical properties of the raw EPDM/waste EPDM/PP blends were determined. The ratio of the rubber content in the waste EPDM (r-W-EPDM) to the raw EPDM (R-EPDM) in the blends was varied from 0 : 100 to 45 : 55. Attempts to replace higher amounts (>45%) of R-EPDM by W-EPDM failed because of processing difficulty. Although a drop in mechanical properties of the blends was observed at lower loadings of W-EPDM, the properties showed improvement at intermediate W-EPDM loadings. The R-EPDM/W–EPDM/PP blends were found to be reprocessable. © 2001 John Wiley & Sons, Inc. J Appl Polym Sci 82: 3304–3312, 2001

Key words: recycling; EPDM waste; PP/EPDM blends; thermoplastic elastomer

INTRODUCTION

The drawbacks underlying the disposal of waste rubbers by methods such as landfills, pyrolysis, and incineration encouraged further research on the recycling technology. Reutilization of powdered waste rubber in mixtures with virgin rubbers[1–7] and plastics[8–12] is an attractive alternative. A recent advancement in this field is the use of ground rubber tire (GRT) in the preparation of thermoplastic elastomeric blends based on rubbers and thermoplastics. Osborn[13] studied the feasibility of using activated tire rubber as a modifying ingredient in thermoplastic elastomers such as Santoprene. Baker et al.[14–17] reported the use of functional polymers as compatibilizers in enhancing the properties of GRT–polyolefin blends. Al-Malaika and Amir[18] found that half of the natural rubber (NR) can be replaced by reclaimed rubber in a thermoplastic elastomeric blend of NR/polypropylene (PP) without adversely affecting the mechanical properties of the blend. Preparation of thermoplastic elastomers consisting of ultrasonically devulcanized GRT and PP was also previously reported.[19] Apart from the tire rubbers, other automotive rubbers such as ethylene propylene diene monomer (EPDM) rubber also contribute to the disposal problems.

The present study deals with the replacement of virgin EPDM by EPDM vulcanizate powder of known composition, abbreviated as W-EPDM, in 70/30 EPDM/PP blend, which is known to behave as a thermoplastic elastomer.[20] Naskar et al.[21] reported replacement of 50% of EPDM by GRT in a thermoplastic elastomeric blend of EPDM/poly(ethylene-*co*-acrylic acid) blend. In the present study, the ratio of W-EPDM to raw EPDM (R-

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Table I Details of the Materials Used

Material	Trade Name	Source	Characteristics
EPDM	Royalene 521	Uniroyal Chemical Co., Naugatuck, CT	ML(1+4)125, 29; E/P ratio 52/48; % ENB 4.9
Polypropylene	Profax 7823	Himont USA Inc., Wilmington, DE	Copolymer with 5% ethylene; sp gr 0.88–0.92; MFI 0.4 (ASTM D1238)
Dicumyl peroxide	Polydispersion DCP-60 (Dicup-R)	Rhein Chemie, Mannheim, Germany	DCP/4 methoxy phenol/ nonhazardous binder
W-EPDM	Waste rubber molded and ground in the laboratory		

EPDM) was varied, keeping the total rubber content constant, to study the effect of replacement of EPDM by waste rubber on the properties of 70/30 EPDM/PP blend.

EXPERIMENTAL

Materials

The details of the materials used are given in Table I. As described in a companion investigation,[22] EPDM vulcanizate of known composition and physical properties, was powdered after aging, using a mechanical grinder to obtain highly aggregated, chainlike waste rubber particles (W-EPDM).

Blending of Plastic and Rubber

The compositions selected for the study are given in Table II. The thermoplastic elastomeric composition of 70/30 EPDM/PP blend was chosen. The ratio of the rubber content of the powdered waste rubber (r-W-EPDM) to raw EPDM (R-EPDM) was varied from 0 : 100 to 45 : 55, keeping the total rubber content constant. The amount of DCP used for the dynamic vulcanization of the blends was 1.0 phr based on the total rubber content in the blend in all the compositions. Because 60% DCP was used, the actual amount of DCP used in the compositions for 70 parts of rubber was 1.2 parts. This means that 70 parts of rubber contains 0.7 parts of 100% DCP or, in other words, 1.0 phr (parts per hundred rubber) of 100% DCP.

The mixing schedule used is shown in Table III. Rubber was mixed with DCP at 70°C and then blended with PP at 180°C in a Brabender Plasticorder PLE 330 (Brabender OHG, Duisburg, Germany).

Determination of Physical Properties of the Blends

Compression molding of the blends was done in an electrically heated hydraulic press at 200°C and 5 MPa pressure and then the platens were cooled under pressure. The tensile properties, tear strength, and tension set of the molded samples were determined as per ASTM D412-98a,

Table II Compositions Showing Variations of W-EPDM Loadings at Constant Rubber-to-Plastic Ratio of 70 : 30 (Parts by Weight)

	Mix Number						
Ingredients	F_0	F_{10}	F_{20}	F_{30}	F_{35}	F_{40}	F_{45}
R-EPDM	70.0	63.0	56.0	49.0	45.5	42.0	38.5
W-EPDM[a,b]	0.0	22.7	45.4	68.0	79.4	90.7	102.1
	(0)	(7.0)	(14.0)	(21.0)	(24.5)	(28.0)	(31.5)
PP	30.0	30.0	30.0	30.0	30.0	30.0	30.0
DCP[c]	1.2	1.2	1.2	1.2	1.2	1.2	1.2

[a] Values in parentheses are the rubber content in W-EPDM (i.e., r-W-EPDM).

[b] Ratios of r-W-EPDM to R-EPDM in different mixes are as follows (total rubber content in each case being constant at 70 parts): F_0, 0 : 100, F_10, 10 : 90, F_{20}, 20 : 80; F_30, 30 : 70; F_{35}, 35 : 65; F_{40}, 40 : 60, F_{45}, 45 : 55.

[c] Dicumyl peroxide. 1.2 parts of 60% DCP in 70 parts is equivalent to 1 part of 100% DCP in 100 parts of rubber (i.e., 1.0 phr).

Table III Mixing Schedule

Description	Mixing Equipment/Temperature	Mixing Time/rpm
Stage I Mixing		
1. Rubber masticated	Brabender Plasticorder (70°C)	1. 2 min at 30 rpm
2. Ground rubber added		2. 2 min at 60 rpm
3. DCP added		3. 2 min at 60 rpm
4. Sheeted out and cut to strips	Two-roll mill (25°C)	—
Stage II Mixing		
5. PP melted	Brabender Plasticorder (180°C)	5. 0 rpm
6. The rubber compound strips added at 50 rpm		6. Kept at 70 rpm until the torque stabilized
7. Blend sheeted out	Two-roll mill (25°C)	—

D624-98, and D412-98a, respectively, as discussed in the companion study.[22] Toughness of the blends was calculated from the area under the stress–strain curve.

SEM Studies of the Blends

Blend Morphology

Cryogenically broken ends of the molded samples were acid etched (boiling concentrated HNO_3, 30 min) to remove the rubber phase, washed repeatedly with water, and dried at 50°C. The etched samples were gold coated and examined under scanning electron microscope (JEOL JSM 5800 SEM; JEOL, Peabody, MA).

Fractography

The fractured surfaces of the dumbbell-shaped samples after tensile testing were gold coated and examined under scanning electron microscope (JEOL JSM 5800 SEM).

Dynamic Mechanical Analyses

The dynamic mechanical properties were determined using DMTA MK-II (Polymer Laboratory, Loughborough, UK) at a frequency of 1 Hz and heating rate of 2°C/min. The temperature range chosen was from −80°C to 120°C.

X-ray Diffraction

X-ray studies of PP and the blends were performed with Philips X-ray diffractometer type PW 1710 (Philips, The Netherlands) using iron-filtered CoK_α radiation from a Philips X-ray generator (type PW 1729). The accelerating voltage and the current were 40 kV and 200 mA, respectively. The ratio of the total area of the crystalline peaks

Table IV Formulations for Studying the Filler Effect and Curative Migration in the Rubber Phase

	Mix Number							
Ingredient	F'_{20}	F'_{40}	FG_{20}	FG_{40}	FG'_{20}	FG'_{40}	F''_{20}	F''_{40}
R-EPDM	56	42	56	42	56	42	70	70
W-EPDM	45.4	90.7	—	—	—	—	—	—
(r-W-EPDM)[a,b]	(14)	(28)	—	—	—	—	—	—
W-EPDM(G)	—	—	15.9	31.8	15.9	31.8	—	—
(r-W-EPDM)[a,c]	—	—	(14)	(28)	(14)	(28)	—	—
GPF carbon black	—	—	—	—	—	—	16.8	33.7
Oil	—	—	—	—	—	—	9.8	19.6
Paraffin wax	—	—	—	—	—	—	1.4	2.8
Factice	—	—	—	—	—	—	1.4	2.8
PP	30	30	30	30	30	30	30	30
DCP	—	—	1.2	1.2	—	—	1.2	1.2

[a] Values in parentheses are the rubber content.
[b] W-EPDM contains fillers along with other additives.
[c] W-EPDM(G) is similar to W-EPDM, but does not contain fillers. G stands for gum; W stands for waste.

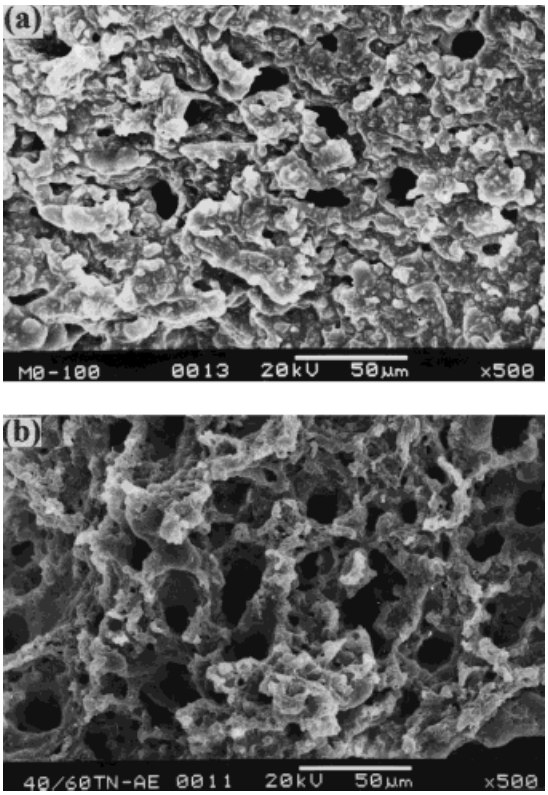

Figure 1 SEM photomicrographs of acid-etched samples: (a) F_0; (b) F_{40}.

to the total area between the curve and the baseline was taken as a measure of the crystallinity of the samples.

Study on Effect of Fillers and Curatives in W-EPDM on the Blend Properties

To study the effect of fillers and unreacted curatives of W-EPDM on the properties of the EPDM/PP blends, compounds based on the formulations (as given in Table IV) were prepared. Rheographs of the rubber compounds were taken at 180°C using a Monsanto rheometer MDR-2000 (Monsanto, St. Louis, MO). Physical properties of the molded blends were determined as per ASTM standards. The control EPDM compound [W-EPDM(G)], without filler and plasticizers, was also prepared and its properties determined.

Reprocessability of the Blends

To ensure the thermoplasticity of the blends, the molded samples were remixed in the Brabender Plasticorder PLE 330 at 180°C for 2 min and sheeted out and remolded. Samples were punched out from the molded sheets and testing was done as per ASTM standards.

RESULTS AND DISCUSSION

SEM Study on Blend Morphology

SEM photomicrographs of the acid-etched samples are given in Figure 1. The blends showed the characteristic thermoplastic elastomeric morphology, consisting of finely dispersed rubber

Table V Effects of W-EPDM on Mechanical Properties of 70 : 30 Rubber : Plastic Blends[a]

	Mix Number[b]						
Property	F_0	F_{10}	F_{20}	F_{30}	F_{35}	F_{40}	F_{45}
Tensile strength, MPa	4.83	4.30	4.20	4.20	4.87	5.50	6.10
	(+6)	—[c]	(0)	(+8)	—[c]	(+8)	—[c]
Elongation at break, %	220	183	215	216	205	239	249
	(+8)	—[c]	(−5)	(+4)	—[c]	(+3)	—[c]
Modulus at 100% elongation, MPa	3.51	3.77	3.32	2.99	3.62	3.79	3.89
	(+3)	—[c]	(+3)	(+8)	—[c]	(+9)	—[c]
Tear strength, kN/m	30.2	25.8	26.4	24.5	29.3	29.0	35.0
	(+3)	—[c]	(+3)	(+8)	—[c]	(−4)	—[c]
Tension set at 100% elongation, %	14	26	18	14	14	14	14
	(0)	—[c]	(0)	(0)	—[c]	(0)	—[c]
Toughness, J/m^2	3323	2794	3079	3136	3093	4039	4516
Hysteresis loss after first cycle, $(J/m^2) \times 10^{-6}$	0.078	0.101	0.078	0.084	0.084	0.084	0.088

[a] The values in parentheses are the percentage increase (+) or decrease (−) in properties after aging at 70°C for 48 h.
[b] The subscripts in the mix number represent the percentage of rubber hydrocarbon from W-EPDM in total rubber.
[c] Aging not done.

phase in a continuous plastic phase. The continuity of PP provides the necessary thermoplasticity and strength to the blends. As reported earlier by Isayev et al.,[19] the net result of dynamic vulcanization could be the formation of small gel particles of rubber in a matrix of PP.

Physical Properties of the Blends

From the results of preliminary experiments, it was found that up to a maximum of 45% of R-EPDM could be substituted by r-W-EPDM, without adversely affecting the processability of the blends.

The mechanical properties of the blends are given in Table V. It is evident that the properties drop initially with the addition of W-EPDM to R-EPDM, but at higher loadings of W-EPDM there is an improvement in properties of the EPDM/PP blend. It is now established that the properties of thermoplastic elastomeric blends and alloys depend on the blend morphology, state of cure, and degree of packing of finely dispersed rubber phase.[23] It was also reported that the addition of filler can strengthen the blend composition.[24,25]

In the present investigation, the variation in properties of the blends may be explained as follows: at lower concentrations of W-EPDM (i.e., F_{10}, F_{20}, and F_{30}), the effects of crosslinking in the matrix R-EPDM is prominent. It can be seen from the rheometric studies of the corresponding rubber compounds, that the Δtorque ($M_H - M_L$) initially decreases on addition of W-EPDM, whereas at higher replacement by W-EPDM, Δtorque remains unaltered; Δtorque can be related to the degree of crosslinking. It is found that when the extent of replacement of R-EPDM by W-EPDM is high (i.e., F_{40} and F_{45}) the effect of degree of crosslinking is overshadowed by the filler effect, giving higher modulus and strength.

SEM Studies of Fractured Surfaces

SEM photomicrographs of the fractured surface of the tensile specimens are given in Figure 2. Loose W-EPDM particles can be seen in F_{20} blend, whereas no such pull out of W-EPDM can be seen in the F_{40} blend, indicating higher extent of matrix–filler interaction. The observations are in agreement with the mechanical properties.

Dynamic Mechanical Analyses

The tan δ versus temperature curves, as given in Figure 3, show the characteristic biphasic structure of the blends. The $(\tan \delta)_{max}$ values decrease with the increase of W-EPDM loading, whereas the glass-to-rubber transition temperatures (T_g) remain almost constant. The presence of PP in the blends is observed as a hump around 15–20°C, which is the T_g of PP.

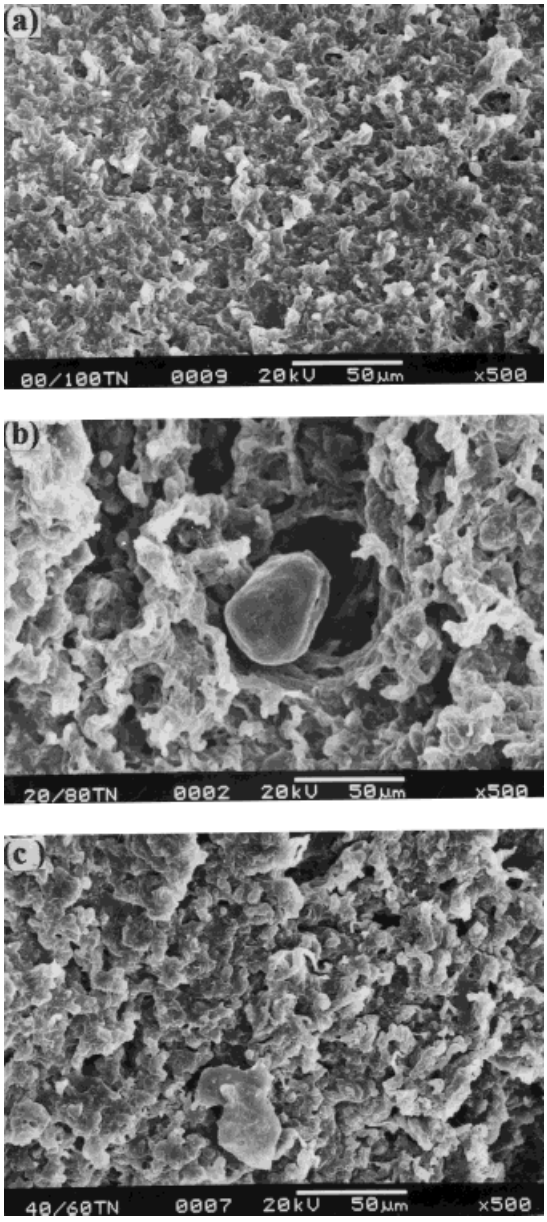

Figure 2 SEM photomicrographs of tensile fracture surfaces: (a) F_0; (b) F_{20}; (c) F_{40}.

Crystallinity of the Blends

The percentage crystallinity of the blends calculated from the X-ray diffractographs is given in Table VI. The crystallinity of PP is 46%. By rule of

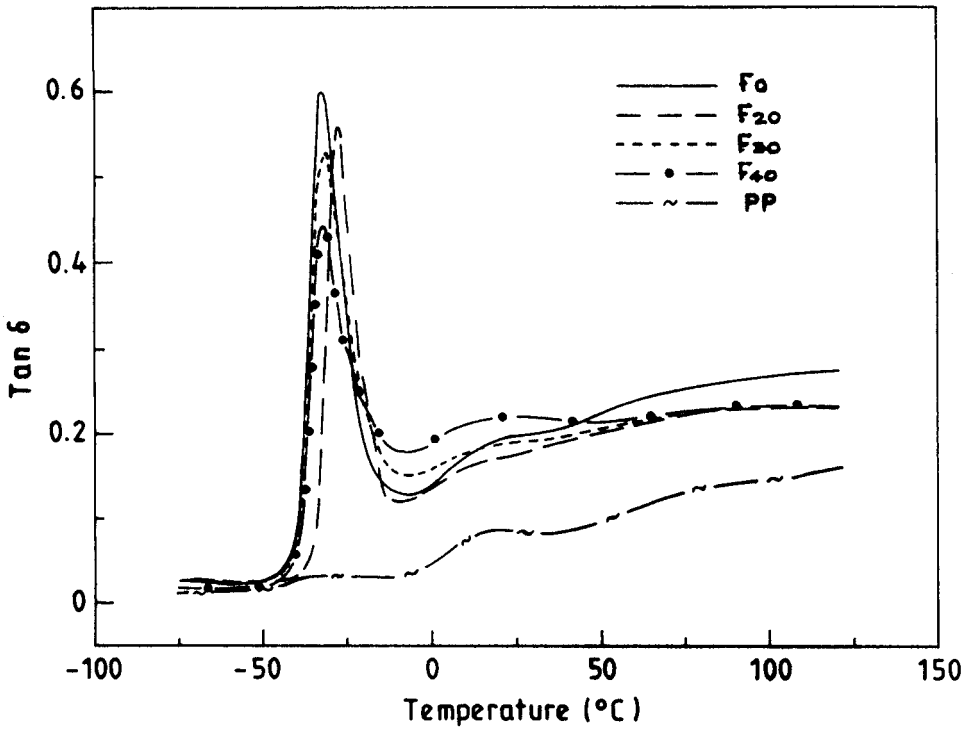

Figure 3 Plots of tan δ versus temperature of the blends.

mixtures the total crystallinity of the blend composition can be given as $C = V_{PP}C_{PP} + V_{EPDM}C_{EPDM}$, where V and C are the volume fractions and percentage crystallinity, respectively. Because EPDM is amorphous, the percentage crystallinity of the blend is given by $C = V_{PP}C_{PP}$. As the W-EPDM content increases in the blend, the volume fraction of PP contributing to the crystallinity decreases; hence, the theoretical value for crystallinity decreases with increase in W-EPDM content. From the X-ray diffraction studies, it is found that the crystallinity of PP drops to 11% (calculated 13%) by the addition of EPDM, as in F_0; however, F_{40} shows a value of 12% (calculated 9%). The exact reason for the marginal increase of crystallinity over the calculated value at higher W-EPDM loading is not clear. This slight increase of crystallinity of the continuous phase may also contribute toward the improved mechanical properties of the F_{40} blend.

Table VI Crystallinity of the Blends

	Percentage Crystallinity	
Mix Number	Experimental	Calculated
PP	46	—
EPDM	0	—
F_0	11	13
F_{20}	11	11
F_{40}	12	9

Effect of Filler and Curatives Present in W-EPDM on the Properties of EPDM/PP Blends

Rheographs obtained for the corresponding rubber compounds in the formulations F_0, F_{20}, F_{40}, FG_{20}, FG_{40}, F'_{20}, F'_{40}, FG'_{20}, FG'_{40}, F''_{20}, and F''_{40} are shown in Figure 4 and the curing parameters are summarized in Table VII. The addition of gum or black-filled waste powder lowers the maximum torque. The Δtorque values of the compounds containing DCP as curing agent (F_{20}, F_{40}, FG_{20}, FG_{40}) are indicative of a higher degree of crosslinking in FG_{20} and FG_{40} than that in F_{20} and F_{40}. This may be ascribed to the effect of carbon black and plasticizers present in W-EPDM, which adsorbs a part of DCP at the mixing temperature (70°C), leaving the matrix R-EPDM less crosslinked at the dynamic vulcanization temperature (180°C). The effect can be also observed in F''_{20} and F''_{40} blends, in which the Δtorque value decreases on addition of carbon black and process aids.[26] The Δtorque values of

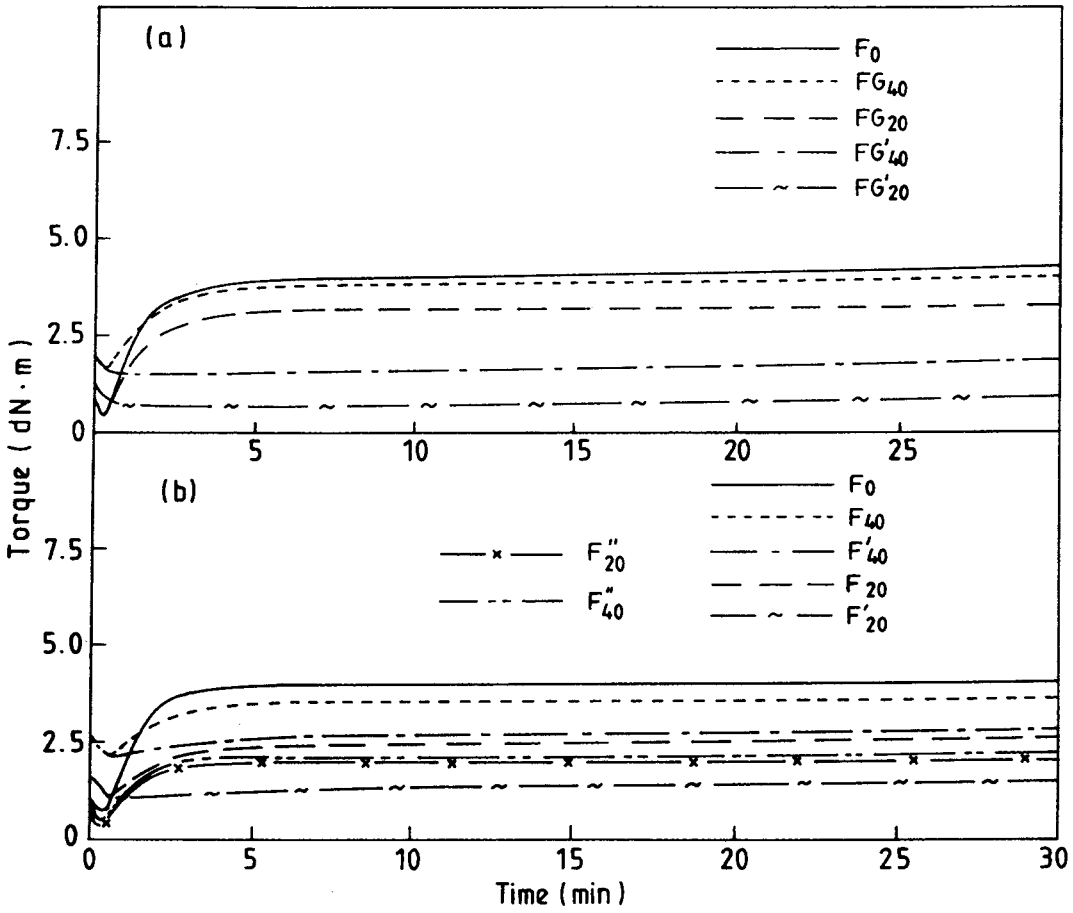

Figure 4 Curing characteristics of the rubber phase in the formulations given in Table IV: (a) Compounds containing W-EPDM(G); (b) compounds containing black-filled W-EPDM.

F'_{20}, F'_{40}, FG'_{20}, and FG'_{40} indicate marginal curing of the compounds even without the addition of DCP. This insignificant level of curing may possibly result from the accelerator (TMTD) migration from W-EPDM to R-EPDM. TMTD is a sulfur donor and acts as a curing agent.[27]

From Table VIII, it can be seen that carbon black–filled W-EPDM enhances the properties, whereas the gum powder can act only as a diluent. The inferior mechanical properties of the compositions without DCP (F'_{20}, F'_{40}, FG'_{20}, and FG'_{40}) can be attributed to the presence of unvul-

Table VII Results of Monsanto Rheometric Studies of Rubber Compounds (Tables II and IV) at 180°C Using MDR-2000

	Mix Number										
Curing Characteristic	F_0	F_{20}	F_{40}	F'_{20}	F'_{40}	FG_{20}	FG_{40}	FG'_{20}	FG'_{40}	F''_{20}	F''_{40}
Minimum torque (M_L), dN · m	0.49	1.29	2.28	1.04	2.16	0.99	1.86	0.78	1.58	0.46	0.51
Maximum torque (M_H), dN · m	4.02	2.58	3.61	1.52	2.86	3.13	3.86	0.86	1.73	1.93	2.19
Δtorque ($M_H - M_L$), dN · m	3.53	1.29	1.33	0.48	0.70	2.14	2.0	0.08	0.15	1.47	1.68

Table VIII Mechanical Properties of Molded Blends (Table IV)

Property	Mix Number							
	F'_{20}	F'_{40}	FG_{20}	FG_{40}	FG'_{20}	FG'_{40}	F''_{20}	F''_{40}
Tensile strength, MPa	3.17	4.23	3.20	3.47	1.43	2.77	3.23	3.73
Elongation at break, %	138	109	155	130	164	109	212	199
Modulus at 100% elongation, MPa	3.01	4.22	2.91	3.34	1.40	2.71	2.54	2.94
Tear strength, kN/m	23.4	28.7	22.7	22.3	16.3	22.9	22.0	25.5
Tension set (at 100% elongation), %	—[a]	—[a]	—[a]	20	54	—[a]	14	12

[a] Sample broke during testing.

canized raw EPDM. Although F''_{20} and F''_{40} contain the same amount of fillers and plasticizers as that in F_{20} and F_{40}, the degree of dynamic vulcanization with DCP alone is insufficient and, accordingly, they are inferior to F_{20} and F_{40} in physical properties. Thus it can be concluded that the fillers and plasticizers present in W-EPDM contribute significantly to the mechanical properties of the blends, whereas the accelerator migration from W-EPDM to R-EPDM plays an insignificant role. Kuriakose et al.[24] reported that reinforcing fillers cause enhancement in mechanical properties of thermoplastic elastomeric rubber/plastic blends and the effect is more pronounced in the case of blends with high rubber content.

Reprocessability of the Blends

The mechanical properties of the blends molded after repeated cycles of mixing (Table IX) show that the blends can be reprocessed and the changes in properties of the recycled blends are within acceptable limits.

CONCLUSIONS

1. The blends of R-EPDM/W–EPDM/PP at a constant rubber/plastic ratio of 70/30 show typical thermoplastic elastomeric morphology with a finely dispersed rubber phase in a continuous PP phase. The virgin EPDM (R-EPDM) and the rubber component present in W-EPDM (r-W-EPDM) together constitute the rubber phase in the EPDM/PP blend.
2. Although addition of W-EPDM to R-EPDM/PP blend causes an initial drop in the mechanical properties, thermoplastic elastomers with enhanced properties were obtained at higher loadings of W-EPDM.

Table IX Mechanical Properties of F_0 and F_{40} After Different Mixing Cycles

Property	F_0			F_{40}		
	Mixing Cycle			Mixing Cycle		
	I	II	III	I	II	III
Tensile strength, MPa	4.83	4.32	4.34	5.50	5.61	4.84
Elongation at break, %	220	252	193	239	255	244
Modulus (at 100% elongation), MPa	3.51	2.93	3.31	3.79	3.54	3.12
Tension set at 100% elongation, %	14	16	16	14	14	12
Tear strength, kN/m	30.2	29.7	30.6	29.0	31.0	27.0
Toughness, J/m^2	3323	3373	2571	4039	4337	3531
Hysteresis loss after first cycle $(J/m^2) \times 10^{-6}$	0.078	0.072	0.081	0.081	0.084	0.068

3. Up to 45% of R-EPDM can be replaced by r-W-EPDM without affecting the processability and the physical properties of the blends. At higher loading (>45%) of W-EPDM, it is not possible to mix W-EPDM with R-EPDM.
4. The blends prepared were found to be reprocessable.
5. Utilization of waste EPDM in making thermoplastic elastomers offers a potential means to recycle waste polymers, keeping economic as well as environmental factors in mind.

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