Plasticization Effect of Transgenic Soybean Oil. I. On Ethylene Propylene Diene Monomer (EPDM), as Substitute for Paraffin Oil

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ABSTRACT: Ethylene propylene diene monomer (EPDM) compounds were prepared with different amounts of transgenic soybean oil (TSO), a renewable and reactive plasticizer. For comparison, similar compounds were prepared with petroleum-based paraffin oil (PO), one of the most common plasticizers for EPDM. The plasticization effects of TSO and of PO were studied by Mooney viscometry, capillary rheometry, differential scanning calorimetry (DSC), and rubber processing analysis (RPA). The results showed that TSO has better plasticization effect than PO on EPDM. In addition, the curing characteristics of the EPDM compounds were studied. TSO was found to react with the curing agent dicumyl peroxide (DCP) during the curing process. Excessive amounts of TSO led to low crosslinking density, which was improved by adjusting the added amount of DCP. The mechanical properties, extraction resistance, and thermal stability of the EPDM vulcanizates plasticized with different amounts of TSO and PO were compared to determine the optimum amount of TSO to replace PO. At the same amount and no more than 15 phr of plasticizer, the TSO-plasticized EPDM vulcanizate has higher tensile and tear strength, elongation at break, extraction resistance, and thermal stability, but lower Shore A hardness than the PO-plasticized EPDM vulcanizate. © 2013 Wiley Periodicals, Inc. J. Appl. Polym. Sci. 130: 4457–4463, 2013

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INTRODUCTION

In the rubber industry, plasticizer is a very important additive that can reduce the viscosity and improve the machinability of the raw rubber, obtain better filler dispersion, as well as provide flexibility, low glass transition, low hardness, and some other improved properties in the end-use product.^{1–3} Plasticizers for rubbers at present are mostly petroleum-based or coal-based chemicals with low molecular weights, including aromatic oils and aliphatic oils that generally have adverse effects on our health.^{4,5} Besides, the petroleum and coal resources may be depleted in several decades. Thus, it is very important to explore alternative renewable plasticizers to meet the requirements of environmental protection and sustainable development.

Plant oils, because of their low cost, large production, nontoxicity, and ready availability, have attracted increasing interests.^{6–9} Compared with other natural resources, plant oils, being liquids, are potential plasticizers to replace mineral oils. Plant oils are triglycerides, which contain three fatty acid chains and each fatty acid chain varies from 14 to 22 carbons in length, with 0–3 double bonds. The special structures containing both polar and non-polar parts promise good compatibility between plant oils and most rubbers. Several studies have shown that plant oils can act as excellent plasticizers in rubbers.^{10–15}

Soybean oil (SO) has become one of the biggest plant oils since transgenic soybean was successfully developed, which ensured a low price of SO about \$750/ton. SO, containing 23.4% of oleic, 53.3% of linoleic, 7.8% of linolenic, and around 15% of palmitic and stearic acids, has been used since the early nineties in many products like soaps, paints, coatings, lubricants, plastics, and elastomers.¹⁷ At present, there is no report on the plasticization of rubber by SO, which may be limited by the high price and the edible requirement of SO. Indeed, the major application of SO is food; however, transgenic soybean oil (TSO), with the same structure as SO, was controversial if TSO is suitable for human consumption because the ingestion of transgenic corn was found lead to cancer.¹⁶ Therefore, a reconsideration of the application of TSO was needed. Anyway, TSO is a renewable plasticizer that fit the requirement of sustainable development.

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Table	I.	Formulation	for	EPDM	Composites
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Ingredient	Grams per hundred grams of rubber (phr)
EPDM 2070	100
Plasticizer	Variable (5, 10, 15, 20, 30)
Carbon black (N 770)	50
DCP	Variable (3, 4, 5, 6, 7, 8, 10, 12)
TAIC	1.3

In this study, we systematically studied the plasticization effect of TSO on different rubbers.

Ethylene propylene diene monomer (EPDM) rubber is one of the most important synthetic rubbers because of its large production, excellent aging characteristics, resistance to oxidation, and chemical resistance. However, the high viscosity of EPDM resulted in poor machinability. Thus, plasticizers need to be used to reduce the viscosity of EPDM and finally increase the elasticity of the end-product. Paraffin oil (PO), about \$900/ton, is the most commonly used plasticizer for EPDM in the peroxide curing system. Compared with PO, TSO is inexpensive and renewable. However, TSO can react with dicumyl peroxide (DCP) during the curing reaction because there are many double bonds in the TSO molecules.¹⁸

In this study, we prepared TSO- and PO-plasticized EPDM compounds to explore the possibility of replacing PO with TSO as the plasticizer. TSO and PO were compared in terms of plasticization effect, plasticization mechanism, and the mechanical properties of the end-product. TSO was found to be a good plasticizer for EDPM and a potential substitute for PO.

EXPERIMENTAL

Materials and Formulation

Transgenic soybean oil (TSO) with an iodine value (the weight of absorbent iodine per 100 g of EPDM) of 120–137 was a gift from Shandong Wandefu Industrial Group Co. Carbon black N770 was purchased from Tianjin Dolphin Carbon Black Development Co. EPDM 2070, with 58% ethylene and ethylidene norbornene as the comonomer was purchased from Jilin chemical factory. The Mooney viscosity (Z100°C 1+4) of EPDM 2070 is 62 and the iodine value is 2.6. DCP, triallyl isocyanurate (TAIC), paraffin oil (PO), and toluene were all analytically pure and were purchased from HWRK Chemical, China.

Mixing and Curing of EPDM Compounds

The blending of EPDM 2070 with carbon black (N770, 50 phr) was carried out on a two-roll mill (Φ 160 mm, Zhanjiang Machinery Factory, Guangdong Province, China) with a roller spacing of 0.5 mm. TSO or PO, TAIC, and DCP were added in turn according to the formulation given in Table I. The ingredients were processed on the mill for about 15 min in order to obtain homogeneous EPDM compounds. The EPDM compounds with different contents of PO or TSO were named EPDM-PO/TSO-*n* (n = 5, 10, 15, 20, 30), and the vulcanizates obtained from EPDM-TSO-20 with different contents of DCP were named EPDM-TSO20-DCP*m* (m = 3, 4, 5, 6, 7, 8, 10, 12).

The rubber compounds were then vulcanized by using a compression molding press (Shanghai Rubber Machinery Factory, Shanghai, China) for the curing time of T_{90} (as determined by a disc rheometer, P355B2, Beijing Huanfeng Chemical Technology and Experimental Machine Plant, Beijing, China) at a pressure of 15 MPa and a temperature of 160°C.

Soxhlet Extraction

A 1 g sample of each vulcanized EPDM composite was extracted for 36 h with 300 mL of refluxing toluene in a Soxhlet extractor. After extraction, the insoluble fractions were dried in a vacuum oven at 60° C, and the dried solids were weighed.

Characterization

Mooney Viscosity. The Mooney viscosity was measured by using a Mooney Viscometer (M3810C, Beijing Huanfeng Chemical Industry Co., China) according to ASTM D 2084 and ASTM D 1646 [Z100°C (1 + 4) min].

Shear Viscosity. The shear viscosity was obtained by an Instron 3211 Capillary Rheometer (Instron Co., UK) at 60°C after the sample was heated for 5 min. The diameter of the capillary was 0.0628 inch.

Rubber Machinability. The rubber machinability was measured by using a Rubber Process Analyzer (RPA2000, Alpha Technologies Services LLC) at 60°C and a scanning frequency of 1 Hz.

Glass Transition Temperature (Tg). The T_g of the vulcanizate was determined by a STARe system DSC1 Instruments (Mettler-Toledo International, Switzerland). The samples in the form of small rectangles about 0.2 mm thick were accurately weighed and sealed in aluminum crucibles. The temperature was first raised to 100°C at 10°C/ min, allowed to stay there for 5 min to eliminate any thermal history and moisture from the sample, and then lowered to -100° C at 10° C/min. Data were recorded in the temperature range -100° C to 50° C at a heating rate of 10° C/min. The nitrogen flow was 150 mL/min.

Cure Characteristics. The cure characteristics were measured on a Disc Rheometer (P355B2, Beijing Huanfeng Chemical Industry Co., China) at 160°C.

Mechanical Properties. The mechanical properties of all vulcanizates were measured at 25°C according to ASTM D638 with a CMT4104 electronic tensile tester (SANS, China) at a crosshead speed of 500 mm/min. The dumbbell-shaped samples (25 mm \times 6 mm \times 2 mm) were prepared according to ISO/DIS 37-1990.

Thermal Stability. The measurements of sample weight loss were carried out on a STARe system TGA/DSC1 thermogravimeter (Mettler-Toledo International, Switzerland) with a Cooling Water Circulator. The testing was done under a flowing nitrogen atmosphere (20 mL/min). All the samples used in the thermogravimetric measurements were similar in weight (10 ± 1 mg) and heated from 30 to 800° C at a heating rate of 10° C/min.

RESULTS AND DISCUSSION

Comparison of Plasticization Effects of PO and TSO on EPDM Compounds

An excellent plasticizer reduces the viscosity and glass transition temperature, and increases the machinability of the compound.



Figure 1. Mooney viscosity of EPDM compounds plasticized by different fractions of SO and PO.

Since PO has proved an excellent plasticizer for EPDM, we compared the plasticization effect of TSO with that of PO on EPDM at the same plasticizer content. The plasticization effects were determined by Mooney viscometry and capillary rheometry, and also studied by RPA and DSC.

Mooney Viscosity. The Mooney viscosities of the TSO- and PO-plasticized EPDM compounds are compared in Figure 1. At the same plasticizer content, the Mooney viscosity of the EPDM compound plasticized by TSO is lower than that of the compound plasticized by PO. This result indicates that TSO can improve the machinability of EPDM better than PO. The Mooney viscosity of EPDM-TSO-10 is lower than that of EPDM-PO-20. Thus, the use of TSO contributes to saving the cost of the end-product.

Shear Viscosity. The shear viscosities of the EPDM compounds were measured by a capillary rheometer to further demonstrate the plasticization effects of PO and TSO on EPDM. Figure 2 shows that the shear viscosity of all the EPDM compounds decreases with increasing shear rate, a typical non-Newtonian behavior. With the low plasticizer content, the shear viscosity of EPDM-PO and EPDM-TSO do not show obvious differences. When the plasticizer content is above 10phr, the shear viscosity of EPDM-TSO is much lower than that of EPDM-PO (shown in Table II).

RPA Measurement. Figure 3 shows that at the same plasticizer content, (i) the initial G' of the TSO-plasticized EPDM is lower than that of the PO-plasticized EPDM, in accordance with the viscosity data, and (ii) $\triangle G'$ (the difference between the initial G' and the final G') of the TSO-plasticized EPDM is also lower



Figure 2. Flow curves of EPDM compounds plasticized by different fractions of TSO and PO. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

than that of the PO-plasticized EPDM, indicating that the Payne effect is lower and the dispersion of the carbon fillers is better in the TSO-plasticized EPDM than in the PO-plasticized EPDM. At a given strain, G' decreases with increasing plasticizer content because the forces between the carbon fillers and EPDM molecules are reduced by the lubrication action of the plasticizer, further indication that the plasticization effect increases with increasing plasticizer content. The G' of the POplasticized EPDM decreases only slightly with strain below 10% strain because there is no change in the forces between carbon fillers and EPDM molecules. With further increases in strain, the EPDM molecules start to slip away from the carbon fillers, resulting in the decrease of the forces between the carbon fillers and EPDM molecules. On the other hand, the G' of the TSOplasticized EPDM decreases with increasing strain even below 10% strain; this decrease becomes sharper with increasing TSO content This last result indicates that TSO can highly reduce the forces between the carbon fillers and EPDM molecules, resulting in a lower viscosity and modulus.

Glass Transition Temperature Determined by DSC. The addition of plasticizer usually leads to a lower glass transition temperature of the rubber compound. The glass transition temperatures of the EPDM compounds plasticized by TSO and PO were determined by DSC in order to study the plasticization effects of the plasticizers. The DSC thermograms of the EPDM compounds are shown in Figure 4. With increasing amount of plasticizer, the glass transition temperature of the EPDM compound dereases slightly, indicating that the addition of plasticizer

Table II. Shear Viscosity Data of the EPDM Compounds Plasticized by Different Fractions of TSO and PO

			EPC	M-PO		EPDM-TSO				
$\log \eta$ (Pa	a s)	5 phr	10 phr	15 phr	20 phr	5 phr	10 phr	15 phr	20 phr	
log γ	1	4.24	4.23	4.14	4.13	4.22	4.15	4.06	3.97	
(s ⁻¹)	2	3.57	3.57	3.49	3.46	3.56	3.56	3.42	3.39	
	3	2.84	2.79	2.75	2.74	2.81	2.75	2.60	2.55	





Figure 3. G'-strain curves of EPDM compounds plasticized by different fractions of TSO and PO. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

results in better plasticization. At the same plasticizer content, the glass transition temperature of the EPDM compound plasticized by TSO is lower than that of the EPDM compound plasticized by PO (Table III), indicating that TSO has a better plasticization effect on EPDM than PO does.

The Mooney viscosity, shear viscosity, storage modulus, and glass transition temperature results all showed that TSO has a better plasticization effect on EPDM than PO does To further explore the possibility of replacing PO with TSO, the effects of PO and TSO on the cure characteristics, mechanical properties, and thermostability of the end-products were studied.

Cure Characteristics

The cure characteristics were determined by a curometer, and the vulcanization curves of the TSO- and PO-plasticized EPDM compounds are shown in Figure 5. It can be seen that at the same plasticizer content, the minimum torque of the TSOplasticized EPDM is lower than that of the PO-plasticized EPDM, indicating lower viscosity of the TSO-plasticized EPDM than that of the PO-plasticized EPDM, in accordance with Mooney viscosity results. With increasing plasticizer content, the maximum torque decreases, indicating a decrease in crosslinking density. The maximum torque of EPDM-TSO decreases significantly with increasing TSO content. At a TSO content of 30 phr, the maximum torque is so low that EPDM-TSO-30 cannot be crosslinked and stay in shape because TSO can consume DCP.¹⁸ Therefore, the higher the TSO content, the smaller the amount of DCP participating in the curing reaction, resulting in lower crosslink density. The decrease in crosslink density adversely affects the mechanical properties of the end-product. Besides, the T_{90} (the time when the torque reaches 90% of the maximum torque) of the TSO-plasticized EPDM is much longer



Figure 4. DSC thermograms of EPDM compounds plasticized by different fractions of TSO and PO. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

than that of the PO-plasticized EPDM, another indication that the addition of TSO adversely affects the curing reaction. To solve this problem, different amounts of DCP were used to adjust the crosslink density and the mechanical properties of the EPDM vulcanizate.

Figure 6 shows the cure behavior of EPDM-TSO-20 with different amounts of DCP. The maximum torque of EPDM-TSO-20 increases with increasing amount of DCP below 10 phr. At amounts of DCP higher than 10 phr, the maximum torque of EPDM-TSO-20 does not increase with further increases in the amount of DCP, indicating that the maximum the crosslink density has been reached. Thus, the crosslinking density can be adjusted by varying the amount of DCP below 10 phr. The mechanical properties of the vulcanized EPDM with different plasticizer contents and different amounts of DCP were then studied.

Mechanical Properties

The stress–strain curves of the vulcanized EPDM plasticized by different amounts of PO and TSO are shown in Figure 7. We can clearly see that at the same plasticizer content, the tensile strength of the TSO-plasticized EPDM is higher than that of the PO-plasticized EPDM at plasticizer contents below 15 phr. At TSO contents above 15 phr, the tensile strength of EPDM decreases with increasing plasticizer content as a result of the decrease in crosslink density. Figure 7 also shows that the elongation at break of the TSO-plasticized EPDM largely increases over that of the PO-plasticized EPDM because TSO has a better lubricating effect than PO on the EPDM matrix to make it easier for the molecular chains of EPDM to stretch. The higher elongation at break also indicates a better plasticization effect of TSO than that of PO on EPDM. The other mechanical

Table III. Glass Transition Temperature of EPDM Compounds Plasticized by Different Fractions of TSO and PO

Amount of		EPD	M-PO	EPDM-TSO				
plasticizer (phr)	5	10	15	20	5	10	15	20
Tg	-53.95	-55.75	-57.25	-57.94	-57.8	-58.12	-58.8	-60.43



Figure 5. Cure behavior of EPDM compounds plasticized by different amounts of TSO and PO. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]



Figure 6. Cure behavior of EPDM compounds plasticized by 20 phr of TSO at different amounts of DCP. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

properties listed in Table IV show that at the same plasticizer content, (i) the tensile modulus at 100% elongation of the TSO-plasticized EPDM is lower than that of the PO-plasticized EPDM, indicating a lower crosslink density of the TSO-plasticized EPDM than that of the PO-plasticized EPDM, and



Figure 7. Stress–strain curves of vulcanized EPDM plasticized by different fractions of TSO and PO. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

(ii) the Shore A hardness of the TSO-plasticized EPDM is much lower than that of the PO-plasticized EPDM, indicating a better plasticization effect of TSO. Though the lower crosslink density can lead to a lower Shore A hardness, we consider the plasticization effect as the major factor according to the Shore A hardness data of the EPDM at low plasticizer contents. The tearing strength of the TSO-plasticized EPDM is much higher than that of the PO-plasticized EPDM because of the better filler dispersion indicated by the RPA results. In summary, (i) at plasticizer contents lower than 15 phr, the mechanical properties of the TSO-plasticized EPDM are better than or at least comparable with those of the PO-plasticized EPDM, and (ii) at plasticizer contents higher than 15 phr, the mechanical properties of the TSO-plasticized EPDM decrease with increasing TSO content because of the increase in crosslink density. In an effort to improve the mechanical properties of EPDM at high TSO contents, different amounts of DCP were used to tune the crosslink density of the EPDM vulcanizate. EPDM-TSO-20 was selected for further studies.

The mechanical properties of vulcanized EPDM-TSO-20 crosslinked by different amounts of DCP are listed in Table V. It can be seen that with increasing amount of DCP, (i) the elongation at break and the permanent deformation both decrease, while

Table IV. Mechanical Properties of Vulcanized EPDM Plasticized with Different Fractions of TSO and PO

Sample	EPDM- PO-5	EPDM- PO-10	EPDM- PO-15	EPDM- PO-20	EPDM- TSO-5	EPDM- TSO-10	EPDM- TSO-15	EPDM- TSO-20
Shore A hardness	57	54	52	48	42	39	36	32
Tensile strength (MPa)	11.5 ± 0.6	9.8 ± 0.5	6.4 ± 0.4	6.9 ± 0.4	12.2 ± 0.6	12.8 ± 0.6	7.5 ± 0.3	3.3 ± 0.3
Tear strength (kN⋅m ⁻¹)	23.3 ± 0.6	28.4 ± 0.5	22.1 ± 0.4	20.2 ± 0.5	26.2 ± 0.6	35.3 ± 0.8	22.1 ± 0.3	18.6 ± 0.4
Modulus at 100% elongation (MPa)	2.9	2.0	1.6	1.4	1.7	1.1	0.9	0.6
Modulus at 300% elongation (MPa)	-	-	-	-	8.9	3.5	1.4	0.8
Elongation at break (%)	214 ± 13	232 ± 11	236 ± 15	262 ± 15	378 ± 21	763 ± 27	927 ± 31	1135 ± 42



Sample	EPDM- TSO20- DPC3	EPDM- TSO20- DPC4	EPDM- TSO20- DPC5	EPDM- TSO20- DPC6	EPDM- TSO20- DPC7	EPDM- TSO20- DPC8	EPDM- TSO20- DPC10	EPDM- TSO20- DPC12
Shore A hardness	32	38	45	49	53	55	57	58
Tensile strength (MPa)	3.3 ± 0.3	8.1 ± 0.4	12.6 ± 0.6	7.3 ± 0.4	6.9 ± 0.4	5.9 ± 0.6	5.0 ± 0.3	3.7±0.3
Tearing strength (kN⋅m ⁻¹)	18.6 ± 0.4	27.4 ± 0.5	32.3±0.6	28.4 ± 0.6	28.0 ± 0.5	17.2 ± 0.3	13.4±0.2	11.7 ± 0.1
Modulus at 100% elongation (MPa)	0.6	0.9	1.2	1.4	1.7	2.7	3.5	3.6
Elongation at break (%)	1135 ± 42	902 ± 21	668±15	372±13	279±11	204 ± 9	116±8	104±8
Permanent deformation (%)	26	21	17	16	11	8	5	4

Table V. Mechanical Properties of Vulcanized EPDM-TSO-20 Crosslinked by Different Amounts of DCP

Table VI. Extraction Data of Vulcanized EPDM at Different Plasticizer Contents with Toluene as Extractant

Sample	Pure EPDM	EPDM- PO-5	EPDM- PO-10	EPDM- PO-15	EPDM- PO-20	EPDM- TSO-5	EPDM- TSO-10	EPDM- TSO-15	EPDM- TSO-20
Initial proportion of plasticizer (%)	0	3.1	6.1	8.9	11.5	3.1	6.1	8.9	11.5
Extracted part (%)	1.8	4.8	7.7	10.4	13.0	1.9	4.6	7.7	11.0

the Shore A hardness and the tensile modulus at 100% elongation both increase, indicating an increase in crosslinking density, and (ii) both the tensile strength and the tear strength first increase and then decrease, and EPDM-TSO20-DCP5 was found to have the best mechanical properties. In other words, a DCP content of 5 phr is optimum for curing EPDM-TSO-20 to obtain a proper crosslink density and good mechanical properties. From the analyses of cure characteristics and mechanical properties, TSO is regarded as a comparable plasticizer with PO when the TSO content is lower than 15 phr. Though the crosslink density and mechanical properties are affected by the TSO content, they can be adjusted by changing the added amount of DCP. Since TSO reacts with DCP during the curing reaction, TSO can be grafted onto the EPDM molecular chains. Therefore, better extraction resistance and thermal stability of the TSO-plasticized EPDM are expected. The extraction resistance was studied by Soxhlet extraction and the thermal stability was studied by TGA.

Extraction Resistance

It can be seen from Table VI that the extracted part of the POplasticized EPDM is approximately equal to the extracted part of pure EPDM plus the initial proportion of PO, an indication that nearly all the added PO can be extracted. However, the extracted part of the TSO-plasticized EPDM is much lower than that of the PO-plasticized EPDM at the same plasticizer content. This result indicates that the TSO-plasticized EPDM has better extraction resistance than the PO-plasticized EPDM at the same plasticizer content because TSO can react with DCP to form macromolecules or graft onto EPDM molecular chains.

Thermal Stability

Figure 8 shows the TGA thermograms of the EPDM vulcanizates plasticized by 5 phr of PO and TSO. Two thermal degradation stages can be identified. The similar weight losses of EPDM-TSO-5 and EPDM-PO-5 in the second degradation stage from 450 to 500°C are attributed to the degradation of EPDM. The first thermal degradation stage of the PO-plasticized EPDM



Figure 8. TGA thermograms of EPDM vulcanizates plasticized by 5 phr of TSO and PO.

is from 190 to 400°C and is attributed to the evaporation of PO, while the first thermal degradation stage is from 360 to 440°C for the TSO-plasticized EPDM These TGA results indicate that the TSO-plasticized EPDM has better thermal stability than the PO-plasticized EPDM.

CONCLUSIONS

TSO was used as a reactive renewable plasticizer for EPDM. At the same plasticizer content, the TSO-plasticized EPDM showed lower Mooney viscosity, shear viscosity, and T_g than POplasticized EPDM. According to the RPA results, TSO could better reduce the modulus G' and the Payne effect of the EPDM compound than PO does, indicating that the addition of TSO can lead to better machinability and filler dispersion than that of PO. All these results confirmed that TSO has better plasticization effect on EPDM than PO.

The mechanical properties of the TSO-plasticized EPDM, such as tensile strength, tear strength, and elongation at break, are better than those of the PO-plasticized EPDM at a plasticizer content of no higher than 15 phr. At a plasticizer content of 20 phr, the reaction between TSO and DCP adversely affects the curing reaction and results in low crosslink density and poor mechanical properties of the end-product. Different amounts of DCP were tried, and the optimum mechanical properties were obtained at a DCP content of 5 phr. In addition, the extraction resistance and thermal stability of the TSO-plasticized EPDM are better than those of the PO-plasticized EPDM. Thus, TSO is an excellent plasticizer for EPDM and a good substitute for PO.

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