

A Comparative Study of the Effect of Some Paraffinic Oils on Rheological and Dynamic Properties and Behavior at Low Temperature in EPDM Rubber Compounds

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ABSTRACT: The influence of some paraffinic oils on rheological properties, dynamic properties, and behavior at low temperature of various ethylene-propylene-diene monomer rubber (EPDM) compounds was studied. Three different types of EPDM, Dutral TER 4049, Dutral Ter 4038, and Nordel IP 4770 R, and five different paraffinic oils were used. The properties of the compounds were evaluated with

reference to the oil characteristics: viscosity, composition, glass transition temperature, and solubility parameter. © 2005 Wiley Periodicals, Inc. *J Appl Polym Sci* 97: 1825–1834, 2005

Key words: rubber; rheology; glass transition; viscoelastic properties

INTRODUCTION

Ethylene-propylene-diene monomer rubber (EPDM) is the most synthetic rubber used for non-tire applications. As a result of the saturated main chain,^{1–4} it is relatively stable toward oxidative, ozone, thermal, and photodegradation, making it very suitable for the following outdoor applications: automotive and building profiles, roof sheetings, tubes, seals, and gaskets. In recent years,^{3,5,6} it has been used in a diverse range of electrical applications, because of its superior electrical properties, its good physical properties, and its flexibility over a wide temperature range.

The greater part of EPDM production is destined to extruded articles. In extrusion applications,^{4,7} the rubber must have ultrafast curing rates to achieve economic cure cycles, parts with low compression set for good sealing, and relatively high molecular weight to provide good shape retention and to maintain dimensional stability.

Plasticizers⁸ are often added during compounding to reduce elastomer viscosity, promote mold flow and release, improve milling and extrusion, facilitate the incorporation of fillers, improve low temperature flexibility, and provide a softer vulcanizate.

EPDM⁸ can incorporate a relatively large amount of plasticizer into its polymer matrix without signifi-

cantly affecting the physical properties obtained in the finished rubber product.

Petroleum⁸ oils, vegetable oils, and synthetic products such as esters and high molecular weight sulfonic acids are often used as plasticizers.

Petroleum oils are used primarily with nonpolar elastomers and are either naphthenic, paraffinic, or aromatic, depending upon elastomer compatibility, volatility, and expected product service conditions. Due to its low polarity, EPDM is compatible with paraffinic or relatively naphthenic oils.

In the present article, we describe a comparative study of the effects of five different plasticizer oils on the rheological characteristics, dynamic viscoelastic properties, and behavior of vulcanized rubber at low temperature, of EPDM rubber formulations.

EXPERIMENTAL

Materials

The following three ethylene-propylene-diene terpolymer rubbers with 5-ethylidene-2-norborene (ENB) as termonomer were used in this study: Dutral* TER 4049, Dutral* TER 4038 supplied by Polimeri Europa (Milan, Italy) and NORDEL[®] IP 4770 R manufactured by DuPont Dow Elastomers (Akron, OH). Their compositions and viscosities are shown in Table I.

Five experimental paraffinic oils supplied by Repsol-YPF were chosen. All oils are blends of hydrocarbon petroleum byproducts, whose physical properties are listed in Table II.

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TABLE I
Composition and Viscosity of Polymers

	A	B	C
Trade name	DUTRAL* TER 4049	DUTRAL* TER 4038	NORDEL ®IP 4770R
Ethylene content (%)	59	73	68
ENB content (%)	4.4	4.4	4.5
Mooney viscosity ML 1+4 (125°C)	76	60	65

Compound preparation and vulcanization

EPDM gum mix, with the recipes given in Table III, were prepared on a laboratory two-roll mill, with a friction ratio of 1 : 1.20, heated at 60°C.

The compounds were vulcanized in a hydraulic press heated by thermofluid to the vulcanization temperature for a specified vulcanization time.

A Monsanto viscometer model MV 2000E was used to measure the Mooney viscosity, which is shown as ML(1 + 4) at a temperature of 100°C.

The vulcanization curves were obtained by using a rotorless Monsanto Rheometer model MDR 2000E, at a temperature of 165°C, with an oscillation arc of 0.5°.

Rheological properties

A high-pressure capillary rheometer model Göttfert Rheograph 2003 was used to study rheological properties. Tests were carried out with two capillary dies with diameters of 2 mm and L/D ratios of 10 and 15, respectively. After a warming up period of 3 min, the melt was extruded through the capillary at 100°C. The shear rates were from 0.1 to 900 s⁻¹.

The barrel pressure and ram rate were converted into the apparent shear stress (τ_a) and shear rate (γ_a), respectively, by using the equation

$$\tau_a = \frac{B_p}{4 \cdot L/D} \quad (1)$$

where B_p is the barrel pressure and L/D is the capillary length-to-diameter ratio and

$$\gamma = \frac{32Q}{\pi D^3} \quad (2)$$

where Q is the volumetric flow rate and D is the capillary diameter.

The true shear rate was calculated from the apparent shear rate by using the Weissenberg–Rabinowitsch correction

$$\gamma = \frac{3n' + 1}{4n'} \gamma_a \quad (3)$$

The term n' is the flow behavior index, which is given by

$$n' = \frac{d \log(\tau_a)}{d \log(\gamma_a)} \quad (4)$$

TABLE II
Physical Properties of Paraffinic Plasticizer Oils

Identification numbers:		1	2	3	4	5
Trade name:		KRISTOL M-70	New 500	New Bs	Extensoil 29	Extensoil 270
Specifications	Method					
Composition (%)						
Aromatic carbons	ASTM D-2140	0	2.12	3.24	7.06	5.60
Paraffinic carbons		68.20	68.28	69.49	70.60	66.33
Napthenic carbons		31.80	29.60	27.26	22.34	28.07
Viscosity 100°C (cSt)	ASTM D-445	8.880	10.78	32.40	36.50	8.236
Viscosity 40°C (cSt)	ASTM D-445	69.33	95.94	504.6	602.6	64.14
Density 15°C (g/cm ³)	ASTM D-4052	0.8689	0.8772	0.8945	0.9074	0.8866
VGC		0.8000	0.8039	0.8055	0.8208	0.8248
Aniline point (°C)	ASTM D-611	120.3	116.3	126.8	119.0	101.8
T _g ' DSC (°C)		-61.1	-57.98	-45.17	-43.18	-61.33
Pour point (°C)	ASTM D-97	-18	—	—	-9	-9
ΔE (cal/g)	DSC	20522	21826	29574	30467	19724
M _w (g/mol)	Gossens	444	473	639	654	425
V _m (cm ³ /mol)		511	539	714	722	480
d (cal ^{1/2} /cm ^{3/2})		6.34	6.36	6.43	6.50	6.41

TABLE III
Formulation of the Compounds (in pphr)

Mix name	A	B	C
Dutral TER 4049	100	—	—
Dutral TER 4038	—	100	—
Nordel 4770	—	—	100
FEF N-550	110	—	—
GPF N-660	—	140	105
Stearic acid	1	1	1
Zinc oxide	5	5	5
Calcium oxide	5	6	6.4
Whiting	25	30	—
Polyethylene glycol	2	5	—
Sulfur	0.5	0.8	2
MBT ^a	—	1.2	—
TMTD ^b	—	0.8	1
DTDM ^c	—	2	—
TBBS ^d	—	—	1
TDEC ^e	—	—	1
ZDBC ^f	—	—	3
Perkadox 14/40 ^g	6	—	—
Trigonox 2940 ^h	4	—	—
Paraffin oil	70	100	75
Paraffin wax ⁱ	—	—	6
Vanfre ^j	—	—	5

^a 2-Mercaptobenzothiazole.

^b Tetramethyl thiuram disulfide.

^c 4,4'-Dithiodimorpholine.

^d *N*-tert-butyl-benzothiazyl sulphenamide.

^e Tellurim diethyldithiocarbamate.

^f Zinc dibutyldithiocarbamate.

^g 1,3-Bis-(*tert*-butyl-peroxy-isopropyl) benzene.

^h 1,1-Di-*tert*-butyl-peroxy-3,3,5-trimethyl cyclohexane.

ⁱ Paraffinic wax Redezon 100.

^j Processing aid.

and was determined by the regression analysis of the values of τ_a and γ_a obtained from the experimental data. The shear viscosity (η) was calculated from the ratio between apparent shear stress and the true shear rate obtained by the Weissenberg–Rabinowitsch correction

$$\eta = \frac{\tau_a}{\gamma} \quad (5)$$

Dynamic properties

The dynamic properties were determined in a Metra-vib Viscoanalyzer RAC 815, based on a principle of nonresonant forced vibrations. Tests were performed over a wide frequency range (5, 15, 50, 100 Hz) and the temperature programs were run from -80 to 130°C under a controlled sinusoidal strain, under a flow of liquid nitrogen. An oscillating dynamic strain of 0.012% was used. The viscoelastic properties, such as the storage modulus (E'), the loss modulus (E''), and the mechanical loss factor ($\tan \delta = E''/E'$), were recorded as a function of temperature and frequency.

Low temperature behavior

To determine the stiffness of vulcanized rubbers at low temperature by means of the Gehman (Torsion Wire) method, a Wallace Gehman Tester was used, according to national standard (UNE 53568:1978). The test specimens were strips measuring 40 ± 2.5 mm \times 3.0 ± 0.2 mm (length \times width) and 2 ± 0.2 mm of thickness. Each mix was tested three times by using different specimens. The lowest temperature was -70°C , and this was increased by steps of 5°C , conditioning the sample for 5 min at each temperature. The temperatures at which the relative modulus are 2, 5, 10, and 100 are designated as T2, T5, T10, and T100, respectively.

RESULTS AND DISCUSSION

Mooney viscosity

Figure 1 shows ML(1 + 4) at 100°C of the three pure gums. EPDM Dutral Ter 4049 shows the higher viscosity so it probably has the highest molecular weight. Mooney viscosity¹ is somehow related to the molecular weight, but it is also affected by other parameters, such as the molecular weight distribution and the degree of branching.

Figure 1 also shows the ML(1 + 4) at 100°C of all compounds. As is well known, the addition of a plasticizer oil diminishes the Mooney viscosity, the higher the oil content, and the greater the decrease in Mooney viscosity.

In the three series, the Mooney viscosity of the compound plasticized with oil-4 (Extensoil 29) is higher than others, independently of rubber type and oil content.

In Dutral TER 4049 and 4038, there is a clear connection between the Mooney viscosity and the oil viscosity. In Nordel compounds, Mooney viscosity is affected by other parameters in addition to oil viscosity. Perhaps the oil composition plays an important role in this case.

Rheological properties

Because EPDM rubbers are extensively used in manufacturing a variety of extruded articles,⁴ it is very important to study rheological properties: swell, smooth surface, flow curves, and viscosity.

For the extrusion process,⁶ processibility of a rubber compound means that the compound can flow easily and the extrudate has dimensional stability. As mentioned, the rheological properties of the compounds were measured by using a capillary rheometer.

Figure 2 shows the plot of log shear stress versus log apparent shear rate of the three EPDM compounds with oil-1 (samples A-1, B-1, and C-1), as an example. To correct the shear rate by using the Weissenberg–

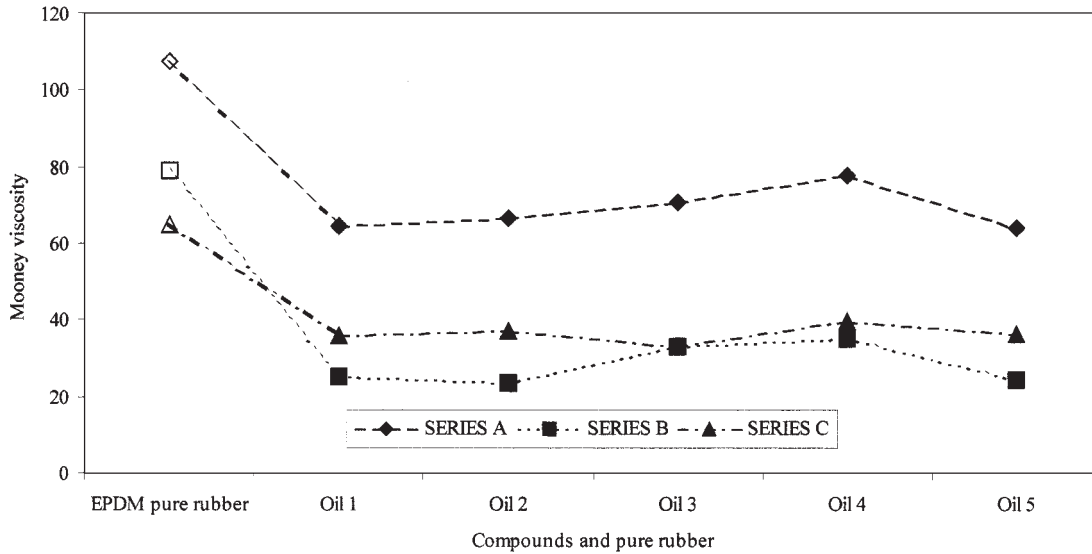


Figure 1 Viscosity Mooney ML(1 + 4) of the three EPDM pure rubbers at 100°C and the three Series A, B, and C, with oil 1, 2, 3, 4, and 5 at 100°C. *ML(1 + 4) of Nordel IP 4770 R at 125°C.

Rabinowitsch method, only A and B series can be fitted to straight lines. Table IV shows the n' values calculated for these series, together with the coefficients of determination R .

Figure 3 shows the log-log plot of shear stress versus the true shear rate, calculated by applying the correction of Weissenberg–Rabinowitsch for the A series with the $L/D = 10$ die. The variations are lineal.

Therefore, the material obeys a power law model⁹ of flow response. The power model of flow is described by the equation

$$\tau_a = K\gamma^n \tag{6}$$

where K and n are constants characteristic of the material. The constant n is usually called a non-Newtonian

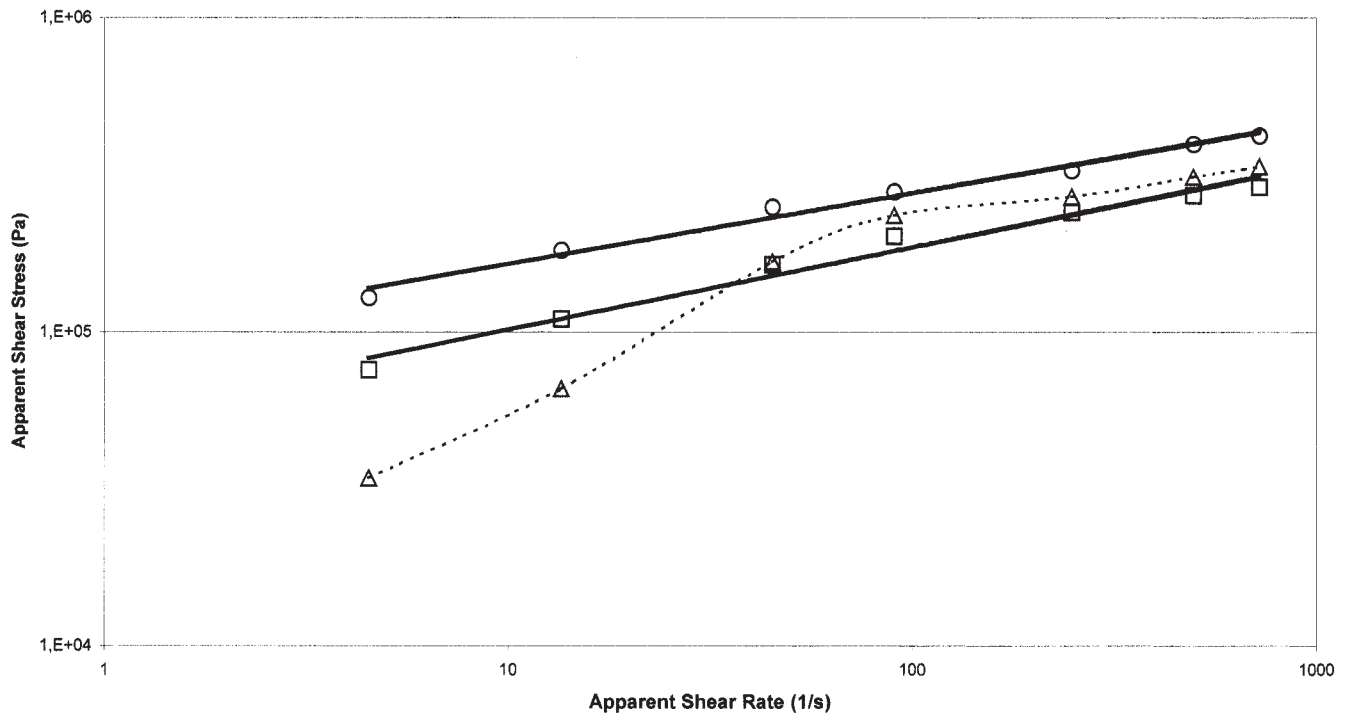


Figure 2 Log-log apparent shear stress versus apparent shear rate plots, $L/D = 10$ die. Oil-1; Samples A-1 (O); B-1 (□), C-1 (△).

TABLE IV
***n'* Values for the Weissemberg–Rabinowish Correction**

Sample	<i>n'</i>	3 <i>n'</i> + 1/4 <i>n'</i>	<i>R</i>	Sample	<i>n'</i>	3 <i>n'</i> + 1/4 <i>n'</i>	<i>R</i>
L/D = 10 die							
A-1	0.2721	1.6688	0.98	B-1	0.1812	2.1297	0.98
A-2	0.2404	1.7899	0.99	B-2	0.6243	1.150	0.97
A-3	0.2306	1.8341	0.99	B-3	0.1616	2.297	0.99
A-4	0.2268	1.8522	0.99	B-4	0.1654	2.2615	0.99
A-5	0.2446	1.7721	0.98	B-5	0.1722	2.2018	0.98
L/D = 15 die							
A-1	0.1793	2.1443	0.99	B-1	0.2022	1.9864	0.99
A-2	0.1769	2.1632	0.99	B-2	0.6445	1.1379	0.99
A-3	0.1902	2.0644	0.99	B-3	0.3124	1.5502	0.99
A-4	0.1810	2.1312	0.99	B-4	0.2675	1.6845	0.98
A-5	0.1860	2.0941	0.99	B-5	0.2845	1.6287	0.98

nian index or flow behavior index and the values of *n* for the different blends can be calculated from the slope of the straight lines in Figure 3. The *n* values of the A and B series blends are shown in Table V. The low values of *n* (<1) are indicative of pseudoplastic behavior of the blends.

In any case, the flow behavior index with the die of L/D = 15 ratio is smaller than with the die of L/D = 10 ratio. For the same oil, the value of the flow behavior index is higher in Series B than in Series A. The greater the L/D ratio of the die, the more similar the flow index values. The influence of oil type on the flow index is dependent on the rubber type. So, in

compounds based on EPDM DUTRAL TER 4049 (Series A), the oil KRISTOL M70 (oil-1) provides the highest flow index value, while, in compounds based on EPDM, DUTRAL TER 4038 is the EXTENSOIL 270 (oil-5).

According to Tables I and II, we can conclude that the flow behavior index is directly related to the ethylene content of rubber and the greater or minor paraffinic and naphthenic character of oil.

These calculations do not apply to NORDEL IP 4770 (Series C) because their log–log plots of shear stress versus shear rate are not fitted to a straight line, probably due to the special characteristic of the elastomeric

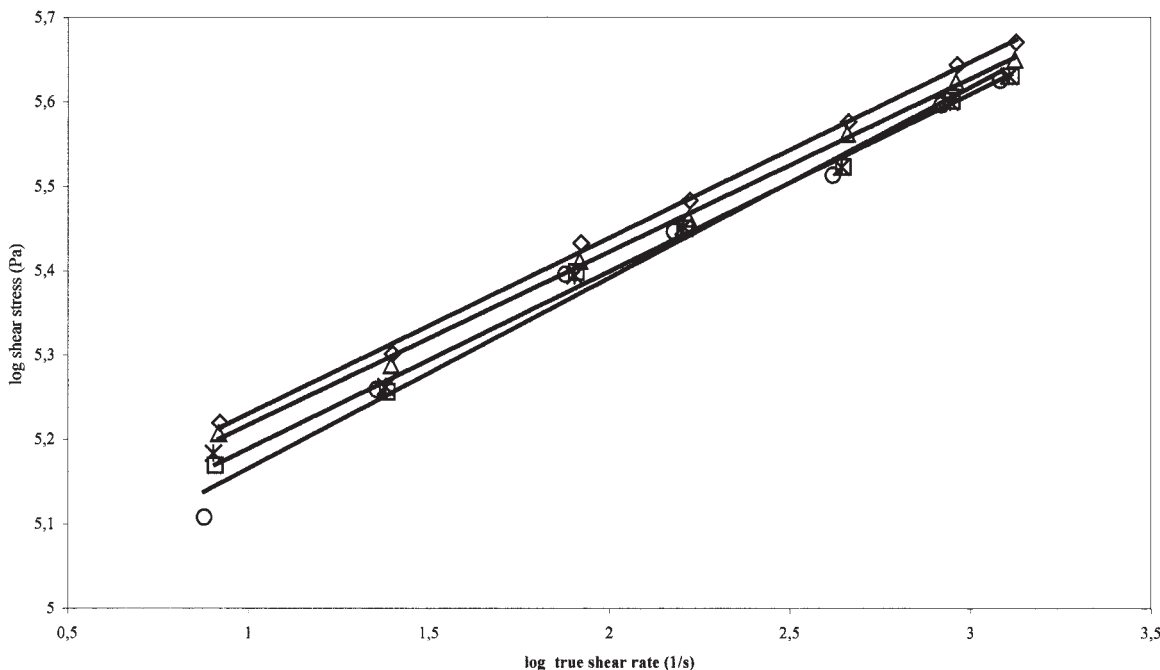


Figure 3 Log–log shear stress versus true shear rate plots, L/D = 10 die in Series A; Oil-1 (○), Oil-2 (□), Oil-3 (△), Oil-4 (224), Oil-5 (*).

TABLE V
Flow Behavior Index of the Compounds

Compounds	Flow behavior index (n)	
	Die	
	L/D ratio = 10	L/D ratio = 15
A-1	0.247	0.195
A-2	0.221	0.190
A-3	0.213	0.190
A-4	0.215	0.181
A-5	0.217	0.186
B-1	0.327	0.306
B-2	0.348	0.278
B-3	0.355	0.276
B-4	0.411	0.268
B-5	0.432	0.306

matrix. NORDEL IP 4770 is a new generation of EPDM based on the INSITE™ catalyst by DuPont Elastomers.¹⁰ The INSITE process uses a combination of a constrained geometry catalyst system and innovative process technology. It is different from other metallocene-type catalysts¹¹ because of its molecular geometry, which forces a high exposure of the active metal site. The behavior of the Series C compounds may be attributed to the fact that EPDM Elastomers produced at INSITE catalyst¹² have a narrower molecular weight comonomer distribution than traditional Ziegler–Natta catalyst EPDM. The literature^{13,14} shows that branched polymers present entanglements that act as knots, hindering slipping of the chains. In

metallocene catalyst EPDM, the branches are longer than traditional Ziegler–Natta catalyst EPDM, resulting in these entanglements possibly not being presented.

One characteristic that defines an oil is the solubility parameter.¹⁵ The solubility parameter is described by the equation

$$d = \sqrt{\frac{\Delta E}{V_m}} \quad (7)$$

where ΔE is the cohesion energy of the molecules per mol and V_m is the volume per mol of the oil. Usually this parameter is calculated only for pure products, but it can be used to make calculations for nonpure substances. The cohesion energy can be calculated with a DSC (strictly, this is the vaporization energy for the oil) and the molar volume can be calculated dividing the average molecular weight (calculated by the formula of ASTM D-2502¹⁶) by the density (calculated by ASTM D-1298¹⁶). The results of d are shown in Table II. Solubility parameter represents the density of the substance's cohesion energy. In general, a polar compound has a very high cohesion energy, giving it a high solubility parameter. A nonpolar compound has a low solubility parameter.

The plot of the solubility parameter versus the flow index (L/D = 10 ratio) for two different EPDMs is shown in Figure 4. The flow index of the mixes depends on the solubility parameter of the oil. For Dutral

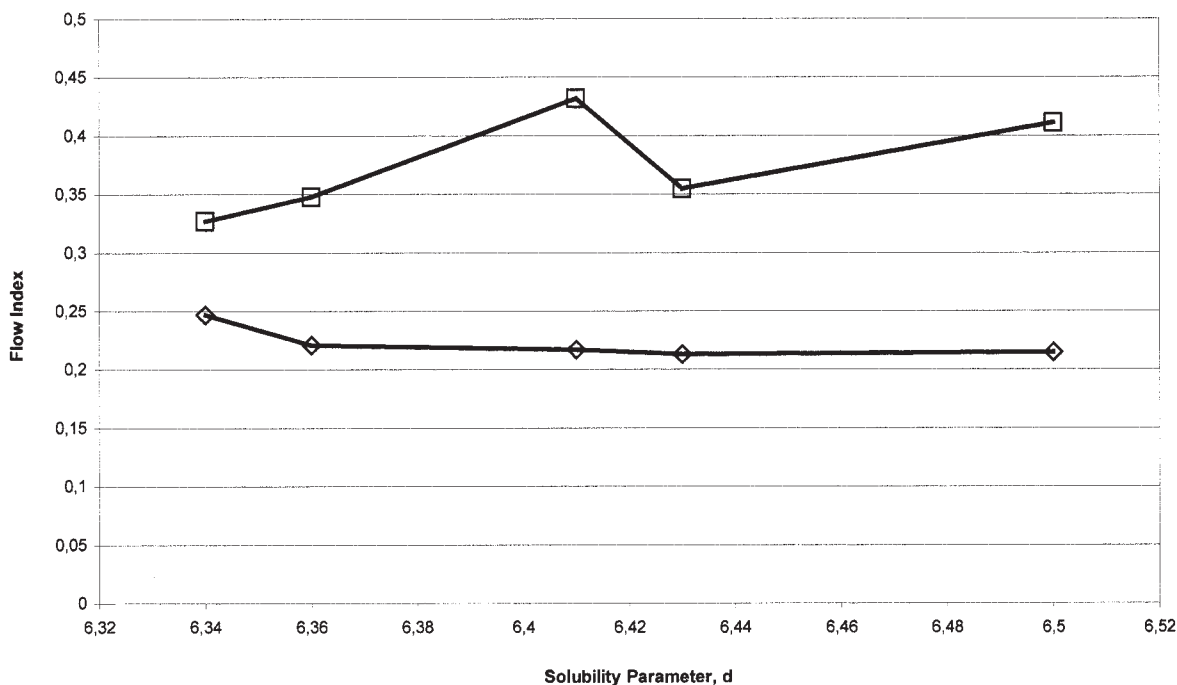


Figure 4 Flow behavior index of the compounds versus solubility parameters, d (cal/cm^3)^{1/2} of the oils; Series A (224); Series B (□).

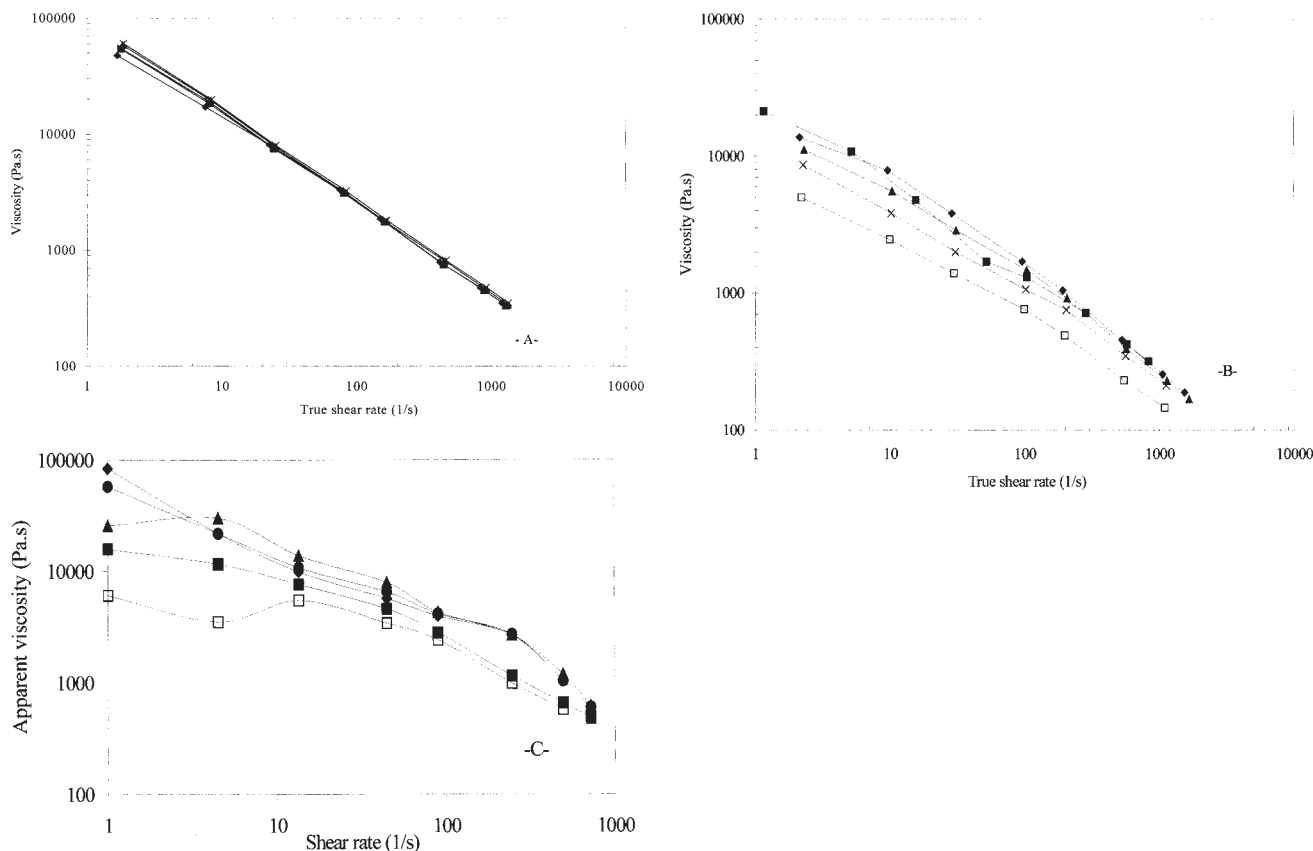


Figure 5 Log-log viscosity versus shear rate plots, $L/D = 10$ die; (A) Series A; (B) Series B; (C) Series C: Oil-1 (\blacklozenge), Oil-2 (\square), Oil-3 (\blacktriangle), Oil-4 (\times), Oil-5 (\square).

TER 4049, a decrease in the solubility parameter increases the flow index (nonpolar oil). By contrast, the flow index for Dutral TER 4038 increases when d is in the range of $6.40 \text{ (cal/cm}^3)^{1/2}$. To increase the flow parameter of the mixes of Dutral TER 4049, we can use an oil with a low-solubility parameter. If we are going to use Dutral TER 4038, we should use an oil with a higher solubility parameter.

Figures 5 and 6 show log-log plots of viscosity versus shear rate at different L/D ratio dies. In Series A and B, the true shear rate and the corrected viscosity values were used. At different L/D ratios, the viscosity decreases¹⁷ with an increasing shear rate in every compound. At a particular shear rate, the viscosity of the compounds in Series B is lower than in the two other series, as a consequence of its higher oil content.

With the $L/D = 10$ ratio die, all blends in Series A show the same trend in their behavior. The viscosity values are very similar in all blends at the same shear rate. At the same shear rate, the differences between oils in Series B are more pronounced, because of higher oil content. In this case, the viscosity of the oil-4 and -5 blends is lower than in the other blends. According to Table II, the aromatic content of these oils are the greatest and, consequently, they seem to behave better as plasticizer than the others.

Owing to blends of Series C having a higher oil content than Series A but lower than Series B, we can observe some differences in the behavior of the five oils. At a high shear rate, the apparent viscosity of 5 and 4 oil blends are lower than the others, as with Series B. The shear rate affects the behavior of the blends in this series.

The log-log plots of apparent viscosity versus shear rates, with the $L/D = 15$ ratio die, are shown in Figure 6. All blends of Series A present apparent viscosity versus shear rate lines similar to the other die, except at very low shear rate. The plots of Series C seem more homogeneous with this die; the die does not seem to influence the behavior and the viscosity values are closer at the same shear rate.

Behavior at low temperature

The study of the behavior at low temperature of the compounds was undertaken by both the Gehman Method and the dynamic mechanical analysis.

The loss factor-temperature curves have a maximum that coincides with the glass transition temperature (T_g) of the compounds. With the use of plasticizer, the maximum loss factor peak shifts to low

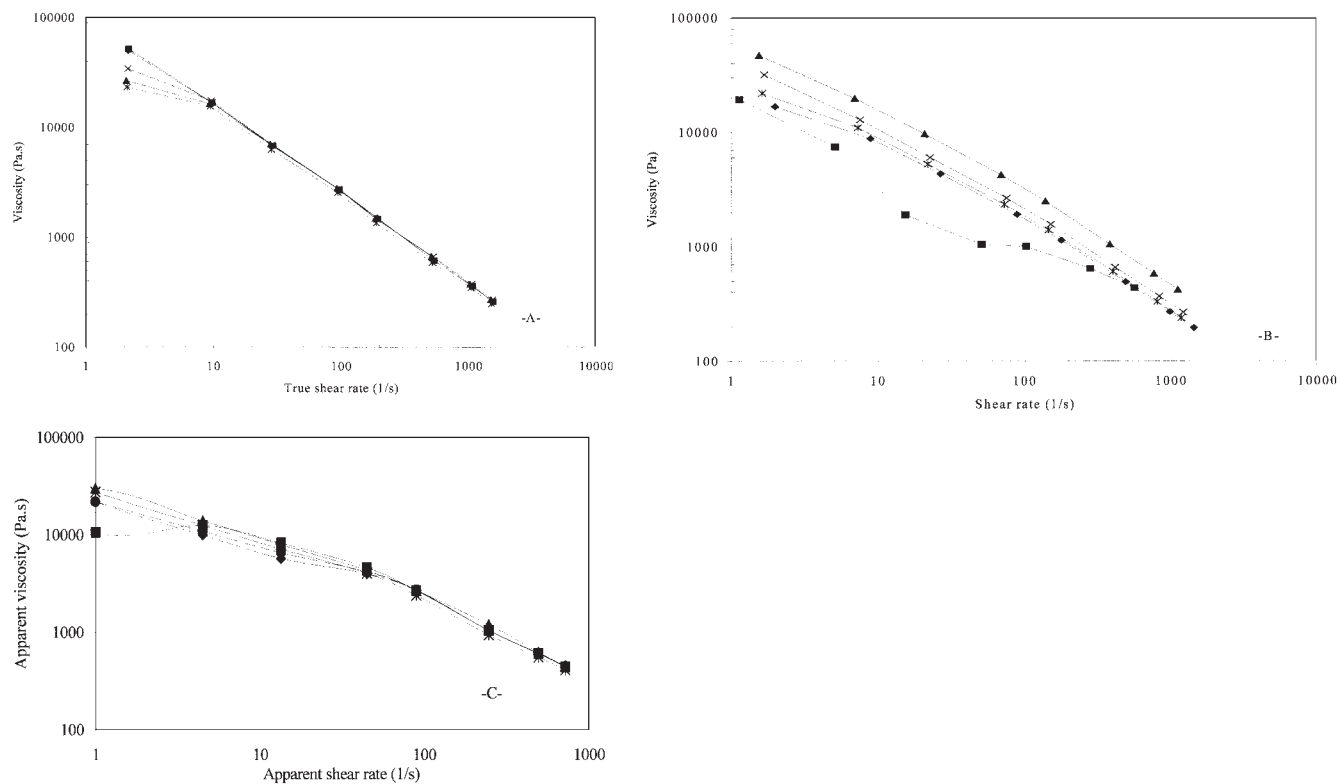


Figure 6 Log-log apparent viscosity versus shear rate plots. $L/D = 15$. Symbols as in Figure 5.

temperature and the peak decreases and expands. Figure 7 shows the peaks of the $\tan \delta$ curves of the compounds as a function of temperature and Table VI

shows the T_g temperatures of the all compounds determined from $\tan \delta$ and E'' curves, respectively. In the three series, we observed that the g 's of 1, 2, and 5

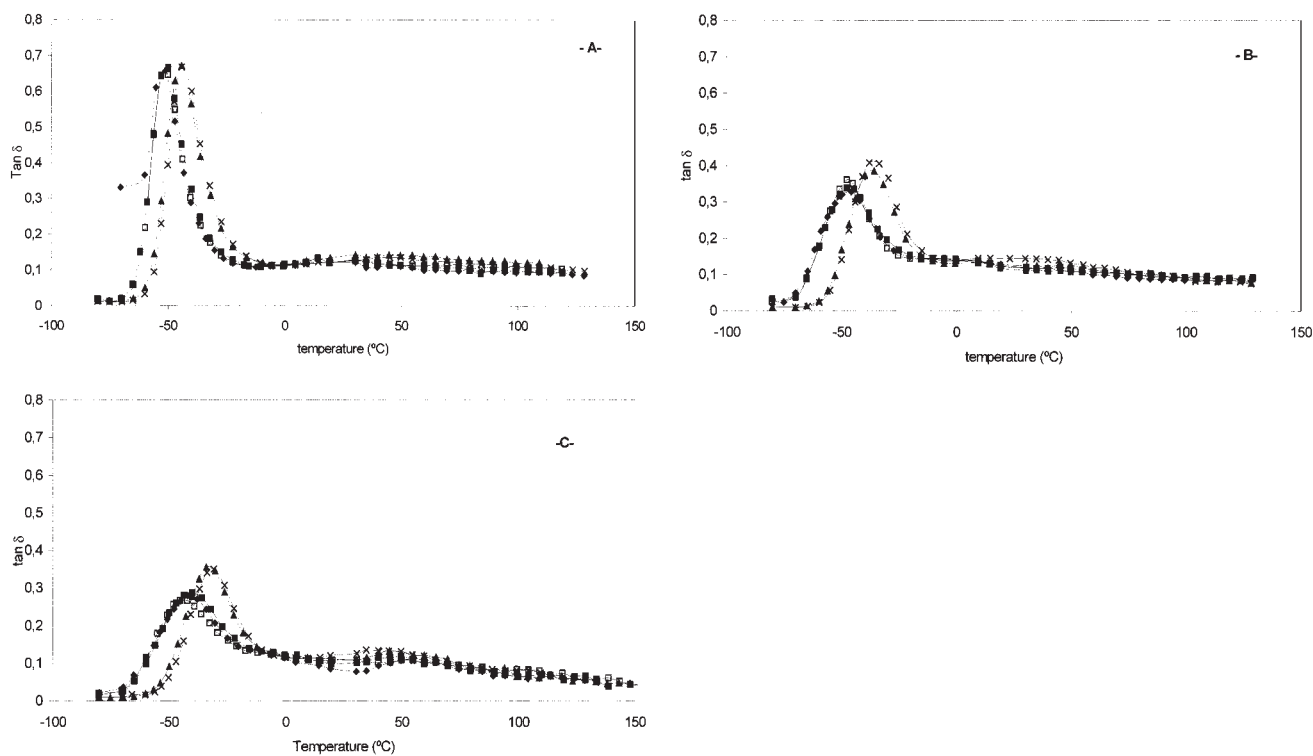


Figure 7 Variation of loss tangent ($\tan \delta$) of the blends at 5 Hz frequency. Symbols as in Figure 5.

TABLE VI
Glass Transition Temperatures of Compounds with Various Paraffinic Oils by Using $Tan \delta_{max}$ and Loss Modulus Max (E'')

Compounds	T_g ($\tan \delta$) (°C)	T_g (E'') (°C)
A-1	-52.5	-56.1
A-2	-51.2	-55
A-3	-45.0	-51.9
A-4	-43.5	-50.5
A-5	-49.0	-54.5
B-1	-47.0	-60.4
B-2	-47.0	-58.2
B-3	-35.8	-47.5
B-4	-35.5	-46.0
B-5	-48.5	-58.4
C-1	-41.6	-56.1
C-2	-40.9	-54.8
C-3	-32.3	-42.7
C-4	-30.2	-41.1
C-5	-41.6	-55.9

compounds are approximately similar but the T_g 's of 3 and 4 compounds are higher than the others. There is an absolute agreement between the T_g of the oil and the T_g of the compound in every series. So, the 1, 2, and 5 compounds show better behavior at low temperature.

We can make a graph of the T_g versus d . The graph of the different oils/rubbers is given in Figure 8. The graph in Figure 8 presents a maximum for $d = 6.35-6.40$ (cal/cm^3)^{1/2} and is similar to that in Figure 4. This indicates that if we use an oil with $d = 6.38$ (cal/cm^3)^{1/2} in the formulation, we can obtain an EPDM

compound with excellent properties at low temperatures and good behavior when processed.

Table VII shows T_2 , T_5 , T_{10} , and T_{100} of all compounds, determined by using the Gehman Method. The lower the temperatures T_2 , T_5 , T_{10} , and T_{100} , the better the properties at low temperature of the vulcanized rubber. The T_5 data indicate the temperature at which the elastomer samples are five times stiffer¹⁸ than their modulus at room temperature. This temperature is closer to the temperature where elastic properties should cease on cooling. The T_5 temperatures¹⁸ are higher than T_g values obtained by the other method, but in the three series, the trend is the same.

T_5 of the 1, 2, and 5 compounds in the three series is smaller than T_5 of the 3 and 4 compounds so the 1, 2 and 5 compounds show the better behavior at low temperature, as we commented earlier when discussing the dynamic mechanical analysis.

CONCLUSIONS

The influence of five paraffinic plasticizer oils on Mooney viscosity, rheological properties, and behavior at low temperature were investigated. The following conclusions can be drawn.

Mooney viscosity of the compounds is dependent on oil viscosity in such a manner that the greater the oil viscosity, the higher the Mooney viscosity.

The oil composition plays an important role in the flow behavior index that is also dependent on the ethylene content of rubber.

The flow index is related to the solubility parameter of the oil. This relation depends on the type of elas-

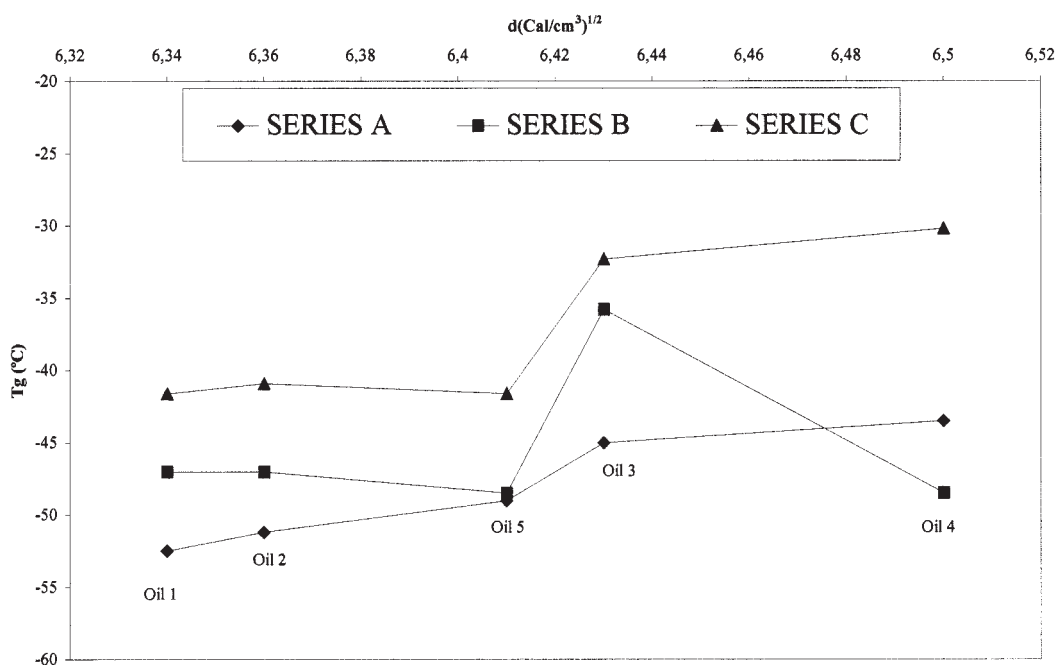


Figure 8 T_g (°C) versus d (cal/cm^3)^{1/2}; Series A (▲); Series B (■); and Series C (▲).

TABLE VII
T₂, T₅, T₁₀, and T₁₀₀ of Compounds with Various Paraffinic Oils by Using Gehman Method

Compounds	α , 25°C	T ₂ (°C)	T ₅ (°C)	T ₁₀ (°C)	T ₁₀₀ (°C)	Apparent torsion modulus, 25°C (mPa)
A-1	166	-24	-45.2	-53.4	-59.4	0.084
A-2	165	-24	-44.2	-52.5	-59.2	0.091
A-3	165	-24	-39	-48.8	-58	0.091
A-4	162	-14.5	-34.8	-42.2	-52.8	0.011
A-5	160	-18.4	-42.2	-49.2	-59	0.125
B-1	161	9.3	-20.8	-35.9	-50.4	0.118
B-2	160	5.8	-22.1	-35	-48.4	0.125
B-3	161	3	-19	-29.3	-41	0.118
B-4	161	5.7	-16	-25.9	-39.2	0.118
B-5	161	9.3	-16.5	-32.7	-48.9	0.118
C-1	144	8.4	-23.2	-33.5	-49.2	0.25
C-2	146	8.1	-21.5	-31.7	-48	0.23
C-3	145	3.5	-20.5	-27	-40	0.24
C-4	143	4.9	-17.2	-23.5	-38.5	0.26
C-5	142	7.1	-21.2	-30	-46.9	0.27

tomer and is inverse for Dutral TER 4049 and direct for the other two.

The viscosity of the compounds is related to the aromatic content of the oil, leading to the compounds with oil-4 and oil-5 having the lowest viscosity.

The behavior at low temperature is related to the T_g of the oil as evaluated by $\tan \delta$ maximum peak and Gehman Method measurements. In this sense, the compounds with oil-1, oil-2, and oil-5 show the best behavior, independent of rubber type.

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