

# Preparation and Characterization of Low-Halogen and Nonhalogen Fire-Resistant Low-Smoke (FRLS) Cable Sheathing Compound from Blends of Functionalized Polyolefins and PVC

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## SYNOPSIS

An attempt is made to develop a novel series of cable sheathing compounds with variation in chlorine content and sufficient fire retardance and unique low-smoke characteristics. These are prepared by blending PVC and functionalized polyolefins in different compositions. PE and EPDM have been functionalized by grafting dibutyl maleate (DBM) using DCP as initiator. FRLS compounds made from PVC-functionalized polyolefin blends possess the special characteristics of low-smoke, low-acid-gas generation, increased fire retardance, and improved volume resistivity, which are much better in comparison with a typical PVC sheathing compound. Thermoplastic elastomer (TPE) based nonhalogen FRLS compounds are also reported. The mechanisms for grafting, polymer-polymer and polymer-filler interactions have been presented.

## INTRODUCTION

The widespread use of electric cables in a large number of areas often causes outbreaks of fire either by accidental short circuit condition or by burning of some extraneous matter that subsequently ignites the cables. For many years many halogen-containing polymers have been used as the base material for fire-resistant sheathing compounds in the manufacture of electric cables. Because poly(vinyl chloride) (PVC) has good flame resistance properties coupled with other desirable properties such as good mechanical properties and easy processing, it is the obvious choice for cables of such applications for a long time. However, recently it has been recognized that conventional cable sheathing compounds based

on PVC can present serious problems in the event of fire due to the formation of large amounts of dense black smoke together with acidic and toxic fumes. This becomes a serious concern particularly when a large number of people gather in a confined areas such as underground railways, hotels, exhibition halls, ships, factories, etc. Moreover, the formation of highly acidic and corrosive fumes can damage costly equipment such as computers, electronic systems, telephone exchanges, etc.<sup>1</sup>

So, it is highly desirable to have a sheathing (protective or jacketing) compound that will be fire resistant on one hand and will have reduced or no hazards from generation of smoke, toxic, and corrosive fumes.<sup>2-4</sup> In 1981, the U.S. Navy Shipbuilding Program Office initiated work for replacement of multiconductor PVC-jacketed cables with low-smoke/low-toxicity (LS/LT) jacketed cables.<sup>5</sup>

This has led to the development of nonhalogen fire-retardant thermoplastic compounds with an aim to manufacture cables that generate only very low

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amounts of smoke and toxic fumes during fire.<sup>1,2,4</sup> But in most of the cases the formulations are not disclosed.

A set of properties such as electrical, mechanical, weather resistance, and very low smoke generation on burning possessed by polyolefins combined with fire retardance properties of PVC are expected to provide better combination of properties such as cable sheathing compounds having significant technological importance. Polyolefins are not compatible with PVC,<sup>6</sup> however; blending of these polymers results in poor mechanical properties.

The improved performance of olefinic thermoplastic elastomers along with its processing advantages have made possible their wider acceptance in the wire and cable industry, and currently they are being used in flexible cables, control cables, welding cables, etc.<sup>7</sup>

In the present investigation efforts are made to develop nonhalogen and low-halogen FRLS thermoplastic elastomer compounds based on functionalized polyolefins. Attempts have also been made to develop a set of compounds for sheathing applications based on functionalized polyolefins-PVC blends having sufficient fire retardance and low-smoke properties to meet the required specification.

## EXPERIMENTAL

**Materials.** Low-density polyethylene (LDPE) (NESTE Polyethylene DFDS, 4445 of Neste Polyeten AB, Sweden) of density 0.915 g/cm<sup>3</sup> and MFI 2.0 g/10 min (ASTM-D 1238), ethylene propylene 1,4 hexadiene terpolymer (EPDM) (Nordel 2760 of DuPont), PVC (Indovin of IPCL, India) of K value 65, acrylonitrile butadiene copolymer (NBR) (Krynac 3450 of Polysar, Canada) were used. Dibutyl maleate (DBM), dicumyl peroxide (DCP), and other ingredients were of standard laboratory grade.

**Graft Modifications and Blending.** Grafting of dibutyl maleate (DBM) on PE and EPDM was carried out in a Brabender Plasticorder (PLE 330) using a cam-type mixing head N 50H. Different blend compositions were made in the same mixer. PVC was used always in stabilized and plasticized form with the formulation as PVC-100; tribasic lead sulphate (TBLS), 6; calcium stearate (C.S.), 1; and trioctyl trimellitate (TOTM), 50.

**Infrared Spectroscopy.** Infrared spectroscopy was carried out in Shimadzu IR-420 using thin film of

polymer. Free DBM was removed by acetone extraction of the film.

**Differential Scanning Calorimetry.** A Mettler DSC-20 of TA-3000 System was used. Heating rate was 10°C/min.

**Mechanical Properties.** Tensile properties were studied by Zwick UTM (Model 1445) using dumb-bell specimen (ASTM D 412-80).

**Limiting Oxygen Index (LOI).** LOI of the samples was determined according to ASTM-D 2863-81 specification.

**Smoke Density Test.** The specification D-2843-81 and NBS Specification ASTM-E-662-83 were followed for smoke density measurement.

**HCl Gas Generation Test.** This test was performed according to the specification IEC 754-1.

**Scanning Electron Microscopy (SEM).** SEM was carried out in Hitachi SEM Model S145A. Samples were prepared by extraction with cyclohexanone.

**Volume Resistivity.** Volume resistivity was measured by a Hewlett-Packard 4329A high-resistance meter, according to the specification ASTM D 257-78.

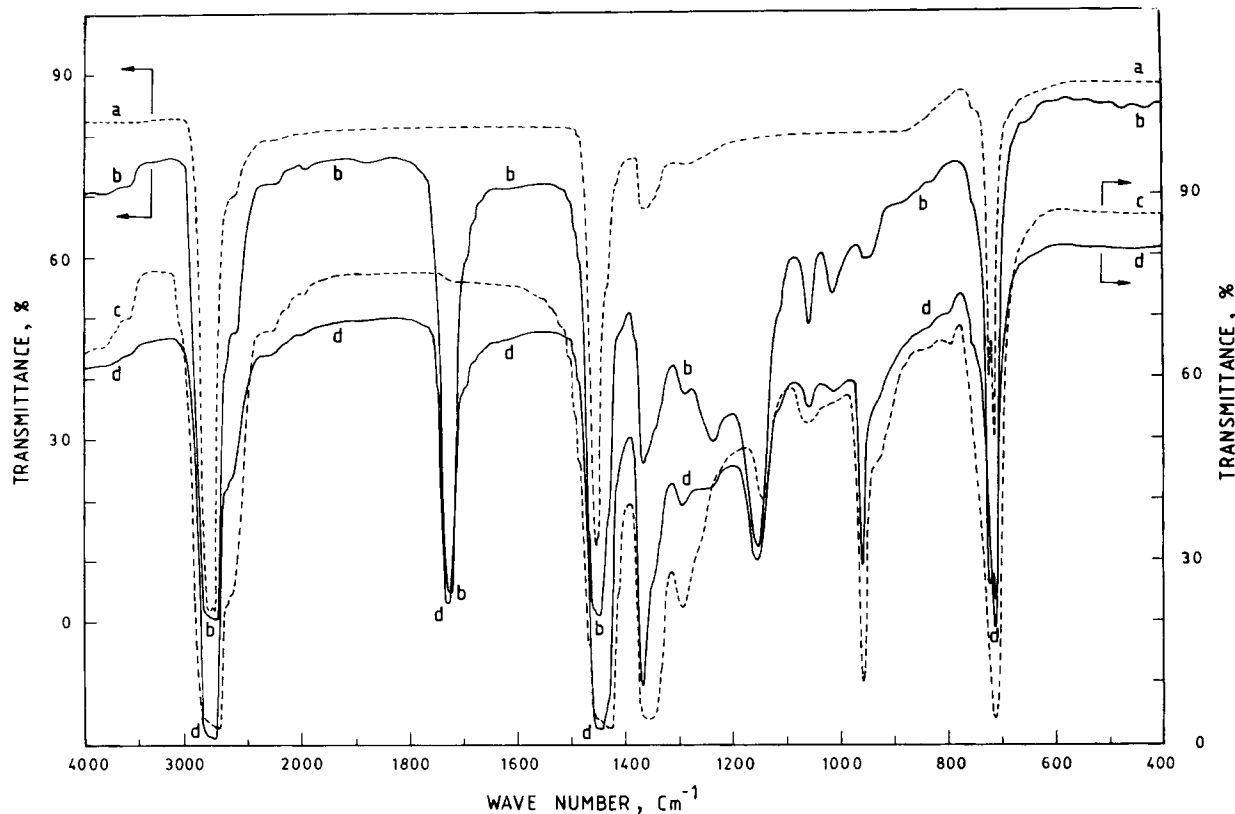
**Static Thermal Stability.** The sample (10 g) was taken in a long test tube and heated to a constant temperature of 200°C in a silicone oil bath. Congo red paper was hung from the mouth of a closed tube with cotton. Stability time was taken as that time when the Congo red paper turned brown.

**Thermogravimetry.** A Mettler TG 50 of a TA 3000 system with TC-10A microprocessor was used throughout the study. The heating rate was 20°C/min.

## RESULTS AND DISCUSSION

### Functionalization of Polyolefins and Its Characterization

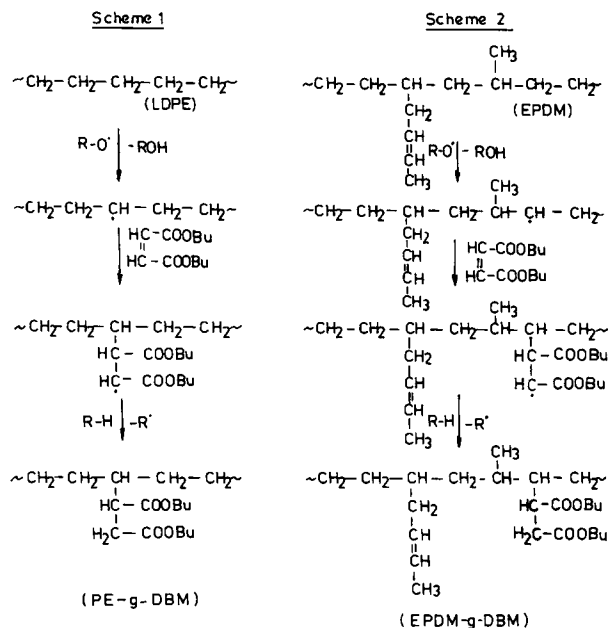
Both polyethylene (PE) and ethylene propylene diene terpolymer (EPDM) were functionalized by grafting dibutyl maleate (DBM) using dicumyl peroxide (DCP) as initiator at the temperature range of 140–200°C. Optimization of the reaction condi-



**Figure 1** Infrared spectra of (a) PE, (b) PEgDBM, (c) EPDM, and (d) EPDMgDBM.

tions and reactant concentrations was also done. The optimum concentration of DBM is 10% for both polymers. The results are in good agreement with that obtained by Greco et al. for ethylene propylene copolymer (EPR).<sup>8</sup> The optimized concentration of DCP is 0.2% for EPDM and 0.5% for PE. Beyond this concentration of DCP, crosslinking of the respective polymers takes place. The conditions for graft modification reactions are 20 min at 160°C.

IR spectra of starting and functionalized PE and EPDM are reported in Figure 1. Both functionalized polymers show a typically strong absorption band at 1738 cm<sup>-1</sup> associated with stretching absorption of >C=O in the ester groups of the attached maleate units. In general, the resulting degree of functionalization is high for both the polymers. However, a higher degree of graft modification for PE is obtained due to the absence of unsaturation and high chain mobility. This result is also in line with that obtained by Benedetti et al. for diethyl maleate grafting.<sup>9</sup> The graft modification reactions of PE and EPDM in the presence of DCP may be visualized in Schemes 1 and 2 (Fig. 2).



**Figure 2** Schematic representation of the graft modification reactions. Scheme 1 for PE and scheme 2 for EPDM. In the case of EPDM grafting may also take place at the allylic position.

**Table I Comparison of Properties between TPEg and TPE**

	Gel Fraction (%)	Tensile Strength (MPa)	EB%	Volume Resistivity ( $\Omega$ -cm)	LOI	Thermal Properties (% Crystallinity)	Peak Melting Point ( $^{\circ}$ C)
TPEg	41	8.6	505	$1.027 \times 10^{17}$	17.2	8.1	104.0
TPE	42	7.5	350	$2.5 \times 10^{17}$	17.0	8.4	104.5

### Preparation and Properties of Thermoplastic Elastomer from Functionalized Polyolefins and Nonhalogen FRLS Compound

Two types of thermoplastic elastomers have been made. The compound made by simultaneous grafting and dynamic curing technique is designated as TPEg. The formulation used for making this compound is PE-25 + EPDM-75 + DBM-10 + DCP 0.35. Grafting and dynamic curing was done at  $160^{\circ}$ C. To compare the properties at similar gel fraction, another thermoplastic elastomer has also been made using the composition PE-25 + EPDM-75 + DCP 0.25 designated as TPE. The physical properties of these compounds are tabulated in Table I.

It is observed from Table I that TPEg has higher tensile strength and percent elongation at break than that of TPE due to the higher interaction between the two components in the presence of the grafted maleate group. Both the compounds have similar volume resistivity and LOI values. The percent crystallinity and melting point are also almost similar for both compounds.

**Table II Formulation of FRLS Compound**

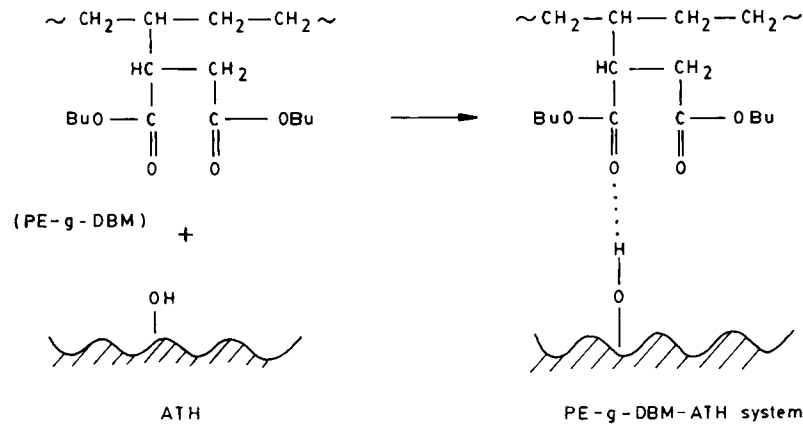
TPEg or TPE	100
Sb <sub>2</sub> O <sub>3</sub>	10
Mo <sub>2</sub> O <sub>3</sub>	5
Pentabromodiphenyl oxide	6
Ethylene bistetrabromodiphenyl oxide	6
ATH	50
Zinc magnesium sulfate	2

Fire-retardant properties of the compounds discussed are very poor as indicated by their low LOI value (Table I). Both the thermoplastic elastomers could, however, be made FRLS compounds by mixing with some common fire-retardant additives such as aluminium trihydrate (ATH), molybdenum trioxide (Mo<sub>2</sub>O<sub>3</sub>), antimony trioxide (Sb<sub>2</sub>O<sub>3</sub>), and special additives such as brominated compounds. As a smoke suppressant zinc magnesium sulfate was added. Through a series of experiments the formulation was selected in such a way so that the FRLS compound made from TPEg could reach an LOI value of 30, which is generally accepted as the minimum value required to meet the fire test according to specification IEE 383, BS 4066 Part 3, and IEC 332 Part 3. In order to compare the performance, TPE was also made into a FRLS compound using the same formulation. The typical formulation is given in Table II.

The mechanical and electrical properties, smoke properties, and resistance to fire of both the low halogen containing FRLS compounds are given in Table III. It is observed from Table III that the TPEg FRLS compound possesses significantly higher tensile strength and percentage elongation at break, higher LOI, and better low-smoke properties compared to the TPE FRLS compound. Unlike the TPE FRLS compound, the TPEg FRLS compound achieves the LOI value of 30.3, which meets the specification mentioned earlier. These differences in tensile and fire resistance properties between the two types of FRLS compounds arise due to compatibilization of ATH with the polymer matrix of the TPEg FRLS compound. The compati-

**Table III Comparison of Properties between TPEg FRLS and TPE FRLS Compounds**

	Tensile Strength (MPa)	EB%	Volume Resistivity ( $\Omega$ -cm)	LOI	Smoke Density	Minimum % of Light Transmission
TPEg FRLS	8.4	240	$8.2 \times 10^{15}$	30.3	108	81
TPE FRLS	6.4	140	$8.4 \times 10^{15}$	27.2	125	75



**Figure 3** Schematic representation of H bond formation between the hydroxyl group of ATH and maleate group of PEgDBM.

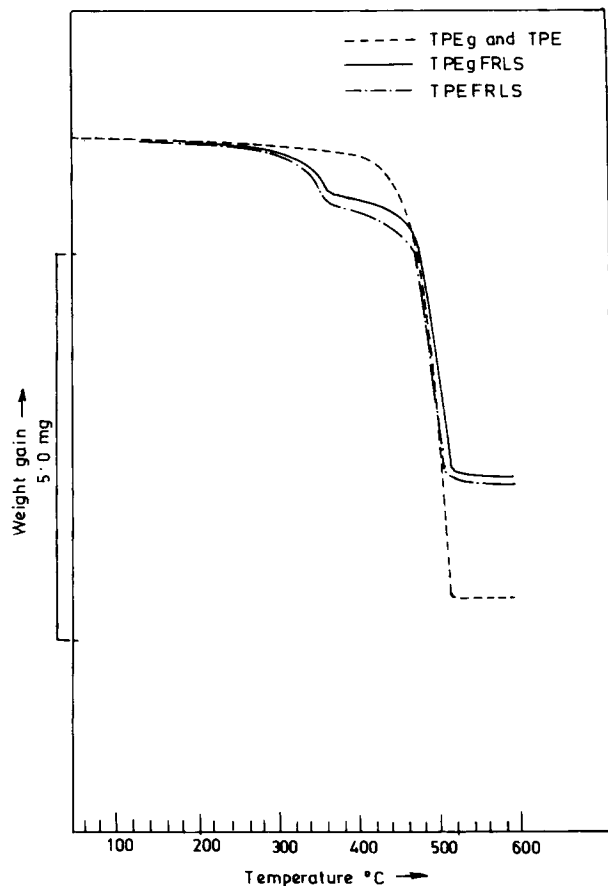
bilization presumably involves the reaction of the pendant ester groups with the hydroxyl groups of the ATH. The interaction between the maleate moieties and ATH hydroxyl group can be depicted as shown in Figure 3.

The decomposition characteristics of TPEg and TPE as well as the FRLS compounds made from them were studied by thermogravimetry (TG) under nitrogen atmosphere at a heating rate of 20°C/min. The TG traces are presented in Figure 4. The decomposition characteristics of both the TPEg and TPE are almost identical. But in the case of FRLS compounds the TPEg FRLS compound possesses higher stability in terms of onset of decomposition and loss in weight. The first peak is mainly due to the loss of H<sub>2</sub>O from ATH. The lower loss in weight in the case of the TPEg FRLS compound may be due to the increased interaction between ATH and the polymer matrix through DBM moiety as described earlier. Hence, it can be concluded that the developed nonhalogen FRLS compound meets the requirements of the cables (specification IEE 383 and IEC 332 Part 3).

#### Preparation of Low-Halogen FRLS Compounds by Blending PVC with Functionalized PE and EPDM

The properties of the blends of PVC with PE and EPDM are given in Table IV. PVC shows a tensile strength of 20 MPa. With the introduction of PE, the tensile strength drops gradually. Similar results are obtained for EPDM systems. The elongation at break also shows a similar trend in both cases. The decrease in strength and elongation may be ascribed to the incompatibility of these polymeric systems.

McGrath et al. reported the incompatibility of these two systems.<sup>6</sup> The SEM photographs of the pure polyolefins and PVC blends after cyclohexanone extraction are shown in Figure 5. The phase (layer)



**Figure 4** Thermogravimetric traces of TPEg, TPE, TPEgFRLS, and TPE FRLS compounds.

**Table IV Comparison of Properties between PVC and PVC–Polyolefins Blends**

Blend System	Tensile Strength (MPa)	Elongation at Break (%)	Volume Resistivity ( $\Omega$ -cm)	LOI	HCl Gas Generation (%)	Static Thermal Stability (min)
PVC-100	20	250	$3 \times 10^{13}$	25.5	32.0	96
PVC-85 + PE-15	5.5	62	$7 \times 10^{13}$	24.8	27.5	103
PVC-75 + PE-25	4.0	48	$2 \times 10^{14}$	24.5	26.0	108
PVC-85 + EPDM-15	5.9	68	$7.5 \times 10^{13}$	24.8	27.6	103
PVC-75 + EPDM-25	5.1	60	$2.4 \times 10^{14}$	24.5	26.2	110

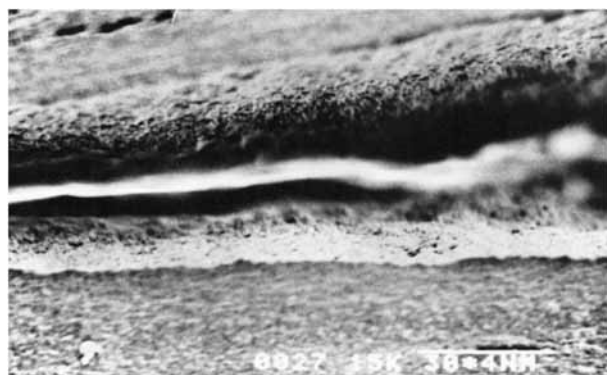
separation and nonuniform dispersion of the blends are apparent from the micrographs.

As expected, the results also show the gradual improvement of volume resistivity, HCl gas generation, and static thermal stability by blending PE or EPDM. However, a decrease in LOI values is observed with the decrease in the PVC content. Smoke density test could not be precisely done due to the excessive smoke generation. However, both LOI and smoke generation can be improved by the addition

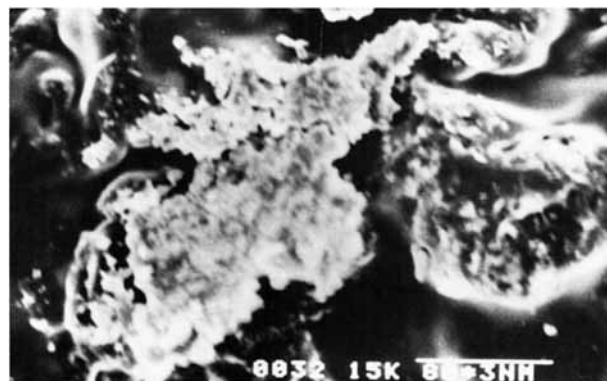
of flame retardants and smoke suppressants. But, the major constrain of these blend systems for its application in the cable manufacture is the very poor mechanical properties. The mechanical properties could be enhanced by improving the physical or chemical compatibility. These are reported for various polymer blends.<sup>10–12</sup> In order to make PVC compatible with PE or EPDM, experiments have been carried out in two ways: (i) Blending functionalized PE or EPDM with PVC (Table V) and (ii) adding functionalized PE or EPDM (5 parts) as compatibilizing agents. Various properties of such blends are reported in Tables V and VI. It may be observed from Table V that the blends containing functionalized PE and EPDM result in considerable improvement in tensile properties over pure blends. Static thermal stability also increases, but other properties remain more or less the same. This improvement in tensile properties is due to the uniform dispersion of the modified blends as shown in Figure 6. Better compatibility is also evident from the lower melting temperature and melting endotherm of functionalized PE in its blends with PVC (Fig. 7). This observation is similar to the PE–EPDM blends<sup>13</sup> reported earlier.

Similarly, a few compositions were studied to observe the role of functionalized polyolefins to make PVC–polyolefins blends compatible. It is observed from Table VI that though the properties are considerably better than normal PVC–polyolefins blends (Table IV), they are almost similar when compared with the properties of PVC–functionalized polyolefin blends. But this comparison suggests that about 5 parts of functionalized polyolefins are sufficient for compatibilization of the PVC–polyolefins blends, at least up to the ratio studied here.

However, the mechanical properties reported in the Tables V and VI are far below the industry norms. As such for further improvement the blend systems were modified with the help of dynamic crosslinking. In a few cases small portion of acrylonitrile butadiene rubber (NBR) was also added



(a)



(b)

**Figure 5** SEM photographs of PVC–PE (75 : 25) blends: (a) phase (layer) separation, (b) nonuniform dispersion.

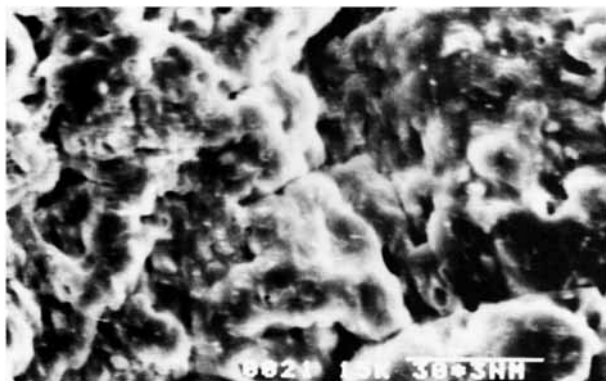
**Table V** Various Properties of PVC-Functionalized Polyolefins Blends

Blend System	Tensile Strength (MPa)	Elongation at Break (%)	Volume Resistivity ( $\Omega$ -cm)	LOI	HCl Gas Generation (%)	Static Thermal Stability (min)
PVC-85 + PEgDBM-15	8.4	93	$6.8 \times 10^{13}$	24.7	24.2	107
PVC-75 + PEgDBM-25	7.5	88	$1.5 \times 10^{14}$	24.5	22.9	116
PVC-85 + EPDMgDBM-15	9.8	105	$7.6 \times 10^{13}$	24.8	23.6	108
PVC-75 + EPDMgDBM-25	9.1	98	$2.6 \times 10^{14}$	24.4	22.0	118

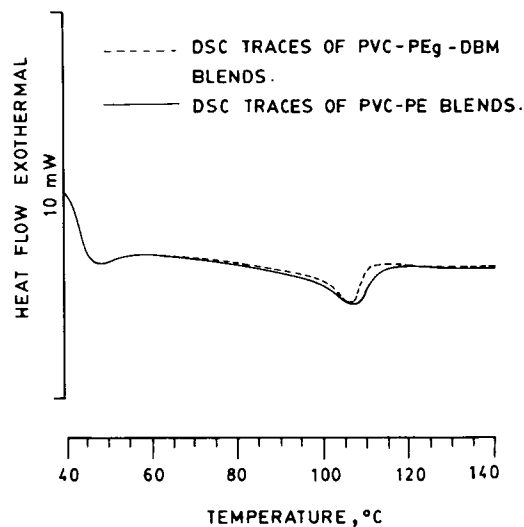
**Table VI** Properties of the PVC-Polyolefins Blends Compatibilized with Functionalized Polyolefins

Blend System	Tensile Strength (MPa)	Elongation at Break (%)	Volume Resistivity ( $\Omega$ -cm)	LOI	HCl Gas Generation (%)	Static Thermal Stability (min)
PVC-85 + PEgDBM-5 + PE-10	8.5	86	$6.9 \times 10^{13}$	24.7	24.6	105
PVC-75 + PEgDBM-5 + PE-20	7.7	80	$1.5 \times 10^{14}$	24.4	23.4	111
PVC-85 + EPDMgDBM-5 + EPDM-10	9.6	104	$7.7 \times 10^{13}$	24.7	23.9	106
PVC-75 + EPDMgDBM-5 + EPDM-20	9.0	94	$2.8 \times 10^{14}$	24.3	22.4	112

for better compatibility. NBR was selected because it forms compatible blends with PVC.<sup>14</sup> The tensile properties of a few optimized systems are given Table VII. Significant improvement in tensile properties has been achieved. The grafting of DBM on PE and EPDM gives rise to interaction mainly through a hydrogen bond between the carbonyl group of DBM and methine H of PVC. Besides, dipole-dipole interactions of the type  $\text{>C=O} \cdots$

**Figure 6** SEM photograph showing uniform dispersion of PVC-PEgDBM (75 : 25) blends.

$\text{Cl-C}$  may also exist. This kind of interaction has been evidenced on the basis of a progressive shift toward lower frequencies and broadening of the carbonyl band as a function of PVC concentration.<sup>8</sup> The NBR act as co-compatibilizer and increases the

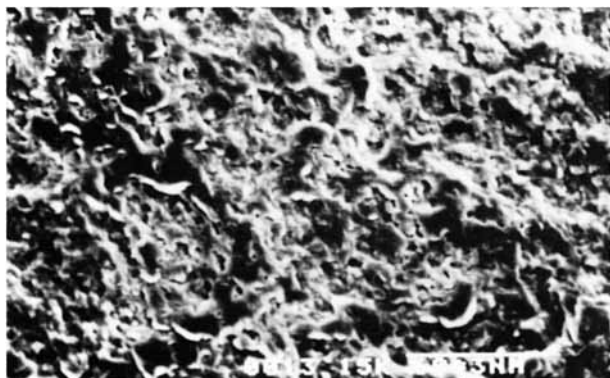
**Figure 7** DSC traces of PVC-PE and PVC-PEgDBM blends.

**Table VII Comparison of Tensile Properties for Different Blend Systems Prepared by Dynamic Vulcanization Techniques**

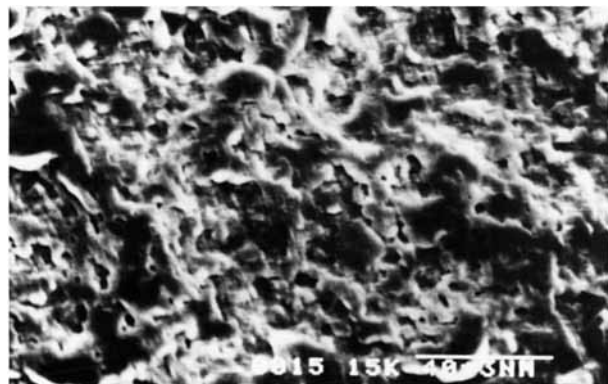
	Tensile Strength (MPa)	Elongation at Break (%)
PVC-85 + XLPE-10 + PEgDBM-5	10.5	107
PVC-75 + XLPE-20 + PEgDBM-5	9.8	100
PVC-85 + XLPE-10 + PEgDBM-5 + NBR-2	13.5	135
PVC-75 + XLPE-20 + PEgDBM-5 + NBR-4	12.2	120
PVC-85 + XLEPDM-10 + EPDMgDBM-5	11.6	125
PVC-75 + XLEPDM-20 + EPDMgDBM-5	11.0	115
PVC-85 + XLEPDM-10 + EPDMgDBM-5 + NBR-2	14.5	150
PVC-75 + XLEPDM-20 + EPDMgDBM-5 + NBR-4	13.8	140

XLPE and XLEPDM are crosslinkable PE and EPDM containing two parts of DCP.

adhesion between functionalized polyolefins–PVC interface during mixing, enabling further improvement in dispersion. Finally the morphology developed during mixing by chemical and mechanical compatibilization becomes permanent by dynamic



(a)

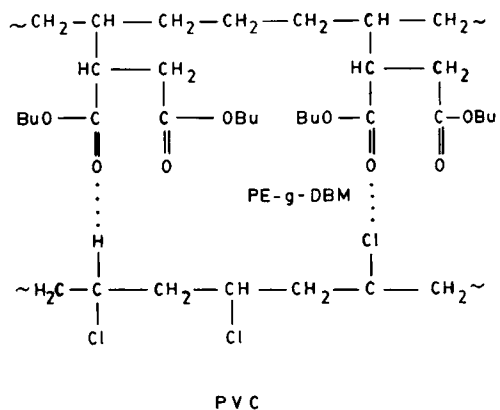


(b)

**Figure 8** SEM photographs showing interpenetration and uniform dispersion of (a) PVC-75 + XLPE-20 + PEgDBM-5 + NBR-4 and (b) PVC-75 + XLEPDM-20 + EPDMgDBM-5 + NBR-4 blends.

crosslinking, resulting in significant improvement in tensile properties. The modified morphology developed by adding NBR and dynamic crosslinking is shown in Figure 8. The compatibilization between the polymers may be visualized as shown in Figure 9. It may be concluded that using the method of compatibilization and dynamic crosslinking, the tensile properties could meet the level of requirement.

The blends optimized for tensile properties do not, however, meet the fire resistance properties. Thus, FRLS compounds were made by mixing with some common fire-retarding additives, such as aluminium trihydrate (ATH), molybdenum trioxide ( $\text{Mo}_2\text{O}_3$ ), and antimony trioxide ( $\text{Sb}_2\text{O}_3$ ). As a smoke suppressing ingredient zinc magnesium sulfate was also used.<sup>15</sup> A typical formulation is given in Table VIII, and the base polymer–polymer blends studied for FRLS compound are given in Table IX.



**Figure 9** Mechanism of compatibilization between PEgDBM and PVC through H bonding and dipole–dipole interactions.



**Table VIII Formulation of FRLS Compound**

Base polymer-polymer blends	100
Molybdenum trioxide (Mo <sub>2</sub> O <sub>3</sub> )	8
Antimony trioxide (Sb <sub>2</sub> O <sub>3</sub> )	6
Aluminium trihydrate (ATH)	45
Zinc magnesium sulfate	2

### Mechanical Properties

The mechanical properties of FRLS compounds and their percentage change after heat air aging at 100°C for 7 days are given in Table X. It is observed from the results that the mechanical properties of the PVC drop marginally [21% of tensile strength (TS) and 28% for elongation break (EB)] on addition of ATH and other ingredients. But in the case of blends containing functionalized polyolefins, there is little improvement in tensile properties. This improvement may result from the compatibilization of ATH with polymer systems. The compatibilization presumably involves the reaction of the pendant ester group with the hydroxyl groups of the ATH as described earlier (Fig. 3).

Note also that the tensile strength and elongation at break meet the industry norms even after the addition of flame retardants. The aging resistance properties are also satisfactory and almost the same for all compounds.

### Fire Resistance, Smoke Density, and Acid Gas Generation Properties

The limiting oxygen index (LOI) values, smoke density, HCl gas generation, and thermal stability for the different compounds are given in Table XI. The results show that LOI values of all the low-halogen FRLS compounds made from PVC-PEgDBM and PVC-EPDMgDBM blends are higher than PVC FRLS compound. The most remarkable

**Table IX Compositions of the Base Polymer-Polymer Blends Used for Making FRLS Compounds**

A <sub>F</sub>	PVC-100
B <sub>F</sub>	PVC-85 + XLPE-10 + PEgDBM-5 + NBR-2
C <sub>F</sub>	PVC-75 + XLPE-20 + PEgDBM-5 + NBR-4
D <sub>F</sub>	PVC-85 + XLEPDM-10 + EPDMgDBM-5 + NBR-2
E <sub>F</sub>	PVC-75 + XLEPDM-20 + EPDMgDBM-5 + NBR-4

Suffix F denotes FRLS compounds made according to the formulation given in Table VIII.

**Table X Mechanical Properties of Different FRLS Compounds**

FRLS Compounds	TS (MPa)	EB (%)	Percentage Change on Aging	
			TS	EB (%)
A <sub>F</sub>	15.8	180	+2.0	-3.5
B <sub>F</sub>	14.0	150	+1.5	-3.0
C <sub>F</sub>	13.1	143	+2.5	-4.0
D <sub>F</sub>	15.1	160	+2.0	-3.0
E <sub>F</sub>	14.3	153	+3.0	-4.5

TS = tensile strength and EB = elongation at break.

improvement is observed for the compounds D<sub>F</sub> and E<sub>F</sub>, i.e., for PVC-EPDMgDBM blends. In general, LOI decreases with the decrease in PVC content, but here this improvement in flame retardancy occurs probably through the enhanced interaction of the maleate groups with the flame-retardant additives ATH, Sb<sub>2</sub>O<sub>3</sub>, and Mo<sub>2</sub>O<sub>3</sub>. The rapid propagation of a fire occurs through the formation of very high energy H<sup>•</sup> and OH<sup>•</sup> radicals.<sup>16</sup> The maleate groups may also act as the scavenger of these radicals and suppress the propagation of the flame. In PVC-PEgDBM blends crystallites may be melted in the presence of high heat and flame, though PE is cross-linked. This may generate fresh surface by dripping for flame propagation. But for PVC-EPDMgDBM blends there is no such possibility of dripping. Probably due to this reason PVC-EPDMgDBM blends result in such an improvement in flame retardancy.

It is also observed from Table XI that minimum percent light transmission significantly increases, i.e., smoke generation on burning decreases, by the replacement of PVC with functionalized polyolefins. These compounds were also tested in the NBS-Smoke Chamber. The data obtained also support the preceding result.

It is very interesting to note from the results given in Table XI that acid gas generation significantly decreases with the partial replacement of PVC, the potential source of liberated HCl. The decrease is above 50%. Static thermal stability of all the compounds increases with the addition of fire-retardant ingredients. Moreover, this property further increases with the partial replacement of PVC in the blends.

### Electrical Properties

Initial volume resistivity of the compounds and its change after water immersion are plotted in Figure

**Table XI** The Limiting Oxygen Index (LOI), Smoke Density, HCl Gas Generation, and Thermal Stability of the Different FRLS Compounds

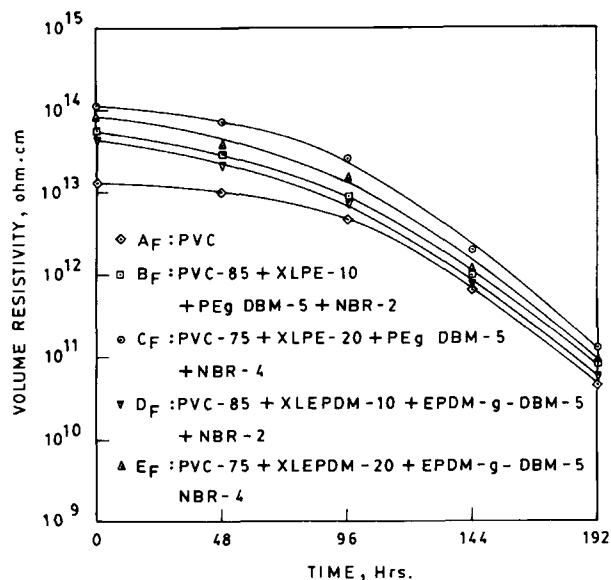
Compound	LOI	Minimum % Light Transmission	Smoke Density (Dm)	% HCl Generated	Static Thermal Stability (min)
A <sub>F</sub>	33.0	41.0	388	15.3	116
B <sub>F</sub>	34.0	50.5	350	7.5	133
C <sub>F</sub>	34.0	58.4	327	6.8	155
D <sub>F</sub>	35.0	51.7	348	7.3	135
E <sub>F</sub>	36.5	60.6	317	6.6	158

10. It is observed that initial volume resistivity increases with the increase in proportion of functionalized PE and EPDM in PVC blends. This is due to the nonpolar nature of PE and EPDM. The volume resistivity measured after immersion of the compound in water decreases with the time of immersion due to the diffusion of water into the systems. The decreasing trend is almost similar for all the blends.

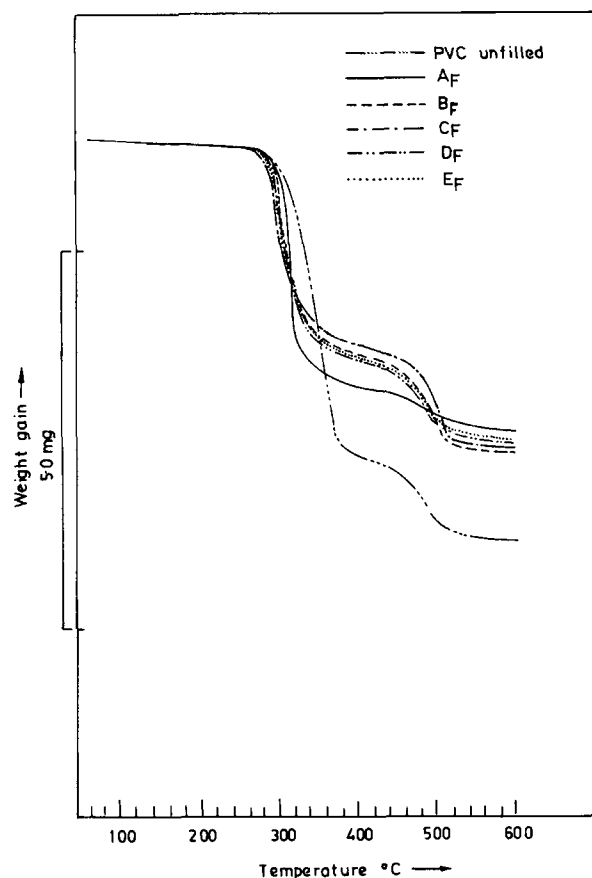
#### Thermogravimetric Analysis

The decomposition characteristics of PVC unfilled compound, PVC FRLS compound, and the other FRLS compounds made by the blends of PVC and functionalized polyolefins were studied by thermogravimetry under nitrogen atmosphere at a heating

rate of 20°C/min. The TG traces are presented in Figure 11, and the results calculated by the built-in software are given in Table XII. The theoretical weight loss due to volatilization and decomposition of different components (ingredients) in the corresponding step is given in the remarks column of Table XII.



**Figure 10** Variation of volume resistivity of different FRLS compounds with time of immersion in water. See Table VIII for FRLS formulation.

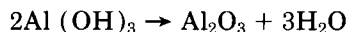


**Figure 11** Thermogravimetric traces of PVC unfilled, PVC filled, and other FRLS compounds. For formulation and composition see Tables VIII and IX.

**Table XII Results of Thermogravimetric Analysis of PVC Unfilled, PVC Filled, and Other FRLS Compounds**

Serial Number	Compound Reference	Step 1				Step 2				Residue At 494 (°C) (%)	Remarks		
		Start Temp. (°C)	Peak Temp. (°C)	End Temp. (°C)	Weight Loss (%)	Start Temp. (°C)	Peak Temp. (°C)	End Temp. (°C)	Weight Loss (%)				
1.	PVC unfilled	166	350	399	70.6	Theoretical weight loss of TOTM = 31.8% HCL due to dehydrochlorination of PVC = 37.2%	399	477	531	17.5	Weight loss due to the decomposition of dehydrochlorinated PVC	10.9	Residue from PVC + residue of inorganic additives
		Total = 69%											
2.	A <sub>F</sub>	180	310	417	56.2	Theoretical weight loss of TOTM = 22.9% HCL = 26.8% (from PVC) H <sub>2</sub> O = 7.1% (from ATH)	490	490	545	9.5	Weight loss due to the decomposition of dehydrochlorinated PVC	32.9	Residue from inorganic additives = 23.7% Residue from PVC = 9.1%
		Total = 56.83%											
3.	B <sub>F</sub>	171	303	406	49.7	Theoretical weight loss of TOTM = 20% HCL = 25.2% (from PVC) H <sub>2</sub> O = 7.4% (from ATH)	406	493	545	16.6	Weight loss due to the decomposition of dehydrochlorinated PVC = 8.6% + Weight loss due to the decomposition of grafted polymer & NBR = 8%	31.3	Residue from inorganic additive = 24.2% Residue from PVC = 8.4%
		Total = 52.6%											
4.	C <sub>F</sub>	165	302	387	48.4	Do	387	484	568	18.1	Do	31.8	Do
		Total = 50.8%											
5.	D <sub>F</sub>	179	300	400	47.3	Theoretical weight loss of TOTM = 18.0% HCL = 20.96% (from PVC) H <sub>2</sub> O = 7.4% (from ATH)	400	496	572	21.2	Weight loss due to the decomposition of dehydrochlorinated PVC = 7.2% + Weight loss due to the decomposition of grafted polymer & NBR = 14.4%	30.2	Residue from inorganic additive = 24.1% Residue from PVC = 7.2%
		Total = 46.3%											
6.	E <sub>F</sub>	176	297	405	45.8	Do	405	494	587	23.1	Do	29.5	Do
		Total = 46.3%											

In step 1, HCl elimination occurs by the dehydrochlorination of PVC, and the plasticizer TOTM is lost due to volatilization. Water elimination also takes place in this step from the ATH of the FRLS compounds ( $A_F$  to  $E_F$ ) by the reaction



For PVC unfilled and other FRLS compounds, the theoretically calculated weight loss in step 1 is almost the same as that obtained experimentally. In step 2 weight loss occurs due to the partial decomposition of dehydrochlorinated PVC and complete decomposition of other polymers present in the systems. After a significant amount of polyene is formed by the dehydrochlorination of PVC, degradation to volatile combustible and crosslinking of the polyene occur.<sup>17</sup> In the step 2 about 50–52% weight loss occurs due to the decomposition of dehydrochlorinated PVC, i.e., from polyene, and the rest remain as residue with other inorganic additives.

It may be noted that the compounds developed would suit the specification for tensile properties, smoke generation, as well as fire resistance properties with lower content of halogen on the polymer.

## CONCLUSION

1. Nonhalogen FRLS compound made by the functionalized polyolefin-based thermoplastic elastomer (TPE) has been developed. This compound possesses good electrical and mechanical properties, very low smoke generation, and sufficient fire retardance properties and is acceptable to the cable industry.
2. A series of low-halogen cable sheathing compounds have also been developed by blending PVC and functionalized polyolefins.
3. Precise comparisons among all the systems with respect to the electrical, mechanical, aging, and above all fire retardance, smoke generation, and acid gas generation properties indicate superiority of the developed low-halogen FRLS compounds over the normal PVC compound. These compounds meet all the specification of the cable industry.

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