

# Flame-Retardant PVC Compound for Cable Applications

P. V. REDDY, S. SRIDHAR, and M. C. RATRA\*

Central Power Research Institute, Bangalore-560 094, India

## SYNOPSIS

The low-melting ternary sulfate and modified sulfate glasses based on transition metals like copper, nickel, and vanadium reduce the smoke generation during burning of the PVC compound. With increase in concentration of these glasses, the smoke-density rating values decrease without appreciably altering the oxygen index values. Although trihydrated alumina and potash alum improve flame-retarding properties, the latter cannot be used as it considerably reduces the thermal stability of the PVC compound. Formulations to meet the flame-retardant requirements of a PVC compound suitable for cable sheathing have been developed.

## INTRODUCTION

Most of the polymer-based materials used for engineering applications are combustible and they release toxic and noxious gases and fumes during combustion. The problem of controlling the fire hazard of polymeric materials has become a subject of worldwide concern in recent years due to increased usage of these materials. The risk involved and the damage caused by fire wherein polymers are present is significantly high considering its use in the construction of high-rise buildings, thermal power plants, oil refineries, and transportation vehicles. All these involve risks of a high degree of occupancy and difficulty of speedy evacuation from the fire zone under critical conditions.

In many research and industrial laboratories, attempts are being made to render plastics fireproof by the addition of suitable compounds or by changing the structure of polymer itself. Poly(vinyl chloride) (PVC) is being used abundantly for insulation and sheathing of electric cables. Unplasticized PVC is inherently flame retardant because of its high halogen content. The addition of plasticizers, however, make the material flammable. The flammability of plasticized PVC can be controlled by the use of suitable additives. The additives used to impart flame retardance with low smoke (FRLS)

characteristics and to minimize toxic gas formation during burning of a PVC compound involve various elements, alloys, inorganic compounds, coordination, as well as several organometallic compounds.<sup>1</sup> Among these, transition-metal compounds have been reported<sup>2</sup> to be the most effective smoke retarders.

According to Lattimer and Kroenke,<sup>3,4</sup> the role of active smoke retarders is to change the thermal degradation pattern of PVC and to promote the formation of char. Lattimer et al.<sup>5</sup> also proposed the mechanisms by which smoke retarders function in PVC. The use of antimony trioxide ( $\text{Sb}_2\text{O}_3$ ) and molybdenum oxide ( $\text{MoO}_3$ ) as flame and smoke retarders for plasticized PVC has been reported.<sup>6-8</sup> Flaherty et al.<sup>9</sup> described the flame-retardant effect of  $\text{Sb}_2\text{O}_3$  in phosphate plasticized PVC. The use of chlorinated paraffins as coplasticizers has also been reported.<sup>10</sup>

Recently, the use of low-melting sulfate glasses and glass ceramics as fire- and smoke-retardant additives for PVC has been described.<sup>11</sup> The aim of our study was to check the effect of such low-melting sulfate glasses on the flame-retardant, mechanical, thermal, and electrical properties of the plasticized PVC compound that are required for power cables.

## EXPERIMENTAL

### Raw Materials Used

The following raw materials were used for this investigation: PVC resin, diisodecylphthalate (DIDP),

\* To whom correspondence should be addressed.

**Table I Effect of Phosphate/Brominated Plasticizers (15 phr) on FRLS Properties of Formulation R**

Property	PVC Ref. Compound	Reofos-50	Reomol-OTM	Reomol-LTM	DE-60F
OI (%)	26.3	31.0	28.0	28.0	33.4
SDR (%)	88-90	88-90	85-88	80-85	88-91

and trioctyl trimellitate (TOTM) as plasticizers; isopropylated phenyl phosphates/mellitates Reofos-50, Reomol-OTM, and Reomol-LTM and an organobromine compound DE-60F as flame-retardant plasticizers; tribasic lead sulfate (TBLS) as stabilizer; precipitated calcium carbonate as filler; and trihydrated alumina (THA) as filler/flame-retardant additive; and cupric oxalate, chromium sulfate, zinc borate, ammonium molybdate, and thermoguard CPA as metal-containing flame-retardant additives. The specifications and the properties of these chemicals are described in the literature.<sup>12-22</sup> Except for plasticizers, all other additives were dried in an air-circulating oven at 378 K for 3 h. The potash alum was ground into fine powder of 200 mesh and dried at 378 K before use.

The low-melting ternary sulfate glass and modified sulfate glasses based on the transition metals copper, nickel, and vanadium were prepared as per standard procedures<sup>11</sup> and the fine powder of 200 mesh was used.

### Formulations and Compounding of PVC

Different PVC formulations were made by carefully examining the function of each of the individual additive and its effect on physical, mechanical, thermal, and flame-retardant properties. Brabender plasticorder PLE 331 was used for compounding the formulations at 413 K.

### Evaluation of FR-PVC Compounds

The oxygen index, smoke-density rating, and hydrochloric acid gas generation were measured as per

ASTM D2863-77, ASTM D2843, and IEC-754, part I, respectively. An average value of four samples was taken for the smoke-density rating. The physical, mechanical, and electrical properties were evaluated as per IS:10810, part 7-1984, specifications. The thermal stability of PVC compounds was measured as per IS:5831-84 specifications using a constant temperature bath maintained at  $473 \pm 2$  K.

## RESULTS AND DISCUSSION

To check the effect of various ingredients systematically, a reference PVC compound was prepared using the following formulation: PVC resin, 100 parts; TBLS, 5-7 parts; DIDP, 30-50 parts;  $\text{CaCO}_3$ , 25-35 parts; calcium stearate, 0.5-1.0 part; and Bisphenol A, 0.5-1.0 part; the formulation is represented as *R*. This PVC compound exhibited an oxygen index (OI) value of 26.3 and smoke-density rating (SDR) of 88-90%.

### Effect of Flame-Retardant Plasticizers

The results obtained by the addition of 15 phr of various flame-retardant plasticizers with DIDP (15 phr) in formulation *R* are shown in Table I. The addition of brominated plasticizer increases the OI value by 27%. A marginal increase in the OI value was observed for other phosphate plasticizers.

Although there was an improvement in OI values with different flame-retardant plasticizers, there was no change in the smoke-density rating values. The thermogravimetric analysis of the PVC compound containing DE-60F indicated an initial decomposi-

**Table II FRLS Properties with Different Metal-Containing Additives**

Property	Thermoguard CPA	Zinc Borate	Cupric Oxalate	Ammonium Molybdate	Chromium Sulfate	Potash Alum <sup>a</sup>
OI (%)	33	31	31	33	32	26
SDR (%)	89	88	91	86	86	64
IDT <sup>b</sup>	508	509	509	514	512	468

<sup>a</sup> At 10 phr level, the thermal stability (473 K) value was 45 min compared to 100 min in its absence.

<sup>b</sup> Initial decomposition temperature by the TGA method at 100 mL/min oxygen flow and 6°C/min heating rate.

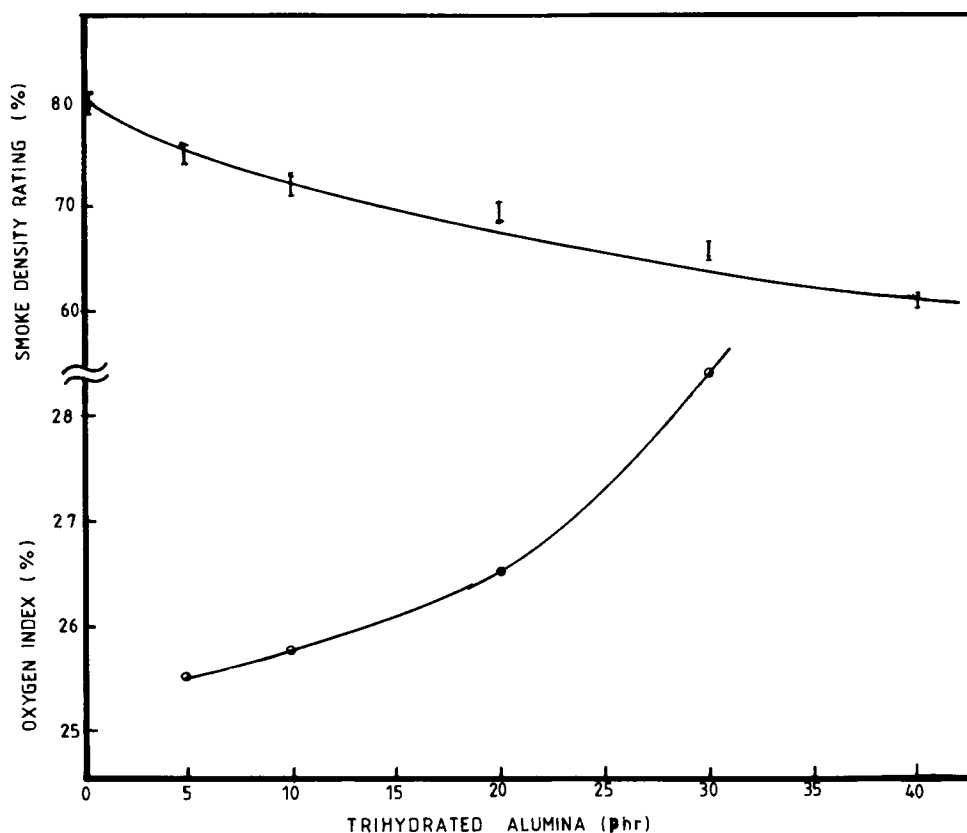


Figure 1 FRLS properties of PVC compound with variation of trihydrated alumina.

tion temperature of 519 K, and this compares favorably with the values of 523 K for the available FRLS compounds elsewhere in the world under similar conditions. PVC compounds with other plasticizers indicated lower initial decomposition temperatures ranging from 503 to 508 K.<sup>23</sup>

#### Effect of Metal-Containing FR Additives

The results obtained for metal-containing flame-retardant additives at the 2 phr level into the PVC formulation are indicated in Table II.

The results indicate improvement in the OI values of the PVC formulation from 26.3 to the range of 32.0, while SDR remains unchanged at the 86–91% level. The addition of alum, while decreasing the SDR value by 30%, also appreciably reduces the thermal stability of the compound.

An increase of OI and a gradual decrease in the SDR value was observed with an increase in the concentration of trihydrated alumina (Fig. 1). An optimum level of 30 phr of THA has been used in the formulation (*R*) for further studies. The addition of THA has little or no impact on the thermal stability of the PVC compound.

Table III Flame-Retardant Properties of PVC Compound with Variation of Sulfate and Modified Sulfate Glasses (phr)

Property	Modified Ref. Compound with THA 30	SG			MSG-Cu		
		10	20	40	10	20	40
OI (%)	30.4	30.6	30.9	31.0	30.8	32.8	30.7
SDR (%)	63–68	59–65	59–62	54–57	56–61	48–52	47–50
Thermal stability at 473 K (min)	100	100	95	90	85	80	65

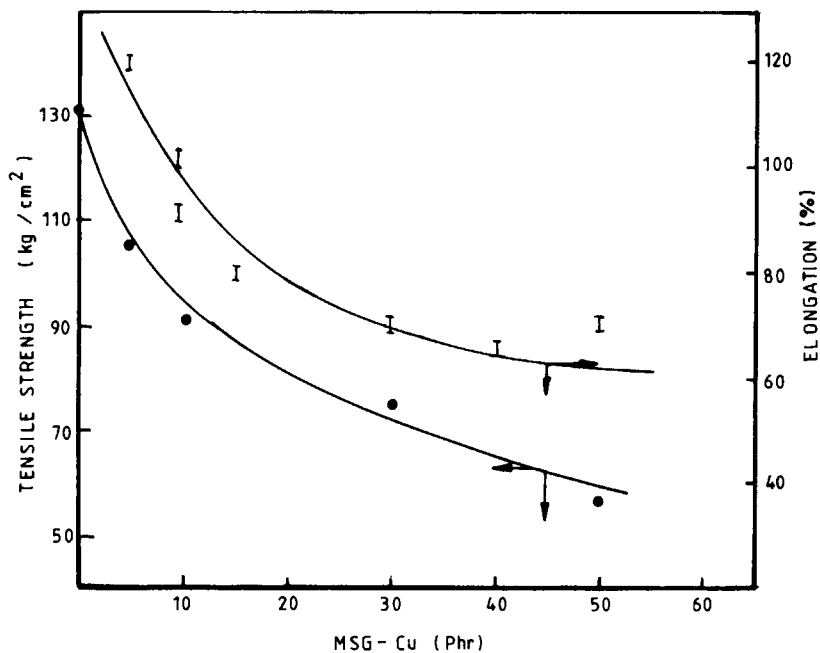


Figure 2 Mechanical properties of PVC compound with the addition of SG based on copper sulfate.

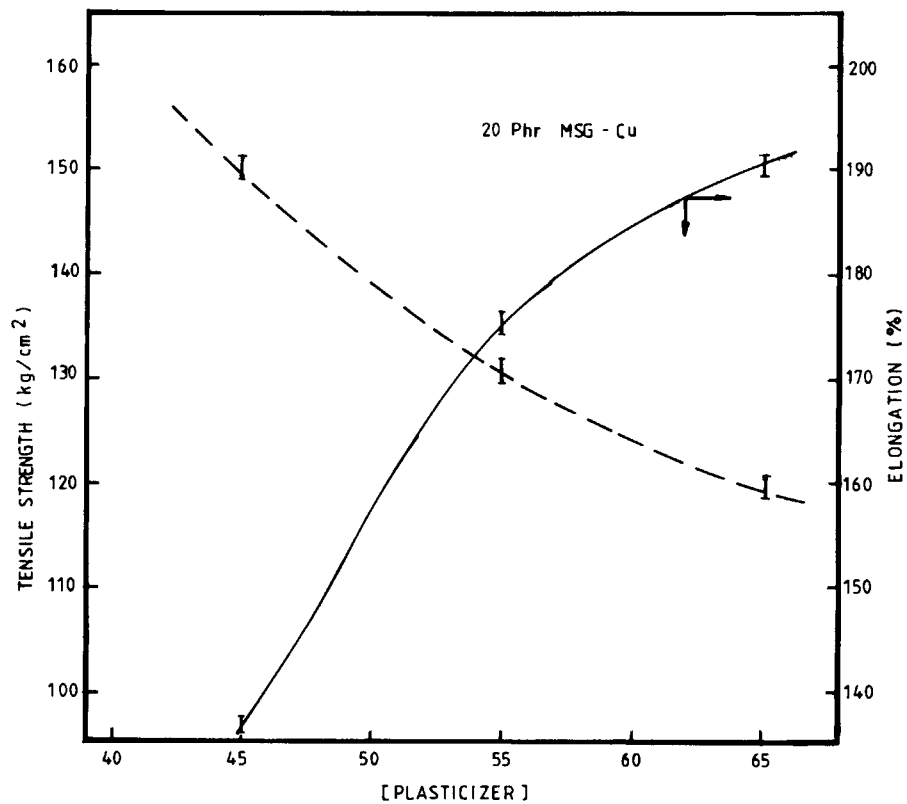


Figure 3 Variation of mechanical properties with plasticizer.

**Table IV** Electrical Properties of PVC Formulations with Variation of Plasticizer

Property	Concentration of TOTM (phr)		
	45	55	65
Tan delta (%)	6.76	6.46	5.89
Dielectric constant	3.99	4.44	4.56
Volume resistivity (ohm-cm) $\times 10^{14}$	0.93	0.49	0.53

### Effect of Low-Melting Sulfate Glasses

The effect of ternary sulfate glass (SG) and the modified sulfate glass based on copper (MSG-Cu) on flame-retardant properties in addition to 30 phr of THA are presented in Table III.

The addition of low-melting SGs, while marginally increasing the OI values, appreciably decrease the SDR values. As the concentration increases, the thermal stability gradually decreases. The tensile strength and % elongation values decrease (Fig. 2) with increase in the content of MSG-Cu. To improve the thermal stability and mechanical properties of the PVC compound, the plasticizer DIDP was re-

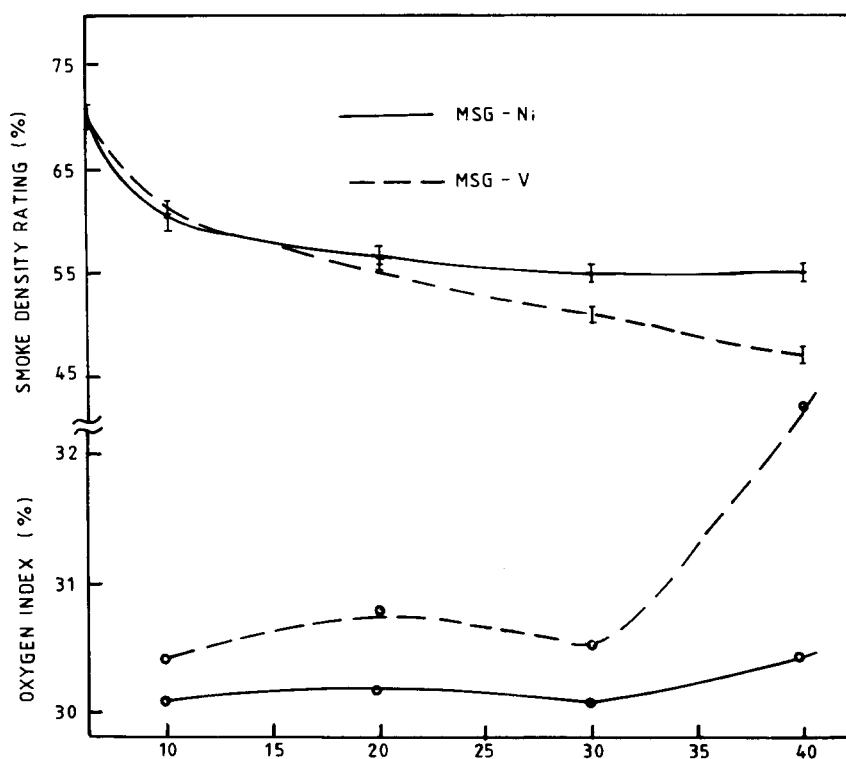
placed by TOTM. The thermal stability of the PVC compound increased up to 130 min at 60 phr of TOTM. The enhanced mechanical properties with an increase in concentration of plasticizer are depicted in Figure 3. The observed electrical properties are tabulated in Table IV.

The results suggest that the loss factor values decrease and dielectric constant values increase with increase in the plasticizer content of PVC formulation.

The studies using the transition-metal-modified SGs nickel (MSG-Ni) and vanadium (MSG-V) into the PVC formulations were similarly carried out. The properties of these formulations (Fig. 4), while being comparable to the MSG-Cu system, were found to be slightly lower than those of the glass based on the copper-added PVC compound.

### CONCLUSIONS

The addition of flame-retardant plasticizers and metal-containing additives into the PVC formulation increased only the oxygen index values. Trihydrated alumina and potash alum appear to be the better smoke retarders of the PVC formulation.

**Figure 4** OI and SDR properties of PVC compound with MSGs.

However, the latter drastically reduces the thermal stability. Low-melting sulfate glass and modified sulfate glasses (Cu, Ni, V) were found to decrease the smoke density rating to greater extent with little loss in thermal stability.

## REFERENCES

1. P. Bajaj, Fire and smoke retarder additives for PVC cables, Workshop on Power Cables and Accessories, Bangalore, December, 1988.
2. W. J. Kroenke, *J. Appl. Polym. Sci.*, **26**, 1167 (1981).
3. R. P. Lattimer and W. J. Kroenke, *J. Appl. Polym. Sci.*, **26**, 1191 (1981).
4. R. P. Lattimer and W. J. Kroenke, in *Analytical Pyrolysis, Techniques and Applications*, K. J. Voorhees, Ed., Butterworths, London, 1984.
5. R. P. Lattimer, W. J. Kroenke, and R. G. Getts, *J. Appl. Polym. Sci.*, **29**, 3783 (1984).
6. R. M. Lum, *J. Appl. Polym. Sci.*, **23**, 1247 (1979).
7. W. H. Starnes and D. Edelson, *Macromolecules*, **12**, 797 (1979).
8. D. Edelson, V. J. Kuck, R. M. Lum, E. Scalco, W. H. Starnes, and S. Kaufman, *Combust. Flame.*, **38**, 271 (1980).
9. R. T. Flaherty, J. P. Hamilton, J. Litwin, and C. A. Lynch, in *SPE Technical Papers (29 ANTEC)*, Vol. XVII, 1971, p. 412.
10. C. C. Colelli and P. V. Loenen, *Plast. Technol.*, **June**, 67 (1979).
11. W. J. Kroenke, *J. Mater. Sci.*, **21**, 1123 (1986).
12. N. L. Perry, in *Encyclopedia of PVC*, L. I. Nass, Ed., 1977, Vol. 2, p. 851.
13. P. Ghera, *Mod. Plast.*, **36**(2), 135 (1958).
14. Technical Service Bulletin, CIBA-GEIGY (UK), No. PL 1.1, 1979.
15. *Flame Retardant Selection Guide*, Great Lakes Chemical Corp., West Lafayette, IN, Catalogue No. 01282.
16. F. Chevassus and R. deBroutelles, *The Stabilization of Polyvinylchloride*, St. Martin's Press, New York, 1963, p. 277.
17. I. Phillips, and P. G. Youde, *Br. Plast.*, **30**(7), 297 (1957).
18. *Chem. Abstr.*, **82**, 112769g, 172767q (1975).
19. *Fire and Flame Retardant Polymers, Recent Developments Chemical*, Technology Review No. 122, Noyes Data Corporation, Park Ridge, NJ, 1979.
20. *Chem. Abstr.*, **102**, 25401h (1985); **82**, 172027y (1975).
21. *Chem. Abstr.*, **87**, 85704b (1977).
22. *Technical Information Data Sheet*, M&T Chemicals, No. 1-4674, Rahway, NJ (1981).
23. Unpublished results, Polymer Laboratory, Central Power Research Institute, Bangalore, India.

Received July 9, 1991

Accepted November 11, 1991