JOURNAL OF PROPULSION AND POWER
Vol. 17, No. 5, September–October 2001

Flexibilized Novolac Epoxy Resin for Inhibition of Composite Propellants

J. P. Agrawal* and N. T. Agawane†
High Energy Materials Research Laboratory, Armament Post, Sutarwadi, Pune 411 021, India

The effect of variation of polyamide hardener (EH411) on a novolac epoxy resin (Dobeckot E4) has been studied in terms of tensile strength and percent elongation. Based on a combination of tensile strength and percent elongation, formulation 9 (Dobeckot E4 : 100 + hardener EH411 : 50) has been selected for studying the effect of liquid polysulfide rubbers (LP-32 and LP-33). The data indicate that gel time, tensile strength, heat resistance, and flame retardance decrease, whereas elongation and dioctyl adipate absorption increase as the proportion of liquid polysulfide rubbers increases. The effect of antimony trioxide and alumina trihydrate fillers on formulation 20 (Dobeckot E4 : 100 + hardener EH411 : 50 + LP-33 : 20) and formulation 21 (Dobeckot E4 : 100 + hardener EH411 : 50 + LP-33 : 30) has also been studied. Based on tensile strength, elongation, dioctyl adipate absorption, heat resistance, and flame retardance data, formulation 27 (Dobeckot E4 : 100 + hardener EH411 : 50 + LP-33 : 30 + Sb$_2$O$_3$: 40) has been selected for inhibition followed by static evaluation of hydroxy-terminated-polybutadine-(HTPB-) based tubular composite propellants. The pressure–time profiles from − 40 to 50°C indicate that the formulation 27 (Dobeckot E4 : 100 + hardener EH411 : 50 + LP-33 : 30 + Sb$_2$O$_3$: 40) is a viable inhibition system for inhibition of HTPB-based composite propellants.

Nomenclature

E = elongation, %
E_{PT} = exotherm peak temperature, °C
G_t = gel time, minute
P–t = pressure–time
r = burning rate, mm/s

Introduction

A NEW generation of epoxy resins, that is, novolac epoxy resins, are now preferred, mainly on account of retention of their properties at elevated temperatures.[1,2] Based on an extensive study in this laboratory, the use of novolac epoxy resin has recently been reported for inhibition of hydroxyl-terminated-polybutadine-(HTPB-) based composite propellants.[3] The inhibition of rocket propellants demands moderate tensile strength (TS) and higher elongation E. Novolac epoxy resins are reported to possess elongation of medium order and as a result, fail to meet mission requirements over a wide range of temperatures, that is, from −40 to 50°C. It is reported in the literature that the elongation of these resins is improved by the incorporation of either long chain molecules (dibutyl phthalate, tricresyl phosphate, etc.) that remain unreacted after curing in the resin (referred as plasticizers)[4] or long chain molecules (epoxy diluents, liquid rubbers, etc.) that react during curing and become an integral part of three-dimensional networks (referred to as flexibilizers).[3,5] The use of reactive epoxy diluents/liquid rubbers is preferred because they become an integral part of three-dimensional networks and, therefore, do not migrate during subsequent aging/storage. The most important members of flexibilizers are liquid polysulfides, which impart flexibility of high order.[6] The object of the present study is to modify properties of a commercially available novolac epoxy resin (Dobeckot E4) with the incorporation of liquid polysulfides (Thiokol LP-32 and Thiokol LP-33) and fillers. The resulting formulations have been characterized for a number of properties, followed by static evaluation of inhibited HTPB-based composite propellants.

Materials and Their Specifications

Materials

1) Novolac epoxy resin, Dobeckot E4, is an epoxy resin based on cashew nut shell liquid that is an amber-colored clear liquid with density (at 25°C) 1.064 ± 0.002 g/ml, viscosity (at 25°C) 17,120 ± 1000 cPs, and epoxide equivalent 330 ± 35.

2) Liquid polysulfide LP-32 is a clear and viscous liquid with viscosity (at 25°C) 35,000–45,000 cPs and number average molecular weight $\bar{M}n = 4000 \pm 500$.

3) Liquid polysulfide LP-33 is a mobile liquid with viscosity (at 25°C) 1400–1650 cPs and $\bar{M}n = 1000 \pm 150$. LP-32 as well as LP-33 are thiol terminated, saturated, linear liquid polymers and consist of 0.5% cross-linking agent.

4) Polyamide hardener, EH 411, is a room-temperature curing, hardener and is a yellowish brown, clear and viscous liquid with density (at 25°C) 0.98 ± 0.01 g/ml and viscosity (at 25°C) 17,750 ± 100 cPs.

5) Alumina trihydrate ($Al_2O_3 \cdot 3H_2O$) is a white free-flowing powder with purity 99.5% minimum and sieve size all passing through 400 BSS.

6) Antimony trioxide (Sb_2O_3) is a white amorphous powder with purity 99.5% minimum and sieve size passing through 200 BSS and retained on 240 BSS or British sieve size. These fillers (alumina trihydrate and antimony trioxide) were dried in oven at 100 ± 2°C for 2 h before use.

7) Composite propellants included HTPB-based composite propellants with 85% solids loading, made by a casting technique and used for inhibition and static evaluation.

Methods

A number of formulations based on the Dobeckot E4, hardener EH411, liquid polysulfides, and fillers (in different proportions) were made and characterized for gel time and exotherm peak temperature,[7] TS and percent elongation,[8] dioctyl adipate (DOA) absorption,[9] heat resistance,[10] oxygen index,[11] flame retardance,[12] and bond strength with composite propellants.[9] A brief description of the methods used for characterization is as follows.

Gel Time G_t and Exotherm Peak Temperature E_{PT}

The term gel time, as used here, is defined as the time corresponding to a sudden buildup of viscosity (known as gelling) of a

---

Presented as Paper 99-2652 at the AIAA 35th Joint Propulsion Conference and Exhibit, Los Angeles, CA, 20–24 June 1999; received 18 March 2000; revision received 20 March 2001; accepted for publication 27 April 2001.

*Associate Director.

†Technical Officer.

standard weight of resin, at a given temperature, that is, 40 ± 1°C, after adding a curing agent.

First, 100 g of the formulation was placed in a plastic beaker (dimensions: 71 mm diameter and 95 mm height), and the thermometer was centered into the resin mass. The plastic beaker was then immersed in a water bath so that the composition level was below that of the water surface. The bath temperature was maintained at 40 ± 1°C. When the formulation attained the temperature 40 ± 1°C, curing agent was added and mixed thoroughly. The temperature of the resin was recorded every minute. The time corresponding to the sudden buildup of viscosity was recorded with a stop watch and noted as gel time. The temperature of the resin was continuously recorded until the temperature was at a maximum (known as exotherm peak temperature) and then declined by a few degrees. A mean of three readings was taken and reported as G_t and E_{PT}. The mean variation for G_t was ±1 min. and for E_{PT} was ±0.2°C.

Bond Strength with Composite Propellants

The bonding properties were determined by measuring the bond strength between composite propellant and different formulations. The propellant samples (dimensions 50 × 20 × 10 mm) were prepared by rubbing their ends with emery paper where bonding with resin formulation was required and covering the remaining portion with aluminium foil. They were kept in a mold with a prepared surface, facing each other at a distance of 10 mm apart. The formulation with curing agent was poured between the gap of the propellant samples. After curing of the resin, the bonded pieces were removed and breaking load was determined on an Instron Machine (Model 1185). The bond strength was calculated as

bond strength, in kilograms per square centimeter
= breaking load/cross-sectional area.

Preparation of Specimens for Various Properties

The novolac epoxy resin is originally in a liquid state. All ingredients of a particular formulation with LP-32/LP-33 and fillers were mixed by mechanical stirring, and different test specimens were made by casting in the corresponding molds. Curing was done at 27 ± 2°C, followed by postcuring for a week at the same temperature before characterization.

Tensile Strength and Percent Elongation

Tensile strength and percent elongation were measured by using an Instron machine (Model 1185). Dumbell-shaped specimens were prepared in a mold. The specimens were then clamped in the jaws of the machine and tension was applied at a constant rate of 5 cm/min. The elongation and maximum load at break were recorded on chart paper. The cross-sectional area of the central part of the dumbell was measured with a micrometer screw gauge, and tensile strength and elongation were calculated from

tensile strength in kilograms per square centimeter
= load at break/cross-sectional area

percent elongation
= extension in length/original gauge length × 100

The mean percent variation for elongation and tensile strength was 1 and 2, respectively.

DOA Absorption

DOA absorption was determined by the liquid absorption method. The specimen (dimensions 50 × 50 × 25 mm) was suspended fully immersed in DOA at 27 ± 2°C. The samples were removed at suitable intervals, dried with filter paper, and the gain in weight was recorded; percent DOA absorption was calculated as

percent DOA absorption
= increase in weight/original weight × 100

The mean percent variation was 0.05.

Heat Resistance

Heat resistance was determined with the specimens of dimensions 28.57 mm diameter and 25.4 mm height. The specimens were kept in an electric oven at 150 ± 2°C. They were removed after heating for 1 h, cooled in a desiccator, and loss in weight was recorded. This was continued for 6 h and loss in weight was calculated. The mean percent variation was found to be 0.05.

Flame Retardance

The specimens of dimensions 127.0 mm length, 12.7 mm width and 3.2 mm thickness were cast and allowed to cure at 27 ± 2°C for seven days. American Society for Testing and Materials (ASTM) D 635, with some minor modifications, was used to measure comparative flame retardance. The mean percent variation was found to be 0.1.

Oxygen Index

The relative flammability or flame retardance of polymeric materials is generally expressed in terms of the oxygen index (OI), which is defined as minimum concentration of oxygen, in a slowly rising mixture of oxygen and nitrogen, that just supports candlelike combustion. The OI was determined using an ONI Flammability Tester, Model CS-178B (Custom Scientific Instruments, Inc.). The details of the method are given in ASTM D 2863. The oxygen index was calculated by

$$\mathrm{OI} = 100 \times \mathrm{O_2}/\mathrm{O_2} + \mathrm{N_2}$$

where O_2 = oxygen and N_2 = nitrogen. Oxygen and nitrogen are the volumetric flows in cubic centimeters per second. The mean percent variation was found to be 0.5.

Inhibition, X-Ray Screening, and Static Evaluation of Composite Propellants

The composite propellants (in tubular mode) were used for static evaluation after inhibition with selected formulation by the casting technique. Industrial x-ray equipment, Model MG 150/300 (M/s. Philips) was used for detection of internal defects of propellant and inhibitor. The inhibited and x-ray cleared propellants were statically evaluated after conditioning at different temperatures and P–t profiles were recorded.

Results and Discussion

Because TS and percent E of polymeric materials are important properties used to judge their suitability for inhibition of rocket propellants, the effect of hardener EH 411 on them is discussed first.

Mechanical Properties

Table 1 lists the effect of hardener EH411 on TS and percent E of Dobeckot E4 and lists 13 formulations. Note that hardener EH 411 gives a high order of TS to the cured products. A combination of maximum TS and minimum percent E is possible when the hardener crosslinks the maximum number of epoxy molecular chains present in the resin. The ratio of epoxy resin to hardener is generally selected on the basis of maximum TS of the cured resin. Accordingly, formulation 9 was selected for further study. Also note that mechanical properties depend on the quantity of hardener. If there is less hardener, percent E is found to be greater because uncured epoxy molecules are present in the cured resin. Also, if the hardener is too high, it results in high E and low TS because the excess of hardener acts as flexibilizer.[13]

The inhibited composite propellants used in rockets and missiles for defense applications are required to function satisfactorily from −40 to 50°C in Indian context, and, therefore, the inhibitor for these rocket propellants should possess a high order of elongation. The liquid polysulfide polymers have been used to modify properties of formulation 9.

Characterization of Dobeckot E4: Liquid Polysulfides Blends

Liquid polysulfides, LP-32 and LP-33 (in different proportions) have been added to formulation 9. The effect on various properties is discussed hereafter.

Gel Time G_t and Exotherm Peak Temperature E_{PT}

The composition of various formulations along with the data on G_t and E_{PT} is given in Table 2. The data show that G_t for formulation 9, which does not contain any liquid polysulfide flexibilizer, is 38 min. G_t decreases from formulation 14 to formulation 18 as well as from formulation 19 to formulation 23 because these formulations contain LP-32 and LP-33 in increasing order. This is because liquid polysulfides accelerate the rate of curing of epoxy resin in the presence of a hardener.[14] The liquid polysulfides have terminal-SH groups, which participate during curing and enhance the rate of reaction. Furthermore, a comparison of G_t data of LP-32 based formulations (from formulation 14 to formulation 18) and LP-33 based formulations (from formulation 19 to formulation 23) (Table 2) indicate that the formulations based on LP-32 possess higher G_t. This is attributed to the higher molecular weight of LP-32 as compared to LP-33, which causes LP-32 to react sluggishly with epoxy resin, resulting in higher G_t. It is reported in the literature[15] that G_t and E_{PT} are inversly related, and, if G_t decreases, E_{PT} increases and vice versa. As G_t decreases from formulation 14 to formulation 18 and from formulation 19 to formulation 23, E_{PT} is expected to increase. This is observed experimentally.

TS and Percent Elongation

TS and percent E data for these formulations are given in Table 2, and it is evident that percent elongation increases as the concentration of liquid polysulfides, LP-32 and LP-33, increases in all formulations whereas TS decreases. This is because the thiol-terminated liquid polysulphides (LP-32 and LP-33) coreact with Dobeckot E4 in the presence of a polyamide hardener to give materials possessing a number of improved properties over either polymer when converted alone. The elastomeric polysulphide segments in the epoxy chain impart flexibility, and, as the concentration of LP-32/LP-33 increases, percent elongation also increases with corresponding decrease in TS.[16] This is in line with the general observations reported in case of epoxy resins and other thermosets.[17]

Table 1 Effect of polyamide hardener EH411 on mechanical properties of novolac epoxy resin, Dobecot E4

	Composition (parts by weight)			
Formulation	E4	EH411	Elongation percent	TS, kg/cm^2
1	100	10	——	——
2	100	15	58.0	12
3	100	20	43.0	53
4	100	25	21.0	102
5	100	30	13.0	153
6	100	35	12.3	167
7	100	40	10.0	198
8	100	45	8.3	215
9	100	50	5.9	266
10	100	55	6.0	240
11	100	60	10.8	208
12	100	65	23.0	158
13	100	70	35.0	102

Furthermore, a comparison of TS of LP-32 based formulations (from formulation 14 to formulation 18) and LP-33 based formulations (from formulation 19 to formulation 23) (Table 2) indicates that the formulations consisting of LP-32 possess higher TS. As recorded earlier, LP-32 has a higher molecular weight as compared to LP-33, and, therefore, LP-32 imparts higher tensile strength to novolac epoxy resin based formulations. This is in agreement with the data reported in the literature.[18]

Plasticizer (DOA) Absorption, Heat Resistance, and Flame Retardance

The data on DOA absorption, heat resistance (as measured in terms of loss in weight at 150 ± 2°C), and flame retardance (as measured in terms of burning-rate r) for these formulations is given in Table 3.

The tensile strength of the formulation 9 is 266 kg/cm^2, which decreases on addition of LP-32 in various proportions resulting in a change from formulation 14 to formulation 18. Similarly, addition of LP-33 to formulation 9 in various proportions results in a change from formulation 19 to formulation 23, where TS also decreases. Because TS is indicative of crosslink density, it means crosslink density decreases from formulation 14 to formulation 18 and also from formulation 19 to formulation 23.

It is seen that the DOA absorption increases from formulation 14 to formulation 18 and from formulation 19 to formulation 23. The higher DOA absorption is due to the decrease in the crosslink density of formulations. The polysulphides act to decrease the crosslink density of the resin formulations, thereby increasing susceptibility to plasticizer (DOA) migration. This is similar to the observations already reported by several researchers.[19,20]

The loss in weight also increases on incorporation of polysulfides. As explained out earlier, the crosslink density (rigidity or compactness of molecular structure) also decreases from formulation 14 to formulation 18 and from formulation 19 to formulation 23. This decrease in rigidity or compactness facilitates loss of volatile matters, and, accordingly, loss in weight increases.[7] In other

Table 3 Effect of liquid LP-32 and LP-33 on DOA absorption, heat resistance, and flame retardance of formulation 9 (E4: 100 + EH411 : 50)

Formulation	LP-32 (parts by weight)	LP-33 (parts by weight)	DOA absorption (after 6 days), %	Loss in weight (after 6 h), %	Burning rate, mm/s
9	——	——	0.597	0.158	0.39
14	10	——	0.661	0.210	0.40
15	20	——	0.795	0.228	0.42
16	30	——	0.819	0.252	0.48
17	40	——	0.880	0.292	0.53
18	50	——	1.172	0.383	0.59
19	——	10	1.081	0.302	0.48
20	——	20	1.289	0.321	0.50
21	——	30	1.614	0.535	0.54
22	——	40	1.677	0.678	0.57
23	——	50	1.725	0.732	0.62

Table 2 Effect of liquid LP-32 and LP-33 on gel time, peak exotherm temperature, elongation and TS of formulation 9 (E4: 100 + EH411 : 50)

Formulation	LP-32 (parts by weight)	LP-33 (parts by weight)	Gel time G_t, min.	Peak exotherm temp. E_{pt},°C	Elongation E, %	TS, kg/cm^2
9	——	——	38	43.4	5.9	266
14	10	——	32	43.6	28.6	142
15	20	——	29	43.8	31.1	140
16	30	——	27	44.2	34.8	132
17	40	——	25	46.6	37.6	109
18	50	——	23	45.6	42.1	100
19	——	10	30	45.4	59.3	87
20	——	20	25	46.0	68.6	70
21	——	30	18	48.0	84.8	43
22	——	40	16	48.4	90.2	30
23	——	50	15	48.6	93.0	21

Table 4 Effect of fillers on elongation and TS of formulation 20 (E4 : 100 + EH411 : 50 + LP-33 : 20) and formulation 21 (E4 : 100 + EH411 : 50 + LP-33 : 30)

Fillers							
Sb_2O_3 (parts by weight)	$Al_2O_3 \cdot 3H_2O$ (parts by weight)	Formulation	Elongation, %	TS, kg/cm^2	Formulation	Elongation, %	TS, kg/cm^2
0	——	20	68.8	70	21	84.8	43
10	——	24	51.9	86	32	62.1	62
20	——	25	44.5	95	33	54.6	67
30	——	26	42.1	98	34	53.5	67
40	——	27	39.5	106	35	52.1	68
——	10	28	48.4	89	36	63.0	52
——	20	29	46.6	90	37	61.2	58
——	30	30	42.6	98	38	59.2	62
——	40	31	43.5	100	39	58.5	64

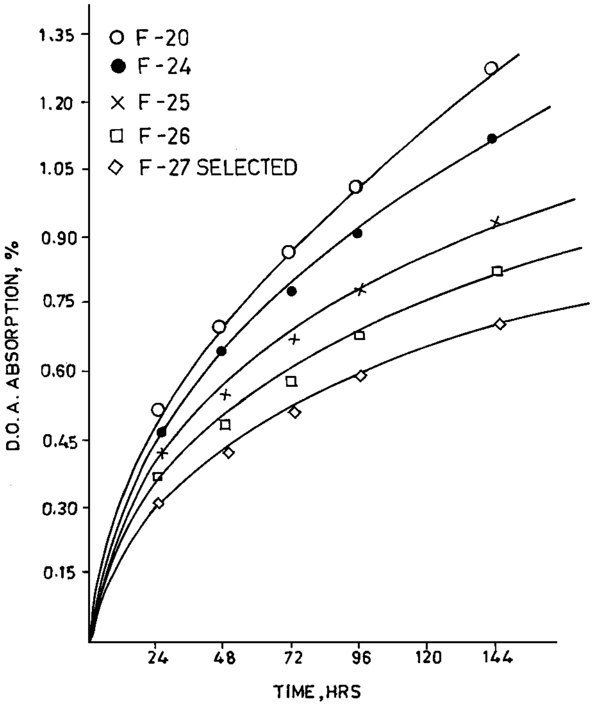

Fig. 1 Effect of antimony trioxide on DOA absorption of formulation 20.

words, heat resistance decreases in this order. Furthermore, it may be because of the thermal stability of LP-32/LP-33 present in these formulations because the thermal stability of polysulphides in air is limited by oxidation of the sulphide group.[21]

The flame retardance data may be explained if we assume that the polymer degradation governs the regression rate. The TS/crosslink density is maximum in the case of formulation 9, and it decreases progressively from formulation 14 to formulation 18 and from formulation 19 to formulation 23 on addition of LP-32/LP-33. Therefore, formulation 9 requires greater energy for breaking the polymer chains, as compared to other formulations in the series, to give degradation products responsible for the flame reactions. Thus, the burning rate increases as the crosslink density/TS decreases from formulation 14 to formulation 18 and also from formulation 29 to formulation 23 (Ref. 22). In other words, flame retardance decreases in this order.

Effect of Fillers on Some Selected Formulations

Based on various properties, formulation 20 and formulation 21 have been selected for studying the effect of fillers. Liquid polysulfide LP-33 has low viscosity and is easily miscible with Dobeckot E4. On the other hand, LP-32 has high viscosity and requires more time for mixing with Dobeckot E4. Also due to higher viscosity of LP-32 based formulations, inhibition of rocket propellants by the casting technique is difficult. Therefore, LP-33 based formulations have been selected for studying the effect of fillers.

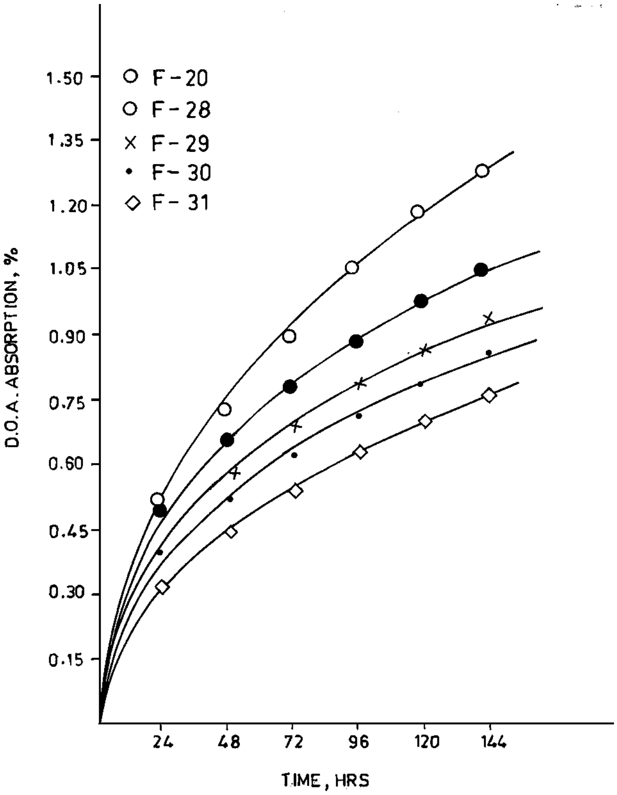

Fig. 2 Effect of alumina trihydrate on DOA absorption of formulation 20.

TS and Percent Elongation

Table 4 lists the data on TS and percent *E* for formulation 20 and formulation 21 with different parts of antimony trioxide and alumina trihydrate fillers. TS of formulation 20 is 70 kg/cm^2, which increases to 106 kg/cm^2 on addition of 40 parts of antimony trioxide, whereas it increases to 100 kg/cm^2 with 40 parts of alumina trihydrate because of its reinforcing action on the resin matrix.[23] Furthermore, there is a corresponding decrease in elongation as the filler concentration increases. This is agreement with the data reported in the literature.[24] Similar trends are also observed in case of formulation 21 with various parts of fillers.

DOA Absorption

The graphical presentation of DOA absorption variation with time for these formulations is given in Figs. 1–4. Note that the DOA absorption decreases as the quantity of antimony trioxide or alumina trihydrate increases. This is because the quantity of epoxy/polysulfide resins, which are mainly responsible of DOA absorption, decreases as the quantity of filler increases.[25,26]

Polyesters and epoxies are macromolecules and form three-dimensional networks on curing. DOA molecules, being small in

Table 5 Effect of fillers on heat resistance and burning rate of formulation 20 (E4 : 100 + EH411 : 50 + LP-33 : 20) and formulation 21 (E4 : 100 + EH411 : 50 + LP-33 : 30)

Fillers							
Sb_2O_3 (parts by weight)	$Al_2O_3 \cdot 3H_2O$ (parts by weight)	Formulation	Loss in weight (after 6 h), %	Burning rate, mm/s	Formulation	Loss in weight (after 6 h), %	Burning rate, mm/s
0	——	20	0.321	0.49	21	0.546	0.54
10	——	24	0.266	0.46	32	0.485	0.51
20	——	25	0.246	0.39	33	0.442	0.45
30	——	26	0.236	0.36	34	0.407	0.38
40	——	27	0.225	0.28	35	0.377	0.31
——	10	28	0.295	0.47	36	0.511	0.52
——	20	29	0.287	0.41	37	0.471	0.47
——	30	30	0.272	0.36	38	0.435	0.38
——	40	31	0.246	0.31	39	0.408	0.32

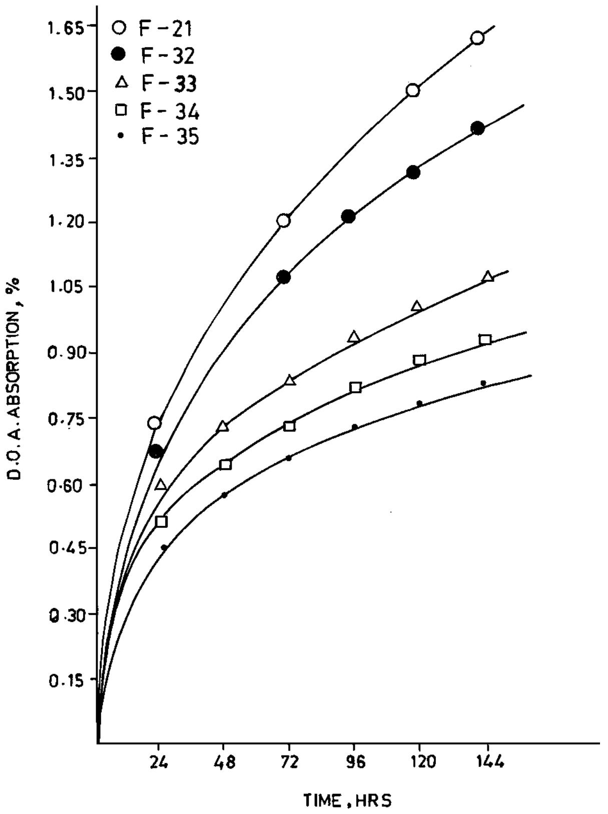

Fig. 3 Effect of antimony trioxide on DOA absorption of formulation 21.

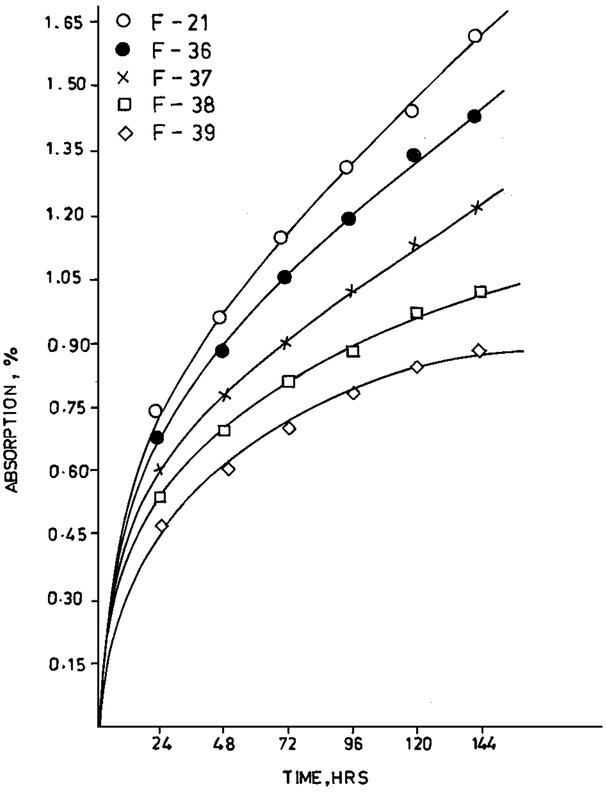

Fig. 4 Effect of alumina trihydrate on DOA absorption of formulation 21.

size, easily seep into the structure of epoxies. The migration of DOA is prevented by physically blocking the pores by adding antimony trioxide or alumina trihydrate fillers. Furthermore, the extent of DOA migration reduces on addition of fillers to polymer/resin and goes on decreasing with the decrease in the quantity of polymer/resin due to the dilution effect.[27]

Heat Resistance and Flame Retardance

Table 5 lists variation of loss in weight with time and burning-rate data of formulation 20 and formulation 21 containing varying quantity of fillers. Note that the loss in weight decreases or heat resistance increases as the quantity of filler increases. Similarly, r decreases, or flame retardance increases, as the quantity of filler increases. These phenomena may be explained along the lines of DOA absorption.

Selection of Formulation for Inhibition of Composite Propellants and Static Evaluation

Based on TS, percent elongation, DOA absorption, and flame retardance, formulation 27, formulation 31, formulation 35, and formulation 39 are considered promising for inhibition. These formulations have been further characterized for oxygen index (n) and bond strength with composite propellants. The OI data are in agreement with the flame retardance data and suggest that the formulation 27 is most flame retardant. A comparative account of their properties is listed in Table 6. Furthermore, the effect of temperature conditioning [i.e., ambient, hot (50°C), and cold (−40°C)] on TS and percent E of selected formulations is given in Table 7. Note that the formulation 27 possesses a good combination of various properties and was selected for the inhibition of composite propellants.

The radiographed composite propellants (tubular) inhibited with the formulation 27 were statically evaluated after conditioning at ambient, hot (50°C for 20 h), and cold (−40°C for 16 h). The pressure–time (P–t) profiles, as shown in Fig. 5, are smooth and show that the bonding between the inhibitor and propellant is intact, and no additional surface is exposed to the flame at the time of combustion. In other words, the formulation 27 is a potential inhibition system for inhibition of composite rocket propellants over a wide range of temperatures, that is, from −40 to 50°C.

The additional data on modulus, thermal expansion, and temperature rate sensitivity of selected cured formulations is also considered important. Furthermore, the aging studies at magazine temperature (27 ± 2°C) as well as elevated temperature (50°C) of HTPB-based

Table 6 Comparison of some properties of promising formulations

	Formulations			
Property	27	31	35	39
Tensile strength, kg/cm^2	106	100	68	64
Elongation, %	39	40	52	58
Bond strength with composite propellants	Bonds remain intact and propellants break first at the time of bond strength determination			
DOA absorption, % (after 144 h)	0.412	0.762	0.813	0.874
Loss in weight, % (after 6 h)	0.225	0.236	0.377	0.408
Flame retardance, mm/s	0.28	0.31	0.33	0.34
OI, %	22.20	21.75	20.83	20.23

Table 7 Effect of conditioning temperature on TS and percent elongation of promising formulations

	Formulations			
Property	27	31	35	39
	TS, kg/cm^2			
At ambient	106	100	68	64
At 50°C after 24 h	130	105	79	72
At −40°C after 24 h	158	131	104	93
	Elongation, %			
At ambient	39	40	52	58
At 50°C after 24 h	32	35	43	48
At −40°C after 24 h	25	26	30	31

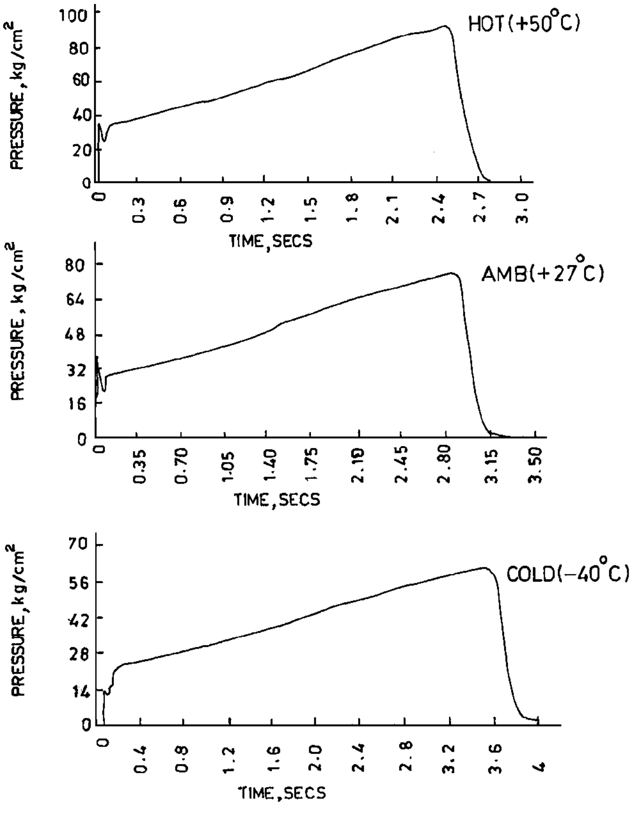

Fig. 5 Pressure-time (*P–t*) profiles of HTPB-based composite propellants inhibited with formulation 27.

composite propellants inhibited with formulation 27 will indicate their useful life. These studies are being planned and will be reported in due course.

Conclusion

This study concludes that the formulation 27 ($E4{:}100 + EH411{:}\ 50 + LP - 33 : 20 + Sb_2O_3 : 40$) is a potential new inhibition system for HTPB-based composite propellants over a wide range of temperatures, that is, −40 to 50°C.

Acknowledgments

The authors are thankful to the director of the High Energy Materials Research Laboratory for permission to publish the data of this investigation.

References

[1] Potter, W. G., *Epoxide Resins*, Butterworths, London, 1970, Chap. 9, p. 190.

[2] Lee, H., and Neville, K., *Epoxy Resins*, McGraw–Hill, London, 1970.

[3] Agrawal, J. P., Gholap, D. H., Bansod, V. P., Satpute, R. S., and Agawane, N. T., "Novolac Epoxy Resin Based Inhibition System for Composite Propellants," *Indian Journal of Engineering and Materials Sciences*, Vol. 2, No. 1, 1995, pp. 34–49.

[4] Lee, H., and Neville, K., *Handbook of Epoxy Resins*, McGraw–Hill, London, 1967, Chap. 13.

[5] Agrawal, J. P., and Satpute, R. S., "Cardanol-Based Epoxy Flexibilizers for Inhibition of Composite Propellants," *Journal of Macromolecular Science Part A*: *Chemistry*, A, Vol. 30, No. 1, 1993, pp. 19–34.

[6] Cranker, K. R., and Breslau, A. J., "Epoxy Casting Resins Modified with Polysulfied Liquid Polymer," *Industrial Engineering Chemistry*, Vol. 48, No.1, 1956, pp. 98–103.

[7] Agrawal, J. P., Chowk, M. P., and Satpute, R. S., "Study on Mixed Glycol Based Semi-Flexible Unsaturated Polyesters for Inhibition of Rocket Propellants," *British Polymer Journal*, Vol. 14, 1982, pp. 29–34.

[8] "Test Method for Tensile Properties of Plastics," *Annual Book of ASTM Standards, Plastics*, Vol. 08.01, American Society for Testing and Materials, Philadelphia, 1984, pp. 231–247; ASTM Designation, D 638.

[9] Agrawal, J. P., and Pokharkar, R. D., "Inhibition of Rocket Propellants," *Journal of Scientific and Industrial Research*, Vol. 39, No. 11, 1980, pp. 633–640.

[10] Robitschek, P., and Bean, C. T., "Flame-Resistance Polyester from Hexachloropentadiene," *Industrial Engineering Chemistry*, Vol. 46, No. 8, 1954, pp. 1628–1632.

[11] "Standard Method for Measuring the Minium Concentration to Support Candle-Like Combustion of Plastics (Oxygen Index)," *Annual Book of ASTM Standards, Plastics*, Vol. 08.02, American Society for Testing and Materials, Philadelphia, 1984, pp. 642–648; ASTM Designation D 2863.

[12] "Standard Test Method for Rate of Burning and/or Extent and Time of Burning of Self-Supporting Plastics in a Horizontal Position," *Annual Book of ASTM Standards, Plastics*, Vol. 08.01, American Society for Testing and Materials, Philadelphia, 1984, pp. 223–227; ASTM Designation D 635.

[13] "Dobeckot Epoxy Resins," Technical Data Sheets 5-67-8205, 5-91-8305, and 5-14-8410, Dr. Beck and Co., Pune, India.

[14] Lee, H., and Naville, K., *Epoxy Resins*: *Their Applications and Technology*, McGraw–Hill, New York, 1957, Chap. 7.

[15] Agrawal, J. P., "Prediction of Gel Time and Exotherm Peak Temperature of Unsaturated Polyesters and Chloropolyesters Blends" *European Polymer Journal*, Vol. 24, No. 1, 1988, pp. 93–97.

[16] Sorg, E. H., and McBurney, C. A., "Polysulphide—Epoxy Compositions," *Modern Plastics*, Oct. 1956, pp. 187–197.

[17] Boenig, H. V., *Unsaturated Polyesters*: *Structure and Properties*, Elsevier, New York, 1964, Chap. 6, pp. 55–132.

[18] Berenbaum, M. B., "Polysulphide Polymers," *Encyclopedia of Polymer Science and Technology*, Vol. 11, edited by H. F. Mark, N. G. Gaylord, and N. M. Bikales, Interscience, New York, 1969, pp. 425–446.

[19] Parker, E. E., and Moffett, E. W., "Physical Properties of Polyester Resins," *Ind. Engg. Chem.*, Vol. 46, No. 8, 1954, pp. 1615–1618.

[20] Agrawal, J. P., and Khare, Y., "Novolac Epoxy Resin-Hydroxy Terminated Adipates Based Inhibition System for Double-Base Rocket Propellants," *Journal of New Polymeric Materials*, Vol. 4, No. 3, 1994, pp. 167–176.

[21] Cassidy, P. E., *Thermally Stable Polymers*:*Synthesis and Properties*, Marcel Dekker, New York, 1980, p. 60.

[22] Agrawal, J. P., Chowk, M. P., Bansod, V. P., Kulkarni, K. S., Satpute, R. S., and Inamdar, A. R., "Dichloro Maliec Anhydride Containing Chloropolyesters as Matrices for Composites," Composites, Vol. 21, No. 4, 1990, pp. 345–349.

[23] Wypych, G., *Fillers*, Chem. Tec. Publishing, Toronto, Canada, 1993, p. 185.

[24] Nametez, R. C., "Self-Extingishing Polyester Resins," *Industrial Engineering Chemistry*, Vol. 59, No. 5, 1967, pp. 99–116.

[25] Caire-Maurisier, M., and Tranchant, J., "La Mogration de La Nitroglycerine les Inhibiteurs de poudres a Double-Base," *Propellants and Explosives*, Vol. 2, No. 5, 1977, pp. 101–104.

[26] Agrawal, J. P., Chowk, M. P., Satpute, R. S., and Kulkarni, K. S., "The Role of Alumina Trihydrate Filler on Semiflexible Unsaturated Polyester Resin-4 and Inhibition of Double-Base Rocket Propellants," *Propellants, Explosives, and Pyrotechnics*, Vol. 10, No. 3, 1985, pp. 77–81.

[27] Agrawal, J. P., Gupta, D. C., Khare, Y., and Satpute, R. S., "Zinc Borate Complex as a Flame Retardant Filler," *Journal of Applied Polymer Science*, Vol. 43, No. 2, 1991, pp. 373–377.