Journal of Macromolecular Science, Part A
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713597274

Epoxy Resin as Inhibitor for Nitramine Containing Composite Modified Double Base (CMDB) Propellants

O. C. Gupta[a]; M. V. Vaidya[b]; S. S. Raomore[b]; V. C. Phadke[b]; P. K. Divekar[b]
[a] Defence R & D Establishment, Gwalior, India [b] Explosive R & D Laboratory, Pune, India

To cite this Article Gupta, O. C. , Vaidya, M. V. , Raomore, S. S. , Phadke, V. C. and Divekar, P. K.(1995) 'Epoxy Resin as Inhibitor for Nitramine Containing Composite Modified Double Base (CMDB) Propellants', Journal of Macromolecular Science, Part A, 32: 1, 1245 — 1255
To link to this Article: DOI: 10.1080/10601329508020346
URL: http://dx.doi.org/10.1080/10601329508020346

MACROMOLECULAR REPORTS, A32(SUPPL. 8), 1245-1255 (1995)

EPOXY RESIN AS INHIBITOR FOR NITRAMINE CONTAINING COMPOSITE MODIFIED DOUBLE BASE (CMDB) PROPELLANTS

D.C.Gupta*, M.V.Vaidya, S.S.Raomore, V.C.Phadke and P.K.Divekar

Explosive R & D Laboratory, Pune - 411 008, INDIA.

Abstract

Epoxy resin was investigated as inhibitor for nitramine containing Composite modified double base (CMDB) propellants. Inhibited CMDB propellants were statically (rocket firing under static condition) evaluated at ambient, hot (+60°C) and cold (-30°C) temperatures. Incompatibility problem between epoxy resin and CMDB propellants were overcome by use of barrier layers of fast-setting isophthalic acid based polyester resin (Acrolite-471). Storage ageing trials at ambient and hot (+60°C) temperatures were also studied for propellant-inhibitor bond failure at above mentioned temperatures.

Introduction

Migration of glycerol trinitrate (nitroglycerine, NG) to the inhibitor which is conventionally a thermoplastic such as ethyl cellulose or cellulose acetate [1, 2] is a problem for composite modified double base propellants [Nitro cellulose (NC) + nitro

* Present Address : Defence R & D Establishment, Jhansi Road Gwalior - 474002 INDIA and Corresponding author.

glycerine(NG) + Ammonium Perchlorate(AP) + Aluminium(Al) + Nitramine, Research and Development Explosive (RDX) (cyclo trimethylene tri nitramine]. To solve this problem, attempts have been made in some laboratories to use rigid thermosetting polymers. For example Polyester resin [2] has been used which is compatible with the propellant as a barrier coating for application as inhibitor. Polyester resin containing poly-vinylchloride (PVC) powder and titanium dioxide as fillers has been claimed as good inhibitor[3] for Double base (DB) propellants. Polyester resin has very good bond strength with DB propellants but NG migration was still observed. Barrier coat of fast setting polyster resin followed by inhibition with polyester resin (EP-4) was developed and reported at ERDL[4]. Inhibition of nitramine containing CMDB propellants was also carried out by giving a coat of Desmodur R (Triphenyl methane triisocyanate solution in methylene dichloride) followed by barrier coats of fast setting polyester resin and final inhibition by ERDL-EP-4 polyester resin containing 30% alumina as filler[4]. However polyester resin as inhibitor has limited shelf life and gets delaminated after a period of 6 months to 1 year. This led to the search for newer type of polymeric materials for their use as inhibitors. Number of polymers have been reported as inhibitors for CMDB propellants such as epoxy resin-butadiene-acrylic copolymer [5], polyester resin[3,6], silicones[7], as inhibitors. However, this information is limited and only available in patent literature.Therefore attempts have been made to develop newer type of inhibitors. We have developed number of inhibitors for composite and CMDB propellants[8-12]. Due to excellent adhesive properties, low shrinkage on curing as compared to polyester resin and ease of handling, epoxy resin[13] was tried as inhibitor for CMDB

propellants. We are now reporting epoxy resin as inhibitor for nitramine containing CMDB propellants in the present paper.

EXPERIMENTAL

MATERIALS:

(i) Dobeckot E-4 epoxy resin*

(ii) Polyamide hardener EH411*

(iii) Diluent C*
(* Propriety products of Dr.Beek & Co., Pune, INDIA)

(iv) Desmodur R (Triphenyl methane triisocyanate- M/s Bayer A.G (Germany).

(v) Acrolite -471 an isophthalic acid based fast setting polyester resin containing styrene as cross -linking monomer . M/s Acrolite polymers, Gurgaon (INDIA).

(vi) Methyl ethyl ketone peroxide solution in dimethyl phthalate (catalyst) and cobalt naphthenate solution in methylene chloride (accelerator) M/S Baketite Hylam, Hyderabad (INDIA)

(v) Antimony trioxide (Sb_2O_3 conforming to IS 38; 1976 purity 99.0 minimum and all passing 200 BSS- M/s UNI Campaine Limited, Pune, INDIA

Characterization of epoxy resin.

Epoxy resin [Dobeckot E4 (100 parts)] was mixed with Diluent C (Mono epoxy resin plasticizer) and filler Sb_2O_3 (15 parts) and cured with polyamide hardener EH-411. This was cast into Dumbell shaped specimens in a mould. Elongation % and tensile strength were measured by using an instron machine (model No.1185) UK (Table 1 & 2). Epoxy resin composition suitable for inhibition purpose (composition No.5) was characterised for gel time, oxygen index, thermal conductivity, heat resistant and brittle temperature as reported earlier[10]

Table 1 : Formulation of Inhibitor composition

Composition	Formulations, amount in gms				
	1	2	3	4	5
Epoxy resin E-4	50	50	50	50	50
Polyamide hardner EH-411	25	25	25	25	25
Monoepoxydiluent (DilC)	--	5	5	5	10
Antimony Trioxide Sb_2O_3	--	--	10	15	15

Table 2 : Mechanical properties of Epoxy resins

Formulation No	gel time hr	Tensile strength Kg/cm^2	E %	Bond Strength*
1	2.45	252.0	5.0	---
2	3.50	110.2	16.4	---
3	3.25	115.9	14.0	20.0
4	3.10	95.0	27.7	19.2
5	3.10	80.4	30.1	21.5
6*	3.10	112.7	13.6	Swells & crack

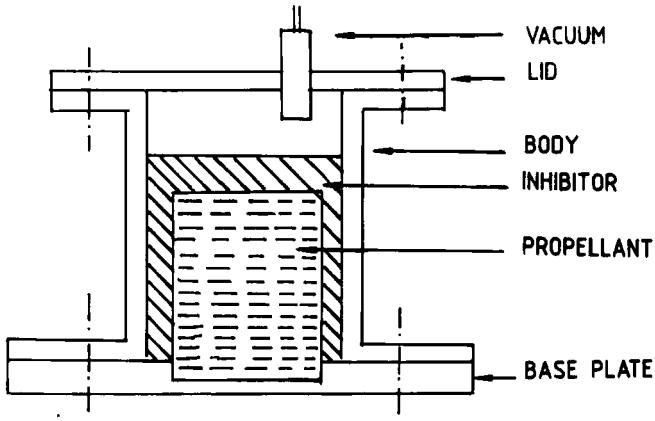
* ensile Strength and % Elongation values at 60°C after 5 days. B-S with nitramine containing CDMB propellent and after giving precoats of Desmodur R and fast setting polyester resin (Acrolite 471) epoxy resin was used as main inhibitor.

Bond Strength.

The bonding properties were determined by measuring the bond strength between CMDB propellant and epoxy resin. The propellant samples (dimension in mm= 50x20x10) were prepared by rubbing their ends with emery paper where bonding with inhibitor was required and covering the remaining portion with aluminium foil. As epoxy resin is known to be incompatible with double base matrix (NC+NG), therefore propellant faces (ends) were given precoats of Desmodur R (triphenyl methane triisocyanate) and of fast setting polyester resin (Acrolite 471). The precoated propellant (faces) pieces were placed in an aluminium mould with prepared surface facing each other at a distance of one cm apart. The epoxy resin composition along with diluent, filler and curing agent was poured between the gap of the propellant samples. After 24 hours the pieces were removed and prepared. Bond strength was determined on the instron machine (Table 2)

Inhibition and static evaluation of CMDB propellants.

A CMDB propallent charge containing RDX (cyclo trimethlene trinitramine) machined to the dimension (OD= 129 mm, L=40mm) was subjected to a brush coat of Desmodur R. After a period of 3-4 hrs, three brush coats of polyester resin of Acrolite 471 (setting time = 10 minutes) one over other were applied. Charge was put in the inhibition assembly mould and epoxy resin (Debeckot E4 + EH-411 + Dil-C + filler Sb_2O_3) was allowed to pour by casting method. Charge was kept overnight at ambient in the assembly (Fig.1). Charge was extracted from inhibition mould after 24 hours. Inhibition charge was machined to OD = 135 mm., L = 50 mm X-rayed and statically evaluated after conditioning for 24 hrs at low ($-30^{o}C$), ambient and high (16 hrs at + $50^{o}C$) temperatures for burning duration of 3-16 seconds.

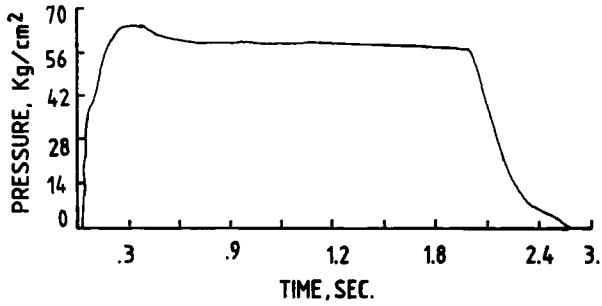


1. Inhibition assembly for nitramine containing CMDB propellants

Storage-ageing trials were also carried out at ambient and hot temperatures (+60°C) by putting inhibited charge at ambient and at 60°C in constant temperature water jacketted oven and observed for the bond failure between propellant and inhibitor.

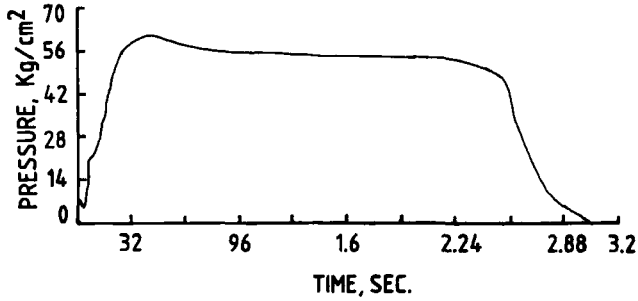
Results and Discussion

Epoxy resin E4 cured with polyamide hardener EH-411 has a very high tensile strength (252 kg/cm^2) and poor elongation (2 %). This resin is very hard and brittle on curing. To increase its elongation, Diluent-C (monoepoxy diluent) was added leading to 16.4 % elongation. Finally Sb_2O_3 was also incorporated in the composition to make it more flame-retardant. Thus composition no.5 was selected because of good balance of mechanical properties along with reasonable setting or gel time. This composition was utilized for inhibition of RDX (nitramine) containing CMDB propellants. Bond strength of the resin with propellant was found to be 21.5 kg/cm^2. It was also observed that break occurs in the CMDB propellant and not at the

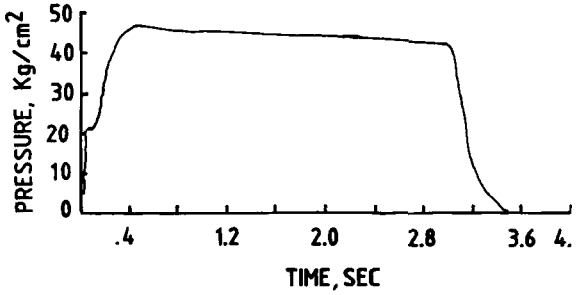


2. Pressure-time profile for nitramine containing CMDB propellants inhibited with epoxy resin at ambient temperature in cigarette burning mode.

propellant-inhibitor interface indicating good bonding of the inhibitor with the CMDB propellant. Static evaluation (propellant in rocket motor fired in static condition) of CMDB propellants in a cigarette burning mode gives presure-time profiles at ambient, hot (+50°C) and cold (-30°C) temperatures (Fig. 2-4) for burning duration of 3.0 seconds. Pressure time profile were near neutral. There was no progressivity or regressivity observed in any of the firings. The pressure-time curve indicates the suitability of epoxy resin as inhibitor for nitramine containing CMDB propellants. Due to these results epoxy resin was considered suitable for inhibition of CMDB propellants. Epoxy resin was having characteristic properties like gel time, oxygen index, thermal conductivity and heat resistance as given in the table 3. These values are comparable with reported values of other inhibitors.[6,10] Further a full sustainer charge (L=160 mm, OD=129 mm) was inhibited in a similar way by epoxy resin and statically evaluated in sustainer mode at ambient temperature for burning duration of 16 seconds.



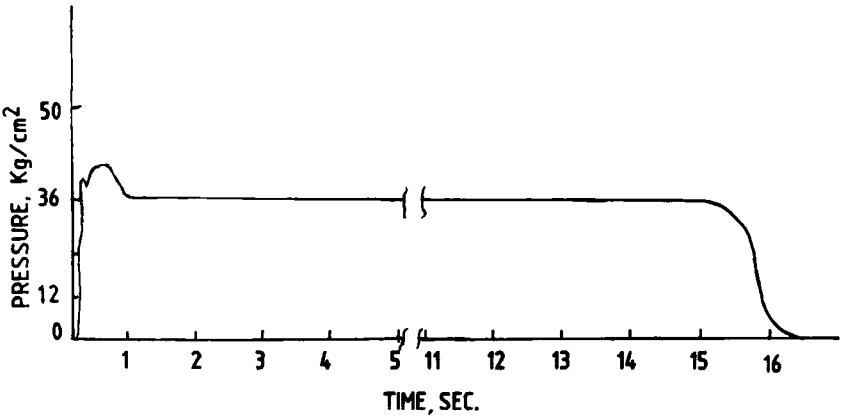
3. Pressure-time profile for nitramine containing CMDB propellants inhibited with epoxy resin at hot (+50°C) temperature in cigarette burning mode.



4. Pressure-time profile for nitramine containing CMDB propellants inhibited with epoxy resin at cold (-30°C) temperature in cigarette burning mode.

Table 3 : Characteristics of Epoxy resin inhibitor (F No.5)

Geltime	3.10 hr
oxygen index	21.5
Thermal conductivity (W/mk)	0.88 at 89°C
Heat resistant (Wt loss %)	0.98 after 10 hr
Brittle temperature	+15°C



5. Pressure-time profile for nitramine containing CMDB propellants inhibited with epoxy resin at ambient temperature in sustainer mode.

The pressure-time curve was near neutral indicating the suitability of epoxy resin as inhibitor for CMDB propellants (Fig. 5).

Storage-ageing trials

Epoxy resin inhibited CMDB propellants and their bond strength pieces were kept at ambient and at 60°C for storage-ageing trials and bond failure between propellant and inhibitor was monitored. At ambient temperature there was no change in bonding between propellant and inhibitor (bond remains intact) for more than three years (07.10.91 to 28.12.94). There was no change in mechanical properties of the inhibitor during this period. However, storage-ageing at 60°C in water-jacketted oven, both CMDB propellant and epoxy resin inhibitor developed crack even after 5 days. This may be due to gasification in the propellant resulting a crack in charge and inhibitor. Secondly

at hot temperature (+60°C) charge may be expanded which was restricted by hard and crosslinked epoxy resin[13] and resulting in to swelling of the charge and crack in the inhibitor and propellant. So the storage-ageing test at hot (+60°C) was discontinued.

Conclusion

Epoxy resin was found suitable as inhibitor for nitramine containing CMDB propellants at ambient, hot (+60°C) and cold (-30°C) temperatures. Incompatibility between double base matrix (NC + NG) and epoxy resin was overcome by applying barrier coats of Desmodur R and fast setting polyester resin Acrolite 471. Storage-aging trials at ambient and hot (+60°C) temperatures suggest that at ambient epoxy resin-propellant bond is intact for more than 3 years (still under observation),however at 60°C bond failure occurs.

Acknowledgments

We are thankful to Dr. H. Singh, Director, ERDL, Pune for his permission to publish this paper. Thanks are due to Sh P G.Shrotri, Group Director and Scientist F, CP Division for encouragement and suggestions.

REFERENCES

1. C.G.Lawson, ERDE(U.K.),Tech.Memo.14/M/65 (1965)
2. R.Stenson, ERDE(U.K.),Tech.Note no.48 (1971)
3. B.Hans, German pat 1206340 (1965) Chem. Abstr 64, 6395 (1966)
4. ERDL work on inhibition of double base and cmdb propellants by EP-4 polyester resin (restricted)
5. O.G. Schaffling, U.S.Pat. 3,947,523 (1976) Chem.Abstr. 85, 35214 (1976)

6. J.P.Agrawal, M.P.Chouk, R.S.Satpute and V.C.Bhale, J. Polym. Sci., Part A, 27, 409 (1989)

7. S.Venugopalan and J.Ather, Inhibition of CMDB propellants by silicone polymers, Paper presented at " HIGH ENERGY-MATERIAL SOCIETY, TRIVENDRUM,INDIA (1994)

8. J.P.Agrawal,D.C.Gupta and Yogesh Khare, J.polym.mat. 8, 121 (1991)

9. J.P.Agrawal, P.G.Shrotri,D.C.Gupta and M.P.Chouk , Composite Manufacturing 2(1) ,48 (1991)

10. J.P.Agrawal, D.C.Gupta, M.P.Chouk and Yogesh Khare , Propellants, Explosives and Pyrotechnics, 18, 155 (1993)

11. D.C.Gupta ,S.S.Deo,D.V.Wast, S.S.Raomore and D.H. Gholap ,J.Appl.Polym.Sci. 55,1151 (1995)

12. D.C.Gupta and P.K. Divekar, Unpublished work on inhibition of CMDB propellants by polyurethanes. (To be sent for publication).

13. H.Lee and K.Neveville "Epoxy resins,their applications and technology" McGraw Hill Co., New York, USA, 1957.