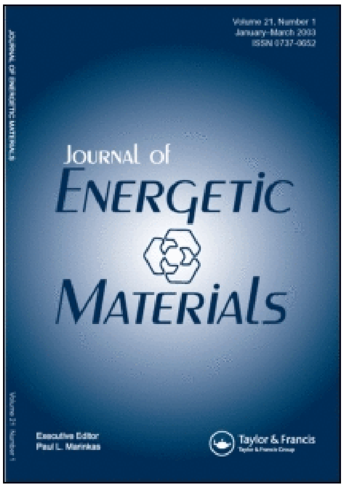

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LEAD SALT OF 2,4,N-TRINITROANILINOACETIC ACID - AN ENERGETIC BALLISTIC MODIFIER FOR DOUBLE BASE PROPELLANTS

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ABSTRACT

Lead Salt of 2,4,N-trinitroanilinoacetic acid has been evaluated as ballistic modifier in composite modified double base matrix. Burn rate measurements showed a plateau effect between 3.43 - 8.82 MPa. The salt-containing propellant composition showed a synergistic effect on decomposition of propellants as evidenced by a reduction in differential thermal analysis exotherm peak temperature. Effect of magnesium oxide-lead salt combination on burn rate of propellant formulation was also evaluated. The combination increased the burn rate

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at 6.86 - 8.82 MPa and decreased at 10.78 MPa. The salt has been prepared by double decomposition of lead nitrate with sodium salt of 2,4,N-trinitroanilinoacetic acid, which in turn is prepared from glycine and 2,4 - dinitrochlorobenzene. The lead salt decomposes at 187°C as shown by differential thermal analysis exotherm. Calorimetric value (+3.14 MJ/kg) of the salt has been calculated. The salt exhibits an impact sensitivity height ($h_{50\%}$) of 97.5 cm and friction sensitivity of >36 kg.

INTRODUCTION

Energetic additives play a vital role in enhancing the performance of propellants for rockets and missiles without affecting processing. One among them is ballistic modifiers, which are generally metal salts of carboxylic acids. Various lead salts like salicylates and stearates[1] have been widely used. It has been observed that lead salts of aliphatic carboxylic acids are effective in cool double base formulations, while lead salts of aromatic carboxylic acids are operative in hot formulations,[2] and at high pressures (beyond 8.82 MPa) they induce a plateau effect. However, the organic moiety in all these salts are inert and tend to bring down the energetics of the system. Salts of carboxylic acids, which contain energetic groups like -

NO_2 could increase the total heat output. Thus, it was of interest to synthesize a carboxylic acid, which contains a picrylamino moiety. 2,4,N-Trinitroanilinoacetic acid (TNAA) is an energetic carboxylic acid synthesized from glycine and 2,4 -dinitrochlorobenzene.[3] Subsequently, Pb^{2+} salt of this acid was prepared from TNAA and $Pb(NO_3)_2$. This salt, to the best of our knowledge, is the first example of an energetic ballistic modifier used in a composite modified double base propellant formulation.

EXPERIMENTAL

Synthesis

Synthesis of Pb^{2+} salt of TNAA has been carried out as reported.[3]

The thermal decomposition characteristics of lead salt were studied by differential thermal analysis (DTA) and thermogravimetry (TGA). These were recorded using a Netzsch thermal analyzer model 409 in static air. Analytical grade alumina, previously dried at 700°C for an hour, was used as reference material. Sample mass used was 10 mg and the heating rate employed was 10°C/min. Impact sensitivity test was carried out on an impact sensitivity apparatus of falling hammer type with a falling weight of 2 kg, fabricated in this laboratory and friction sensitivity was done on Julius Peters friction sensitivity apparatus.

Propellant Processing

Ingredients used in double base propellant compositions were dense nitrocellulose (DNC) (12.2% nitrogen content) in spheroidal form of 20-25 μm size, energetic plasticizer nitroglycerine (NG), inert plasticizer diethylphthalate (DEP) and thermal stabilizer 2-nitrodiphenyl amine (2NDPA). In composite modified double base (CMDB) formulations, ammonium perchlorate (AP) of 8-10 μm was used. In addition, toluene di-isocynate (TDI) was used as curing agent for DB composition in 0.5 parts in all formulations. Ingredients were dried to a moisture level of less than 0.5% before processing. NG desensitized by mixing with DEP was deaerated under vacuum for half an hour at less than 10 mm of mercury before use. Propellant processing was done by slurry cast process in 1 kg batch size by mixing thoroughly all the ingredients and casting in aluminum moulds. The slurry was evacuated for half an hour at less than 10 mm of mercury before curing in an oven at 60°C for minimum three days. Cured propellant samples were cut into strands to generate data on calorimetric value, stability, burn rate etc. Compatibility tests were done by conducting heat test at 71°C as per British

Standard. Thermal stability was determined by carrying out methyl violet test (MV Test) as per standard procedure followed for DB propellant. An average of five runs are routinely done to determine the stability. Burning rate was determined using 6 mm propellant strands in an acoustic strand burner apparatus. Calorimetric value was determined using a Parr adiabatic bomb calorimeter at 0.01 g/cc loading. Mechanical properties were determined using universal testing machine, INSTRON, on samples prepared as per the ASTM standard.

RESULTS AND DISCUSSION

DTA of the salt shows an exothermic peak with inception temperature at 170 °C and peak temperature at 182.5 °C (Fig.1). The TGA curve shows a single step weight loss with an inception temperature of 169 °C and final temperature of 180 °C, with a total weight loss of 70.3%. This corresponds to a residue of 29.7%, which is in agreement with the calculated residue weight of 28.71% due to PbO. The energetics of the reaction possibly leads to the formation of PbO from the salt without the formation of any intermediate. The calorimetric value of lead salt was calculated to be 3.14 MJ/kg. The impact sensitivity of the salt is 97.5 cm and friction sensitivity is >36 kg. These values indicate that salt is insensitive and safe for processing in propellant formulations. DTA of the propellant sample shows an exotherm peak at 166°C and is identical to that of the lead salt, which indicates that lead salt is compatible with the DB matrix. This is further confirmed by heat test results (Table 1).

TABLE 1

Effect of Pb Salt on DB Propellant - Compatibility Studies(Control Composition (%):DNC-50, NG-40, DEP-9, 2-NDPA-1)

Pb salt (parts)	Cal-val. (MJ/kg)	Heat test (min.)	DTA exotherm (°C)		
			inception	peak	end
Nil	3.85	8	165	198	215
2	3.84	9	166	190	207

Plots of pressure Vs burn rate are shown in Fig.2. The formulation containing Pb^{2+} salt shows higher burn rate and a plateau effect is observed between 3.43 and 6.86 MPa. The lead salt containing formulation was found to be less stable by methyl violet test (Table 2). To moderate this, MgO was incorporated in the formulation. The stability and thermal analysis data of MgO-moderated samples also show that these formulations are compatible with the DB matrix (Table 2). The burn rate Vs pressure data of MgO-moderated formulation is given in Table 3. The behaviour at this pressure range was found to be the expected post plateau behaviour as in control formulation. The mechanical properties (Table 4)in the presence of the salt were found to be more or less the same as that of control. If we compare the mechanical properties of leaded versions (i.e. first two rows of Table 4.) we can see that the tensile strength, % elongation and modulus remain more or less the same. When MgO is incorporated, more so when MgO and lead salt are present, they may be showing some reinforcing characteristics, and hence the higher values of TS and modulus compared to the control.

CONCLUSION

Lead salt of TNAA, a new energetic ballistic modifier has been evaluated in a double base formulation. Calorimetric value of the salt indicates the compound is energetic. The salt is insensitive, thus safe for processing, and is compatible with double base propellant matrix. The salt induces a plateau burning of the double base propellant formulation in the pressure range 3.43 -

8.82 MPa. Lead salt effected an increase in burn rate at 6.86 - 8.82 MPa. The MgO moderated formulation exhibited lower pressure index compared to the formulation containing lead salt alone.

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TABLE 2

Effect Of Pb Salt On CMDB Propellant -Thermal Decomposition and Stability Studies. (Control Composition (%): DNC-45, NC-36, DEP-8, 2NDPA-1, AP-10)

Modifier (parts)		Methyl violet test (min)		DTA exotherm (°C)		
Pb salt	MgO	Colour Change	Explosion time	inception	peak	end
--	--	30	120	172	200	215
2	--	45	195	170	200	240
--	2	45	>300	172	200	228
1	1	45	>300	170	185	205

TABLE 4

Effect of Pb Salt on CMDB Propellant - Mechanical Property (Control Composition (%): DNC-45, NG-36, DEP-8, 2NDPA-1, AP-10)

Modifier (parts)		Mechanical properties		Youngs modulus
Pb salt	MgO	TS (MPa)	%Elongation	(MPa)
--	--	6	84	32
2	--	6	77	34
--	2	7	66	44
1	1	9	57	65

TABLE 3

Effect of Pb Salt on CMDB Propellant - Calorimetric Value and Burn Rate Studies (Control Composition (%) : DNC-45, NG-36, DEP-8, 2NDPA-1, AP-10)

Modifier (parts)		Cal-val. (MJ/kg)	Burn rate (mm/s) pressures in (MPa)					Pressure index for pressure range (MPa)			
Pb salt	MgO		3.43	4.90	6.86	8.82	10.78	3.43-4.90	4.90-6.86	6.86-8.82	8.82-10.78
--	--	4.20	6.1	7.6	8.7	10.1	11.5	0.62	0.40	0.59	0.65
2	--	4.20	8.1	8.2	8.4	10.7	13.1	0.03	0.07	0.96	1.01
--	2	4.17	5.8	7.0	8.3	10.2	10.9	0.53	0.51	0.82	0.33
1	1	4.17	7.9	8.0	8.6	10.9	12.1	0.04	0.21	0.94	0.52

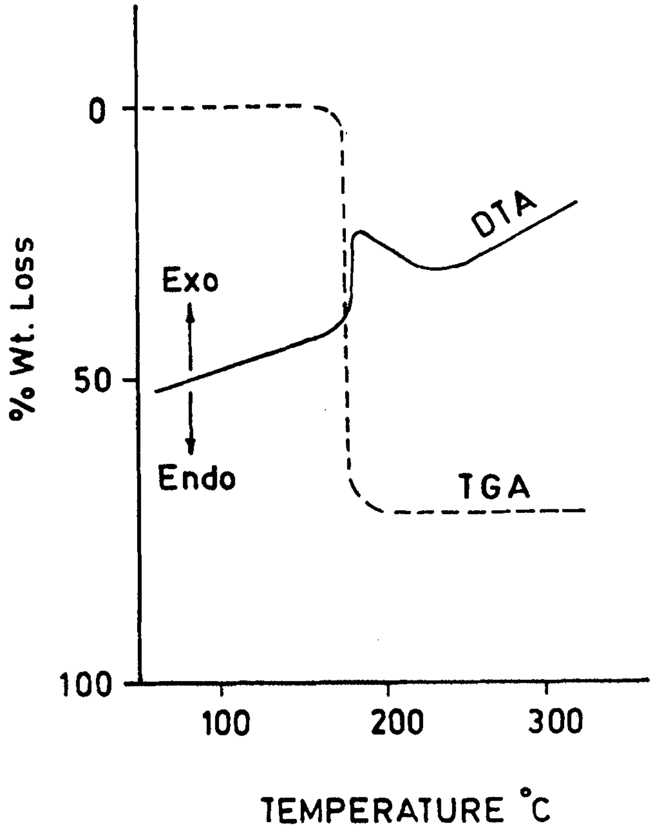

Figure 1

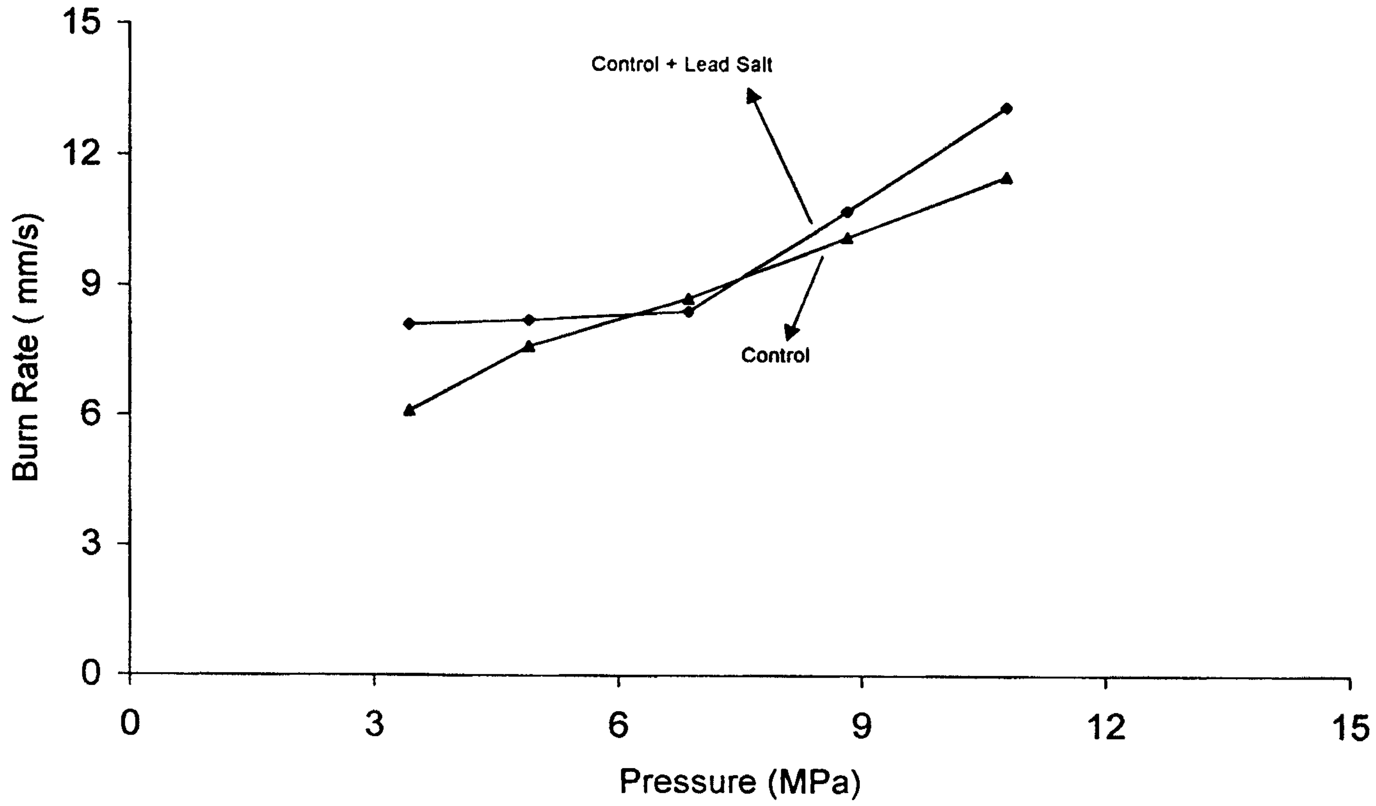

Fig. 2 : Plot of Pressure versus Burn Rate