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Evaluation of Energetic Plasticisers for Solid Gun Propellant

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ABSTRACT

This paper reports the evaluation of four different energetic plasticisers, viz., glycidyl azide polymer (GAP, MW = 390), 1,5-diazido-3-nitrazapentane (DANPE), ethylene-glycol-bis-azido-acetate (EGBAA) and *N*-*n*-butyl-*N*-(2 nitroxyethyl) nitramine (*n*-Bu-NENA) separately into high energy gun propellant containing 28 per cent NC (13.1 N %), 65 per cent RDX, 6 per cent di-octyl-phthahate (DOP) and 1 per cent carbamite. Four different propellant compositions based on the energetic plasticiser have been formulated separately with the replacement of non-energetic plasticiser, DOP. The propellants were processed by standard solvent method and evaluated experimentally along with the control composition to determine the ballistic parameters, cal-val, sensitivity, thermal characterisation, thermal stability and mechanical properties. The performance of the propellants containing the energetic plasticiser has been compared with that of the control composition containing the non-energetic plasticiser, DOP so as to assess the suitability of the energetic plasticiser for the futuristic gun propellant formulations. It has been found out that *n*-Bu-NENA is the superior plasticiser among the four energetic plasticisers evaluated in this study.

Keywords: Energetic plasticiser, flame temperature, force constant, mechanical properties, burning rate characteristics

1. INTRODUCTION

Plasticiser is a key ingredient of gun propellant which plays a vital role in controlling the mechanical properties. Moreover, it imparts the homogeneity and plasticity to the propellant dough and thus, it facilitates the processing of the propellant[1]. Plasticisers exist in liquid state and are generally high molecular weight esters compatible with nitrocellulose (NC) and nitroglycerine[2]. However, sometimes solid plasticisers like camphor, centralite, dinitrotoluene, etc. are also used in gun propellant formulations[3]. It has been observed that the lower viscosity and glass transition temperature (T_g) of the plasticiser improve the processability of the propellant whereas the availability of long carbon-carbon chains and conjugation in its molecular structure contribute for the better mechanical properties[2]. It is, therefore, the organic phthalates such as dioctyl-phthalate (DOP), dibutyl-phthalate (DBP), dioctyl-adipate (DOA), etc. have been extensively used as plasticisers in the conventional gun propellants[4-6]. However, it has been observed that the incorporation of organic phthalates into propellant composition do not contribute in the energy output. In contrast, energetic plasticisers invariably contain the explosophoric groups such as nitro, nitrato, fluoronitro, fluoroamino, azido, etc. in the carbon backbone of the molecule and thus they significantly contribute to the energy of the propellant[7]. Literature survey reveals that a

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large number of energetic plasticisers have been synthesised and characterised all over the globe[8-10]. However, it appears that no systematic work has been done on the evaluation of energetic plasticisers into solid gun propellant. Hence, this work has been undertaken.

Four different energetic plasticisers, viz., glycidyl azide polymer (GAP),1,5-diazido-3-nitrazapentane (DANPE), ethylene–glycol–bis–azido–acetate (EGBAA) and *N*-*n*-butyl-*N*-(2nitroxyethyl) nitramine (*n*-Bu-NENA) have been selected for the present study. Selection of the plasticiser was made on the basis of thermodynamic data and rheological properties which have great influence to the energy output and mechanical properties of the propellant. Thermodynamic parameters like flame temperature, mole number, mean molecular weight, heat of formation, density, and oxygen balance have been considered as the key determinants of energy whereas viscosity and glass transition temperature of the plasticiser were measured to improve the processing and ultimately the mechanical properties of the propellant. In addition, factors like availability, cost, ease of synthesis, and yield of the plasticiser were also taken into consideration while assessing the suitability.

Flanagan[11], *et al.* explored the possibility to increase the force constant of gun propellant by replacing the non-energetic plasticiser PEG-4000 with an energetic plasticiser blend consisting of trimethylol ethane trinitrate (TMETN) and diethylene glycol dinitrate (DEGDN). In a similar way, an attempt to replace the non-energetic plasticiser, DOP, separately with the energetic plasticisers GAP, DANPE, EGBAA, and Bu-NENA has been made. It has been reported that the propellant composition containing 28 per cent NC (13.1 N %), 65 per cent RDX, 6 per cent DOP and 1 per cent carbamite exhibits an ideal performance for the tank gun ammunition in respect of higher force constant (1200 J/g) at relatively lower flame temperature (3210 K), reasonably good burning rate characteristics (β^1 = 0.14 cm/s/MPa, α = 0.90) and mechanical properties (UTS = 180 kgf/cm^2, compression = 13.10 %)[12]. In view of this, it has been referred as the control composition for the present study. Attempts to replace the non-energetic plasticiser, DOP, with an energetic plasticiser are aimed to increase the force constant of the propellant so as to achieve a higher muzzle velocity.

2. EXPERIMENTAL

2.1 Materials

The energetic plasticisers, viz., GAP, DANPE, EGBAA, and *n*-Bu-NENA have been synthesised in the laboratory on the lines of the reported procedures[13-16], whereas the non-energetic plasticiser, DOP, was procured from trade. The plasticisers have been characterised on the basis of instrumental analysis, viz., FTIR, H^1-NMR, HPLC, thermal analysis, and elemental analysis.

2.2 Methods

The control composition contains 28 per cent NC (13.1 N %), 65 per cent RDX, 6 per cent DOP, and 1 per cent carbamite. Four different propellant compositions have been formulated with the replacement of non-energetic plasticiser, DOP (6 %), separately by the energetic plasticisers, viz., GAP, DANPE, EGBAA, and Bu-NENA (Table 1). Theoretical performance of the propellants was computed using "THERM" Programme[17] and presented in Table 1. The compositions were processed on a laboratory scale (1 kg batch) using the standard solvent method[18] with 30 per cent solution of acetone and alcohol in 70 : 30 ratio. The propellants were made into multi-tubular configuration and dried in an oven by passing hot air blow at 45 °C till the volatile matter reduced to ~1 per cent. The dried propellant samples were tested for physical measurements like web size, density and finally subjected to evaluation tests.

2.3 Evaluation

The propellants were fired into 700 cc high pressure closed vessel at the loading density of 2.0 g/cc in order to determine the ballistic parameters. Impact sensitivity was determined by standard Fall Hammer setup according to the Bruceton Staircase Approach[19], whereas friction sensitivity was assessed on the Julius Peter apparatus[20] by incrementally increasing the load from 0.2 kg to 36.0 kg till there was no ignition/explosion in five consecutive test samples. Thermal stability was studied on the basis of evolution of gaseous nitrogen oxide taking place during the heating of propellant sample by applying both qualitative (Abel heat test and methyl violet test) and quantitative (Bergmann and Junk test) methods as per standard procedure[21]. The calorimetric value of propellant composition was determined in Julius Peter's adiabatic bomb calorimeter. Ignition temperature was recorded in Julius Peters Furnace whereas the decomposition temperature was determined on the basis of DTA analysis. Mechanical properties of the propellants were determined on Universal Testing Machine (Instron-1185).

3. RESULTS AND DISCUSSION

Theoretical calculations made in respect of thermo-chemical properties of the propellant compositions indicate that the replacement of non-energetic plasticiser, DOP (6 %), separately with the energetic plasticiser, viz., GAP, DANPE, EGBAA, and Bu-NENA increases the force constant by around 75–90 J/g and flame temperature about 300–500 K (Table 1). The results of ballistic evaluation obtained from closed vessel tests are given in Table 2. The calorimetric values determined by adiabatic bomb calorimeter are presented in Table 3.

It has been observed that the experimentally determined values of force constant and cal-val are in good agreement with the theoretically calculated values. It is seen that the replacement of DOP with GAP increases the force constant by about 7 per cent, cal-val by about 15 per cent, T_F by about 13 per cent, and β_1 by about 7 per cent. However, the value of α found to decreased by about 11 per cent (Tables 2 and 3). A higher force constant (as compared to the control composition)

Table 1. Theoretically calculated thermochemical parameters of the propellant compositions

Propellant composition	Force constant (J/g)	Flame temp. (K)	'n' value (mole/g)	MW (g/mole)	Co-volume (ml/g)	Sp. heat ratio (ã)
NC (13.1 N %)/RDX/GAP/Carbamite 28 65 6 1	1280	3600	0.0462	23.46	0.9535	1.2448
NC (13.1 N %)/RDX/DANPE/Carbamite 28 65 6 1	1290	3700	0.04198	23.82	0.9442	1.2473
NC (13.1 N %)/RDX/EGBAA/Carbamite 28 65 6 1	1288	3705	0.04195	23.83	0.9440	1.2470
NC (13.1 N %)/RDX/Bu-NENA/Carbamite 28 65 6 1	1275	3500	0.04284	23.25	0.9545	1.2515
NC (13.1 N %)/RDX/DOP/Carbamite 28 65 6 1	1200	3210	0.04499	22.93	0.9843	1.2588

Table 2. C.V. test results of the propellant compositions

Propellant composition	Force constant (J / g)		Flame temp. (K)	Linear rate of burning coefficient, â₁ (cm/s/MPa)	Pressure exponent (á)
	Theoretical	Exptl.			
NC(13.1 N%)/RDX/GAP/Carbamite 28 65 6 1	1280	1275	3600	0.15	0.80
NC(13.1 N%)/RDX/DANPE/Carbamite 28 65 6 1	1290	1289	3700	0.15	0.86
NC(13.1 N%)/RDX/EGBAA/Carbamite 28 65 6 1	1288	1288	3705	0.16	0.88
NC(13.1 N%)/RDX/Bu-NENA/Carbamite 28 65 6 1	1275	1275	3500	0.14	0.80
NC(13.1 N%)/RDX/DOP/Carbamite 28 65 6 1	1200	1200	3210	0.14	0.90

exhibited by the GAP-based propellant is attributed to lower molecular weight of the combustion gases, higher density (1.3 x 10^3 kg/m^3) and positive heat of formation (ΔH_f = +957 kj/kg) of GAP[22] as compared to that of DOP whereas the higher level of flame temperature and cal-val exhibited by the GAP-based propellant is attributed to the higher oxygen balance of GAP than that of DOP [OB_{100} (GAP) = –121 and DOP = –257][22]. The ratio of specific heat of gases for GAP-based propellant was found to be lower than that of DOP-based propellant (Table 1). Hence, the GAP-based propellants exhibit higher flame temperature than the DOP- based propellants. However, GAP-based propellants show lower level of sensitivity as compared to DOP-based propellants (Table 4).This finding suggests that the sensitivity of GAP-based propellant is not predicted on the basis of oxygen balance alone but is a combination of several factors such as friction shear and thermal behaviour in addition to oxygen balance[23]. High-speed photographic study carried out by Agarwal[24], *et al*. also supports this finding.

Table 3. Calorimetric value of the propellant compositions

Propellant composition	Calorimetric value	
	Theoretical (cal/g)	Experimental (cal/g)
NC (13.1 N %)/RDX/GAP/Carbamite 28 65 6 1	1130	1127
NC (13.1 N %)/RDX/DANPE/Carbamite 28 65 6 1	1155	1155
NC (13.1 N %)/RDX/EGBAA/Carbamite 28 65 6 1	1160	1156
NC (13.1 N %)/RDX/Bu-NENA/Carbamite 28 65 6 1	1140	1138
NC (13.1 N %)/RDX/DOP/Carbamite 28 65 6 1	990	988

Table 4. Sensitivity tests of the propellant compositions

Propellant composition	Impact (h_{50} % expl) (cm)	Friction (kg)
NC (13.1 N %)/RDX/GAP/Carbamite 28 65 6 1	50	25
NC (13.1 N %)/RDX/DANPE/Carbamite 28 65 6 1	40	36
NC (13.1 N %)/RDX/EGBAA/Carbamite 28 65 6 1	28	20
NC (13. N %)/RDX/Bu-NENA/Carbamite 28 65 6 1	40	35
NC (13.1 N %)/RDX/DOP/Carbamite 28 65 6 1	45	25

GAP-based propellant was found to have lower value of pressure exponent: (α) even though it exhibits higher value for the linear rate of burning coefficient (β_1) (Table 2). Higher value of β_1 can be attributed to the large amount of heat evolved at the burning surface of the propellant because of the energetic additive, GAP. This is due to the fact that the heat flux generated at the burning surface of GAP as a result of exothermic scission of C-N_3 bond of GAP is higher than the heat flux transferred back from the gas phase to the burning surface during the combustion of DOP. However, the lower value of α is attributed to the higher stand-off distance between the luminous flame and the burning surface of GAP-based propellant resulting due to the large amount of carbonaceous matter produced at the burning surface of GAP.

The carbonaceous matter acts as a heat sink keeping the luminous flame away from the propellant surface, though the reaction rate in dark zone is increased by the addition of GAP[25]. Decomposition temperature of GAP-based propellants was found to be below 200 °C and slightly lower than the DOP-based propellants (Table 5). This is attributed to an exothermic scission of *C*=*N* bond available within the GAP molecule. Results of thermal stability measured by Abel Heat Test, Methyl Violet Test, and Bergmann and Junk Test indicate that GAP-based propellant is thermally stable (Table 6). This can be attributed to adequate nitrogen content (22.01 %) within the GAP molecule. However, the addition of GAP decreases the mechanical properties of the propellant in terms of tensile strength from 180 kgf/cm^2 to 112 kgf/cm^2 and compression strength from 13.10 per cent to 10.20 per cent (Table 7). This may be attributed to the higher viscosity (5000 CPS) and T_g value (–50° C) of GAP. Moreover, the availability of pendant alkyl groups and the lack of carbon-carbon chain in its molecular structure are also responsible features for the lower mechanical properties[2].

Replacement of DOP with DANPE and EGBAA separately helped to increase the force constant, flame temperature and cal-val of the propellant to higher level than that of GAP (Table 1). Moreover, composition No. 2 containing DANPE and composition

No. 3 containing EGBAA were found to exhibit slightly better mechanical properties as compared to composition No.1 containing GAP (Table 7). Hence, DANPE and EGBAA are considered to be better plasticisers than GAP. However, among the two plasticisers, EGBAA was found to accelerate the burning rate characteristics (β_1 and α) and lower the mechanical properties of the propellant as compared to DANPE (Tables 2 and 7). The higher burning rate characteristics (β_1 and α) exhibited by the EGBAA-based propellant are attributed to the relatively higher oxygen balance of EGBAA than that of DANPE and which enhances the combustion potential of the EGBAA-based propellant as compared to DANPE.

The relatively better mechanical properties exhibited by the DANPE-based propellant over EGBAA-based propellant are attributed to lower glass transition point (T_g–90° C) and viscosity (12.215 c/s) of DANPE as compared to that of EGBAA (T_g=–66.7 ^{0}C and viscosity= 19.3 c/s) demonstrating the good plasticising ability for the propellant dough and making processing much easier enabling higher filler loading. In view of this, DANPE has been considered to be a better plasticiser than EGBAA. However, Bu-NENA has been found to be a suitable plasticiser among the four energetic plasticisers evaluated, because it increases the force constant of the propellant almost to the level reached by using other plasticiser (GAP, DANPE, and EGBAA). But, it exhibits relatively lower flame temperature and burning rate characteristics (Table 2). This can be attributed to the lower molecular weight of the combustion gases produced by Bu-NENA.

Table 5. Thermal characteristic tests of the propellant compositions

Propellant composition	Decomposition temp (°C)
NC (13.1 N %)/RDX/GAP/Carbamite 28 65 6 1	195
NC (13.1 N %)/RDX/DANPE/Carbamite 28 65 6 1	202
NC (13.1 N %)/RDX/EGBAA/Carbamite 28 65 6 1	197
NC (13.1 N %)/RDX/Bu-NENA/Carbamite 28 65 6 1	203
NC (13.1 N %)/RDX/DOP/Carbamite 28 65 6 1	199

Table 6. Thermal stability tests of the propellant compositions

Propellant composition	Abel heat test at 65.5 °C (min)	MV test at 120 °C (5 h heating) (min)	B & J test at 120 °C (ml/5 mg)
NC(13.1 N%)/RDX/GAP/Carbamite 28 65 6 1	14	NBF	1.0
NC(13.1 N%)/RDX/DANPE/Carbamite 28 65 6 1	15	NBF	0.6
NC(13.1 N%)/RDX/EGBAA/Carbamite 28 65 6 1	13	NBF	1.2
NC(13.1 N%)/RDX/Bu-NENA/Carbamite 28 65 6 1	15	NBF	1.0
NC(13.1 N%)/RDX/DOP/Carbamite 28 65 6 1	12	NBF	0.25

Table 7. Mechanical properties of the propellant compositions

Propellant composition	TS (kgf/cm^2)	Elongation (%)	Flexural displacement (mm)	Compression (%)
NC (13.1 N %)/RDX/GAP/Carbamite 28 65 6 1	112	1.44	0.20	10.20
NC (13.1 N %)/RDX/DANPE/Carbamite 28 65 6 1	120	2.60	0.80	12.00
NC (13.1 N %)/RDX/EGBAA/Carbamite 28 65 6 1	116	2.00	0.60	11.00
NC (13.1 N %)/RDX/Bu-NENA/Carbamite 28 65 6 1	160	2.90	0.90	13.00
NC (13.1 N %)/RDX/DOP/Carbamite 28 65 6 1	180	3.66	1.00	13.10

Table 8. Principle parameters to ascertain better performance of the Bu-NENA-based propellant over other three propellants

Parameter	Value	Remarks
Ballistic parameters		
Force constant (J/g)	1275	Comparable to other three propellants; comparable muzzle velocity
Flame temperature (K)	3500	Less than other three propellants; comparatively less erosion of barrel
Linear rate of burning coefficient, $\hat{a}_1$ (cm/s/MPa)	0.14	Less, hence lower web size of the propellant. Higher loadability
Pressure exponent (α)	0.80	Less, hence safe for gun barrel
Sensitivity		
Impact ($H_{50\ \%}$ expl, cm)	40	Better than EGBAA and comparable to GAP and DANPE propellants and safe
Friction (kg)	35	Better than EGBAA and GAP and comparable to DANPE propellants and safe
Thermal stability		
Abel heat test (min)	15	Comparable to other three propellants; acceptable for production, better life
B&J test (ml/5mg)	1.0	Better than DANPE and comparable to GAP-and EGBAA-based propellants. Acceptable for production; better life
Mechanical properties		
Tensile strength (kgf/cm^2)	160	Superior to GAP-, DANPE-, and EGBAA-based propellants. Hence safe for processing and evaluation
Elongation (%)	2.90	
Compression (%)	13.0	

The combustion gases enriched with hydrogen and nitrogen increases the ratio of specific heat of gases (γ) which ultimately contributes towards the lower flame temperature and keeping the force constant at higher level. The propellant composition based on Bu-NENA has been found to exhibit relatively lower values for the burning rate characteristics (β_1 and α) as compared to the rest of three compositions (Table 2). This may be due to the typical combustion behaviour as followed by the GAP-based propellant[25]. Moreover, Bu-NENA-based propellant was found to pass all the thermal stability tests and shows low vulnerability in respect of sensitivity and decomposition temperature. In addition, Bu-NENA propellant exhibits reasonably good mechanical properties almost similar to the control composition (Table 7). This is mainly due to the relatively lower viscosity, availability of long carbon-carbon linkage of the alkyl group (*n*-butyl) and good plasticity tendered by the nitrate ester group[2].Thus, Bu-NENA has been found to be suitable energetic plasticiser meeting the stringent requirements of the gun propellant. Moreover, its synthesis was found to be quite easy and with higher yield. Table 8 gives the parameters of Bu-NENA-based propellant under which it has given better performance for productionisation over GAP-, DANPE-, and EGBAA-based propellants in respect of processing, gun erosion, safety and life of the propellant.

4. CONCLUSIONS

It is concluded that *N*-*n*-butyl-*N*-(2 nitroxyethyl) nitramines (*n*-Bu-NENA) is a promising energetic plasticiser for the solid gun propellants.This is followed by 1, 5-diazido-3-nitrazapentane (DANPE), ethylene-glycol-bis azido-acetate (EGBAA), and glycidyl azide polymer (GAP), respectively studied in this paper. Hence, *n*-Bu-NENA has got a potential for replacement of non-energetic plasticiser like DOP in the futuristic solid gun propellant.

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