Feasibility Study of Processing Estane-based LOVA Gun Propellant


High Energy Materials Research Laboratory, Sutarwadi, Pune-411 021

ABSTRACT

Low vulnerability ammunition (LOVA) propellant are currently being developed globally to replace all types of single-base, double-base and triple-base gun propellants, because LOVA propellants possess advantage like low vulnerability without compromising on ballistic properties. The objective of present study is to establish processing of thermoplastic elastomer (TPE); estane-based LOVA gun propellant in cord/heptatubular geometry. Keeping in view various advantages of TPE such as simple processing, greater dimensional stability, lower production losses, superior insensitivity, and mechanical properties of the propellants, estane 5731 (polyurethane-ester-MDI, aromatic polyester) is selected for feasibility study as a binder in LOVA-based gun propellant composition, processed by solvent and semi-solvent methods and compared feasibility of processing as well as their evaluation wrt ballistics, vulnerability, and mechanical properties. The results indicate that gun propellants processed by semi-solvent method and extruded at elevated temperatures exhibit better ballistics, mechanical, and vulnerability properties.

Keywords: LOVA gun propellant, RDX, vulnerability, thermoplastic elastomer, estane, solvent method, semi solvent method

1. INTRODUCTION

Conventional ammunitions are sensitive to external unplanned stimuli because of sensitive ingredients present in their propellant compositions. As a result of sensitive behaviour of these ingredients, many accidents have been reported not only in war but also during transport and storage of explosives. This led to the concept of development of LOVA propellant. In the present study, LOVA propellant formulations are made with thermally stable nitramines cyclotrimethylene trinitramine (RDX) dispersed in thermoplastic elastomeric binder, estane.

Thermoplastic elastomers (TPEs) are physically cross linked (i.e., intermolecular forces of attraction) polymeric systems having superior strain capability in low-temperature region like elastomers, while these can be processed as thermoplastic at elevated temperatures as they also regain their original physical characteristics on cooling. Basically, TPEs are copolymers of ABA or AB types, where A and B are hard and soft segments respectively. The hard segment is capable of crystallisation or association leading to thermoplastic behaviour, whereas soft segment provides elastomeric characteristics to the copolymer. Thermoplastic elastomers offer advantages to the formulator of solid propellants like simplified processing, greater dimensional stability control and lower production losses. TPE-based RDX propellants also have the advantage of high insensitivity to impact and friction stimuli. In the present study, TPE-based LOVA propellants...
are processed by both solvent and semi-solvent methods and the feasibility of processing such propellants is studied. The ballistics, mechanical, and vulnerability properties of these propellants have been examined.

2. EXPERIMENTAL

2.1 Materials

The formulations with solid loading up to 78 per cent were processed with fine RDX of average particle size 5 µm as an energetic oxidiser and estane (in the form of solid granules) as a polymeric binder. Cellulose acetate (CA) and low percentage of nitrocellulose (NC) is also used as an energetic binder in order to improve the burning characteristics as well as structural integrity. Triacetin (source-MERK, Mumbai) is employed as a plasticiser. Formulations were processed by both solvent and semi-solvent methods. Thermochemical parameters were theoretically computed by applying computer software (Therm) developed by HEMRL\(^5\). Table 1 gives details of formulations and the theoretical thermochemical data. The properties\(^6\) of estane are shown in Table 2.

3. METHOD OF PREPARATION OF PROPELLANT

The propellant mixing was carried out using Sigma blade mixer of 2.5 kg capacity. The stages of processing of propellant and sequence of addition were as follows:

(a) *Wet mix preparation*: Volatile matter-corrected wet RDX was coated with plasticiser (triacetin) in sigma blade incorporator for half an hour. The volatile matter-corrected wet NC and CA were added and incorporator was further run for one and a half hour. Resulting mix (wet mix) was kept for maturation for 5 days and then drying (40 °C) to keep volatile matter < 1 per cent was carried out.

(b) *Weighed out estane* was kept soaked in a glass beaker with 15 per cent of solvent (acetone and alcohol in 80:20 ratio) of total weight of dry propellant powder. Next day, glass beaker kept in a water bath was stirred at elevated temperature (55 °C–60 °C) for 35 to 40 min to get a clear solution.

### Table 1. Propellant formulations (Theoretical thermochemical data at 0.18 g/cc loading density)

<table>
<thead>
<tr>
<th>Ingredients</th>
<th>Ref composition</th>
<th>TPE I</th>
<th>TPE II</th>
<th>TPE III</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fine RDX</td>
<td>78</td>
<td>78</td>
<td>78</td>
<td>78</td>
</tr>
<tr>
<td>CA</td>
<td>12</td>
<td>8</td>
<td>4</td>
<td>--</td>
</tr>
<tr>
<td>TA</td>
<td>6</td>
<td>6</td>
<td>6</td>
<td>6</td>
</tr>
<tr>
<td>NC (12.2 % N)</td>
<td>4</td>
<td>4</td>
<td>4</td>
<td>4</td>
</tr>
<tr>
<td>Estane</td>
<td>--</td>
<td>4</td>
<td>8</td>
<td>12</td>
</tr>
<tr>
<td>F/C (J/g)</td>
<td>1114</td>
<td>1102</td>
<td>1090</td>
<td>1091</td>
</tr>
<tr>
<td>FT (C)</td>
<td>2916</td>
<td>2834</td>
<td>2755</td>
<td>2709</td>
</tr>
<tr>
<td>Co volume (g/cc)</td>
<td>1.0130</td>
<td>1.0257</td>
<td>1.0393</td>
<td>1.0549</td>
</tr>
<tr>
<td>Gamma</td>
<td>1.2616</td>
<td>1.2656</td>
<td>1.2695</td>
<td>1.2724</td>
</tr>
<tr>
<td>Cal-val (cal/g)</td>
<td>894</td>
<td>861</td>
<td>828</td>
<td>813</td>
</tr>
<tr>
<td>(P_{\text{max}}) (MPa)</td>
<td>245</td>
<td>243</td>
<td>241</td>
<td>242</td>
</tr>
</tbody>
</table>

### Table 2. Properties of estane

<table>
<thead>
<tr>
<th>Property</th>
<th>Estane</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mol. wt (Mn)</td>
<td>70000-72000</td>
</tr>
<tr>
<td>Softening point</td>
<td>55 °C</td>
</tr>
<tr>
<td>Decomposition temperature</td>
<td>380 °C</td>
</tr>
<tr>
<td>Density</td>
<td>1.18 g/cm(^3)</td>
</tr>
<tr>
<td>Shore ‘A’ hardness</td>
<td>94</td>
</tr>
<tr>
<td>Tensile strength</td>
<td>&gt; 64 kg/cm(^2)</td>
</tr>
<tr>
<td>Percentage elongation</td>
<td>620</td>
</tr>
</tbody>
</table>
(c) Dry propellant powder was poured in a sigma blade incorporator with continuous addition of estane solution.

(d) Sigma blade incorporator was run for four and a half hour to get homogenised propellant dough.

### 3.1 Solvent Method

(a) Half quantity of dough was taken for extrusion with the help of a vertical hydraulic press using suitable die-pin assembly. Cylindrical sample was extruded for mechanical properties while propellant strands in cord geometry were extruded for study of ballistics.

(b) Extruded strands were pre-dried and cut into length of 100 mm.

(c) The propellant cords were dried at elevated temperature (40 °C) by blowing hot air and then subjected to various tests like mechanical properties, ballistic, and various vulnerability tests.

(d) Percentage compression and compression strength were determined by Hounsfield testing machine using cylindrical propellant samples of ASTM Standard L/D ratio 2.

(e) Density was determined by Bianchi densimeter.

(f) To study the ballistic behaviour, all the propellant batches were subjected to closed vessel (CV) firing tests. The CV capacity was 700 cc and the loading density in all cases was 0.18 g/cc. All samples were conditioned at 27 °C for 24 h before firing. From CV firing tests, the ballistic parameters such as force constant, $P_{\text{max}}$, $dP_{\text{max}}$, rise time, pressure exponent and linear burning coefficient ($\beta_1$), etc were computed as per the internal ballistic solution$^7,8$

(g) The impact sensitivity was determined on a 20 mg sample using 2 kg free fall weight by applying Bruceton Staircase method. Friction sensitivity was obtained by testing 5 mg sample in Julius Peter's Apparatus. Ignition temperature was determined by Julius Peter's Apparatus at a heating rate of 5 °C/min. The methods used for determination of impact sensitivity, friction sensitivity and ignition temperature has been given in reference$^9$.

### 3.2 Semi-solvent Method

(a) Remaining half quantity of dough was removed from the incorporator and flattened on an aluminium tray to evaporate solvent at room temperature till all its traces are removed to maximum. The resulted hard dry mass of propellant was then passed through the warm roller maintained at 55 °C–60 °C. After giving minimum 5 number of passes, a propellant sheet was obtained. Propellant discs of 60 mm dia were then punched from the sheet. Hand-operated vertical extrusion press of 60 ton capacity was employed for the extrusion using two different brass die configurations of 14 and 3 mm.

(b) Jacketed press cylinder along with die-pin assembly was preheated by hot water circulation to 55 °C–60 °C. The punched discs were also pre-heated to same temperature were loaded into the press cylinder and extruded.

The extrusion pressure was found to be of the order of 50-55 kg/cm$^2$ for 14 mm dia and 70-80 kg/cm$^2$ for 3-4 mm strands. Propellant flow during extrusion was uniform and no irregular flow patterns were observed. The extrusion rate was observed to be 15-17 mm/min. The extruded propellant strand was soft and had smooth and uniform surface. The propellant strands were subject to evaluation similar to the solvent method.

### 4. RESULTS AND DISCUSSION

Feasibility of processing of TPE-based LOVA gun propellant by both solvent and semi-solvent methods has been assessed and it is evaluated wrt ballistics, vulnerability and mechanical properties (Tables 3, 4 and 5). TPE I compositions are not studied as these compositions could not form sheets after passing through warm rollers, consequently no further hot extrusion was carried out. This may be attributed to the presence of relatively higher percentage of CA, which is thermoplastic in behaviour. Tables 3, 4 and 5 indicate superior characteristics.
of propellant processed by semi-solvent method. This may be attributed to the better colloiding and gelatinising power of TPEs in the presence of a solvent during incorporation stage, then hot rolling and hot extrusion, which further improved the gelatinisation of propellant. Estane exhibited superior compression strength and percentage compression combination at low temperature, i.e., compression strength 204 kg/cm$^2$, compression 19 per cent for TPE II and compression strength 122 kg/cm$^2$, compression 16 per cent for TPE III than propellant processed by solvent method (compression strength 106 kg/cm$^2$, compression 7 per cent) for TPE II and (compression strength 130 kg/cm$^2$, compression 7 %) for TPE III at –20 °C.

Improvements in mechanical properties in general can also be correlated with the presence of aromatic ring in the polymer backbone and involvement of urethane linkage (soft block) in hydrogen bonding between polymer chains. Semi-solvent-processed, TPE-based propellant also exhibits low vulnerability wrt impact ($h_{50} = 99$ cm for TPE II and 70 cm for TPE III, friction sensitivity 36 kg, and ignition temperature > 360 °C) for both the compositions as compared to the solvent method, i.e., $h_{50} = 53$ cm for TPE II and 75 cm for TPE III, friction sensitivity 36 kg and ignition temperature >220 °C for both the compositions. Superior insensitivity characteristics can be attributed to elastomeric nature conferred on these by soft blocks and effective coating of polymer. Increase in the elasticity of TPE during hot spot formation by impact/friction stimuli may be responsible to the reduction of inter-crystalline friction.

In case of a polymer capable of coating of explosive particles, the reaction front meets the surface coating prior to high-energy materials, leading to decrease in probability of appearance of hot spots, and thereby decrease in impact sensitivity. The effective coating can contribute towards efficient quenching of the hot spots because of the formation of the molten layer around RDX particles. As regards ballistics, both the compositions gave higher force constants (1090 J/g and 1091 J/g–theoretical and 1109 J/g and 1095 J/g–experimental) at pressure exponent value < 1 at a flame temperature of
2755 K and 2709 K for semi-solvent method. The values of \( dP_{\text{max}} \) for solvent-processed compositions are very high, i.e., 134 MPa/ms and 90 MPa/ms as against semi-solvent method (12 MPa/ms). Low values \( dP_{\text{max}} \) may be attributed to high degree of gelatinisation in semi-solvent method. This trend can also be explained on the basis of the correlation between burning rate and mechanical properties proposed by Barnes and Fisher\(^1\). They have opined that when the propellant has superior mechanical properties, the flame propagation occurs by rather relatively slow laminar mechanism. In case of propellant with relatively lower mechanical properties, the higher burning rate, and hence, high \( dP_{\text{max}} \) may result due to compressive ignition on stress-induced in-depth ignition.

5. CONCLUSIONS

It can be concluded that estane-based gun propellant processed by semi-solvent method and extruded at elevated temperature exhibits superior ballistics, mechanical and vulnerability properties as compared to that processed by solvent method.

REFERENCES


Contributors

Mr M.A.R. Shaikh received his MSc (Chemistry) from the University of Pune. Presently, he is working as Scientist C at the High Energy Materials Research Laboratory (HEMRL), Pune. He has made significant contributions in the field of inhibition of rocket propellants. Presently, he is working on the processing and development of gun propellants for high caliber guns.
**Dr R.R. Sanghavi** obtained his MSc (Physical Chemistry) from the University of Pune in 1984, MMS (Management Science) from the Symbiosis Institute of Business Management in 1987, and PhD (Chemistry) from the University of Pune in 2004. He has been working as Scientist at the HEMRL, Pune, since 1992. His areas of research include development of propellants for tank and artillery guns.

**Mr S.D. Shelar** obtained his BE (Mechanical) from the University of Pune. He joined HEMRL, Pune in 1996. He has made significant contribution in design, development and fabrication of dies required for different types of gun propellant. Presently, he is engaged in the processing and development of LOVA gun propellant. He is a life member of the Aeronautical Society of India, Pune and Indian National Society for Aerospace & Related Mechanisms (INSARM).

**Dr T.K. Chakraborty** is working for the development of gun propellants having better performance characteristics in the form of higher energy content, better mechanical properties, and higher peizometric efficiency. He has also worked for the development of propellant for HESH and FSAPDS ammunition for MBT Arjun and development of liquid gun propellant. At present, he is engaged in the development of propellant for modular charge system, and low temperature sensitive propellant.

**Dr Amarjit Singh** obtained his MSc (Organic Chemistry) and PhD from the University of Pune. He has more than 85 publications and 3 patents to his credit. He is currently Associate Director HEMRL. His major contributions include: Improved picrite propellant, and low vulnerability propellants for gun ammunition, energetic binders and plasticisers for gun applications, bi-modular and unimodular charge system for 155 mm gun, etc.