Research paper

Studies on the Ballistic Parameters of a Deterred Triple Base Propellant Used in Large Caliber Ammunition

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Abstract: In this work, the energetic plasticizer *N*-n-butyl-*N*-(2-nitroxyethyl) nitramine (Bu-NENA) was used as a deterrent coating for a triple base propellant (TBP) that finds application in large caliber guns. Closed vessel (CV) evaluation was performed to study the ballistic performance of the deterred propellant. A TBP containing nitrocellulose (NC), nitroglycerine (NG) and nitroguanidine (NQ) was coated with an energetic plasticizer using a solution of Bu-NENA. The Bu-NENA coated propellant exhibited inhibition to burning resulting in a reduction in the values of the ballistic parameters *viz.* $(dP/dt)_{max}$, burning rate coefficient (β) and relative vivacity (RV), while the pressure index (α) and rise time were increased. The trends obtained during this work display the potential of Bu-NENA as deterrent.

Keywords: progressive burning, deterred propellant, Bu-NENA, pressure index, force constant, burning rate, chamber pressure

Nomenclature:

Bu-NENA	*N*-n-Butyl-*N*-(2-nitroxyethyl)nitramine
CV	Closed vessel
NC	Nitrocellulose
NG	Nitroglycerine
NQ	Nitroguanidine
RV	Relative vivacity
RF	Relative force
TBP	Triple base propellant

1   Introduction

It is reported in the literature that deterrent coating of propellant surfaces broadens the pressure-time curve maxima and provides increased muzzle velocity (810-840 m/s) by keeping the pressure within gun barrel limits. Deterrents lower the burn rate and thus the rate of gas generation is initially slow [1]. It is well reported that deterred propellant grains meet the need for the higher velocity of kinetic energy projectiles for a given grain geometry and loading density. Deterrents should be chemically compatible with the base propellant, able to adequately penetrate into the grains of the propellant and free of diffusion over long storage periods without adversely affecting the ignitability due to the deterrent coating [2]. According to the literature, layered propellant grain coating can be achieved by dipping propellant into a water emulsion of an energetic plasticizer and polymeric deterrent. Freshly extruded propellant thus becomes impregnated with an energetic plasticizer and polymer [3]. It is also reported that freshly extruded propellant strands are coated on the exterior surface with a deterrent using ethanol as a solvent [4]. Glycidyl azide polymer (GAP), along with a curing catalyst, is also used for gun propellant coating as has been reported by Northrup [5]. Polymer in water slurries have also been reported by Canterberry [6] for propellant coating and lead to reductions of initial burn rate and chamber pressures. Progressive-burning propellant grains, having multiple bases with multi-tubular geometry, have reportedly been prepared by Aaronson *et al.* [7] by heating with a resin during tumbling, to soften the propellant grains and to form a continuous, adherent coating on the grains. The energy lost due to deterrent coating can be compensated for by the addition of suitable energetic materials during processing of the propellant [1].

Stanley *et al.* [9] reported that an energetic deterrent coating should contain at least one slow burning compound on the propellant grain, which would reduce

the burn rate at the grain surface so that progressive burning is realized. It was found that a deterred single base propellant normally shows better ballistic stability at elevated temperatures under extended storage conditions. However, double base propellants exhibit an increase in quickness in the case of an energetic plasticizer due to the diffusion of the deterrent through propellant grains under similar conditions [9]. Diffusion studies of di-butyl phthalate (DBP) in ball powder using autoradiography has been carried out by Brodman *et al.* [10, 11]. It was observed that there is a sudden drop in concentration of deterrent through a region of the ball powder propellant grain. It was also observed that the depth of penetration of the deterrent into the propellant grain increases on increasing the concentration of the deterrent. Bu-NENA has been reported as a deterrent, alone or together with a selected polymer, for TBP [3].

The rate of burning (r) is proportional to the pressure acting on the surface of the propellant [12]. It can be calculated using the exponential form of the burn rate law known as Vieille's law, $r = \beta P^\alpha$, where r is the linear burning rate (cm/s), P is the pressure (MPa) at the surface of the composition at a given instant, β is the burning rate coefficient and α is the burning rate index [13, 14]. The values of β and α (in general r) depends on the loading density, ignition system and temperature of the propellant [12, 15-21]. The rate of change of pressure with respect to time, *i.e. dP/dt*, is obtained from closed vessel (CV) firings results of pressure-time data for TBP samples [16, 17]:

$$dP/dt = (P_2 - P_1)/(t_2 - t_1) \tag{1}$$

where P_1 and P_2 are any two pressure values at two different time intervals t_1 and t_2, respectively, and $(dP/dt)_{max}$ is the maximum value of dP/dt recorded for each firing. The force constant F, (J/g), defined as the energy imparted when 1 g of propellant is burnt, is given by:

$$F = nRT_o = P_m[(1/\Delta) - \eta\,][1 + K/100] \tag{2}$$

where P is the instantaneous pressure (MPa), n is the number of moles of gaseous products per unit mass of propellant, T_o is the adiabatic flame temperature, P_m is the maximum pressure, Δ is the loading density (g/cm^3), η is the co-volume of the propellant gases (cm^3/g), and K is a cooling correction factor applied to the measured P_m due to heat loss inside the CV body. Vivacity is defined by the way in which the propellant behaves with respect to its mass burning rate and is calculated by β/D where D is the web size of the propellant grain [13, 14, 16]. Various techniques have been proposed [15] in order to map the deterrent

profiles in propellants. Propellant grains having the nitrocellulose matrix coated with various concentrations of a deterrent have been studied [2, 15, 25] in order to evolve a method for calculating the depth of deterrent penetration and to explain the concentration profile by a diffusion with interaction mechanism. Some information about the deterrents penetration depth may be obtained by staining and optical methods, but these methods are not capable of directly measuring the concentration profiles. Specially prepared propellants containing radioisotope-labelled deterrent can be detected by autoradiographic and scintillation counting procedures. Other methods are based on gradually removing small layers of the propellant grains by centrifugal sanding or alkaline hydrolysis. However, most of these techniques do not give very reliable quantitative results.

In the present work, Bu-NENA coated TBP [17, 18], which finds application in large caliber guns, has been studied. CV evaluation has been conducted to study the ballistic performance of the deterred propellant. The results obtained will be valuable as limited information is available on Bu-NENA coated propellants. An NC dope containing Rhodamine B pigment marker was used to locate the depth of penetration of Bu-NENA into the propellant surface. Rhodamine B is a well-known staining agent. It imparts a pink colour to the propellant grain. The dye floresces when light from the microscope accessories falls on it and can thus be detected easily under a microscope. It was established as a marker for Bu-NENA by conducting a series of experiments due to its well-known and already established applications in NC dope. It is soluble in ethanol (solubility 1.5 g/100 mL). Its diffusion coefficient is $(3.2 \pm 0.2) \cdot 10^{-10}$ cm^2/s in water at 25 °C [22]. Other dyes are not uniformly dispersed in the NC dope and are found to agglomerate to form lumps that do not give a uniform coating on the propellant grain surface. Coated conventional TBP for gun applications develops pressure in the range 310-350 MPa and imparts kinetic energy to give a muzzle velocity of 800-850 m/s to a projectile having a mass of 42.2-44.5 kg.

2   Materials

The following materials were used:
–   acetone: purity 99.8%, Sigma Aldrich,
–   Bu-NENA: density 1.20-1.22 g/cm^3 at 27 °C, purity >96%, Bharat Explosives, India,
–   Carbamite: purity 99.8%, Director General, Ordnance Factory,
–   dibutyl phthalate: density 1.04 g/cm^3 at 27 °C, purity >99.9%, Aldrich,
–   ethanol: purity >96%, S D Fine Chemicals, India,

- graphite: purity 99.98%, particle size <20 μm, Sigma Aldrich,
- NC: 12.6%N, ether-alcohol solubility >95%, Director General, Ordnance Factory,
- NC dope: viscosity 200-$300 \cdot 10^{-6}$ cm^2/s, pH 5.5-7.5, Prasanna Industries, India,
- NC-NG paste: ratio 54.3:45.7, Bharat Explosives, India,
- NG: 18.3%N, Bharat Explosives, India,
- NQ: purity 98.8% by mass, specific area 19798 cm^2/cm^3, Director General, Ordnance Factory,
- potassium sulphate, K_2SO_4: purity 98.8%, Director General, Ordnance Factory,
- Rhodamine B: purity ≥95%, melting point 210 °C, Sigma Aldrich.

3   Methods

During the present work, TBP having grain length about 22 mm, grain diameter about 10.6 mm and hole diameter about 0.4 mm with rosette-shaped, was processed by the well-known solvent method (acetone/ethanol = 80:20) as listed in the composition given in Table 1 and then coated with energetic material Bu-NENA in ethanol as solvent with the aim of studying its ballistic properties. Different batches of deterrent coated TBP were processed at 1 kg level in a stainless steel blender/mixer (known as sweetie pan) mounted on a shaft that can rotate at a speed of 35 rpm. Different amounts of propellant were immersed in the Bu-NENA (deterrent) solution (in 100 g ethanol as solvent containing a fixed quantity of 8 g NC dope with Rhodamine B as coloring agent). Details of the composition of the deterrent are listed in Table 2 [3, 15].

Table 1.   TBP composition on analysis by HPLC

Sr No.	Ingredients	Composition [%]					
		DS0	DS1	DS2	DS3	DS4	DS5
1	NC^a (12.6%N)	30.24	30.14	30.2	29.24	29.04	28.24
2	NG	25.46	25.16	25.12	24.4	25.4	25.42
3	NQ	38.2	38.1	37.2	37.5	36.2	36.15
4	Carbamite	1.7	1.5	1.7	1.7	1.2	1.3
5	Dibutyl phthalate	2.4	2.2	2.3	2.27	2.31	2.17
6	K_2SO_4	2.0	1.97	1.6	2	1.9	1.8
7	Graphite (part)	0.05	0.05	0.05	0.05	0.05	0.05
8	Bu-NENA	–	0.93	1.88	2.89	3.95	4.92

^{a} NC was determined by difference.

Table 2.   Details of composition of deterrent in 100 g of ethanol

Sr No.	Composition No.	NC dope containing Rhodamine B [g]	Bu-NENA [%]
1	DS0		0
2	DS1		1
3	DS2	8	2
4	DS3		3
5	DS4		4
6	DS5		5

The mixture of propellant grains and deterrent solution in ethanol was tumbled in a sweetie pan for a definite period (about 1 h) until the propellant grains were completely free from each other on evaporation of most of the solvent used to disperse the deterrent. The propellant batches were unloaded from the sweetie pan and air dried for 24 h. The propellant was then oven dried for approx. 50 h at 40 °C or until the volatile matter (VM) content of the propellant had reached about 0.4 wt.%.

The physical measurements of the dried propellant grains were carried out using a Vaiseshika Zoom Stereoscopic Microscope Type 7004 TNC Sr. No. 99075 having a resolution of 7 X to 60 X, to ascertain the depth of deterrent coating into the surface of the propellant. The microscope was connected to a computer. This microscope is manufactured by Vaiseshika Electronic Devices, Ambala Cantt, Haryana, India. In order to measure the penetration of Bu-NENA into the surface of the propellant, the coated grains were cut in transverse section and fixed on a microscope stage with a temporary fixture pointing the cut surface of the grain towards the high power objective lenses of the microscope. An image of the cut surface of the grain was frozen on the computer screen and the depth of penetration of the Bu-NENA was measured (in mm) by moving the crosswire from one point to another point on the frozen image. The movement of the crosswire across the image gives the length or distance between two points in the image.

The Bu-NENA coated propellant batches were subjected to chemical analysis using high performance liquid chromatography (HPLC), model No. Ultimate 3000, manufactured by Dionex Softron GmbH (Germany). The HPLC column had a neutral stationary phase containing C18, octadecasilane, length 250 mm, diameter 4.6 mm and pore size 5 µm. Methanol/water (70:30) mixture was used as the mobile phase. An ether extract of the propellant sample was injected onto the HPLC column. The system was run for 30 min by conditioning at 35 °C at a standard mobile phase flow rate of 1 mL/min and 64 bar (6.4 MPa) pressure in reverse phase. The contents of NC, graphite and K_2SO_4 in the propellant was

calculated by difference. In order to obtain the NC content of the propellant, the residue of the ether extract was subjected to hot water treatment. Hot water dissolves K_2SO_4 that was estimated by well-known methods. Graphite was estimated by adding acetone to dissolve the NC.

The deterred propellant was subjected to CV evaluation at 0.2 g/cm^3 loading density in a 700 cm^3 CV after conditioning at –20, 21, or 55 °C for 24 h to determine ballistic parameters such as P_{max}, $(dP/dt)_{max}$, force constant, $α$, $β$, rise time (in ms), relative force (RF) and relative vivacity (RV) [16, 17, 19-21, 23-25]. In a CV firing, a tourmaline piezo-electric gauge was used for pressure measurement. The propellant charge was ignited by a small quantity (1 g) of gunpowder "puffer" charge. The gunpowder itself was ignited by passing a current (*ca.* 3-5 A) through a short length of fine nichrome wire soldered across the firing pins [16, 17].

4   Results and Discussion

Microscopic examination revealed that the deterrent had penetrated the hepta-tubular rosette-shaped propellant grain surface. As the percentage of deterrent was increased, the extent of penetration in the propellant surface increased. Images of grains taken using the Vaiseshika Zoom Stereoscopic Microscope are shown in Figure 1. The thickness of the coating on the propellant grains using NC dope (8 g) without any deterrent was 0.501 mm. The thickness of the coating increased from 0.632 to 0.907 mm on addition of deterrent in the NC dope, increasing with the amount of deterrent (1-5 wt.%) in solution (Table 3 and Figure 2).
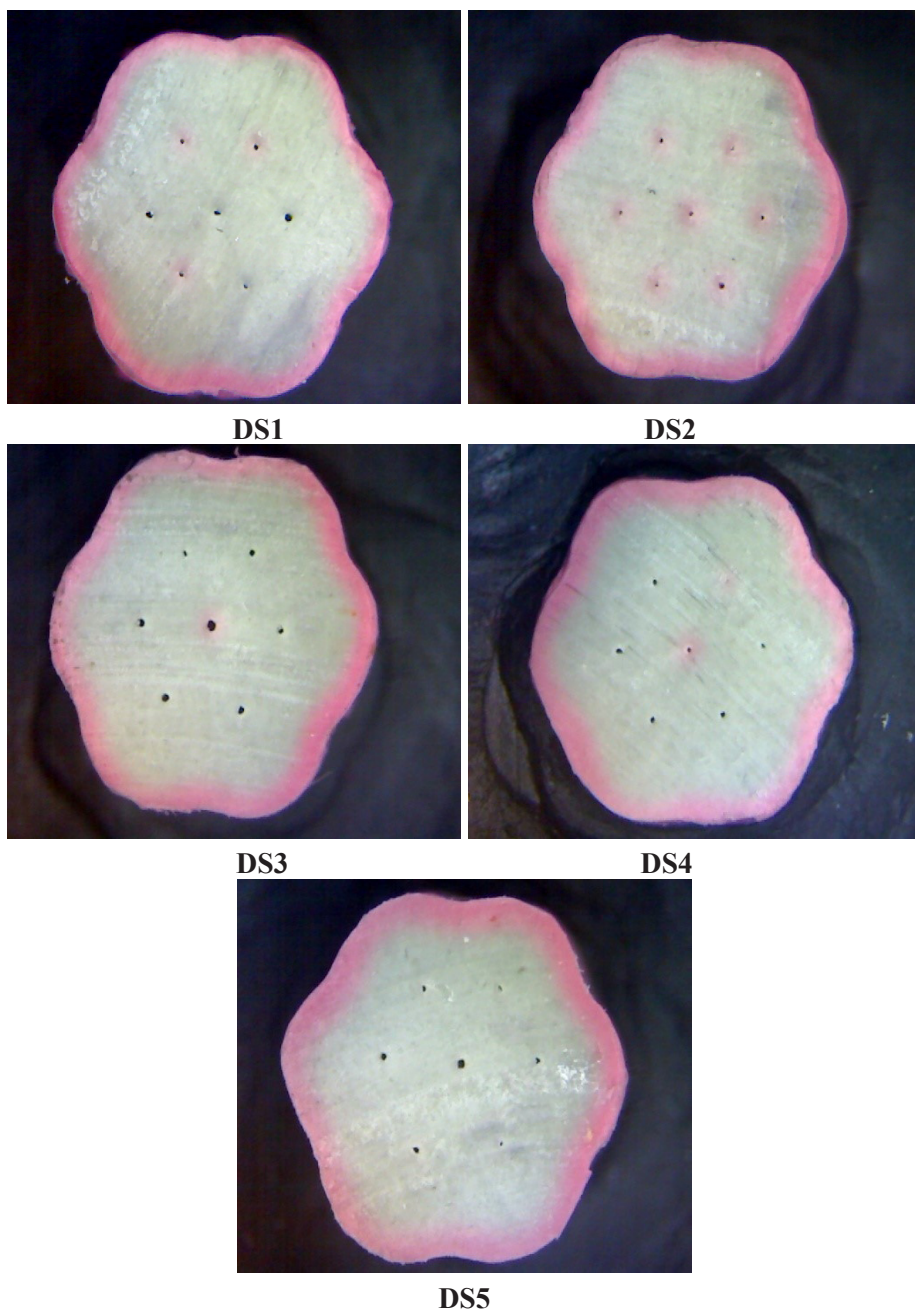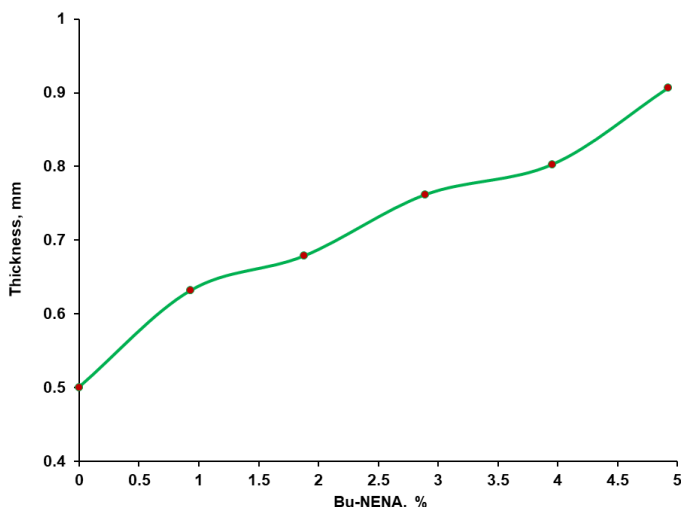

Uncoated TBP   **DS0**

DS1

DS2

DS3

DS4

DS5
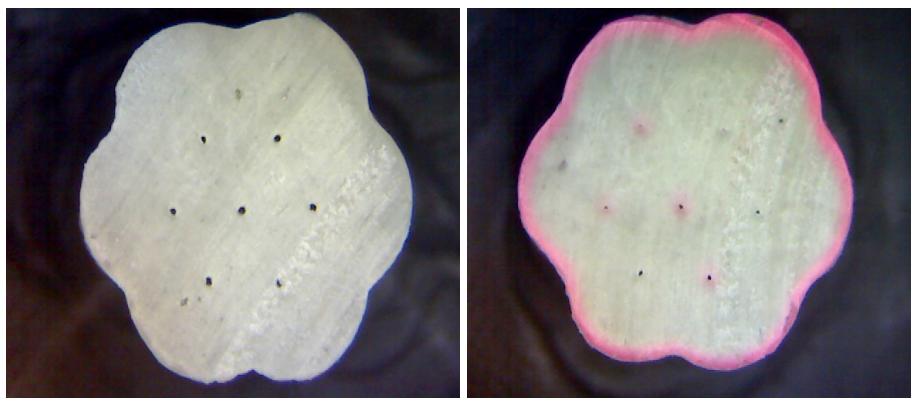
Figure 1.   Vaiseshika Zoom Stereoscopic Microscope Type 7004 TNC images
of deterred TBP

Table 3.   Thickness of deterrent coating

Parameter	Composition					
	DS0	DS1	DS2	DS3	DS4	DS5
Bu-NENA [%]	0	0.93	1.88	2.89	3.95	4.92
Thickness [mm]	0.501	0.632	0.679	0.762	0.803	0.907

Figure 2.   Plot of coating thickness *vs.* content of deterrent Bu-NENA

Rhodamine B in the NC dope solution was used exclusively as a marker for the boundary of the deterrent. The NC dope with Rhodamine B has a penetration of its own into the propellant surface in the absence of Bu-NENA. In the case of DS0 it was 0.501 mm, as shown in Figure 1, whereas it increased from 0.632 to 0.907 mm for a 1 to 5 wt.% increase in Bu-NENA content in the coating solution (Table 3). These results demonstrated that the penetration depth increased for the same quantity of ethanol solution of NC dope with Rhodamine B with increasing Bu-NENA content. Since the quantity of NC dope with Rhodamine B was fixed at 8 g along with the same quantity of ethanol solvent (*i.e.* 100 g), whatever increase in penetration into the propellant surface is observed must be due to the increase in Bu-NENA content. This shows that it is the Bu-NENA that penetrates inside the surface of the propellant grain.

The ballistic parameters observed on CV firing of TBP coated with 1-5% Bu-NENA in the NC ethanol dope, after conditioning at –20, 21 or 55 °C, are listed in Tables 5-7. It was observed that the propellant coated

with Bu-NENA gave lower values of P_{max} and $(dP/dt)_{max}$, whereas the rise time for these compositions was greater than for uncoated propellant. For comparative purposes, data from a CV test of gun propellants are usually displayed in the form of the rate of pressure rise *versus* pressure; $dP/dt = (P_2 - P_1)/(t_2 - t_1)$ is plotted against pressure. The maximum value of dP/dt corresponding to a certain pressure in every firing is denoted by $(dP/dt)_{max}$ [16, 17]. The burning rate coefficient (β) and linear burn rate (β_1) were found to decrease with increasing amounts of deterrent whereas the pressure exponent (α) was found to be almost constant, while at the same time the vivacity was reduced. However, the *RV* for almost all compositions was on the higher side at –20 °C and on the lower side at 55 °C (Table 4 and Figure 3). This may be due to the coated propellant exhibiting brittle fracture, because of which the surface area becomes enlarged at sub-zero firing temperatures and results in an increase in the vivacity; compression of the soft grain at high firing temperatures, thus decreasing the surface area of the holes, leads to a reduction in the vivacity [2, 26, 27].

The DS0 batch was coated with only the NC dope with Rhodamine B. It does not contain Bu-NENA. This coating also gave rise to brittle fracture at sub-zero firing temperatures (–20 °C), because of which the surface area of the propellant grain increased and resulted in an increase in the *RV* of the DS0 batch (> 100) compared to the uncoated propellant.

Table 4.   Relative vivacity of deterred propellant compositions[a]

Composition	Temperature [°C]		
	–20	21	55
DS0	100	98.1	98.3
DS1	83.36	77.24	77.9
DS2	74.67	74.4	76.2
DS3	71.16	67.7	71.6
DS4	68.57	63.36	64.85
DS5	62.47	58.55	62.1

[a] The results are reported with respect to *RV* of the base propellant (uncoated TBP).
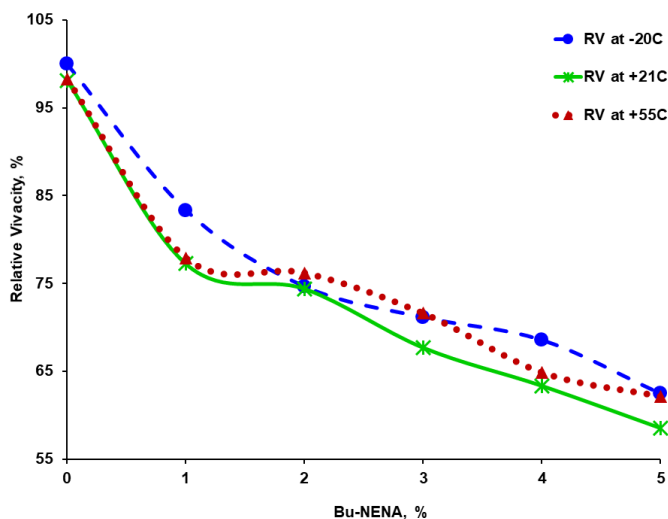
Figure 3.   Plot showing effect of coating of deterrent on *RV* at –20, 21 and 55 °C

The composition DS1, coated with the 1 wt.% Bu-NENA dope, gave an *RV* of 83.4% at –20 °C, 77.2% at 21 °C and 77.9% at 55 °C. The DS1 batch gave the best combination of *RV* and β_1 compared to the other coated compositions evaluated in this work. This indicates that at –20 °C, the gluing effect of the plugs of deterrent and the nitrocellulose matrix is strongly reduced. Consequently the shock wave immediately drives the plugs of deterrent into the perforations of the propellant grain and pulverizes these plugs, because the brittleness, particularly of the plugs, is increased at sub-zero temperatures. As a result more surface is made available for burning, thereby increasing the rate of gas generation at –20 °C. By contrast, at 55 °C the gluing effect of the plugs of deterrent and the nitrocellulose matrix are considerably increased, the coating becoming soft and flexible. Consequently the shock wave experiences difficulty in driving the plugs of deterrent into the perforations of the propellant grain, making comparatively less surface area available for burning, which in turn reduces the rate of gas generation [2, 26, 27]. This behavior shows a trend of coated propellant towards temperature independent characteristics.

Table 5. CV firing results of propellant deterred with Bu-NENA in NC dope, at –20 °C

Parameter	Uncoated TBP	DS0	DS1	DS2	DS3	DS4	DS5
F [J/g]	1010.5	1011	1021.3	998.3	997.4	996.3	987.9
P_{max} [MPa]	243.0	243.0	245.5	240.3	239.9	240.0	237.9
$(dP/dt)_{max}$ [MPa/ms]	22.9	23.0	19.4	17.4	16.3	16.0	14.4
α	0.75	0.79	0.80	0.78	0.74	0.78	0.74
β [cm/s]	0.479	0.388	0.298	0.285	0.347	0.269	0.301
β_1 [cm/(MPa·s)]	0.13	0.133	0.106	0.093	0.087	0.085	0.077
Rise time [ms]	15.3	15.7	19.45	22.15	23.0	24.35	26.25
Vivacity [1/MPa·ms]	5.32E–4	5.33E–4	4.49E–4	4.09E–4	3.90E–4	3.65E–4	3.34E–4

Table 6. CV firing results of propellant deterred with Bu-NENA in NC dope, at 21 °C

Parameter	Uncoated TBP	DS0	DS1	DS2	DS3	DS4	DS5
F [J/g]	1027.4	1024	1011.6	1006.7	1013.3	997.0	993.8
P_{max} [MPa]	246.9	246.0	243.2	242.0	244.1	240.1	239.1
$(dP/dt)_{max}$ [MPa/ms]	25.3	24.2	19.8	19.4	17.1	15.9	14.3
α	0.72	0.77	0.76	0.78	0.73	0.74	0.71
β [cm/s]	0.56	0.449	0.359	0.301	0.366	0.316	0.341
β_1 [cm/(MPa·s)]	0.129	0.132	0.101	0.095	0.087	0.080	0.073
Rise time [ms]	14.60	15.25	19.80	21.55	22.10	24.7	26.35
Vivacity [1/MPa·ms]	5.67E–4	5.58E–4	4.45E–4	4.24E–4	3.84E–4	3.53E–4	3.35E–4

Table 7. CV firing results of propellant deterred with Bu-NENA in NC dope, at 55 °C

Parameter	Uncoated TBP	DS0	DS1	DS2	DS3	DS4	DS5
F [J/g]	1027	1028	1024	1019.2	1018.1	1014.5	1015.1
P_{max} [MPa]	249	247	246.0	245.3	244.8	244.3	244.4
$(dP/dt)_{max}$ [MPa/ms]	27.0	26.8	22.0	21.5	20.0	19.1	17.1
α	0.74	0.77	0.77	0.79	0.76	0.75	0.71
β [cm/s]	0.53	0.456	0.351	0.296	0.319	0.30	0.349
β_1 [cm/(MPa·s)]	0.133	0.133	0.103	0.101	0.09	0.082	0.078
Rise time [ms]	14.60	15.05	19.50	20.50	21.55	24.35	24.7
Vivacity [1/MPa·ms]	5.95E–4	5.83E–4	4.63E–4	4.46E–4	4.16E–4	3.75E–4	3.61E–4

Figures 4(a), 4(b) and 4(c) show the plots of burn rate and pressure at temperatures of –20, 21 and 55 °C, respectively. The burn rate was found to increase with the conditioning temperature for all of the deterred propellant compositions.
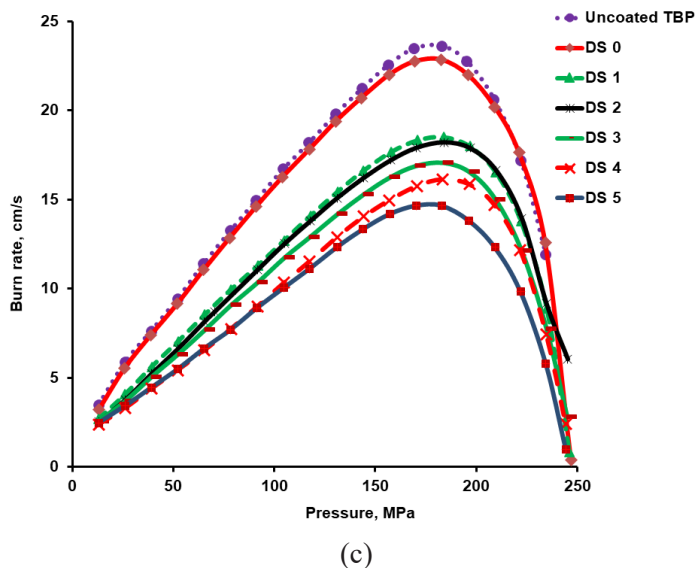

(a)

(b)

(c)
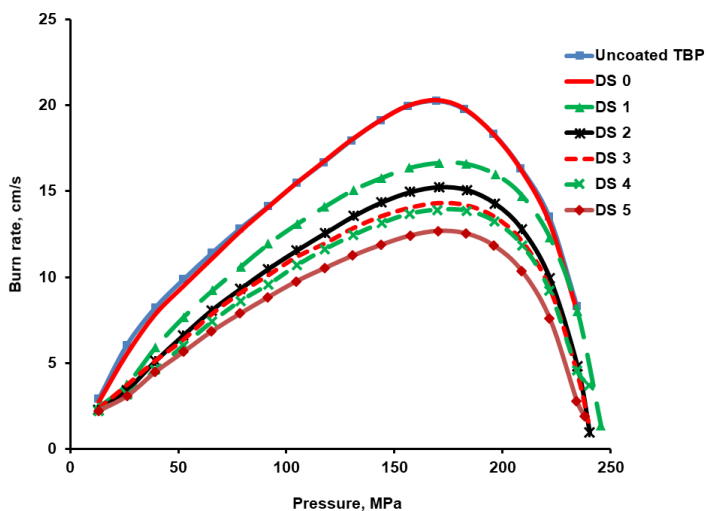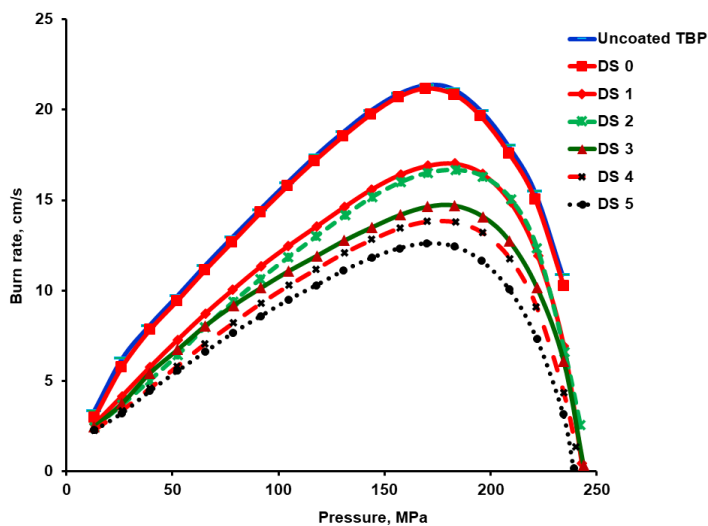
**Figure 4.**   Variation of burn rate with pressure at (a) –20 °C, (b) 21 °C and (c) 55 °C

It may be observed from these figures that burning for all propellant compositions commences at almost the same lower pressure and ends at almost the same higher pressure. This may be because the same quantity (1 g) of gunpowder was taken for ignition of all the propellant batches, however the burn rate differs as time progresses due to the different quantities of deterrent and the maxima of the curve is broadened and shifted towards longer duration times [10]. As a result P_{max}, $(dP/dt)_{max}$ and vivacity are reduced and the rise time is increased. During the burning process of a deterred propellant grain, an increased surface area is available for combustion because of the delayed burning on the surface. However, the inner surface area of the grain increases during combustion as it is less deterred or remains undeterred. This may be attributed to progressive burning of the deterred propellant grains.

This implies that by increasing the coating percentage of Bu-NENA, plasticization on the TBP increases causing the burn rate to be reduced, thereby decreasing the production of gas. It is also observed from Figures 4(a), 4(b) and 4(c) at each temperature that by increasing the deterrent quantity on the propellant grain, the maxima of the burn rate curve is reduced in height. This shows that by deterrent coating of the propellant, the burn rate is reduced to give a lower pressure, which continues acting on the base of the projectile for a longer time to give a higher muzzle velocity [2]. The dynamic vivacity, defined as $[(1/P)(1/P_{max})(dP/dt)]$ is plotted against the normalized pressure (P/P_{max})

of the coated TBP batches, is on the lower side as compared to uncoated TBP [1], as shown in Figures 5(a), 5(b) and 5(c) at –20, 21 and 55 °C, respectively. This may be due to a diminished rate of gas generation as a result of the deterrent coating [8]. However, it was found to increase as the temperature was increased from –20 to +55 °C.
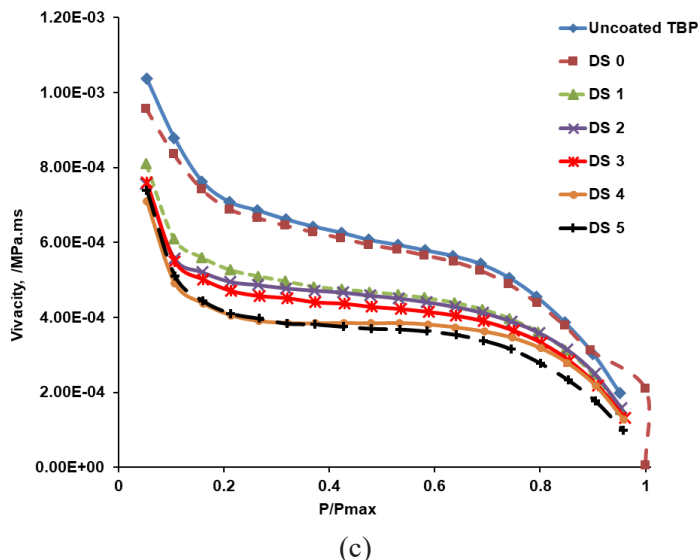

(a)

(b)

(c)
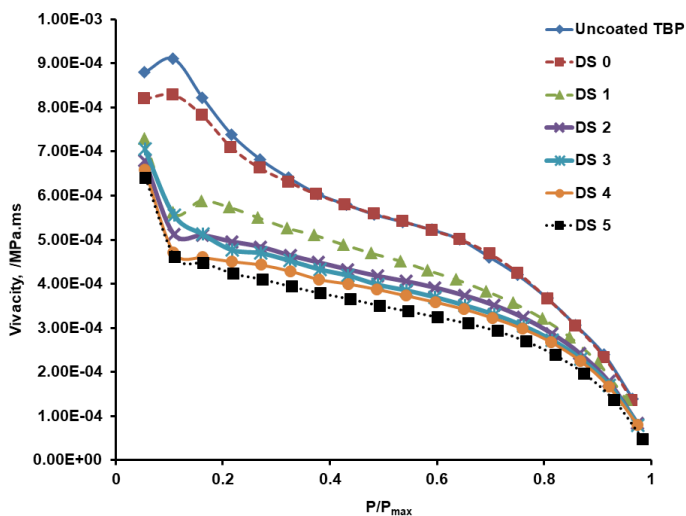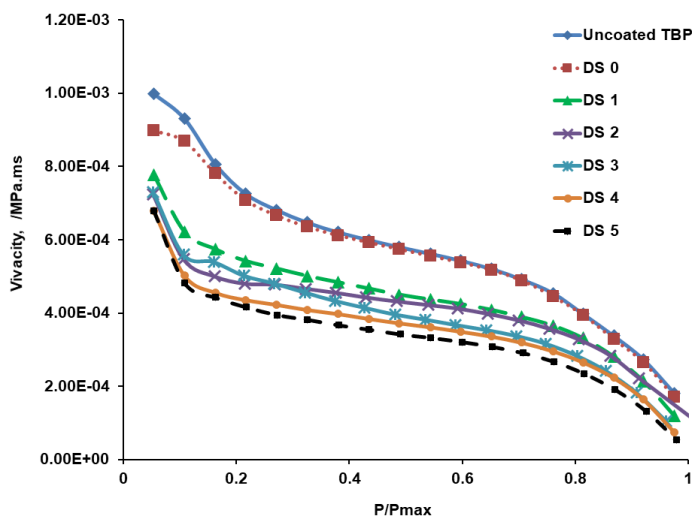
Figure 5. Plot of vivacity *vs.* normalized pressure for different content of deterrent at (a) –20 °C, (b) 21 °C and (c) 55 °C

The observations regarding the ballistic parameters in the case of Bu-NENA deterred TBP are in agreement with the findings reported by Chakraborthy *et al.* [23] and Damse *et al.* [24]; wherein these authors incorporated Bu-NENA at the time of kneading in the propellant composition.

5   Conclusions

The technique of coating TBP reduces the peak pressure (P_{max}), burn rate, vivacity, and differential pressure $(dP/dt)_{max}$, but increases the rise time and pressure index. Coating with a 1% Bu-NENA dope gives the optimum results of *RV*, as 83.4% at –20 °C, 77.2% at 21 °C and 77.9% at 55 °C. This shows that the coating becomes hard with superficial cracking at –20 °C, creating more surface area available for burning and hence the rate of gas generation increases. On the other hand the coating becomes soft and flexible at higher temperature, such as 55 °C, making comparatively less surface area available for combustion and consequently the rate of gas generation is reduced, reducing the peak chamber pressure. This behaviour shows a trend of coated propellant towards temperature independent characteristics.

From this study it may be concluded that deterred burning of TBP is possible by the use of an energetic plasticizer. Such a propellant can be useful for large caliber 155 mm artillery guns. With a higher loading density of deterred propellant, the muzzle velocity can be increased while keeping the chamber pressure within the gun barrel limit. Furthermore, the barrel life can be increased by reducing the barrel erosion because the flame temperature and chamber pressure will be lower compared to uncoated propellant.

Acknowledgement

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