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An Energetic Binder for the Formulation of Advanced Solid Rocket Propellants

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Abstract: Many promising elastomers and energetic binders are reported in the literature for their probable application in propellant formulations. However, their application in actual systems is still restricted due to many inherent limitations. Therefore, an attempt has been made to study the performance of an advanced energetic solid propellant based on an energetic binder comprising a polyol with high hydroxyl functionality plasticized with 1,2,4-butanetriol trinitrate (BTTN). In order to examine the physico-chemical properties of the BTTN/Polvol binder system, the polyol and a series of binders have been formulated and cured with different di-isocyanates viz. TDI, IPDI and H₁₂MDI, and the cured binder sheets were evaluated for their mechanical properties, decomposition temperatures and glass transition temperatures (Tg). The pot life of a representative binder system with various di-isocyanates has also been determined using a gelation timer apparatus. The overall result of this study establishes that the energetic binder system under study has potential applications in the formulation and processing of advanced energetic solid propellant compositions for case bonded rocket motors, offering higher density, higher energy and better low temperature strain capability as compared to conventional HTPB-based composite propellants.

Keywords: BTTN, polyol, low temperature strain capability, glass transition temperature, gel-time

1 Introduction

Solid rocket propellants have come a long way in their development and are being extensively used in almost all operational rockets, missiles and satellite and space shuttle launch vehicles. There are continuous attempts by scientists worldwide to develop more advanced high performance propellants to meet the ever-growing and challenging mission requirements, to carry higher payloads for longer distances. In addition to achieving higher density and higher specific impulse propellants, researchers are also focusing on propellants with new energetic binders, producing reduced smoke levels and combustion products that are less detrimental to the environment. Hydroxyl terminated polybutadiene (HTPB) prepolymer is the most widely used binder system for the processing of composite propellants (CP). However, because HTPB, is an inert binder and also immiscible with energetic nitrate ester plasticizers, the energy of propellant compositions processed with HTPB plasticized with a non-energetic plasticizer like dioctyl adipate (DOA) cannot be further improved upon. Therefore, in the recent past, much of the research work has been oriented towards advanced high performance solid rocket propellants exploring various new energetic binders [1, 2]. Energetic binders like GAP (glycidyl azide polymer), poly-BAMO (poly-3,3-bis-azidomethyloxetane), poly-NIMMO (poly-3-nitratomethyl-3methyloxetane) and poly-GLYN (polyglycidyl nitrate) are being extensively studied worldwide for improvements in the performance of rocket propellants. However, because of the inherent limitations of these materials, together with practical problems like high cost and availability of starting materials in bulk, these binders have received limited applications in actual systems [3-6].

Therefore, an attempt has been made to study the feasibility of an energetic binder comprising a polyol with high hydroxyl functionality plasticized with 1,2,4-butanetriol trinitrate (BTTN) for formulating an advanced high energy solid propellant. Most of the operational solid propellants use ammonium perchlorate (AP) as the oxidizer to the extent of 68-72%. During combustion, an enormous quantity of HCl and other chlorine containing compounds are released into the atmosphere, which is detrimental for the environment. A high energy solid propellant processed with the BTTN/Polyol binder matrix under study, and using nitramines like 1,3,5-trinitro-1,3,5-triazacyclohexane (RDX) and 1,3,5,7-tetranitro-1,3,5,7-tetraazacyclooctane (HMX), could result in an appreciable reduction in the HCl smoke level in the exhaust and thus would be an attractive choice for rocket propulsion systems [7].

The polyol under study was a polycaprolactone based, hydroxyl terminated prepolymer having high hydroxyl functionality, and is completely miscible with

energetic nitrate ester plasticizers like nitroglycerine (NG), 1,2,4-butanetriol trinitrate (BTTN), trimethylolethane trinitrate (TMETN), diethyleneglycol dinitrate (DEGDN) and triethyleneglycol dinitrate (TEGDN), or combinations of these in all proportions. BTTN and TMETN are often used in propellants as a replacement for NG. These nitrate esters have lower densities and energy than NG but offer improved stability and reduced sensitivity. From the density and energy point of view, BTTN has a higher density and energy compared to DEGDN, TEGDN and TMETN [8, 9]. Hence, BTTN has been chosen and used in this study as an energetic plasticizer for the formulation of the energetic binder (Table 1) [10, 11].

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Plasticizer	M.P.	Density	O.B.	ΔH_{f}	$\Delta H_{\text{combustion}}$	$\Delta H_{explosion}$
				[]/8]	[]/8]	[]/8]
NG	2.2 & 13.2	1.59	+3.5	- 1632	6761	6669
BTTN	- 27	1.52	- 16.6	- 1682	9071	6021
TMETN	- 15	1.46	- 34.5	- 1665	11054	4945
DEGDN	-10.9	1.38	- 40.8	- 2226	11707	4565
TEGDN	- 19	1.33	- 66.7	- 2619	14343	3318

Table 1.Physical properties of some nitrate esters

2 Experimental

2.1 Materials

The polyol under study has been synthesized in-house and it is a soft white waxy solid having an hydroxyl functionality of 3.7-3.9, OH value 38-40 mg KOH/g and molecular weight, *Mn*, in the range of 5200-6200 determined by vapor-phase osmometry (VPO). All of the other major ingredients like BTTN, AP, aluminum (Al) and HMX were of Indian origin, whereas the minor ingredients like toluene di-isocyanate (TDI), isophorone di-isocyanate (IPDI), and dicyclohexylmethane-4,4'-di-isocyanate (H₁₂MDI), chain extenders such as butane-1,4-diol (*n*-BD), cross linkers such as trimethylol propane (TMP) and curing catalysts such as ferric acetylacetonate (FeAA), triphenyl bismuth (TPB) and dibutyltin dilaurate (DBTDL), were procured from Sigma-Aldrich and used directly without further purification.

2.2 Characterization Methods

For the preliminary studies, the polyol and BTTN-plasticized polyol only were cured with different di-isocyanates (TDI, IPDI and $H_{12}MDI$), and the cured

polymer sheets and energetic binder sheets were evaluated for their mechanical properties under various temperature conditions, using a Hounsfield (Model H25KS) UK-Universal testing machine at a cross-head speed of 50 mm/min, using test pieces as per ASTM-D638 MII. Decomposition temperatures were measured using a Mettler-Toledo thermo-gravimetric analyzer (TGA); and a Perkin-Elmer differential scanning calorimeter (DSC) and a DuPont, USA make Dynamic Mechanical Analyzer (DMA) were used for the determination of glass transition temperatures (T_g). A Techne, UK gelation timer was used for studying the gel-time of the binder. The propellants were processed in a 10 L capacity planetary mixer, cured at 50 °C and characterized for their physical, mechanical and ballistic properties.

3 Results and Discussion

3.1 Characterization of the cured polyol

Initially, the prepolymer was cured with the di-isocyanates TDI, IPDI and $H_{12}MDI$, in order to evaluate the mechanical properties of the cured polymer sheets. From Table 2, it can be seen that the mechanical properties of the TDI cured polymer at different test temperatures are not attractive, considering the processing of case bonded motors, particularly the elongation and modulus. The mechanical properties of the polymer sheets cured with IPDI and $H_{12}MDI$ are comparable at all temperatures, following a similar trend. However, IPDI does offer more flexibility and a lower T_g compared to both TDI and $H_{12}MDI$. An interesting observation is that in all of the cases the cured binder shows a greater elongation at temperatures lower than ambient conditions, which is different from the usual trend.

Curing agont	Tensile strength [kg/cm ²]			El	longati [%]	on	E-modulus [kg/cm ²]			
(NCO:OH=1)	+27 °C	- 40 °C	+55 °C	+27 °C	- 40 °C	+55 °C	+27 °C	-40 °C	+55 °C	
TDI	108	137	92	22	28	19	576	772	546	
IPDI	91	109	74	212	276	198	226	386	214	
H ₁₂ MDI	87	102	88	232	247	225	256	343	252	

 Table 2.
 Mechanical properties of the cured polymer

The decomposition temperature and T_g of cured polyol and cured BTTNplasticized polyol, *i.e.* energetic binder sheet (EBS), were also determined.

The TGA of cured polymer and cured EBS were recorded in the temperature range 50-500 °C at a heating rate of 10 °C/min in a N₂ atmosphere. The cured polymer showed good thermal stability up to 250 °C and a maximum weight loss of 97.5% was observed at 440 °C (Figure 1). The TGA of the cured EBS samples showed two stages of decomposition: the first stage shows an average weight loss of 60% in the temperature range 150-230 °C, which may be due to the decomposition of BTTN, whilst the second step shows an average weight loss of 30% in the temperature range 300-460 °C, due to decomposition of the polymer (Figure 2). The decomposition patterns of the cured polymer and the cured EBS show similar trends, irrespective of the isocyanate used. T_g of the cured polyol and the cured EBS were determined by DSC and are illustrated in Figures 3 and 4, respectively. IPDI cured samples exhibit the lowest T_g, that is -55.96 °C for the cured polymer and -62.24 °C for the cured EBS. There were no significant changes in the T_g of the cured polymer and the cured EBS when these were cured with TDI or $H_{12}MDI$ (that is $T_g = -53.45$ °C and -54.55 °C respectively for the cured polymer, and -61.60 °C and -61.83 °C respectively for the cured EBS). The use of IPDI also has other advantages during processing, such as lower EoMV (end of mix viscosity) and longer pot life of the propellant slurry, as compared to TDI and H₁₂MDI.



Figure 1. TGA of the cured polymer.



Figure 2. TGA of the cured EBS.



Figure 3. DSC of the cured polymer.



Figure 4. DSC of the cured EBS.

 T_g of cured polyol and cured EBS were also determined by Dynamic Mechanical Analyzer (DMA) to verify the effect of different di-isocyanates on T_g . DMA is a more sensitive technique than DSC for measuring T_g . Extrapolated onset temperature of storage modulus was taken as the T_g . Extrapolated onset temperature of storage modulus is the temperature at which the material's strength begins to decrease such that the material may no longer be able to bear a load without deforming. The comparative data on T_g are presented in Table 3.

Table 3.	T _g of cured po	olyol and	EBS using	different	di-isocyanates
	(NCO:OH=1.0)				

Samples	Formulations	T _g by DSC [°C]	T _g by DMA [°C]
Cured Polyol	Polyol + Adduct [*] + Catalyst [#] + TDI	-53.45	-55.36
	$Polyol + Adduct + Catalyst + H_{12}MDI$	-54.55	-57.29
	Polyol + Adduct + Catalyst + IPDI	-55.96	-58.87
Cured EBS	Energetic Binder [¥] + Adduct + Catalyst + TDI	-61.60	-61.44
	Energetic Binder + Adduct + Catalyst + $H_{12}MDI$	-61.83	-64.31
	Energetic Binder + Adduct + Catalyst + IPDI	-62.24	-68.16

* Adduct : nBD + TMP (2:1 ratio) and a quantity of approx. 0.15% was used in all formulations # Catalyst : FeAA + TPB (1:1 ratio) and a quantity of approx. 0.008% was used in all formulations * Energetic Binder : Polyol (~ 30%) + BTTN (~ 69.5%) + 2-NDPA (~ 0.5%)

Energetic Binder . Folyof (~ 50/6) + B1114 (~ 09.5/6) + 2-MDFA (

3.2 Selection of plasticizer to prepolymer ratio

The selection of the optimum level of plasticizer (Pl) and prepolymer (Po) percentage is important to maintain the right balance between solid loading and ease of processing, as well as achieving the highest possible density, energy level and desired mechanical properties of the propellant. For the selection of the binder composition, a few energetic binder formulations with different Pl/Po ratios were cured with different isocyanates at NCO/OH ratios of 1.0, and were studied for their mechanical properties under different temperature conditions. The results are presented in Table 4. A higher Po content in the energetic binder contributes towards better mechanical properties, whereas a higher BTTN content contributes towards easier processing and higher energy. At a Pl content of 75% or more, the tensile properties of the cured binder are drastically reduced, as found in compositions I and II. The tensile properties of the cured binder increase with an increase in the Po content, as found in compositions IV and V. It was observed that composition III, where Pl:Po=70:30, has the optimum levels of Pl and Po for the formulation of the energetic binder. This trend is also reflected

		0 H ₁₂ MDI	3033	2.21	2.32	2.28	347	424	443		3.16	3.45	2.93		
	Λ	60:4	IDI	929 – (1.39	1.53	1.43	430	618	409		1.19	1.57	1.48	
			IDI	7	1.23	1.35	1.18	274	361	316		2.81	1.68	1.53	
		5	H ₁₂ MDI	3138	2.15	2.26	2.20	487	511	536		2.12	2.27	2.09	
IS I-V	IV	65:3	IDDI	033 -	1.30	1.42	1.32	655	838	586		0.94	1.37	1.42	
ositior			IDI	ε	1.14	1.25	1.08	394	473	419		2.65	1.53	1.47	
s (compo		0	$H_{12}MDI$	3264	2.0	2.14	2.0	725	730	745		1.98	2.15	1.90	
o ratio	III	70:3	IDDI	138 - 3	1.27	0.92	0.75	805	1110	764		0.85	0.96	0.81	
PI:PC			IUT	Э	0.74	1.20	1.00	464	715	682		1.77	1.46	1.39	
different		5	H ₁₂ MDI	3430	0.95	0.76	0.64	314	321	334		1.18	1.31	0.92	
s with	Π	75:2	75:2	IDI	264 – (09.0	0.33	0.28	376	515	337		0.72	0.83	0.67
f EBS			IQT	3	0.35	0.42	0.32	217	232	198		1.11	1.07	0.98	
operties c		0	H ₁₂ MDI	3556	0.38	0.34	0.20	115	119	127		0.74	0.82	0.69	
cal pro	Ι	80:2	IQAI	430 -	0.24	0.15	0.08	130	179	123		0.57	0.72	0.51	
chani			IDI	m	0.14	0.19	0.10	75	86	58		0.73	0.81	0.67	
Table 4. Me	Composition	Pl : Po, [%]	Curator (NCO:OH = 1)	$\Delta H_{explosion}, [J/g]$	TS, [kg/cm ²] at +27 °C	at -40 °C	at +55 °C	E, [%] at +27 °C	at -40 °C	at +55 °C	E-Mod, [kg/cm ²]	at +27 °C	at -40 °C	at +55 °C	

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during the formulation and processing of the propellant composition using this energetic binder. Composition III also reveals that IPDI cured formulations offer better elongation at all temperatures compared to the formulations cured with either TDI or  $H_{12}$ MDI.

## 3.3 Role of NCO/OH ratio

The mechanical properties of the cured energetic binder were determined after curing with three different di-isocyanates and at different NCO/OH ratios. The energetic binder used for the study contained Polyol (30%), BTTN (69.5%) and 2-nitro diphenylamine (2-NDPA, 0.5%). n-BD and TMP were used in the ratio 2:1, in minor quantities, as chain extender and cross-linker, respectively. Minor quantities of the curing catalysts (FeAA + TPB at a 1:1 ratio) were also added to the composition. Although the variation of tensile strength and E-modulus with respect to the NCO/OH ratios were not very conclusive, the percentage elongation was observed to be appreciably higher with IPDI at all temperatures for all of the NCO/OH ratios studied. However, a NCO/OH ratio of 0.9 would be the optimum from the propellant formulation and processing point of view (Table 5).

Table 5. Weenum	icui pi	opertie	5 01 th	e cure						
Curing agent	TDI			IPDI			H ₁₂ MDI			
NCO:OH ratio	0.8	0.9	1.0	0.8	0.9	1.0	0.8	0.9	1.0	
TS, $[kg/cm^2]$										
+27 °C	1.9	1.6	0.74	1.16	1.13	1.27	2.9	2.7	2.0	
- 40 °C	2.3	1.8	1.20	1.23	1.19	0.92	1.5	1.3	2.14	
+55 °C	1.5	1.1	1.00	0.84	0.78	0.75	1.7	1.6	2.0	
E-Mod, [kg/cm ² ]										
+27 °C	4.3	3.6	1.77	0.78	0.67	0.85	2.80	2.60	1.98	
- 40 °C	2.6	2.0	1.46	1.18	1.14	0.96	1.50	1.30	2.15	
+55 °C	2.0	1.5	1.39	0.86	0.79	0.81	1.66	1.56	1.90	
E, [%]										
+27 °C	472	480	464	796	834	805	678	652	725	
- 40 °C	660	653	715	1165	1086	1110	723	743	730	
+55 °C	513	585	682	753	810	764	615	690	745	

**Table 5.**Mechanical properties of the cured EBS

# 3.4 Studies on gel-time

The propellant slurry mix should have an adequate pot life and a reasonably low viscosity to enable smooth casting of the propellant slurry into a rocket motor. The gel-time is a critical parameter determining the pot life of the binder after

the addition of the curing agent. The viscosity of the binder and also that of the propellant slurry mix increase with time due to cross-linking and formation of the urethane network. The point at which the viscosity reaches a level where the binder ceases to flow is considered to be the gel-point or gel-time. The gel-time depends on the nature of the isocyanate, the NCO:OH ratio, the temperature of the slurry mix and the amount of curing catalyst. An instrument known as a gelation timer measures the gel-time of the polymer-isocyanate reaction mixture [12].

The aim of this study was to get a clear understanding of the rate of viscosity built-up with respect to the isocyanates used, keeping the other parameters unchanged, and also for the selection of a specific curing system, *i.e.* isocyanate and curing catalyst. The reaction of isocyanates with hydroxyl groups is very slow in the absence of a catalyst. Organometallic compounds are often used as catalysts to achieve sufficient reaction rates during curing for a variety of polymeric systems, including propellants. The rate of viscosity built-up of a polymeric binder system cross-linked with aliphatic isocyanates is dependent upon the specific catalyst used. Aromatic isocyanates are generally more reactive than aliphatic ones and require the use of catalysts only under certain conditions.

Studies on the gel-time, as well as on the behaviour of the curing catalysts, were carried out at two different temperatures, viz. 50 and 60 °C. The catalysts used for these studies were FeAA, TPB, FeAA+TPB (1:1 ratio) and DBTDL. Three different di-isocyanates viz. TDI, IPDI and H₁₂MDI, were used for the curing of the energetic binder. The quantity of all of the catalysts in all of the compositions was kept at 0.008% and the NCO:OH ratio was maintained at 1.0 during the gel-time studies (Table 6). The reaction of TDI with the binder is faster than IPDI and H₁₂MDI, with the reaction of IPDI being the slowest, *i.e.* IPDI gives a longer pot life irrespective of the type of catalyst used. For all of the di-isocyanates, the curing reaction in the presence of DBTDL is at a higher rate, giving a shorter pot life. For IPDI, all of the catalysts respond towards achieving a longer pot life, however, a combination of FeAA and TPB at a 1:1 ratio works out to be the optimum one. A mixture (0.15%) of n-BD and TMP, at a 2:1 ratio, was used as the chain extender and cross-linker respectively, and improves the mechanical properties. It was also observed that the rate of the curing reaction almost doubles when the curing temperature is raised from 50 to 60 °C.

Composition	Curing Agent (NCO/OH = 1)	Curing catalysts	Gel time (h at 50 °C)	Gel time (h at 60 °C)
		FeAA	6.7	3.5
		TPB	14.0	7.5
	IDI	FeAA + TPB	10.5	6.0
		DBTDL	5.9	3.2
Energetic binder	IPDI	FeAA	52.3	28
+		TPB	78.6	45.2
		FeAA + TPB	65.5	36.5
Catalyst		DBTDL	43.0	25.1
Catalyst		FeAA	38	22
		TPB	45	27.2
		FeAA + TPB	42	25.3
		DBTDL	24.5	15

**Table 6.**Data on gel-time for the EBS

### 3.5 Theoretical performance evaluation

For the selection of a representative energetic propellant composition, the theoretical performances of a series of propellant compositions, based on BTTN/ Polyol binder mix (25%), HMX (0-40%), AP (17-57%) and Al (18%), in respect of their thermo-chemical properties were computed (Table 7). The theoretical calculations show that it is possible to achieve a propellant composition, with the binder system under study, that has a density greater than 1.8 g/cm³, a characteristic velocity (C^{*}) greater than 1600 m/s and a specific impulse (Isp) greater than 250 sec.

Composition	Density [g/cm ³ ]	Flame temp. [K]	C* [m/s]	Isp ₇₀₋₁ [s]
HMX 0, AP 57, Al 18	1.845	3855	1565	263
HMX 10, AP 47, Al 18	1.841	3849	1612	269
HMX 20, AP 37, Al 18	1.837	3823	1622	270
HMX 30, AP 27, Al 18	1.834	3813	1631	271
HMX 40, AP 17, Al 18	1.830	3739	1648	273

 Table 7.
 Theoretical performance evaluation

### 3.6 Evaluation of the propellant compositions

One of the compositions based on the BTTN/Polyol binder loaded with HMX (20%), AP and Al has been processed and the most significant properties derived

after evaluation are given in Table 8. The results confirm the fact that the advanced energetic propellant, processed by using the energetic binder system under study, exhibits higher density, C* and Isp when compared to conventional CPs. An elongation of 95% at -40 °C demonstrates the excellent low temperature strain capability of the energetic propellant under study. RDX and HMX are quite similar in their structure and properties, however HMX has a higher density, yields more energy per unit volume and has a higher melting point. Hence, to achieve a higher performance index, HMX was preferred to RDX during the study [13, 14]. The replacement of AP with HMX in the energetic propellant formulation can reduce the HCl smoke level in the atmosphere to an appreciable extent when compared with a conventional CPs.

Properties	Data obtained
Density, [g/cm ³ ]	1.83
C*, [m/sec]	1610
Isp ₇₀₋₁ , [s]	255
Burn rate, [mm/s @70 kg/cm ² ]	9.8
Pressure index, n _{50-90 kg/cm²}	0.45
TS, $[kg/cm^2]$	5.4
E at -40 °C, [%]	95
E-Mod, $[kg/cm^2]$	3.9
Peel strength, [kgf/cm]	1.25
Tensile bond strength, [kgf/cm ² ]	4.75

**Table 8.**Properties of the energetic propellant

The BTTN-plasticized Polyol binder matrix allows a moderately high percentage (~75%) of solid ingredients, like HMX, AP and Al, to be incorporated in the processing of propellants to achieve a higher density (> 1.8 g/cm³) and a higher specific impulse (> 250 s), along with an excellent low temperature strain capability (> 70% elongation at -40 °C). In spite of the presence of a high percentage of energetic plasticizer as one of the major ingredients, that is, even at high Pl/Po ratio (> 2), the energetic binder under study can be effectively cured with di-isocynates and can be used for the processing of case bonded motors. Moreover, the propellant slurry mix offers lower EoMV: 3000-7000 poise at 30 °C depending on the levels of fine and coarse particulate ingredients) and longer pot life (> 5 h), and hence it is suitable for the processing of case bonded rocket motors.

#### 3.7 Safety and hazard classification

Isocyanates are highly toxic by inhalation and moderately toxic through the skin. They are also powerful irritants to the mucous membranes of the eyes and gastrointestinal tracts. During the present study, the three most commonly known isocyanates, TDI, IPDI and  $H_{12}$ MDI, have been used and their effects on the mechanical properties of the cured prepolymer have been studied. However IPDI was selected for the processing of propellant compositions. The use of isocyanates could not be avoided in this study, as the prepolymer used here for the formulation of an energetic binder is a hydroxyl terminated polycaprolactone based prepolymer, which can only be cured with isocyanates. Hence, due care had to be taken during the processing of the propellant, for example using proper exhaust ventilation in the process room and using personal protective equipment such as impervious clothing, face masks, butyl gloves and safety goggles.

For the safe handling and transportation of CP, it is necessary to ascertain its dangerous properties classification usually into Hazard Divisions HD 1.1 or HD 1.3. Advanced energetic solid propellants, which contain nitramines and nitrate ester plasticizers, are reported to be in class HD 1.1 [15]. Hazard classification of the newly developed energetic propellant composition will be validated in a future endeavour by carrying out the super large scale gap (SLSG) test.

# 4 Conclusions

The energetic binder system under study is suitable for the formulation of advanced energetic solid propellant compositions containing nitramines, metal fuel like aluminum powder and oxidizer ammonium perchlorate, demonstrating excellent low temperature strain capability. The density, specific impulse and characteristic velocity of the propellant composition based on the BTTN/ Polyol binder have been found to be significantly higher than those of HTPB-based composite propellants. Moreover, because of the reduction of AP in the propellant formulation, by approximately 50% when compared with conventional composite propellants, there will be a reduction in corrosive gas formation and the propellant compositions made using the new energetic binder will give reduced smoke levels in the exhaust.

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