

Ballistic Modification of Nitramine Propellants with Special Reference to NG-PE-PCP-Based High Energy Propellants

Shrikant M. Pande,^{*,[a]} Vaibhav S. Sadavarte,^[a] Debdas Bhowmik,^[a] Dashrath D. Gaikwad,^[a] and Haridwar Singh^[a]

Abstract: The burning rate pressure relationship is one of the important criteria in the selection of the propellant for particular applications. The pressure exponent (η) plays a significant role in the internal ballistics of rocket motors. Nitramines are known to produce lower burning rates and higher pressure exponent (η) values. Studies on the burning rate and combustion behavior of advanced high-energy NG/PE-PCP/AP/Al- and NG/PE-PCP/HMX/AP/Al-based solid rocket propellants processed by a conventional slurry cast route were carried out. The objective of present study was to understand the effectiveness of various ballistic modifiers viz. iron oxide, copper chromite, lead/copper oxides,

and lead salts in combination with carbon black as a catalyst on the burning rate and pressure exponent of these high-energy propellants. A 7–9% increase in the burning rates and almost no effect in pressure exponent values of propellant compositions without nitramine were observed. However, in case of nitramine-based propellants as compared to propellant compositions without nitramines, slight increases of the burning rates were observed. By incorporation of ballistic modifiers, the pressure exponents can be lowered. The changes in the calorimetric values of the formulations by addition of the catalysts were also studied.

Keywords: PE-PCP binder · Burning rate · Pressure exponent · Ballistic modifier

1 Introduction

Ballistic modification plays an important role in achieving a desired burning rate in order to meet the mission requirements. Minor quantities of ballistic modifiers (0.1 to 1%) are incorporated in the propellant formulations to achieve a desired burning rate and to lower the pressure exponent over a certain pressure range without altering the ballistic parameters.

The literature [1–4] shows that lead and copper salts are potential candidates as ballistic modifiers. Yount and Musso [5] reported that nitramines do not burn at the surface but rather ignite and burn in the gas stream above the burning surface. Sumi et al. [6] observed that in the gas phase just above the burning surface, the decomposed gases of HMX were found to be less reactive with surrounding gaseous DBP. Cohen et al. [7] reported that at low pressure, the burning rate of the binder is controlled by the exothermic reactions in the condensed phase and at high pressures by reactions in the flame zone. Fifer's review [8] mentioned that catalysts are not significantly effective in nitramine propellants in the lower pressure region and thus a distinct correlation can not be established for combustion of the nitramine propellants. Cohen et al. [9] have found that the HMX combustion is controlled by exothermic vapor phase decomposition and it is difficult to achieve a large increase in the burning rate by catalysis, because the controlling step already takes place very close to the surface.

Reports [10–18] on the burning rate modification of nitramine based propellants show that lead, copper salts and carbon black are useful for lowering the pressure exponent value of the propellant. The authors have explained that the concentration of nitramine, the type of ballistic modifier, the incorporation of fine AP, and the nature of binder have an effect on value of the pressure exponent. Zimmer and Galler [19] studied the deflagration characteristics and surface properties of nitramine-based propellants. Machida [20] reported the effect of a polyester-TDI polymer and lead and copper salts catalyst on the pressure exponent in a CMDB propellant formulation.

McCarty [21] observed a modest increase in the burning rate and a decrease in the pressure exponent at low pressure (less than 10 MPa) for a HMX-cross-linked DBP containing Pb_2O_3 or $\text{Pb}_2\text{O}_3/\text{SnO}_2/\text{C}$ modifiers.

The studies carried out by various scientists; imply that the catalyst reacts at the burning surface and in the succeeding gas phase reaction zone, which is responsible for an increase in the burning rate of the catalyzed energetic

[a] S. M. Pande, V. S. Sadavarte, D. Bhowmik, D. D. Gaikwad, H. Singh
High Energy Materials Research Laboratory
Sutarwadi
Pune-411021, India
*e-mail: smpande@sify.com

binder. It can be concluded that for high-energy nitramine based propellants, among the available modifiers, the most effective ballistic modifiers are the lead salts. However, none of the ballistic modifiers studied produced a plateau effect.

Pentaerythritol polycaprolactone prepolymer (PE-PCP) is a polyester polyol having excellent miscibility with energetic nitrate esters (NE). The NG plasticized PE-PCP can take a moderate percentage of solid loading of AP, Al, and HMX to produce advanced high energy propellants with higher density and I_{sp} [22]. No detailed study has been reported so far in the catalysis/platonization of NG plasticized PE-PCP-based high-energy propellants filled with HMX-AP-Al. Based on the literature survey for nitramine DBP and CMDB propellants and to avoid an increase in the end of mix viscosity (EOMV) by using these ballistic modifiers, particularly lead salts/oxides, which may act as cross-linking catalyst, were used. Limited studies were carried out using one part of ballistic modifiers. In continuation of earlier studies on the burning rate modification of NG-PE-PCP based propellants [23], the present study describes the effect of selected lead salts and oxides as catalysts for these high energy propellants.

2 Experimental Section

2.1 Formulation and Raw Materials

Various propellant compositions, as shown in Table 1, Table 2, and Table 3, were formulated using a hydroxyl-terminated pentaerythritol polycaprolactone prepolymer (PE-PCP) plasticized with nitroglycerine (NG) and 2-NDPA as a stabilizer. The PE-PCP prepolymer ($d = 1.15 \text{ g cm}^{-3}$, $M_n = 5700\text{--}6200$ by VPO and OH value = 38–40 mg KOH per g) was synthesized in-house [22]. Nitroglycerine (N 18.2%), AP (200 μm , 50 μm , and 6.5 μm in the ratio 63.6:22.7:13.6), aluminum metal powder (15 μm), β -HMX (purity > 99%, acidity < 0.019%) from indigenous sources were used. A mixture of trimethylol propane and *n*-butanediol (Make: M/s Acros Organics) was used as adduct in the composition. The ballistic modifiers Fe_2O_3 (2 μm) Cu_2O (63 μm), PbO (30 μm), lead salicylate (75 μm), basic lead stearate (BLS) (37 μm), and carbon black (0.9 μm) were used. All other chemicals are purchased from Sigma-Aldrich Chemicals Pvt. Ltd. and used directly without further purifications.

2.2 Processing

All propellant formulations were processed (batch size 9 kg) using a vertical planetary mixer of 10 L capacity and cast under vacuum by a slurry cast technique [24,25]. The propellant was cured at 50 °C for 7 d in a water jacketed oven.

2.3 Characterization Methods

The strand burning rates of the propellants were determined (as an average of 5 readings; Standard deviation: 0.1–0.25) in the pressure range of 3.5–11.0 MPa by employing an acoustic emission technique [26]. The calorimetric values of the propellant compositions were determined by a Parr adiabatic bomb calorimeter.

3 Results and Discussion

The objective of this presented study is to understand the effects of various ballistic modifiers on the burning rate and pressure exponent of NG-PE-PCP-HMX based high energy propellants. Different series of propellant compositions based on PE-PCP/NG/HMX/AP/Al and burn rate modifiers were formulated

3.1 Burning Rate Catalysis of the PE-PCP-NG-AP-Al-based Composition (70% Solids)

In the first series, propellant compositions with 70% solid loading (AP/Al based) were processed by a slurry cast route (Table 1, Figure 1). On average, the incorporation of 0.5 parts of iron oxide or copper chromite resulted in an increase in the burning rate by 6–12% (Sr No. 2 and 3, Table 1) compared to the control composition. This is similar to CP and AP-CMDB propellants processed using these modifiers. However, there was almost no effect in the pressure exponent values when Fe_2O_3 and copper chromite were used as catalyst.

3.2 Burn Rate Catalysis of the PE-PCP-NG-AP-Al-based Composition (75% Solids)

Subsequently, a propellant formulation with 75% solid loading (AP/Al based) was processed and studied with 0.5

Table 1. Results of burning rate catalysis of the PE-PCP-NG-AP-Al-based composition (70% solids).

Sr No.	Burning rate modifier (parts per 100 parts)	Cal-val/J g ⁻¹	Average burning rate/mm s ⁻¹ at pressures/MPa					Pressure exponent (η) over the pressure range/MPa		
			3.5	5	7	9	11	$\eta_{3.5-7}$	η_{5-9}	η_{7-11}
1	Control ^{a)}	7902	10.8	12.7	14.8	16.2	18.4	0.45	0.42	0.48
2	Control + Fe_2O_3 (0.5)	7885	11.7	13.8	15.7	18.0	19.3	0.43	0.45	0.46
3	Control + CuOCr_2O_3 (0.5)	7889	12.1	14.0	16.4	17.7	19.7	0.44	0.46	0.41

a) Control composition: PE-PCP:7.5 NG:21.5 2-NDPA:0.12 Al:18 AP:52 cured with IPDI.

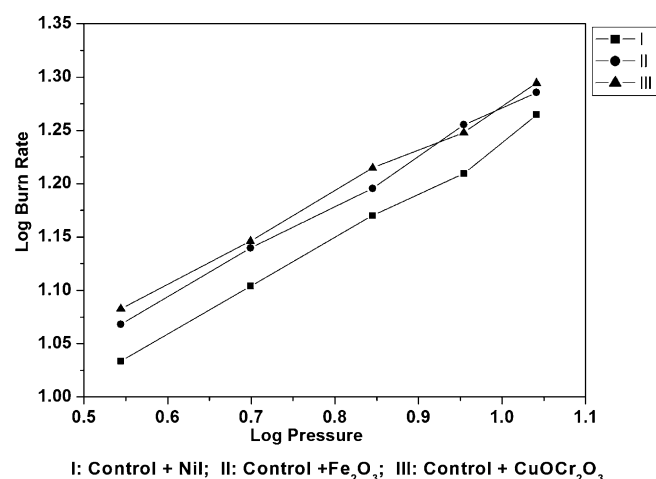


Figure 1. Burning rate behavior of the NG-PE-PCP-AP-Al based propellant (70 % solids) with inorganic oxides.

parts of the oxides. The effect of a higher concentration (1 part per 100 parts) of the catalyst was also studied (Table 2, Figure 2).

Even with 5 % higher AP concentration, the pressure exponent values show a similar trend (no change over different pressure ranges). Thus, it can be seen from the above two examples that for the NG-PE-PCP-AP-Al propellants, the fine particle size of AP and additionally its concentration in the propellant composition play significant roles in determining the “ η ” value, which is around 0.45. Though inclusion of iron, copper, and chromium oxides resulted in an increase in the burning rate of about 8–10 % (Sr. No. 2–5, Table 2), none of the ballistic modifiers studied produced a plateau effect with PE-PCP-based high energy propellants in the overall pressure range. It was observed that the composition with 70 % solid loading has a slightly higher calorimetric value (cal-val) compared to that with 75 % solid. This may be due to the 5 % higher NG content in the former composition.

3.3 Burning Rate Catalysis of the PE-PCP-NG-HMX-AP-Al based Composition (75 % Solids)

The available literature suggests that reactions, which take place at the burning surface of a propellant, play a major

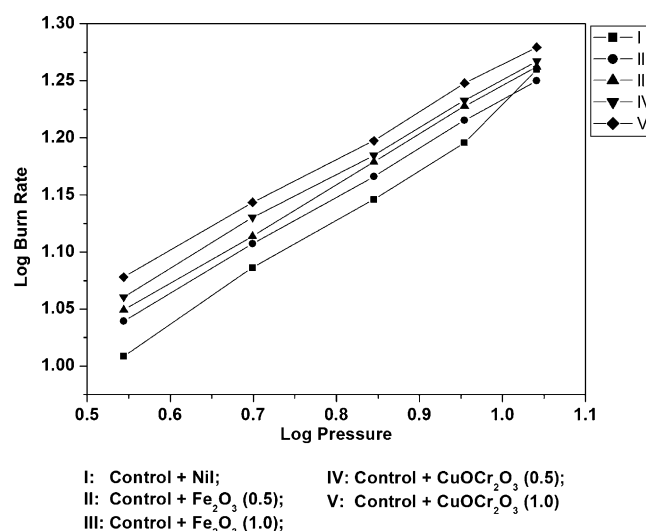


Figure 2. Burning rate behavior of the PE-PCP-NG-AP-Al based propellant (75 % solids) with the inorganic oxides Fe_2O_3 and CuOCr_2O_3 .

role by the release of energy close to the surface that would otherwise only be released further away from the surface. Further, pressure exponents can be brought down by incorporation of catalysts e.g. lead/copper oxides, lead salts in combination with carbon black (Table 3, Figure 3).

The calorimetric value (cal-val) is a measure of the intrinsic energy of the propellant formulation. Upon adding non-energetic additives such as lead/copper oxides, lead salts, the cal-val of the nitramine propellant composition is lowered, which may be due to negative oxygen balance of oxides/salts (Table 3).

The effects of iron oxide and copper chromite as ballistic modifiers were also studied for a propellant formulation with an HMX content of 25 % and 75 % of total solid loading. Incorporation of iron oxide and copper chromite had only small effects on the burning rates of propellants with nitramine concentrations in the order of 25 %. About one part inclusion of these oxides gave almost 3–5 % (Sr. No. 2 and 3, Table 3) increase in the burning rates as compared with the control composition. Compared to iron oxide, copper chromite has a somewhat better effect, particularly in the pressure range 7–11 MPa (0.50 against 0.53) (Sr. No.3,

Table 2. Results of burning rate catalysis of the PE-PCP-NG-AP-Al based composition (75 % solids).

Sr No.	Burning rate modifier (parts per 100 parts)	Cal-val/ J g^{-1}	Average burning rate/ mm s^{-1} at pressures/MPa					Pressure exponent (η) over the pressure range/MPa		
			3.5	5	7	9	11	$\eta_{3.5-7}$	η_{5-9}	η_{7-11}
1	Control ^{a)}	7855	10.2	12.2	14.0	15.7	17.2	0.46	0.43	0.45
2	Control + Fe_2O_3 (0.5)	7843	11.1	13.1	15.0	16.8	18.2	0.43	0.42	0.43
3	Control + Fe_2O_3 (1.0)	7814	11.2	13.0	15.1	16.9	18.3	0.43	0.44	0.43
4	Control + CuOCr_2O_3 (0.5)	7830	11.5	13.5	15.3	17.1	18.5	0.41	0.40	0.42
5	Control + CuOCr_2O_3 (1.0)	7818	11.7	13.6	15.4	17.3	18.6	0.40	0.41	0.41

a) Control composition: PE-PCP:7.5 NG:16.5 2-NDPA:0.25 Al:18 AP:57 cured with IPDI.

Table 3. Results of burning rate catalysis of the PE-PCP-NG-HMX-AP-AI propellants (75% solids).

Sr No.	Additives	Parts per 100 parts	Cal-val/ J g ⁻¹	Burning rate/mm s ⁻¹ at pressures/MPa					Pressure exponent (η) over the pressure range/MPa		
				3.5	5	7	9	11	$\eta_{3.5-7}$	η_{5-9}	η_{7-11}
1	Control ^{a)}	Nil	6814	7.2	8.4	10.2	11.8	13.1	0.52	0.56	0.53
2	Control + Fe ₂ O ₃	1.0	6780	7.5	8.8	10.6	12.2	13.4	0.50	0.52	0.52
3	Control + CuOCr ₂ O ₃	1.0	6764	7.6	8.9	10.8	12.3	13.5	0.50	0.55	0.50
4	Control + CB	0.15	6768	7.1	8.5	10.4	11.6	13.0	0.55	0.53	0.49
5	Control + (PbO + Cu ₂ O + CB) (3 : 3 : 1)	1.0	6646	7.4	8.8	10.4	11.8	13.2	0.49	0.50	0.52
6	Control + (PbO + Cu ₂ O + CB) (3 : 1 : 1)	1.0	6634	7.4	8.7	10.3	11.6	13.0	0.48	0.49	0.51
7	Control + Pb-salicylate	1.0	6676	7.3	8.6	10.1	11.7	12.6	0.47	0.52	0.49
8	Control + Pb-stearate	1.0	6638	7.3	8.6	10.2	11.7	12.7	0.48	0.52	0.49
9	Control + (Pb-Salicylate + CB) (1 : 0.3)	1.3	6688	7.4	8.6	10.2	11.6	13.7	0.46	0.52	0.49
10	Control + (Pb-stearate + CB) (1 : 0.3)	1.3	6622	7.3	8.5	10.1	11.6	12.6	0.47	0.53	0.49

a) Control composition: PE-PCP:7.5 NG:16.5 2-NDPA:0.1 AI:18 HMX:25 AP:32 cured with IPDI.

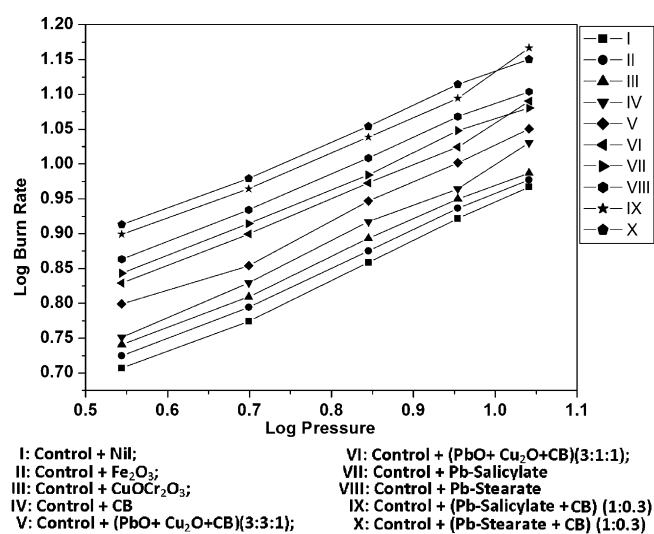
**Figure 3.** Burning rate behavior of the PE-PCP-NG-HMX-AI-AP based composition with various ballistic modifiers.

Table 3). Thus, iron oxide and copper chromite are found to be ineffective for the catalysis of nitramine based PE-PCP propellants.

Incorporation of lead/copper oxide (PbO, Cu₂O) either marginally increased the burn rates or maintained same burn rate and produced marginally lower pressure exponents across all the pressure ranges (Table 3). Carbon black alone had no catalytic effect on HMX filled NG plasticized PE-PCP based matrix. All lead salts and their combination with carbon black increased or maintained burn rates at up to 9 MPa pressures but had decreased the burn rates at 11 MPa pressure resulting in lower pressure exponents over 7–11 MPa pressure range. By and large there was no significant effect on the pressure exponent values. A combination of lead and copper oxides along with carbon black in the 3:1:1 ratio increased burn rates marginally, but brought down the pressure exponent value to 0.48 between 3.5–5 MPa and to 0.49 in the 7–9 MPa pressure regions (Sr. No. 6, Table 3) as compared to control. Similarly among differ-

ent lead salts, Pb-salicylate and carbon black combination marginally offer better option for reduction in pressure exponent.

Of all the oxides and salts studied as ballistic modifiers, the combination PbO/Cu₂O/CB in a ratio of 3:1:1 (Sr. No. 6, Table 3) and the combination Pb-salicylate/CB (Sr. No. 9, Table 3) were found to be slightly more effective in NG plasticized PE-PCP based propellant systems compared to other combinations. The limited effect on the enhancement of the burning rate and lowering of the pressure exponent may also be due to the coarser particle size of the ballistic modifiers.

Kubota et al. [18] proposed that the initial action of the catalyst is in the surface reaction zone. The decomposition process of the propellant is altered to form carbonaceous material on the burning surface. This implied that the gaseous species ejected to the first stage reaction zone changed and the reaction pathway was believed to enhance the reaction rate in the gas phase. The experimental results indicated that by addition of the catalyst the reaction rate in the gas phase is increased more effectively than the reaction rate in the condensed phase.

A model developed by Cohen et al. [9] for active binders supposes a separated degradation for binder and HMX. From experimental observations, it can be concluded that the HMX is completely melted at the surface. In rocket motor conditions, the liquid-gas transition is mainly due to vaporization (95%). Consequently, the heat balance in the condensed phase is endothermic. On the other hand, the chemical reactions of the energetic binder degradation are exothermic. At low pressures, the burning rate of the binder is controlled by the exothermic reactions in the condensed phase and at high pressures by the flame reactions. A mixing between HMX and binder decomposition products significantly decreases the total exothermicity. In the flame zone, there is no chemical interaction between HMX and the binder. Consequently, for low energy binders, the addition of HMX increases the burning rate of the propellant and for high-energy binders, the burning rate decreases.

es. The findings of this study are in agreement with those observations. If additives are present, the lower exothermicity of the condensed phase added to a thinner combustion zone for the propellant, as compared to the binder, induces poor degradation of the additives.

It seems that the ballistic modifiers studied in the present case with a higher concentration of nitramine are not able to significantly change the rate of reaction or energetics (total heat output in the condensed phase), which results in limited or poor catalysis. Some catalytic effects observed with lead salts may be due to their effect on the NG plasticized PE-PCP matrix decomposition and combustion near to the burning surface.

4 Conclusions

From these studies, it can be concluded that for the NG-PE-PCP-HMX-AP-Al propellant containing a moderate level of HMX, the only ballistic modifiers that showed an enhanced performance are the lead salts, whereas Fe_2O_3 and $\text{CuO/Cr}_2\text{O}_3$ are almost ineffective. Incorporation of a Pb/Cu-oxide slightly increased the burning rates and produced lower pressure exponents in the 3.5–7 MPa pressure region. Among the various oxides/salts/combinations studied over the different pressure ranges, a PbO/Cu₂O/CB combination in 3:1:1 ratio and a lead salicylate/CB combination were found to have a better performance in NG-plasticized PE-PCP based propellants.

The addition of a combined catalyst and reduction of the AP particle size significantly improved the combustion characteristics of the propellant in the overall pressure range. The combustion catalyst exerted catalytic effect on the burning rate at relatively low pressure, whereas an increase in the catalyst content did not significantly lead to an increased combustion rate. However, in all these cases with various catalysts evaluated no significant or appreciable effect on the burning rate and pressure exponent values was observed. A combination of Pb-salicylate:CB increased the burning rate at 11 MPa by about 4.6%. Incorporation of CB in addition to other catalysts augmented the catalytic effect.

Symbols and Abbreviations

AP	Ammonium perchlorate
Al	Aluminium powder
Cal-val	Calorimetric value
CB	Carbon black
CMDB	Composite Modified Double Base
DB	Double Base
HMX	1,3,5,7-Tetranitro-1,3,5,7-tetraazacyclooctane
IPDI	Isophorone di-isocyanate
Isp	Specific impulse
2-NDPA	2-Nitrodiphenylamine

NE	Nitrate ester
NG	Nitroglycerine
PE-PCP	Pentaerythritol-polycaprolactone prepolymer
TDI	Toluene diisocyanate
TMP	Trimethylol propane

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