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COMBUSTION AND THERMAL BEHAVIOUR OF BALLISTICALLY MODIFIED EVA AND ESTANE BASED RDX-AP PROPELLANTS RR Sanghavi, SN Asthana, JS Karir and Haridwar Singh

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ABSTRACT

This paper reports the findings of the combustion and thermal studies on RDX-AP based propellants with ethylene vinyl acetate (EVA) and polyurethane-ester-MDI (Estane) binder systems plasticised with triacetin (TA) and glycidyl azide pre-polymer (GAP). Effect of incorporation of selected ballistic modifiers namely iron oxide, copper chromite, basic lead salicylate + cuprous oxide + carbon black combination and sodium borohydride (adsorbed on aluminium oxide) was also studied during this work. Incorporation of GAP as plasticizer in place of TA led to improvement in F by 36-52 J/g. Closed vessel evaluation results were in close agreement with the theoretically predicted performance. Burn rate enhancement by ballistic modifiers was observed in both acoustic burn rate experiments as well as closed vessel test. A major finding was the realization of pressure index value of the order of 0.65 - 0.68 with copper chromite in the high pressure regions (14 - 200 MPa) which is not reported earlier. DTA and DSC studies indicate that RDX decomposition is the major process during propellant degradation and ballistic modifiers catalyse condensed-phase/near-surface reactions involving GAP.

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Thermoplastic elastomers (TPEs) are basically non-homogeneous polymeric systems in which an elastomeric unit has been polymerized with a thermoplastic unit to produce a co-polymer having a central block of elastomer terminated with thermoplastic blocks. The end result of this molecular construction is a material which has the properties of vulcanised rubber and can be processed in a manner similar to thermoplastics¹. Basically, TPEs offer the advantages of desired cohesive strength upto moderately high temperature and flexibility even at sub-zero temperatures. The system may be remelted and reprocessed a number of times providing consequent advantages to the formulator like simplified processing and lower production losses². Owing to these typical characteristics, TPEs have potential of use as binder for rocket/gun propellants and explosives³⁻⁶. Moreover, major advantage of TPEs over chemically cured and crosslinked hydroxy/carboxy terminated polybutadiene binders in current use is the ease in demilitarizing of filled munitions⁶.

During present work, RDX-AP based formulations with TA/GAP plasticized EVA and Estane binder systems were subjected to combustion studies. Effect of inclusion of selected ballistic modifiers was also evaluated. DTA & DSC data on all the systems was generated in the course of this study. An attempt has been made to propose prevailing mechanism for combustion pattern observed on the basis of the thermal and ballistic data generated during this work as well as findings of other researchers.

Propellant formulations evaluated in this study comprised 70 - 60% RDX (~ 5µ), 10-20% AP (~10 µ), 16% TPE binder (EVA/Estane) and 4% plasticizer (TA/GAP). In ballistically modified compositions, iron oxide/copper chromite/basic lead salicylate + cuprous oxide + carbon black combination and sodium borohydride (adsorbed on aluminium oxide) were incorporated at the level of 2 parts (by weight) over and above 100 parts (by weight) of formulation. Solvent technique was adopted to process the formulations⁷. TPEs were solvated by mixing with plasticizer and required quantity of solvent (toluene/acetone) in a sigma blade mixer. RDX was added in installments to the solvated binder matrix. The contents were incorporated for two hours. Subsequently, the mix was dried at 40°C to remove the solvent. Dried crumbs were hot rolled at 60-80°C to obtain extrudable propellant sheets. Computer software (Therm) developed by HEMRL was used to theoretically compute thermochemical parameters⁶. The propellant samples in ribbon form were subjected to combustion in a 100 cc closed bomb at 0.2 g/cc loading density to determine the delivered impetus. Data obtained on closed vessel (CV) firing was acquired using piezoelectric gauge in conjunction with indigenously developed data acquisition system. All the experiments were performed on propellants conditioned at $30 \pm 2^{\circ}$ C. Strand burn rates were determined in the pressure range of 3.4 -10.8 MPa by combusting strand (6 x 6 x 100 mm) in the nitrogen pressurized steel bomb. Acoustic signals generated by the deflagrating sample were unidirectionally transmitted through the water medium and sensed by the piezo-electric transducer of resonance frequency of 200 kHz. Thermal studies were carried out at a heating rate of 10°C/min on indigenously fabricated Differential Thermal Analyser (DTA) in air and Differential Scanning Calorimeter (DSC) of Perkin Elmer Make in N₂ atmosphere.

Results & Discussion :

BALLISTICS:

Closed Vessel Combustion

The theoretical computation of ballistic parameters revealed that the propellants with 70% RDX and 10% AP combination based on TA plasticised EVA binder give F of the order of 1128 J/g with T_f 2763 K and those based on Estane binder give F of the order of 1086 J/g with T_f 2716 K. On increase in AP content to 20% (at the cost of RDX) F increased to the extent of 1136 and 1103 and T_f to 2869 – 2824 respectively. GAP plasticised formulations exhibited higher F (by 36 – 52 J/g) and T_f (by 115-119 K) than corresponding TA plasticized formulations. CV firing experiments gave F values in close agreement with the theoretically computed data (Table 1). The pressure burn-rate relationships obtained for various systems are summarized below and depicted in Fig.1-4.

 $r = 0.113 P^{1.06} r = 0.155 P^{0.95} r = 0.133 P^{0.91} r = 0.162 P^{0.90}$ 70% RDX 10% AP 60% RDX 20% AP
TA GAP

EVA Based Formulations

 $r = 0.092 P^{0.98} r = 0.126 P^{0.90}$ $r = 0.105 P^{0.95} r = 0.137 P^{0.90}$ 70 % RDX 10% AP 60% RDX 20% AP TA GAP

Estane Based Formulations

As the results obtained for composition based on 60% RDX and 20% AP in EVA binder system were superior to those for other compositions studied, in terms of relatively higher burning rate and lower pressure index, it was selected for ballistic modification. The pressure-burn rate relationships of the ballistically modified systems obtained are given below (Fig.5 & 6):

- $r = 0.183 P^{0.89}$ $r = 0.191 P^{0.82}$
 - TA

TA

Fe₂O₃

GAP

- $r = 0.191 P^{0.68}$ $r = 0.212 P^{0.65}$
 - GAP

Cu Chromite

 $r = 0.170 P^{0.82}$ $r = 0.177 P^{0.80}$

TA GAP

BLS + Cu₂O + C-black

- $r = 0.175 P^{0.93}$ $r = 0.187 P^{0.90}$
 - TA

GAP

Sodium Borohydride (on Al₂O₃)

Among the ballistic modifiers, copper chromite appears to be the most effective while BLS + Cu_2O + C-black and sodium borohydride (on Al₂O₃) appear to be the least effective in enhancing the burn rates. Fe₂O₃ gives intermediate results. Copper chromite exhibits the best results with respect to pressure index also as revealed by the value of 0.65 - 0.68 for the composition modified by it compared to 0.8 - 0.93 for BLS + Cu₂O +

C-black, Fe_2O_3 and sodium borohydride on (Al_2O_3) . Such low index values are not reported earlier in the high pressure regions (14 - 200 MPa). As expected, inclusion of ballistic modifier led to marginal reduction in F (Table 2).

Acoustic Burn Rate

TA plasticised EVA binder based formulations with 70 - 60% RDX and 10 - 20% AP exhibited stable combustion in the pressure range studied (8.8 - 10.8 MPa) with the burning rate of $\sim 6 - 8$ mm/s, while corresponding formulations with Estane binder did not exhibit combustion in this pressure range. GAP plasticised Estane binder based systems with 10 - 20% AP revealed stable combustion in the pressure range of 6.8 - 10.8 MPa like EVA based formulation containing 10% AP albeit with relatively lower burning rates (5 - 7 mm/s) than latter (8 - 11.5 mm/s). In case of GAP plasticised EVA based formulation with 20% AP, pressure limit of stable combustion was extended to the level of 3.4 MPa (Table 3). These results further confirm overall superior burning characteristics of EVA binder based formulation with 60% RDX and 20% AP. The pressure-burn rate relationships bring out lower pressure index value of GAP plasticised formulations (Fig. 7 & 8) in the range of 3.4 to 10.8 MPa as observed during closed vessel firings (in the pressure range of 14 to 200 MPa).

Incorporation of ballistic modifiers namely copper chromite, BLS + Cu_2O + C-black and sodium borohydride (on Al_2O_3) in TA plasticized formulations led to marginal increase in burning rates (4 – 6%). Copper chromite was relatively more effective (8-10%). In case of GAP plasticised formulation also burning rate enhancement was marginal (2-6%) with BLS + Cu_2O + C-black and sodium borohydride (on Al₂O₃) while addition of copper chromite resulted in burning rate increase to the order of 30 – 60% in the lower pressure region (3.4 – 4.9 MPa). Fe₂O₃ gave intermediate results (Table 4). As in close vessel test conducted in high pressure region, copper chromite gave the best results in terms of pressure-index (0.32 – 0.45) during acoustic burning tests in the pressure range of 3.4 – 10.8 MPa (Fig.9 & 10).

DSC

As reported earlier by the authors⁹, RDX-EVA and Estane based formulations with TA as plasticizer give endotherm at 196-200°C immediately followed by an exotherm at 241-244°C with Δ H of 1753-1757 J/g. Incorporation of 10-20% AP in TA plasticized EVA and Estane formulations brought down Tm of exotherm by about 10-30 °C with increase in Δ H by ~500 - 900 J/g. Replacement of TA by GAP resulted in increase in Tm by 20°C and decrease in Δ H by 400 - 800 J/g in case of EVA based formulations while it did not have any effect on Estane based formulations (Table 5 & Fig.11-22). Inclusion of Fe₂O₃, Cu chromite, BLS + Cu₂O + C-black, sodium borohydride (on Al₂O₃) as ballistic modifiers in the selected TA plasticizer based formulations (with 60% RDX and 20% AP) led to increase in Tm and decrease in Δ H of exotherm, while in case of GAP plasticised formulation decrease in Tm of exotherm without much effect on Δ H was observed (Table 6, Fig.23-32).

In order to generate additional information, ballistically modified propellants were subjected to TG (Table 7 and Fig.33-48). The TA plasticised formulation with 60% RDX and 20% AP gave weight loss of 90% in temperature range 180 - 210°C. Subsequent 10% weight loss was observed in the temperature range of 210 - 600°C. Ballistic modifiers based formulations also revealed more or less similar weight loss pattern. In case of GAP plasticized formulations 84% weight loss was observed in the temperature range of 180 - 240°C and subsequently 16% weight loss occurred in the temperature range of 240 - 600°C. Fe₂O₃ based formulations exhibited 85% weight loss in the temperature range 180 - 230°C. NaBH₄ (on Al₂O₃) and BLS + Cu₂O + C-black modified formulation exhibited 85% weight loss in the temperature range 180 - 210°C. Copper chromite based formulations revealed relatively higher weight loss (90%) in the same temperature region.

Mechanistic Approach

Authors observed during recent work⁹ that, EVA and Estane based RDX formulations exhibit an endotherm at about 205°C and an exotherm at 225°C. These temperatures are close to melting and decomposition temperatures of RDX respectively, suggesting that RDX plays a major role in the decomposition process as reported by various researchers¹⁰. AP based TA/GAP plasticized formulations exhibited a drop in Tm and an increase in heat release suggesting that AP catalyses condensed phase reactions. These results are in line with those reported by other researchers¹¹ and are attributed to the intensified oxidative reactions on/near surface due to positive oxygen balance of AP.

As regards ballistically modified TA plasticized compositions, heat evolved in DSC was less and Tm was relatively higher compared to those for corresponding unmodified compositions. Much lower ΔH for Fe₂O₃ based formulation may be an

outcome of the need to heat it upto ~1000°C to form the proposed active species CC ~ Fe₂O₂^{12.13}. Although BLS and Cu2O also need to be heated to 700 - 800°C to obtain effective catalytic species¹⁴, it is widely reported that carbon black functions as flame holder and alongwith lead salt catalyzes interaction between NO/NO2 and carbonaceous species near the surface leading to pronounced heat feed back to the condensed phase¹⁵. Copper chromite is proposed to undergo exothermic redox reactions at the propellant surface¹⁶. Boron hydrides act as fuel producing boron and hydrogen which readily undergo exothermic oxidation. Boron is also reported to undergo nitridation¹⁷. It may be suggested on the basis of the observations that the incorporation of ballistic modifiers did not influence the AH in DSC and weight loss pattern in TG despite burn rate enhancement that the site of catalytic action of ballistic modifiers is mainly gas phase as generally reported for AP based compositions¹⁸. Decrease in Tm in case of GAP plasticised formulations (unlike for TA plastisized compositions) on incorporation of ballistic modifiers suggests that these modifiers catalyse interaction between decomposition products of GAP and oxidizer decomposition species in the condensed phase as suggested by Oyumi etal¹⁹ in case of AP - 3,3'Bis Azido Methyl Oxetane (BAMO) / Nitrato Methyl 3 - Methyl Oxetane (NMMO) propellant. Weight loss patterns observed in TG further support these reasoning.

CONCLUSION:

Inclusion of GAP results in an increase in energetics as well as burn rates and reduction in pressure index. EVA based formulations exhibit superior combustion behaviour. Inclusion of ballistic modifiers leads to further improvement in ballistics in terms of enhanced burn rates. The best results are obtained with copper chromite. It was found effective not only in lower pressure region i.e. 3.4 - 10.8 MPa but also in higher pressure region i.e. 14 - 200 MPa. Thermal and ballistic data bring out that the site of action of ballistic modifiers is basically near-surface gas phase reactions, however they appear to catalyse the condensed phase decomposition reactions involving GAP. This study establishes that TPEs have potential for application as binder for not only futuristic gun propellants but also for rocket propellants (Table 8).

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TABLE-1: THEORETICAL AND CLOSED VESSEL RESULTS OF RDX-AP PROPELLANTS

		Esta	ne			EV	A	
	L	A	Ö	AP	TA		GAP	
PARAMETERS	10% AP	20% AP	10% AP	20% AP	10% AP	20% AP	10% AP	20% AP
Theoretical								
Flame Temp, K	2716	2824	2835	2942	2763	2869	2880	2984
Force Constant, J/g	1086	1103	1138	1139	1128	1136	1173	1181
Pmax, MPa	218	220	227	220	226	228	235	237
Mol.Wt (products), g/mol	20.8	21.4	20.8	21.5	20.3	20.9	20.4	21.0
CV Firing								
Force Constant, J/g	1072	1087	1118	1122	1114	1123	1160	1169
Pmax, MPa	245	260	257	264	228	257	247	274
ß, cm/s/MPa	0.092	0.126	0.105	0.137	0.133	0.155	0.133	0.162
۵	0.98	0.90	0.95	0.9	1.06	0.95	16.0	0.00
n, mole/g	0.0481	0.0466	0.04800	0.0465	0.0491	0.0477	0.0490	0.0476

SULTS OF BALLISTICALLY MODIFIED 60% RDX, 20% AP PROPELLANTS WITH EVA	BINDER
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RESULTS O	
ESSEL	
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ABLE-2:	

					E	A				
			TA					GAP		
	Nil	Fe ₂ O ₃	BLS +	NaBH4 on	Ū	Nii	Fe ₂ O ₃	BLS +	NaBH4	IJ
-	BM		Cu ₂ 0.+	Al ₂ O ₃	Chromite	BM		$Cu_2O + Cu_2O + Cu_2$	on Al ₂ O3	Chromite
r'arameter			C-black					C-DIACK		
Force constant, J/g	1123	1102	6601	1092	6011	1169	1110	1106	6601	1115
Pmax, MPa	257	250	249	246	252	274	259	253	251	263
β, cm/s/MPa	0.155	0.183	0.170	0.175	0.191	0.162	0.191	0.177	0.187	0.212
Ø	0.95	0.89	0.82	0.93	0.68	0.90	0.82	0.80	0.90	0.65

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TABLE-3 : STRAND BURN RATES (ACOUSTIC) OF RDX-AP PROPELLANTS

		Es	lane			EV	A	
	L	A	² 0	P.	T	A	07	Р
	10% AP	20% AP	10% AP	20% AP	10% AP	20% AP	10% AP	20% AP
Pressure, MPa				Burn rate,	mm/s			
3.4	-		-	ſ	,	ı	'	5.2
4.9	۰ -	,	ı	r	,	,	,	7.2
6.8	,	'	4.8	5.4	I	I	8	8.4
8.8	,	,	5.7	6.3	6.0	6.3	10.8	11.8
10.8	ı	,	6.6	7.2	7.5	7.9	11.4	12.3
Pressure exponent, n	,	•	0.69	0.62	11.1	-	0.78	0.76
RDX Based Compositic	on did not und	ergo combustio	on in the acous	tic strand burn	÷			

TABLE-4 : STRAND BURN RATES (ACOUSTIC) OF BALLISTICALLY MODIFIED AP/RDX/EVA PROPELLANTS

Burn rate,					EV	A'				
mm/s at			TA					GAP		
pressure, MPa	20% AP	Fe ₂ O ₃	BLS + Cu ₂ O + C-black	NaBH, on Al ₂ O ₃	Cu Chromite	20% AP	Fe ₁ O ₃	BLS + Cu ₂ O + C-black	NaBH4 on Al ₂ O ₃	Cu Chromite
3.4		,	5.4	5.4	5.6	5.2	6.0	5.5	5.4	8.1
4.9	,		5.9	5.8	6.1	7.2	7.7	7.4	7.5	9.0
6.8	•	6.2	6.3	6.3	6.5	8.4	8.9	8.7	8.7	9.8
80.	6.4	6.5	6.6	6.5	6.8	11.8	12.2	12	12.1	12.7
10.8	7.9	8.2	8.3	8.3	8.5	12.3	12.8	12.5	12.6	13.3
Pressure exponent, n	1.0	0.60	0.35	0.32	0.32	0.77	0.68	0.73	0.75	0.45

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		20%	AP	195	68	234	2450
	GAP	10%	AP	961	93	234	2432
2		N:I	AP	202	87	240	1648
Estar		20%	AP	961	57	229	2483
	TA	10%	AP	197	116	231	2416
		lin	AP	203	6	241	1753
		20%	AP	196	58	224	1837
	GAP	10%	٩P	661	89	226	1830
/A		Nil	AP	206	173	239	1634
EV		20%	AP	201	189	208	2667
	TA	10%	AP.	200	257	210	2247
		Nil	AP	206	110	244	1757
Parameters				Endotherm, °C	ΔH, H/g	Exotherm, °C	ΔH, J/g

TABLE-6: DSC OF BALLISTIC MODIFIED AP/RDX/EVA PROPELLANTS

					E	'A				
			TA					GAP		
_	0/M	Fe ₂ O ₃	BLS +	NaBH.	ĩ	0/M	Fc ₂ O ₃	BLS +	NaBH,	Ū
	Ballistic		Cu ₂ 0 +	on Al ₂ O ₃	Chromite	Ballistic		Cu20 +	on Al ₂ O ₃	Chromite
	Modifier		C-black			Modifier		C-black		
Endo, °C	861	200	201	200	200	197	202	199	201	200
ΔH, J/g	57	100	443	189	250	68	235	112	68	107
Exo, °	208	216	214	223	212	224	216	218	217	213
ΔH, J/g	2667	1580	2474	2361	2027	1837	1860	1844	1863	1839

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GA OF BALLISTICALLY MC	
TGA OF BALLISTICALLY MO	
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7 : TGA OF BALLISTICALLY MG	
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LE-7 : TGA OF BALLISTICALLY MO	
BLE-7 : TGA OF BALLISTICALLY MO	
BLE-7 : TGA OF BALLISTICALLY MC	
ABLE-7 : TGA OF BALLISTICALLY MO	
TABLE-7 : TGA OF BALLISTICALLY MO	

EVA	GAP	n Cu Chromite Nil BM Fe ₂ O ₃ BLS + Cu ₂ O + NaBH ₄ on Cu Chromite C-black Al ₂ O	L Temp. WL Temp. WL Temp. WL Temp. WL Temp. WL Temp. WL	<u>30 190-210 90 180-240 84 180-230 85 180-210 85 180-210 85 180-210 90</u>	0 210-600 10 240-600 16 230-600 .15 210-600 15 210-600 15 210-600 10
		Fe ₂ 0,	Temp. WL	180-230 85	230-600 .15
SVA		Nii BM	Temp. WL °C %	180-240 84	240-600 16
E		Cu Chromite	Temp. WL	190-210 90	210-600 10
		NaBH4 on Al ₂ O,	Temp. WL •ر %	180-210 90	210-600 10
	ΤA	BLS + Cu ₂ O + C-black	Temp. WL °۲	190-230 90	230-600 10
		Fe ₂ O ₃	Temp. WL •ر	180-210 90	210-600 10
		Nil BM	ر المسلم ML	80-210 90	01 009-013

WL – Weight loss BM – Ballistic modifier

TABLE 8 : PROMISING ROCKET PROPELLANT COMPOSITION

Composition	Isp. S	Force constant (F). J/g
65% AP + 20% AI + 12% EVA + 3% TA	261	1160
65% AP + 20% AI + 12% EVA + 3% GAP	263	1175
65% AP + 20% AI + 12% Estane + 3% TA	258	1128
65% AP + 20% AI + 12% Estane + 3% GAP	260	1144
65% RDX + 20% Al + 12% EVA + 3% TA	254	1057
65% RDX + 20% Al + 12% EVA + 3% GAP	256	1067
65% RDX + 20% AI + 12% Estane + 3% TA	252	1033
65% RDX + 20% AI + 12% Estane + 3% GAP	253	1044























DSC PROFILES OF RDX/AP/EVA-GAP BASED PROPELLANTS



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DSC PROFILES OF RDX/AP/ESTANE-GAP BASED PROPELLANTS



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TGA PROFILES OF RDX/AP/EVA-GAP BASED PROPELLANTS



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TGA PROFILES OF RDX/AP/ESTANE - GAP BASED PROPELLANTS



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TGA PROFILES OF BALLISTICALLY MODIFIED AP/RDX/EVA-GAP BASED PROPELLANTS