Effect of Ammonium Dinitramide (ADN) on the Characteristics of Hydroxyl Terminated Polybutadiene (HTPB) Based Composite Solid Propellant

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***Abstract*-Ammonium dinitramide (ADN), $NH_4N(NO_2)_2$, can be used in the smokeless composite propellants by replacing AP. It is being considered as one of the most potential energetic oxidizers for propellants. The physical chemistry characteristics of ADN were investigated. The energetic performances, burning characteristics at various pressure ranges, mechanical sensitivity and SEM surfaces observation of HTPB-based propellant with ADN were determined and detected in details. The calculated results showed that the specific impulse and the adiabatic flame temperature are increasing with an increase in the content of ADN. The burning rate and pressure exponent of propellant with a change of pat content of ADN can be boosted higher than those of the AP formulations. The surface structures of composite propellants were little affected by the content of ADN.**

Keywords- Composite Propellants; ADN; Thermal Decomposition; Energetic Property; Mechanical Sensitivity; Combustion Characteristics

I. INTRODUCTION

The development of environmental friendly propellants with low signature characteristics and high energetic properties has been of great interests for researchers[1-4]. Compared with nitramines, such as cyclotrimethylenetrinitramine (RDX) and cyclotetramethylenetetranitroamine (HMX), ammonium dinitramide (ADN), a new kind of high energy density material, has been widely studied and used in the field of solid propellants and high explosives[5-8]. A large number of papers have been published on the combustion mechanism of pure ADN, e.g.[9-14]. Most of these papers have been based on experiments on ADN batches from different sources. Important physicochemical properties and combustion characteristics of ADN and ADN-based propellants were published for the first time in Ref.[15]. It was experimentally found that an advantage of ADN over ammonium perchlorate (AP) (the common oxidizers of HTPB composite propellants) is its cleanness of the production, in addition to its higher energy, meaning that the energetic properties of propellants can be increased by ADN, because ADN has a higher heat of formation than AP[16-20]. Also, it was found that the burning rate of ADN is controlled by reactions in the condensed phase and a multizone flame structure has been established. At present, however, there are only a few papers that are devoted to the study of the combustion characteristics and combustion mechanism of composite ADN-based propellants with different types of binders[21-26]. Though the development of ADN based solid propellants has faced a number of challenges, the research has continued motivated by the high potential. Throughout the years, many technical problems have been solved, such as synthesis, stability, compatibility etc, and there are some reports on the synthesis, spectroscopy, combustion characteristics, and thermal behavior of ADN[27-31]. Whereas, one of the most critical problems that have hampered further development has been the production of ADN particles with acceptable morphology and its application in the solid propellant. There is little report in literature about the mechanical sensitivity and combustion properties on the HTPB-based composite propellants with ADN. Also, it is thus very important to know the properties of ADN based composite propellant compared with AP based one. This is especially important for studies of its energy, sensitivity, thermal decomposition compatibility. In this work, coated ADN particles with energetic binder were used in the hydroxyl terminated polybutadiene (HTPB) based composite propellant by replacing AP, and the energetic performances, combustion characteristics and mechanical sensitivity of the propellants with ADN were studied in details.

II. PAGE LAYOUT

A. Materials and Specimen

ADN can be synthesized by different methods and an overview of these has been described by Venkatachalam, et al[3]. The samples here were prepared from Xi'an Modern Chemistry Research Institute. Hydroxy terminated polybutadiene (HTPB, purchased from LiMing Chemical Engineer Research Institute, China) cured with 2,4-toluene diisocyanate (TDI, purchased from ZhengZhou, China) and aluminum powder (Al, ≥99.8%) were used as components of composite propellant. Two types of ammonium perchlorate (purchased from Dalian Potassium Chlorate Factory, China) were utilized in propellant formulation. The first consisted of pure research grade (> 99 % pure) ammonium perchlorate with an average particle size of 0.105-0.147 mm. The second type of ammonium perchlorate was made by grinding ammonium perchlorate (> 99 % pure) in a fluid energy mill to an average particle size of around 1-5 μm. The average diameters of prepared ADN prills, given by granularity apparatus, are 0.147 μm to 0.205 μm.

All the samples involved in this investigation, which were prepared by mould process at the temperature of 35^0C and then solidified for 96 h (70^0C), were machined to fixed dimension (shape: length: 100-150 mm; width: 2-5 mm, highness: 2-5 mm).

B. Preparation of Composite Propellant

The sample used in the experiments is HTPB-based composite propellant with and without ADN, which basic composition in mass fraction show in Table 1. Propellant batches were run in a 500 g scale in an vertical mixing system HKV 2 at 50^0C and 20 rot·min^{-1}, average mixing time 2-3 h, and cured at 50^0C in the oven for 5 d.

TABLE I THE MAIN INGREDIENT CONTENTS OF COMPOSITE PROPELLANTS

Samples	HTPB	Al	AP	ADN*	Additives
HP-1	13	18	64	0	5
HP-2	13	18	54	10	5
HP-3	13	18	49	15	5
HP-4	13	18	44	20	5

Note: * particles are coated ADN with polymer binder.

C. Apparatus and Measurement Methods

1) Physical Chemistry Characteristics of ADN:

Particle size and size distribution of samples were measured by Master Sizer Instrument. The morphologies of ADN particles were examined by a scanning electron microscope (SEM) technology.

2) Theoretical Calculation:

The theoretical performance of HTPB-based composite propellants with and without ADN were calculated with the minimum free energetic method at the combustion pressure 7.0 MPa, exit pressure 0.1 MPa, and the initial temperature 298 K.

3) Burning Rate Test Method:

A metal fine wire (0.1 mm in diameter) is threaded through the top of the strand with a alternating voltage of 100 V to ignite the propellant strands (diam. = 5-6 mm, length = 140 mm) at the initial temperature of 20^0C. The samples were placed vertically on the combustion rack and sealed in a chamber which was filled with nitrogen atmosphere (Fig. 1).

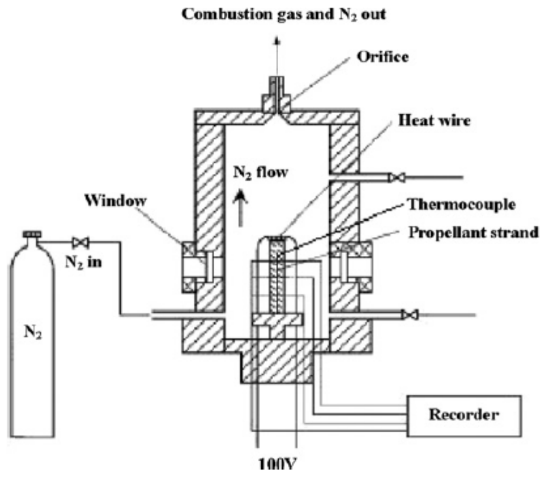


Fig. 1 The apparatus of chimney-type strand burner with observation windows[32]

When a propellant strand is ignited under the nitrogen gas purge conditions, the pressure in the strand burner increases, which is because of the addition of the gaseous products. However, the pressure valve attached to the nitrogen gas supplier is regulated automatically to reduce the nitrogen gas flow rate in order to maintain the pressure constant. Thus, the pressure in the burner is maintained at the desired pressure. Burning rate is measured by determining the instant of melting of each of 5 low-melting-point fuse wires of lead metal, 0.25 mm in diameter, threaded through the strand at accurately known separation distances (15 mm). These 5 fuse wires, each in series with a resistor, form 5 parallel arms of an electrical circuit, whose output voltage changes discontinuously as soon as a fuse wire melts. The temperature of the strand can also be measured by a calibrated copper-constantan thermocouple threaded through the strand and the bead of the thermocouple is placed in the center of the strand. The real time data were recorded by a computer which can process it and figure out the burning rate. The experiment will be repeated for 5 times at each test pressure and the average experimental results were obtained with the standard deviation of 0.13-0.25.

4) Mechanical Sensitivity Test Method:

The mechanical sensitivity of the propellant compositions to impact stimuli was determined by applying the fall hammer method (2 kg drop weight) in a Bruceton staircase apparatus[33] and results are given in terms of statically obtained 50% probability of explosion (H_{50}). Friction sensitivity was measured on a Julius Peter apparatus[34] by incrementally increasing the load from 0.2 to 36 kg, till there was no ignition or explosion in five consecutive test samples.

5) Density and Explosion Heat Test Method:

Explosion heat measurements were made with a Model ZDHW-2 temperature constant automatic calorimeter with sample mass of about 5.0 g and reference sample of standard double-based gun propellant (No. 95-7) in vacuum.

Density measurements were carried out on a Model AG 104 METTLER TOLEDO balance with samples of 30 mm ×30 mm ×10 mm rectangular stick s in steeping medium of liquid paraffine at the temperature of $(20\pm2)^0$C.

III. PAGE STYLE

A. Characteristics of ADN

ADN is a solid white salt of the ammonia cation (NH_4^+) and the dinitramide anion ($N(NO_2)^{2-}$), Figure 2. Its high oxygen balance, +25.79 %, and the absence of halogen and metal atoms make it attractive as an oxidizer in minimum-smoke rocket propellants. It melts at 93^0C and starts to decompose at approximately 150^0C at a heating rate of 10 K·min^{-1}, as seen in Figure 3.

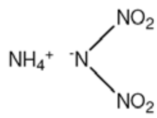


Fig. 2 The chemical structure of ADN

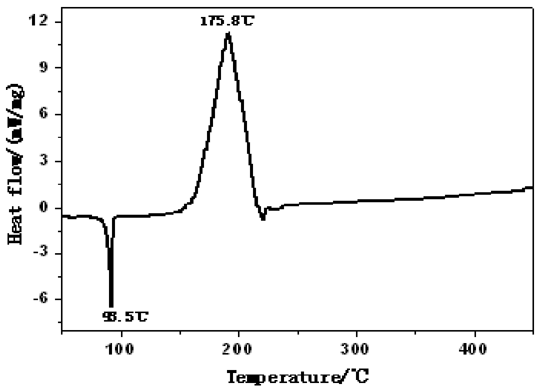
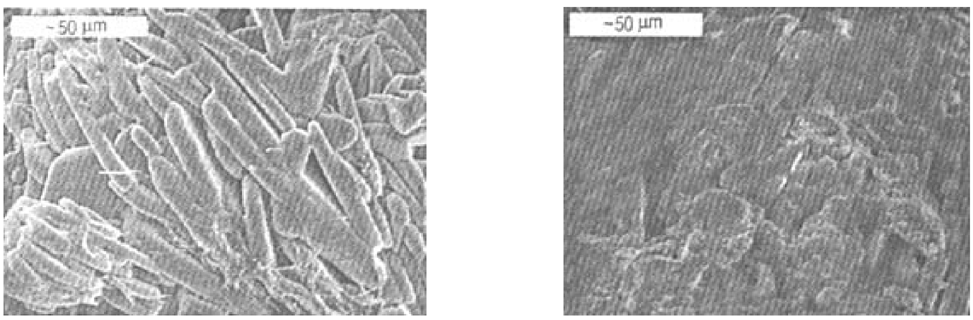


Fig. 3 DSC curve of ADN (heating rate: 10 K·min^{-1})

Two types (coated before and coated with polyurethane binder) of well-dried ADN particles were more free dried. Microstructures of ADN particles (before coating and after coating with polyurethane binder) are shown in Figures 4. The particle size distribution of ADN particles (before coating and after coating) are shown in Figure 5 and Table 2, respectively.



(a) (b)

Fig. 4 Microstructures of ADN (×500, a: before coating, b: after coating)

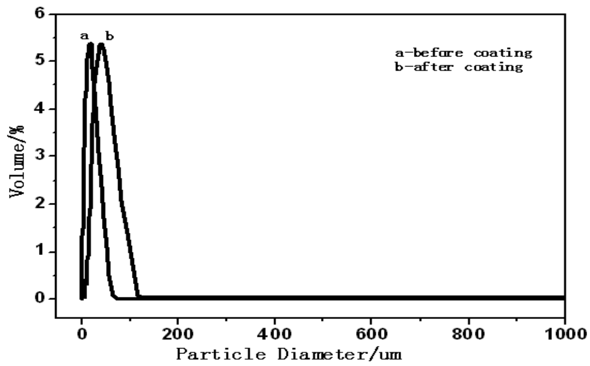


Fig. 5 Particle size distribution of ADN particles

TABLE II THE DIAMETERS OF ADN PARTICLES

Items	unit	ADN	
		Before coating	Coated
d_{10}	μm	2.653	5.648
d_{50}	μm	12.755	22.986
d_{90}	μm	34.097	54.367
Span	-	2.465	2.120
Density	$g\cdot cm^{-3}$	1.812	1.814
Vol. weighted mean	μm	15.927	26.965
Specific surface area	$m^2\cdot g^{-1}$	1.130	0.698

In the table, d_{10}: particle diameter corresponding to 10% of the cumulative undersize distribution, μm; d_{50}: median particle diameter, μm; d_{90}: particle diameter corresponding to 90% of the cumulative undersize distribution, μm; Span= $(d_{90}-d_{10})/d_{50}$; and specific surface area refers to the particle size distribution determined by Malvein Mastersizer.

It can be seen from the results mentioned above that the microstructures of ADN particles after coating with polyurethane binder are ball shaped with a rough surface, whereas ADN before coating shows irregular shapes, the coated ADN particles are more uniform than coated before. The median diameters d_{50} of coated ADN particles (22.986 μm) is much larger than that of before coating particles (12.755 μm). Corresponding to the larger values of d_{50}, the specific surface area of coated ADN (calculated by the Mastersizer) is 0.698 $m^2\cdot g^{-1}$ and much less than that of particles coated before (1.130 $m^2\cdot g^{-1}$). The span of coated particles is 2.120, which is much lower than that of particles coated before (2.465).

Similarly to ammonium nitrate, ADN is hygroscopic and readily soluble in water and other polar solvents, but scarcely soluble in non-polar solvents. The solubility of ADN in different solvents and its basic properties are shown in Table 3 and Table 4, respectively.

TABLE III ADN SOLUBILITY AT 20 ^{0}C

Solvent	Solubility in 100 g solvent (g)
Water	357
Methanol	86.9
Butyl acetate	0.18
n-heptane	0.005
Dichloromethane	0.003

TABLE IV PROPERTIES OF ADN

Molecular weight	124.07 g/mol
Density	1.81 g/cm^3
Melting point	93 ^{0}C
Heat of formation	-148 kJ/mol
Heat of combustion	424 kJ/mol
Oxygen balance	+25.79 %
Critical relative humidity	55.2 %

To obtain a castable propellant formulation with low viscosity and high solid loading, particles with minimum spatial extension are required. For this reason spherical particles are preferred. The particle shape of ADN as received from the synthesis is needle shaped and thus not suitable for formulation. The prills are produced by spraying molten ADN though a nozzle. In the nozzle the molten ADN is atomized to form droplets which then solidify to the desired prills[6]. Currently the prills are produced in batches using up to 250 g of ADN per batch. However, with minor modifications this method can be run continuously making the technology suitable for industrial production.

B. Theoretical Performance of Composite Propellant with ADN

The energy characteristics of the composite propellants with ADN particles were thoroughly calculated by the minimum free energy method in our computer program, whose algorithm was based on the fundamental thermodynamics of minimum free energy[35]. The calculated and determined results are listed in Table 5.

TABLE V THE ENERGETIC CHARACTERISTICS OF HTPB-BASED COMPOSITE PROPELLANT WITH ADN (COMBUSTION PRESSURE: 7.0 MPA, EXIT PRESSURE: 0.1 MPA)

Samples	ρ/g·cm^{-3}	I_{sp}/N·s·kg^{-1}	T_c/K	C*/m·s^{-1}
HP-1	1.718	2519.58	2895	1518. 5
HP-2	1.692	2531.24	2883	1530.2
HP-3	1.681	2540.16	2866	1537.4
HP-4	1.674	2551.84	2851	1540.3

Note: I_{sp} is specific impulse (atmosphere); C* is characteristic velocity; T_c is adiabatic flame temperature; ρ is density.

From the results in Table 5, it can be seen that there are a little difference between the propellant HP-1 and HP-2 - HP-4. The specific impulse and adiabatic flame temperature increase with an increase in the content of ADN at the replacement of each of AP content, respectively. Whereas, the density of composite propellant with ADN decreases in each mass content of AP, which maybe attributes to the density of ADN (1.81 g·cm^{-3}) is lower than that of AP (1.95 g·cm^{-3}).

C. Combustion Characteristics

Considerable high burning rate and appropriate pressure exponents are observed for both ADN/GAP and AP/GAP formulations. Burning rate and pressure exponents of ADN formulation correspond to the values have been found[1, 2]. The measurements of burning rate and pressure exponent are used to study the combustion characteristics of the composite propellant containing ADN as part of oxidizer. The detailed combustion characteristics (burning rates and pressure exponents) of HTPB-based composite propellants with coated ADN particles at the pressure range of 1.0-15 MPa are shown in Table 6. The burning rates of the ADN based propellant samples were measured for five times, and the data in Table 6 are those of the average, each of the result error is in the range of 3.0%.

TABLE VI THE BURNING RATES AND PRESSURE EXPONENTS DATA OF HTPB-BASED COMPOSITE PROPELLANTS WITH AND WITHOUT ADN PARTICLES AT VARIOUS PRESSURE RANGE (PRESSURE RANGE: 1-15 MPA; INITIAL TEMPERATURE: T_0=293 K)

Samples	Burning rate (u/mms^{-1}) at different pressure				
	1 MPa	4 MPa	7 MPa	10MPa	15 MPa
HP-1	2.25	5.44	7.17	8.21	9.36
HP-2	2.44	6.26	9.35	11.88	15.64
HP-3	2.57	6.58	9.51	12.40	16.01
HP-4	2.80	6.62	10.38	13.37	19.43
Samples	Pressure exponent (n) at different pressure range				
	1-4 MPa	4-7 MPa	7-10 MPa	10-15 MPa	1-15 MPa
HP-1	0.48	0.51	0.38	0.35	0.41
HP-2	0.73	0.62	0.67	0.68	0.66
HP-3	0.61	0.78	0.74	0.63	0.68
HP-4	0.72	0.81	0.71	0.65	0.71

From Table 6, it can be seen that although the AP formulation exhibits low burning rate here, a change to pat content of ADN may boost the burning rate higher than that of the AP formulation. Thus, it can be indicated that the increasing content of ADN oxidizer in the formulation not only can increase the specific impulse but also can increase the burning rate for ADN formulations. An interesting feature is that the burning rate and pressure exponent values depend on the formulation of the content of ADN oxidizer, whereas, the influence of the particle size of ADN oxidizer for HTPB composite propellant could be investigated further.

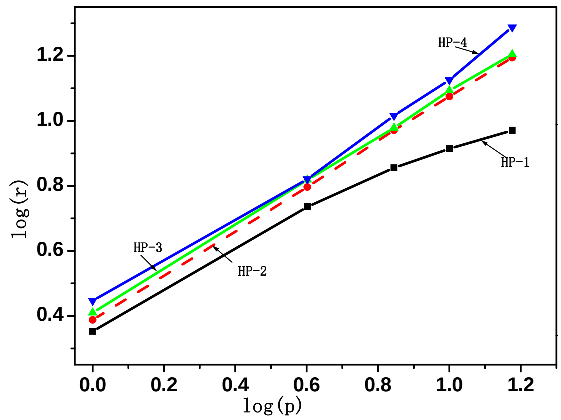


Fig. 6 Effects of different contents of ADN particles on the burning rate of the propellants

Lastly, one point we must emphasize is that the decomposition at lower temperature indicates that the gaseous products formed during decomposition exert lower feedback to the deflagrating propellant surface[36]. Hence, much energy is released in combustion process (at surface) in the same temperature, which supports for the accelerating of burning rate.

D. Mechanical Sensitivity

When formulating propellants or other explosive materials with new energetic ingredients, the major drawbacks to occur are bad chemical stability or significantly enhanced sensitivity to external stimuli. For HTPB-based composite propellants with ADN formulations the values from small scale sensitivity testing are outlined in Table 7 and Table 8, respectively. The results of the friction sensitivity test are expressed by the explosion probability (P) and the impact sensitivity by the drop height (H_{50}). The results indicate that the greater P is, the higher the friction sensitivity is, while the higher H_{50} is, the lower the impact sensitivity is.

TABLE VII MECHANICAL SENSITIVITY DATA OF ADN PARTICLES

Samples	5 s explode point/ ^{0}C	Emit gas volume/mL	H_{50}/cm
Coated before	187.5	1.8mL/g	24.1
Coated particles	200.1	1.7mL/g	33.5

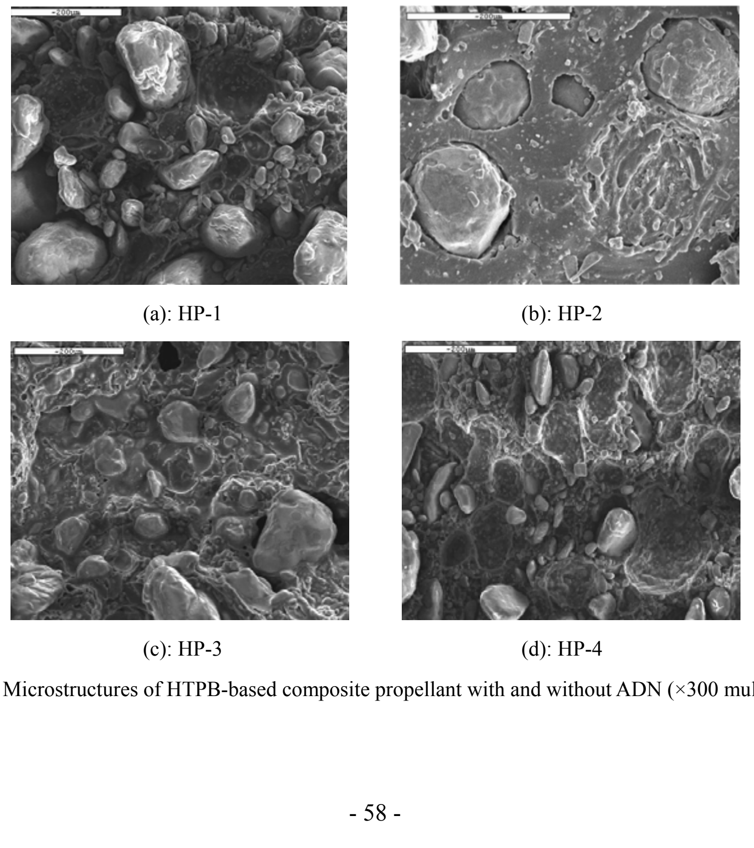
TABLE VIII EFFECT OF ADN ON THE MECHANICAL SENSITIVITY FOR HTPB-BASED COMPOSITE PROPELLANTS

Samples	P/%	H_{50}/cm
HTPB/Al(18%)/AP(67%)	36	112.2
HTPB/Al(18%)/AP(57%)/ADN(10%)	40	80.1
HTPB/Al(18%)/AP(52%)/ADN(15%)	52	67.7
HTPB/Al(18%)/AP(47%)/ADN(20%)	71	54.7

It can be seen from the results in Table 7 that the impact and friction sensitivity for coated ADN particles (33.5 cm and 10.5 %) show higher sensitivity than that of ADN coated before (24.1 cm and 21.4 %), both of the data listed are lower than that of AP (60.5 cm and 96 %)[37]. The propellant formulations containing ADN were sensitive to impact and friction except based propellant with AP, which is more insensitive to friction as compared to the compositions with ADN (in Table 8). The sensitiveness may be attributed to the active groups on the ADN surface and sensitivity of ADN in contrast with AP particles. The data reveal that the use of coated ADN particles incorporation of granulated and shaped in the composite propellant lead to major increase in friction and impact sensitivity, so, researches must be done in the future to decrease the mechanical sensitivity of the composite propellant with ADN.

E. SEM Micrographs of Composite Propellants

SEM observation of the propellants surface is one of the most important means to study the physical structures of ADN oxidizer in composite propellant. Figure 6 shows the SEM micrographs of HTPB-based composite propellants with ADN.



(a): HP-1 (b): HP-2

(c): HP-3 (d): HP-4

Fig. 6 Microstructures of HTPB-based composite propellant with and without ADN (×300 multiple)

From Figure 6, it can be seen that there are many spherical coated particles and a little irregular small particles on the surface of cured composite propellants, and the small particles are changed among the bigger ones. The plain part is corresponding to the molten oxides and the linked networks of binder systems, which were composed of hydroxyl terminated polybutadiene (HTPB, binder) with 2,4-toluene diisocyanate (TDI, curing agent) curing agent (a), which indicate that AP particles are compatible with the ingredients of the composite propellant systems. The foam part due to the holes coming from the hygroscopic of ADN oxidizer at atmosphere or the un-linked binder systems (d), so the application of ADN additives in the propellant formulation need to be farther researched.

In conclusion, the general appearance and dispersing behavior of the ADN particles are similar to that of free flowing spherical particles. The method mentioned above and the HTPB binder might be a suitable modification to the traditional process of the coating technology for ADN particles.

IV. CONCLUSIONS

(1) The specific impulse and characteristic velocity of HTPB-based composite propellant with ADN particles increase with an increase in the content of ADN at the replacement of each of AP content, whereas, the density and adiabatic flame temperature are decrease.

(2) Coated ADN particles can improve the combustion characteristics of composite propellant in the pressure range of 1.0 to 15 MPa. Addition of coated ADN particles to the composition of HTPB - based composite propellants can increase the burning rate effectively with a little increase in the pressure exponent.

(3) Both the impact and friction sensitivities are increased in the content of ADN oxidizer.

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