



Synthesis and Characterization of a High Energy Combustion Agent (BHN) and Its Effects on the Combustion Properties of Fuel Rich Solid Rocket Propellants

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Abstract: A high energy combustion agent (tetraethylammonium decahydro-decaborate, BHN) was prepared by means of an ion exchange reaction (IER), and the prepared samples were characterized by the advanced diagnostic techniques of Scanning Electron Microscopy (SEM), X-ray diffraction (XRD), Thermogravimetric Analysis (TGA), and Differential Scanning Calorimetry (DSC) *etc.* The effects of BHN particles on the hazard and combustion properties of fuel rich solid propellants were investigated. The results showed that the BHN samples and fuel rich propellants containing BHN particles can be prepared successfully and solidified safely. The peak temperature of thermal decomposition and the heat of decomposition of the BHN samples prepared were 305.8 °C and 210.9 J·g⁻¹ at a heating rate of 10 K·min⁻¹, respectively. The burning rate and pressure exponent of fuel rich solid propellants decreases with increases in the fraction of BHN particles in the propellant formulation. Compared with the reference formulation (sample BP-1), the burning rate of the propellant with 10% mass fraction of BHN particles (sample BP-4) had decreased 30% at 3.0 MPa, and the pressure exponent had dropped from 0.44 to 0.41.

Keywords: fuel rich solid propellant, BHN, DSC, TG-DTG, burning rate, combustion properties

Nomenclature

a :	pre-exponential factor of burning rate law
Al:	aluminum powder
AP:	ammonium perchlorate
BHN:	tetraethylammonium decahydrododecaborates
$D_{[3,2]}$:	the surface area mean
$D_{[4,3]}$:	the volume moment mean
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
DOS:	dioctyl sebacate
DSC:	differential scanning calorimetry
FRP-1:	fuel rich propellant of sample 1
GFP:	catocene
HTPB:	hydroxyl - terminated polybutadiene
H_u :	mass heat of combustion, $\text{MJ}\cdot\text{kg}^{-1}$
IPDI:	isophorone diisocyanate
Mg:	magnesium powder
n :	pressure exponent
NEPE:	nitrate ester plasticized polyether
p :	pressure, MPa
r :	strand burning rate, $\text{mm}\cdot\text{s}^{-1}$
SEM:	scanning electron microscope
Span:	$(d_{90}-d_{10})/d_{50}$
SSA:	specific surface area
TG-DTG:	thermogravimetry-derivative thermogravimetry
V_u :	volume heat of combustion, $\text{MJ}\cdot\text{cm}^{-3}$
XRD:	X-ray diffraction
ρ :	density, $\text{g}\cdot\text{cm}^{-3}$

1 Introduction

A solid propellant ramjet is an interesting concept as it utilizes atmospheric air during its operation. It has great advantages in terms of increases in the range or payload capacity of the missile. Fuel rich solid rocket propellants are designed, prepared and intended for ramjets. Using high specific impulse (I_{sp}) fuel, solid rocket propellants are able to generate high thrust despite a small nozzle throat area. Propellants containing highly energetic materials are able to generate high I_{sp} [1-3]. Boron (B) is a potential ducted rocket metal fuel to be used in fuel rich propellants, being superior to beryllium (Be), magnesium (Mg) and aluminum (Al), *etc.* However, the preparation process for fuel-rich solid propellants containing boron powder is difficult due to the presence of a viscous H_3BO_3 and B_2O_3 layer on the surface of the amorphous boron powder, which can react

with the $-OH$ groups of the HTPB binder [4-6]. This restricts the application of boron particles in solid propellants. The highly energetic combustion agent, tetraethylammonium decahydrodecaborate ($[(C_2H_5)_4N]_2B_{10}H_{10}$, BHN), a new energetic material with a high heat of combustion ($49.5 \text{ MJ}\cdot\text{kg}^{-1}$) and low mechanical sensitivity ($H_{50} > 128 \text{ cm}$, $P = 0\%$), can be used as a main ingredient in cast explosives and propellants [7-9]. Its crystal density and detonation velocity are $0.92 \text{ g}\cdot\text{cm}^{-3}$ and $9094 \text{ m}\cdot\text{s}^{-1}$, respectively [10]. Energetic compounds with high heats of combustion can provide high temperature combustion products and improve the propulsion performance of ducted rockets. There are a few reports on the synthesis and evolution of combustion catalysts in propellants and explosives. The most commonly used, in a large range of applications, are obtained by co-precipitation, of decahydrodecaborate salts, together with some oxidizers, such as $K_2B_{10}H_{10}$, $Cs_2B_{10}H_{10}$, $(NH_4)_2B_{10}H_{10}$, $Cs_2B_{10}H_{10}\cdot CsNO_3$ and $Cs_2B_{10}H_{10}/KNO_3$, *etc.* [11-13]. The compatibility of BHN with some energetic components and inert materials is one of the most important aspects of BHN use in practical applications, and it was reported by Pang W.-Q. [14] that prepared BHN particles have good compatibility with the main ingredients of some energetic components and inert materials. Moreover, Chen F.-T. *et al.* [15] reported the effects of $[N(C_2H_5)_4]_2B_{12}H_{12}$ on the combustion properties of nitrate ester plasticized polyether (NEPE) propellants. Their results showed that it is not an effective catalyst for the decomposition of AP, whereas it can increase the decomposition of nitramines, the burning rate of NEPE propellants can be increased by the addition of this compound, and also that a “platform” appears over the high pressure range 7-11 MPa. Thus, from the point of view of the high performance mentioned above, it has potential for possible use as an energetic ingredient in fuel rich solid propellants and explosives [16, 17]. However, there are few reports on the combustion properties of fuel rich solid propellants containing BHN. In the present work, the tetraethylammonium decahydrodecaborate (BHN) particles were prepared. The characteristics of the BHN particles were analyzed by using the diagnostic techniques of scanning electron microscopy (SEM), X-ray diffraction (XRD), thermogravimetric analysis (TGA), and differential scanning calorimetry (DSC). Different mass fractions of BHN particles were added to the formulations and four different propellant compositions with and without BHN were produced. The focus of this paper is on how BHN affects the combustion properties of fuel rich solid rocket propellants, placing the emphasis on the burning rate and pressure exponent performances of the solid propellants, which could be used for solid rocket motor applications.

2 Experimental

2.1 Materials and specimens

Sodium borohydride (purity $\geq 99\%$) and tetraethylammonium chloride (purity $\geq 99\%$), industrial grade, Shandong Chemical Company. Methyl alcohol, methylene dichloride and tetrahydrofuran, analytically pure, Chengdu Kelong Chemical Reagent Company; paroline, analytically pure. HTPB binder cured with IPDI, plasticized by DOS, and micron-sized dual metal powders (Al and spherical Mg) were used as fuel components in the propellants. Two types of AP were utilized in the propellant formulations. The first consisted of research grade AP (purity $> 99\%$ pure) with an average particle size of 105-147 μm . The second type was obtained by grinding AP in a fluid energy mill to an average particle size of around 1-5 μm . BHN particles prepared in the Xi'an Modern Chemistry Research Institute were used. Except where otherwise stated, all propellants were manufactured, processed, and tested at the Xi'an Modern Chemistry Research Institute under identical conditions and using identical procedures.

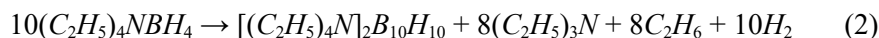
UV spectrophotometer, ZF-II Shanghai; Fourier transform infrared spectrometer, NEXUS 870, American; NMR spectrometer with superconducting magnet, AV500 (500MHz), BRUKER Switzerland; Elemental analysis apparatus, VARIO-EL-3, EXEMENTAR Germany; DSC, Q-200, TA American.

The following are the mass percentages of the ingredients used in the four different propellant formulations: (1) FRP-1: HTPB/16.8%, GFP/5.0%, Al/21%, Mg/21%, AP/34%, others/2.2%; (2) FRP-2: HTPB/16.8%, GFP/5.0%, Al/21%, Mg/18%, BHN/3%, AP/34%, others/2.2%; (3) FRP-3: HTPB/16.8%, GFP/5.0%, Al/21%, Mg/15%, BHN/6%, AP/34%, others/2.2%; (4) FRP-4: HTPB /16.8%, GFP/5.0%, Al/21%, Mg/11%, BHN/10%, AP/34%, others/2.2%.

The propellant formulations were mixed in 500 g batches using a 2 L vertical planetary mixer. All of the samples involved in this investigation, which were prepared by the slurry cast technique at 35 $^{\circ}\text{C}$ and then solidified during 120 h (50 $^{\circ}\text{C}$) in a water jacketed oven, were machined to the fixed dimensions to be used.

2.2 Processing and structures

The BHN particles were prepared according to the following reactions.



The preparation process for BHN was as follows: tetraethylammonium tetrahydroborate (65 g, 0.45 mol) and paraffin oil (60 mL) were added to the pyrolysis reactor under the protection of nitrogen, the oil bath was heated to 185 °C for 16 hours, and then cooled to room temperature. The product was filtered off, washed and dried, and then recrystallized from water and methanol, to give BHN (7.1 g, 39.8%).

The molecular structure of BHN is shown in Figure 1.

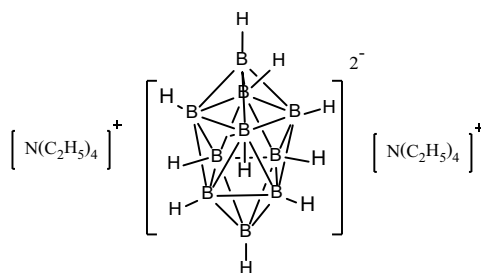


Figure 1. The molecular structure of BHN.

2.3 Characterization

2.3.1 Microstructure and Granularity Distribution of the BHN particles prepared

The particle size and size distribution of the BHN particles were measured with a Master Sizer Instrument. The morphologies of the prepared particles were examined with a scanning electron microscope (SEM).

2.3.2 Thermal decomposition analysis

Thermal analysis (DSC and TG-DTG experiments) of the prepared samples was carried out on a Q-200 TA instrument (made in USA) at a heating rate of 10 °C min⁻¹ at 0.1 MPa under N₂ atmosphere (sample mass 0.5-1.0 mg).

2.3.3 Hazard tests

The sensitivity of the BHN samples and the propellant compositions to impact stimuli was determined by applying the fall hammer method (2 kg drop weight) in a Bruceton staircase apparatus [18] and the results were given in terms of the statistically obtained 50% probability of explosion (*H*₅₀). Friction sensitivity was measured on a Julius Peter apparatus [19] by incrementally decreasing the load from 36 to 0.2 kg, until no ignition was noted in five consecutive test samples.

2.3.4 Burning rate test

The strand burning rate of the propellants was determined in the pressure range $0.5 \text{ MPa} < P < 5.0 \text{ MPa}$ by means of the fuse-wire technique [20-22]. The method involved the combustion of strands (ignited by means of a Nichrome wire) of dimensions $150 \times 5 \times 5 \text{ mm}$ in a nitrogen pressurized steel bomb. The burning rates were computed from the time that was recorded for the tests conducted at each pressure for each sample.

3 Results and Discussion

3.1 Characterization of the prepared samples

3.1.1 Nuclear magnetic resonance spectral analysis of BHN

The nuclear magnetic resonance spectrum of a BHN sample is shown in Figure 2.

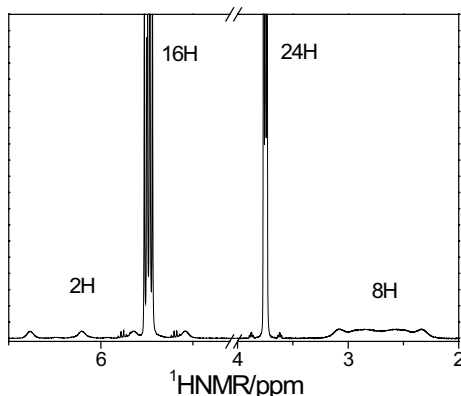


Figure 2. ^1H NMR of a BHN sample [17].

In Figure 2, ^1H NMR (CDCl_3 , 500 MHz), δ : 3.37-3.77 (m, 24 H), 5.72-5.76 (m, 16 H), 5.54-6.38 (q, 2 H), and 2.33-3.09 (m, 8 H); ^{11}B NMR (DMSO, 500 MHz), δ : -28.67 (d, 8B, $J = 130 \text{ Hz}$), -0.83 (d, 2B, $J = 130 \text{ Hz}$).

3.1.2 Infrared spectroscopy (IR) and elemental analysis of BHN samples

Figure 3 shows the IR spectra of BHN samples at different temperatures.

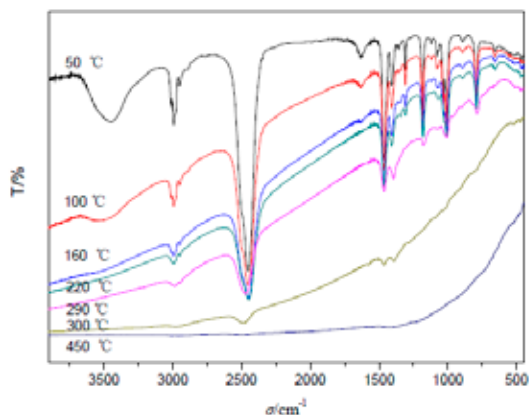
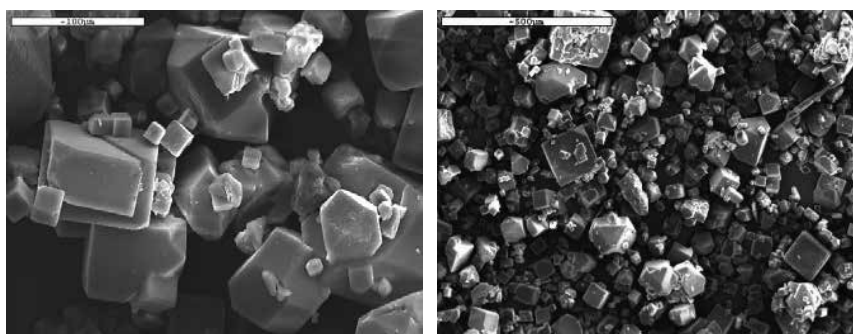


Figure 3. Infrared spectra (IR) of BHN at different temperatures [17].

It can be seen from Figure 3 that the IR spectral analysis of the sample shows: IR (KBr), ν/cm^{-1} : 2991 ($\nu_{\text{C-H}}$), 2450 ($\nu_{\text{B-H}}$), 1460 ($\nu_{\text{C-H}}$), 1401 ($\nu_{\text{C-H}}$), 1309 ($\nu_{\text{C-N}}$), 1184 ($\nu_{\text{C-N}}$), 1034 ($\nu_{\text{B-B}}$), 1008 ($\nu_{\text{B-B}}$). Comparing the elemental analysis of ($\text{C}_{16}\text{H}_{50}\text{B}_{10}\text{N}_2$): calculated B 28.5%, C 50.5%, H 10.5% and N 7.4%, measured values B 29.5%, C 50.6%, H 10.5%, and N 7.3%.

3.1.3 Particle size and size distribution of the prepared BHN particles

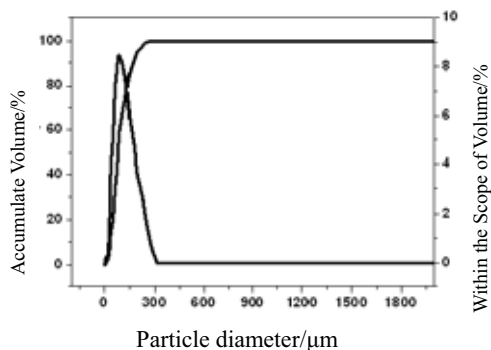
The surface photographs and particle size distribution of the BHN particles were analyzed by means of SEM and a Laser particle analyzer, and the results are shown in Figure 4. The relevant parameters are listed in Table 1.



(i) $\times 100$

(ii) $\times 500$

(a) SEM photographs



(b) Particle size distribution

Figure 4. SEM photographs and particle size distribution of the BHN particles.**Table 1.** Particle size parameters of BHN

Sample	$D_{[3,2]}$ [μm]	$D_{[4,3]}$ [μm]	d_{10} [μm]	d_{50} [μm]	d_{90} [μm]	Span	SSA [$\text{m}^2 \cdot \text{g}^{-1}$]	P [$\text{g} \cdot \text{cm}^{-3}$]
BHN	73.1	106.7	43.9	94.8	189.4	1.536	0.08	0.92

It can be seen that the microstructure of the BHN particles is irregular in shape, and the corresponding diameters of the BHN powder (d_{50}) = 94.8 μm . Corresponding to the large value of d_{50} , the specific surface area and the span of the particles is 0.08 $\text{m}^2 \cdot \text{g}^{-1}$ and 1.536 $\text{m}^2 \cdot \text{g}^{-1}$, respectively. Thus it can be deduced that in order to improve the processing properties of the propellants, the prepared particles need to be coated or prilled to have a rounded surface [23, 24].

3.1.4 Thermal analysis of the BHN particles

Figures 5 and 6 show the DSC and TG-DTG curves of BHN samples at a heating rate of 10 $\text{K} \cdot \text{min}^{-1}$ at 0.1 MPa.

The DSC and TG-DTG curves (Figures 5 and 6, respectively) of the prepared BHN samples show an exothermic change and a single main stage weight loss, respectively. The exothermic peak is very sharp and the peak temperature, together with the heat of decomposition of the BHN sample, are 305.8 $^{\circ}\text{C}$ and 210.9 $\text{J} \cdot \text{g}^{-1}$ at a heating rate 10 $\text{K} \cdot \text{min}^{-1}$, respectively. The DSC curve shows no endothermic changes before the onset of the exotherm, and indicates that this compound is decomposed in the solid state.

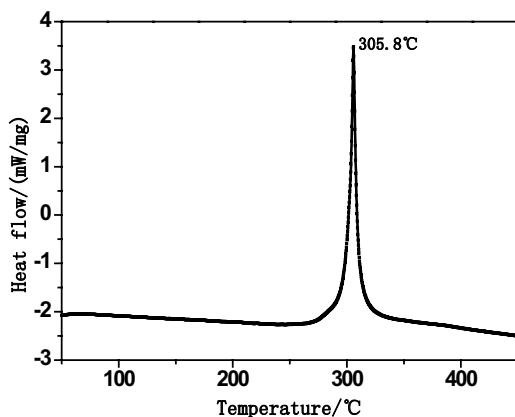


Figure 5. DSC curve of BHN (0.1 MPa).

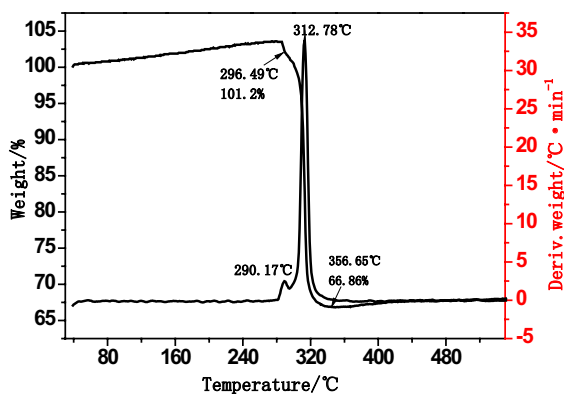


Figure 6. TG-DTG curves of BHN (0.1 MPa).

3.2 Effects of BHN particles on the properties of fuel rich propellants

3.2.1 Effects of BHN particles on the energetic properties of fuel rich propellants

BHN, as one of the high combustion agents, gives much improvement to the energetic properties of solid propellants. The energetic properties (density, mass heat of combustion and volume heat of combustion) were measured and calculated and the data are shown in Table 2.

Table 2. The energetic properties of fuel rich propellants with and without BHN particles

Samples	ρ^* [g·cm ⁻³]	H_u^* [MJ·kg ⁻¹]	V_u^{**} [MJ·cm ⁻³]
BHN	0.94	49.5	46.53
FRP-1	1.63	21.8	35.55
FRP-2	1.59	22.6	36.06
FRP-3	1.56	23.3	36.28
FRP-4	1.53	23.8	36.30

Note: * measured data; ** calculated data.

The data in Table 2 indicate that the mass heat of combustion and volume heat of combustion of fuel rich propellants increase with increasing mass fraction of BHN particles in the propellant formulation, whereas the density decreases. Compared with the reference formulation (sample FRP-1), the mass heat of combustion and volume heat of combustion of the fuel rich solid propellant containing 10% mass fraction of BHN (sample FRP-4) are increased by 9.0% and 2.1%, respectively, whilst the density is decreased by 6.3%, which would have a large influence on an engine's propellant loading, with its finite volume.

3.2.2 Effects of BHN particles on the hazard properties of fuel rich propellants

The mechanical sensitivity of a solid propellant reflects the degree of difficulty of initiation by external mechanical action, which is one of the most important parameters to assess the safety of solid propellants. Therefore, it was necessary for us to investigate the mechanical sensitivity of fuel-rich solid propellants containing BHN particles. Table 3 shows the results of fuel rich propellants with and without BHN particles.

Table 3. The hazard properties of BHN and fuel rich propellants with and without BHN particles

Samples	Impact [N·m]	Standard deviation S (log value)	Friction [N]	Level of confidence 95%
BHN	> 24.68	-	0% at 250 N	(0%, 14%)
FRP-1	21.99	0.30	26.2	(76%, 99%)
FRP-2	6.33	0.37	44.2	(69%, 98%)
FRP-3	3.82	0.23	23.1	(80%, 100%)
FRP-4	2.47	0.10	44.8	(69%, 98%)

It can be seen from the data in Table 3 that the impact and friction sensitivity of the prepared BHN samples are rather low, especially the friction sensitivity which is 0% at 250 N, and indicates that it is safe with respect to mechanical stimulation and it is feasible for use in fuel rich solid propellants. The impact sensitivity increases significantly (from 21.99 to 2.47 N·m) with increases in the BHN mass fraction in the propellant formulation, whereas the friction sensitivity changes little, with only marginal increases. Compared with the data for sample FRP-1, the higher the impact sensitivity value is, the safer the impact sensitivity of the propellant sample is, which may be attributed to the crystal structure of the BHN and the active groups on the surface of the BHN particles, which can react with some of the ingredients added in the fuel rich solid propellant formulations.

3.3 Effects of BHN on the combustion properties of the fuel rich propellants

3.3.1 Burning rate and pressure exponent of propellant with and without BHN particles

BHN, as one of the highest energy combustion agents, has a significant effect on the combustion properties of solid propellants [25-27]. The effects of different mass fractions of BHN on the burning rate and pressure exponent of fuel rich solid propellants are shown in Table 4.

Table 4. Burning rate and pressure exponent of fuel rich propellants with and without BHN particles

Samples	Burning rate, [mm·s ⁻¹]				Pressure exponent (<i>n</i>)			
	0.5 MPa	1 MPa	3 MPa	5 MPa	0.5-1 MPa	1-3 MPa	3-5 MPa	0.5-5 MPa
FRP-1	14.11	17.81	32.68	37.04	0.34	0.55	0.25	0.44
FRP-2	11.63	15.87	25.30	30.49	0.45	0.42	0.37	0.42
FRP-3	11.07	14.45	23.73	28.85	0.38	0.45	0.38	0.42
FRP-4	11.04	14.32	22.73	28.70	0.38	0.42	0.46	0.41

The data in Table 4 show that the combustion behaviour of the fuel rich propellants changes significantly on addition of BHN particles to the propellant formulation, compared with sample FRP-1 (reference formulation). The burning rate and pressure exponent (from 0.44 to 0.41) of fuel rich propellants decrease with an increase in the mass fraction of BHN particles in the propellant formulation, in the pressure range 0.5-5 MPa, in particular there is an obvious reduction for sample FRP-2 compared with the reference formulation. This may be attributed to the low oxygen content of the BHN itself, meaning that its

high energy cannot be released sufficiently when a large amount is added to the propellant formulation. Another interesting feature is that the burning rate and pressure exponent values for the fuel rich solid propellants are dependent on the mass content of BHN in the propellants, but the influence of the particle size of the BHN should be investigated further.

3.3.2 Combustion flame structures of fuel rich propellant with and without BHN particles

The combustion flame structures of the fuel rich solid propellants containing different amount of BHN at 3 and 5 MPa were recorded, and the pictures are shown in Figure 7.

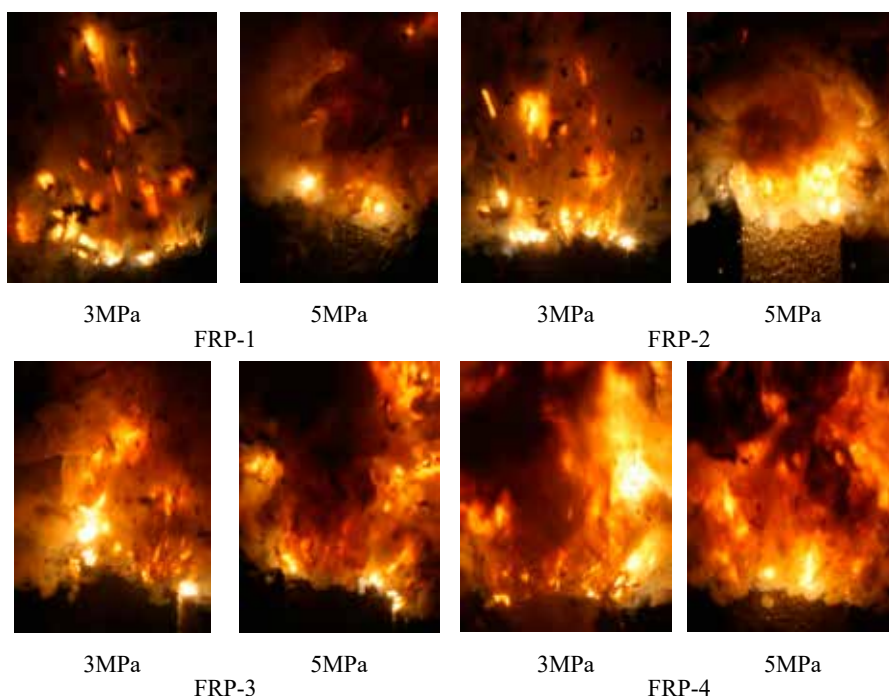


Figure 7. Combustion flame structures of the fuel rich propellants with and without BHN particles.

It can be seen from Figure 7 that the combustion behaviour of the fuel rich propellant containing BHN is similar to that of common composite propellants, which appears as a “multiple-flame structure”. The bright flame is closer to the combustion surface and it becomes much brighter when the measured pressure

is increased, in agreement with the combustion flame characteristics of common composite propellants [27]. Also, there are many sparks on the propellant surface during the combustion process, which can be attributed to the addition of metal particles to the propellant formulations.

3.3.3 Surface morphologies of fuel rich propellant with and without BHN particles

The high energy combustion agent, BHN, when added to a fuel rich solid propellant, has a significant influence on the curing properties and the surface morphology of the propellant. In order to analyze the effects of the BHN particles on the physical structure of the fuel rich solid propellant, the microstructure of the propellants, with and without BHN, is shown in Figure 8.

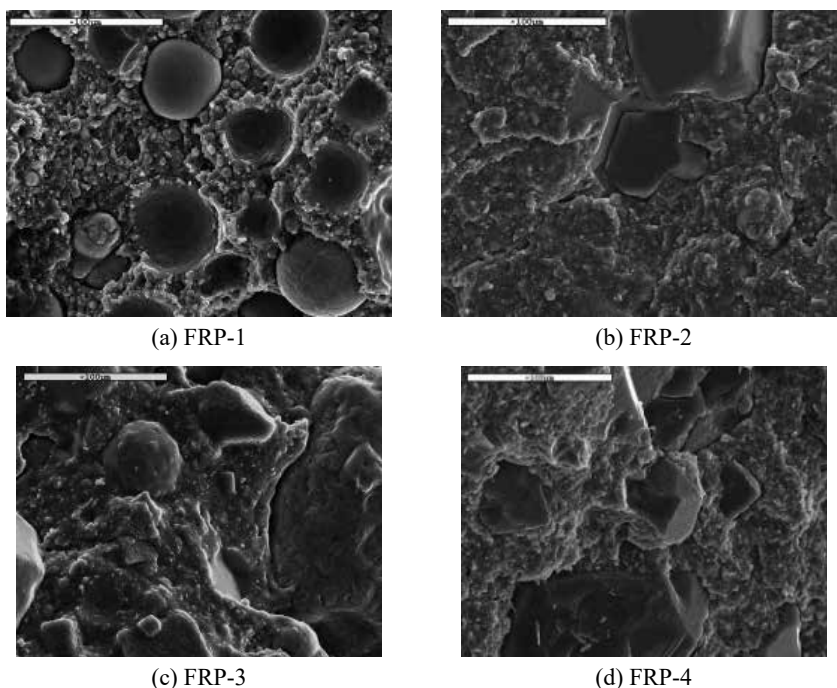


Figure 8. The surface morphologies of the fuel rich propellants with and without BHN particles ($\times 500$).

Figure 8 indicates that there are obviously many approximately spherical and granulated particles on the surface of the cured fuel rich solid propellant, which may be attributed to the addition of spherical magnesium particles to the

propellant formulation. The BHN particles are compatible with the ingredients of the propellant systems, and additionally the granulated particles with smaller diameters can adequately fill the spaces between the larger grains. With the increased BHN content in the propellant formulations, there is an increase in irregular particles on the cured surface of the propellant, caused by the approximately spherical magnesium particles being replaced by an equal mass fraction of irregular BHN particles.

4 Conclusions

(1) BHN can be prepared by means of an ion exchange reaction. The particles prepared exhibit irregular shapes, which need to be coated or prilled to have a desirable rounded surface.

(2) The peak temperature of thermal decomposition and the heat of decomposition of the BHN samples prepared were 305.8 °C and 210.9 J·g⁻¹ at a heating rate of 10 K·min⁻¹, respectively. The heats of combustion of the fuel rich propellant samples increases with increasing mass fraction of BHN particles in the formulation, whereas the density decreases.

(3) The burning rate and pressure exponent of the fuel rich propellant samples decrease with an increase in the mass fraction of BHN in the formulation. The burning rate of solid propellant containing 10% BHN was decreased by 30% compared with that of the reference propellant, and the pressure exponent was decreased from 0.44 to 0.41.

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