

Properties and Application of a Novel Type of Glycidyl Azide Polymer (GAP)-Modified Nitrocellulose Powders

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Abstract: GAP-modified nitrocellulose powders were prepared by an internal solution method and applied in cross-linked modified double base (XLDB) propellants. It was found that GAP-modified nitrocellulose powders exhibit high round, no bonding between the particles and excellent fluidity. When the amount of GAP increased from 10.0% to 30.0%, the median diameter (d_{50}) of powders decreased from 134.53 μm to 94.54 μm . The thermal decomposition process of GAP appeared also in the GAP-modified nitrocellulose powders, but the thermal decomposition peak temperatures of $-\text{N}_3$ and the GAP main chain were found to be lower for the 10.0% and 20.0% GAP-modified

samples than the corresponding peak temperatures for pure GAP, respectively. The plasticizing properties of GAP-modified nitrocellulose powders are better than that of pure nitrocellulose powders, and the drop weight impact sensitivity of the modified powders is reduced as the mass ratio of GAP increases. It was experimentally shown that GAP-modified nitrocellulose powders can improve the mechanical characteristics of the propellant with a maximum tensile strength (σ_m) between $0.36 \text{ MPa} < \sigma_m < 1.10 \text{ MPa}$ and an elongation at maximum tensile strength (ε_m) between $28.8\% < \varepsilon_m < 51.8\%$ at temperatures of -40 , $+20$ and $+50^\circ\text{C}$.

Keywords: Glycidyl azide polymer (GAP) • GAP-modified nitrocellulose spherical powders • Particle size distribution • Thermal decomposition • Mechanical properties

1 Introduction

Nitrocellulose (NC) is one of the important components of double-base or modified double-base propellants. However, the viscosity of propellant's slurry will increase significantly and the preparation technology will be difficult to carry out if NC was added to the slurry of a castable double-base propellant directly [1,2]. Thus, as early as 1936, Olsen proposed the manufacturing principles of nitrocellulose spherical powders. After that, the NC spherical powders have been successfully commercialized [3,4]. However, the nitrocellulose spherical powders cannot be plasticized well, which affects the mechanical properties of XLDB propellants.

Propellants with high-energy, reduced sensitivity and low signature have become a main target because they are the power source for rocket and missile weapons [5]. In propellant formulations, the binder system plays an important role as the base body and skeleton. Using some new types of energetic binders, the propellants can be endowed with perfect energy performance and good mechanical properties with low amounts of solid oxidizers. Additionally, the sensitivity and vulnerability of the propellant system can be reduced [6–8]. Therefore, many countries are widely exploring various types of energetic binders. Glycidyl azide polymers (GAP) are promising candidates for energetic binders in future composite solid propellants with minimum smoke signature, reduced pollution, and low sensitivity [9–13]. The pendant azide groups present in the structure of GAP are

expected to contribute to the energy required from the binder significantly [10]. There have been numerous studies on GAP, including its synthesis [14], structure [15], thermal behavior [16], physical, chemical, and explosive properties [17]. The novel XLDB propellants which contain GAP components have been investigated by Kubota and their mechanical properties and energy performance have been improved significantly [18].

In this paper, in order to improve the plasticizing performance of NC spherical powders, GAP was absorbed on NC, and a new type of GAP-modified single-base spherical powders was prepared by an internal solution method for the first time. Besides, the significance of this way lies in the purpose of premixing GAP and NC, and also improving the uniformity of the propellant composition and physical compatibility. It is thus beneficial for improving the curing of GAP propellants and their mechanical properties.

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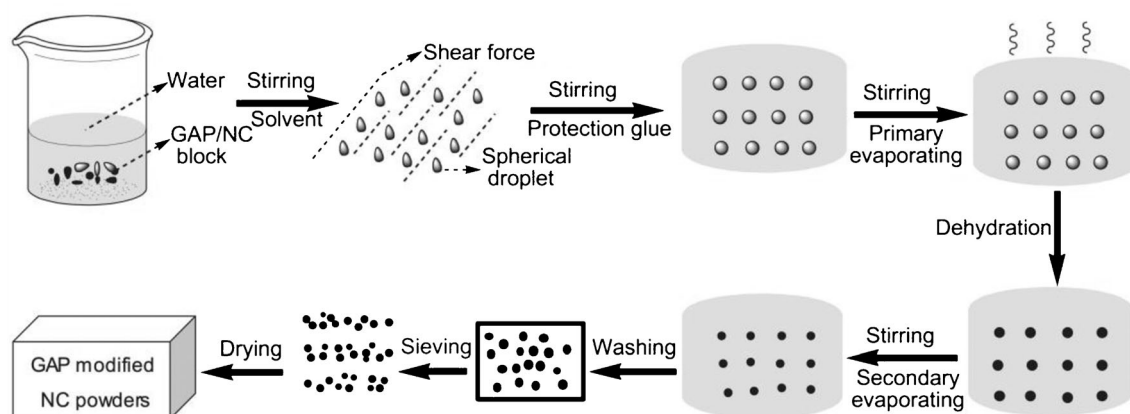


Figure 1. Manufacturing process of GAP-modified nitrocellulose spherical powders.

2 Experimental

2.1 Materials

GAP polyol, copolyether of ethylene oxide, and tetrahydrofuran [P(EO-THF)] were received from Hubei Aviation Institute of Chemical Technology. They are diols with average molecular weights of 5770 and 4500 g mol⁻¹ and hydroxyl values of 0.646 mmol g⁻¹ and 0.577 mmol g⁻¹, respectively. Nitrocellulose (pure NC fibers, 12% nitrogen content), nitroglycerin (NG), dinitroxyethylnitramine, RDX (mean particle size is 90.0 μm), carbon black, and ballistic modifiers were all received from Shanxi Northern Xing An Chemical Industry Co., Ltd. Ethyl acetate, gelatine, and sodium sulfate were supplied from Sinopharm Chemical Reagents Co., Ltd. Triisocyanate (N-100) and triphenylbismuthine (TPB) were obtained from Bayer.

2.2 Synthesis of GAP-Modified Nitrocellulose Spherical Powders

Water (about 360 mL in volume at a temperature of 60 °C) was added into a three-neck flask with a mixing propeller inside, then GAP diol (1.0 g) and pure NC fibers (9.0 g) (take

10% GAP as example) were put into the three-neck flask, and stirred for 30 min at 65 °C. Afterwards, acetic ester (133.3 mL) was added and stirred under reflux for 60 min. After that, bone glue (5.4 g) were added and stirred for another 50 min. The flask was vacuumized at 0.08 MPa for 60 min to evaporate half of the acetic ester. Afterwards, Na₂SO₄ (14.4 g) was added and stirred for 20 min. To remove the residual acetic ester, the flask was vacuumized again for 15 min at 0.08 MPa to distil the remaining acetic ester in the flask. Finally, the mixtures of modified powders, bone glue, and Na₂SO₄ solutions were washed intensively six times with water at a temperature of 50 °C. The powders were dried at 45 °C for 5 d, finally sieved through 180 μm opening. The specific process is outlined in Figure 1.

2.3 Application of the GAP-Modified Nitrocellulose Spherical Powders in XLDB Propellants

The propellant compositions selected to evaluate GAP-modified nitrocellulose spherical powders are illustrated in Table 1. Pure nitrocellulose spherical powders were substituted by GAP-modified nitrocellulose spherical powders in the formulations unchanged premix, and obtained propel-

Table 1. XLDB propellant compositions used for evaluation.

Composition		Weight [%]
NC	(GAP-modified) nitrocellulose spherical powders	8.0–12.0
NG	Nitroglycerin	18.0–25.0
DINA	Dinitroxyethylnitramine(plasticizer)	3.0–5.0
GAP	Glycidyl azide polymer diol	4.5–5.5
P(EO-THF)	Copolyether of ethylene oxide and tetrahydrofuran	4.5–5.5
RDX	Cyclotrimethylenetrinitramine (mean particle size: 90.0 μm)	35.0–45.0
CARBON	Carbon black	0.5–1.0
Ballistic modifiers	Lead and copper compounds	1.5–3.0
C ₂	Centralite II	0.5–1.5
N100	Triisocyanate	1.0–1.5
TPB	Triphenylbismuthine	0.02–0.05

lant samples. The propellant was prepared in a 5 L planetary vertical mixer. Polymer and plasticizer, stabilizers and burning rate modifiers, non nitramine, and nitramine solids and (GAP-modified) nitrocellulose spherical powders were added and mixed until well wetted, and afterwards the mixer was vacuumized. After mixing for 60 min, the curing agent (N100) and catalysts (TPB) were added and mixed for another 30 min under vacuum. The slurry was casted under vacuum into block moulds and cured at 60 °C for 7 d.

2.4 Characterization

The GAP-modified nitrocellulose spherical powders were characterized by using scanning electron microscopy (SEM), FT-IR spectroscopy, density measurements, particle size analysis, thermal gravimetric analysis (TGA), and drop weight tests. Before characterization, the samples were dried for 6 h in a 50 °C water bath oven. SEM micrographs were obtained with a Hitachi S4800 cold field emission scanning electron microscope set with a 15.0 kV acceleration voltage. Infrared analysis was carried out with a Bruker Equinox 55 FTIR spectrometer with a map resolution of 4 cm⁻¹. The particle size distributions of powders were determined by Malvern, UK 2000 laser diffraction instrument, the samples were dispersed in water. Thermogravimetry measurements were performed with a TAQ 500 apparatus with a scanning rate of 5 Kmin⁻¹ until 600 °C. The standard impact tester was used with exchangeable anvil, the amount of tested substance was 50 mm³; drop hammers of 2 kg weight were used. The probit analysis [19] was used to determine the probability levels of initiation. The sensitivity observed was expressed as the drop energy, E_{dr} , versus percentage of initiation. Only the 50 % probability of initiation is used in this article. Mechanical properties of the propellants were determined with an Instron 4505 tensile tester. Cured propellants were cut into slices, from which JANNAP dog bones were stamped. The tests were carried out at temperatures of -40, +20, and +50 °C with 100 mm min⁻¹ cross-head speed.

3 Results and Discussion

3.1 The Morphology of the GAP-Modified Nitrocellulose Spherical Powders

The morphologies of pure nitrocellulose spherical powders and GAP-modified nitrocellulose spherical powders were observed by SEM, the results are shown in Figure 2. It is obvious that GAP-modified nitrocellulose spherical powders exhibit high roundness rate, no bonding between the particles and excellent fluidity. Their surfaces become no longer smooth, but slightly fluffy as the mass ratio of GAP increases. The reason of this phenomenon was that the solvent seeped out from the sphere in the driving solvent stage, and part of the GAP molecules were brought to the spherical powders surface with the solvent. This situation played

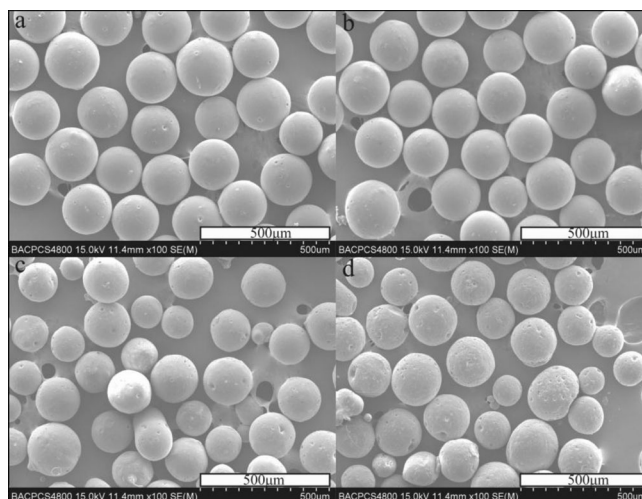


Figure 2. SEM micrographs of GAP-modified nitrocellulose spherical powders with different mass ratio of GAP (a: 0.0 % GAP; b: 10.0 % GAP; c: 20.0 % GAP; d: 30.0 % GAP).

a certain impediment for forming smooth spherical powders. However, this phenomenon was not very obvious, and did not affect the application of GAP-modified nitrocellulose spherical powders in propellants.

3.2 FT-IR Spectroscopic Characterization of GAP-Modified Spherical Powders

The GAP-modified nitrocellulose spherical powders were characterized by FT-IR spectroscopy, the main purpose is to test whether the characteristic functional groups (such as -N₃) of GAP can be found in the spherical powders. The results are shown in Figure 3.

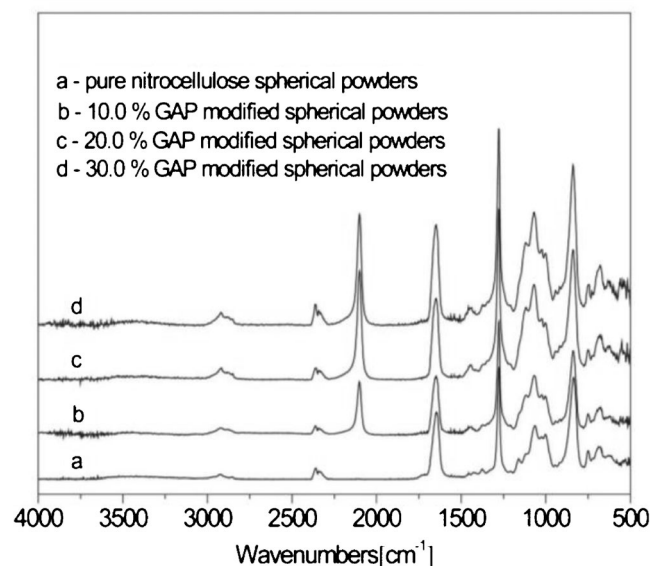


Figure 3. FT-IR spectra of GAP-modified nitrocellulose spherical powders.

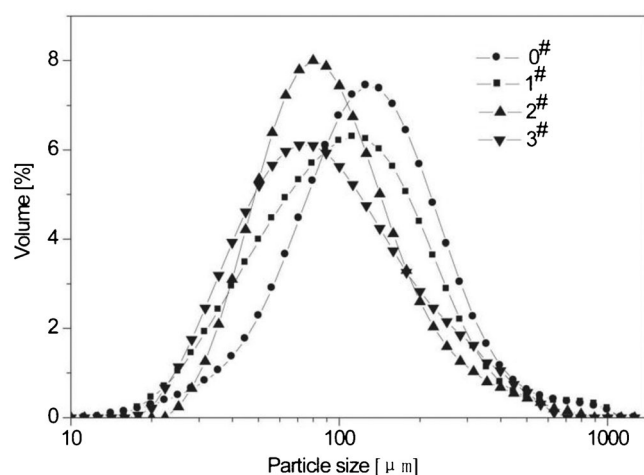


Figure 4. Particle size distribution of samples under different amount of GAP (0#: 0.0% GAP; 1#: 10.0% GAP; 2#: 20.0% GAP; 3#: 30.0% GAP).

Table 2. d_{50} Values, densities, and sensitivities to impact of GAP-modified NC spherical powders.

Sample number	0#	1#	2#	3#
GAP content [%]	0	10	20	30
d_{50} [μm]	134.53	109.96	94.23	94.54
Density [g cm ⁻³]	1.62	1.58	1.54	1.52
Sensitivity to impact [J]	7.12	8.56	10.40	10.90

As shown in Figure 3, there are characteristic bands at 3500 cm⁻¹ (ν(OH)), 1067 cm⁻¹ (ν(CO)), 832.5 cm⁻¹ (ν(O–NO₂)) and 747/681 cm⁻¹ (τ(O–NO₂)) for sample *a*. In the spectrum of sample *b*, in addition to the presence of the above characteristic bands, there are characteristic bands at 2100 cm⁻¹ and 1279 cm⁻¹ (asymmetrical and symmetrical –N₃ stretching). This effect is the same for the samples *c* and *d*. This is a proof that there are characteristic functional groups (such as –N₃) of GAP in the GAP-modified nitrocellulose spherical powders, and that GAP and nitrocellulose were composited together in the NC powders.

3.3 Particle Size Analysis and Density of GAP-Modified Nitrocellulose Spherical Powders

Particle size and particle size distribution are important indicators for the evaluation of spherical powders. The influences of the amount of GAP on the particle size and particle size distribution of GAP-modified powders were studied by laser particle size analysis. The test results are given in Figure 4 and Table 2.

Table 2 indicates that the mass ratio of GAP has a certain effect on d_{50} of modified powders. The surface tension of NC viscose droplets is reduced as the mass ratio of GAP increases, which led d_{50} of NC powders to smaller values. When the content of GAP in the NC powders reached

30.0%, the changing trends began to mitigate. Besides, with increasing amount of GAP, the density of modified powders is reduced due to the fact that the density of NC (1.66 g cm⁻³) [20] is higher than that of GAP (1.30 g cm⁻³). The particle size distribution (Figure 4) showed a single peak respectively and conformed to a normal distribution, which showed that the particle size range could be controlled preferably by the synthesis process.

3.4 TGA Characterization of GAP-Modified Nitrocellulose Spherical Powders

The samples modified with 10.0% GAP, and 20.0% GAP, and pure NC powders were subjected to TG-DTG measurements. The results are shown in Figure 5 and Table 3.

The first decomposition stage of the pure GAP showed a peak temperature at 250.8 °C in the DTG and a mass loss of 42.6%, this degradation step can be attributed to the side chain –N₃ decomposition reaction of GAP. The second decomposition stage referred to a carbonization process. The thermal decomposition process of pure NC powders only has one apparent stage and the onset of thermal decomposition takes place at 192.1 °C. The O–NO₂ group split

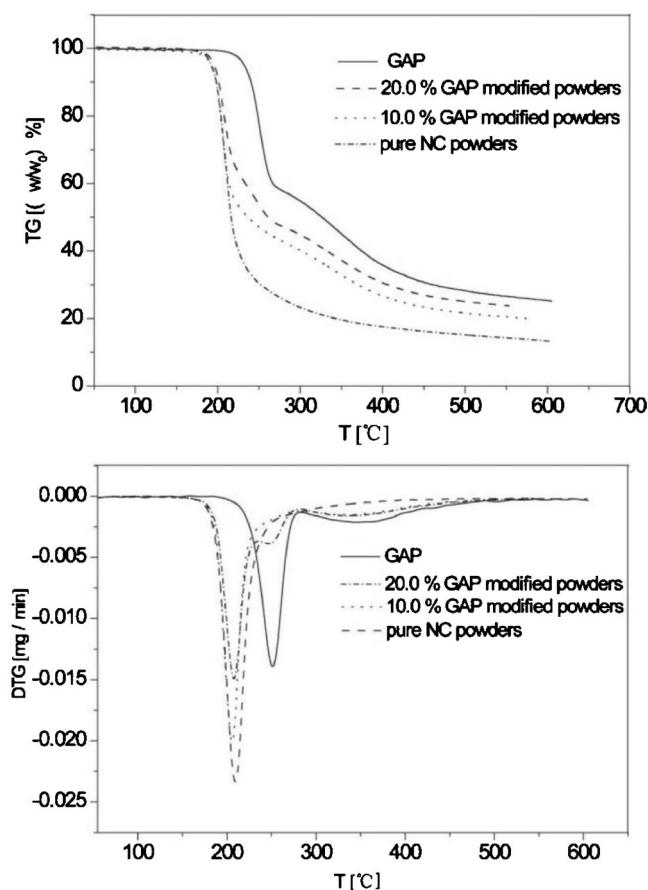


Figure 5. TG-DTG curves of GAP-modified powders (heating rate 5 K min⁻¹).

Table 3. The TG-DTG results of pure GAP, pure NC powders, and GAP-modified powders (heating rate 5 K min⁻¹)^a.

Decomposition stages	Pure GAP		Pure NC powders		10% GAP-modified NC powders		20% GAP-modified NC powders	
	T_p [°C]	Mass loss [%]	T_p [°C]	Mass loss [%]	T_p [°C]	Mass loss [%]	T_p [°C]	Mass loss [%]
1	250.8/ 232.0 ^b	42.6	209.4/ 192.1 ^b	86.3	206.2/ 192.0 ^b	56.6	205.1/ 191.6 ^b	40.7
2	352.9	15.5	–	–	349.4	23.2	249.0	12.2
3	–	–	–	–	–	–	347.5	–

a) T_p = decomposition peak temperature. b) Onsets of thermal decomposition temperature.

into NO₂ at the decomposition peak temperature at 209.4 °C with a mass loss of 86.3% up to 580.0 °C, followed by a slow decomposition of their skeletons. The thermal decomposition process of 10% GAP-modified powders has two apparent stages and the onset of thermal decomposition takes place at 192.0 °C. The first decomposition stage showed a peak temperature at 206.2 °C in the DTG. A mass loss of 56.6% in this degradation step can be attributed to the split of the O–NO₂ group in NC. This should be followed immediately by the thermal decomposition of GAP–N₃ groups, but since the amount of GAP is small, the peak is not obvious. The second decomposition stage shows a peak temperature at 349.4 °C in the DTG and a mass loss of 23.2%. This degradation step can be attributed to the main chain decomposition reaction of GAP and NC. The decomposition peak temperature (352.9 °C) from the main chain of pure GAP is 3.5 °C higher than the decomposition peak temperature (349.4 °C) from the GAP main chain contained in 10% GAP-modified powders. The phenomenon can be attributed to the decomposition reaction of NC, which had reached the maximum rate at 209.4 °C. A large number of NO₂ or HONO groups remained in the GAP and NC chain skeleton, which catalyze the thermal decomposition of the GAP skeleton by splitting the ether bonds. In addition, the heat released by the decomposition of the O–NO₂ group might also catalyze the thermal decomposition of the GAP skeleton, and shift the peak temperature to lower values. For 20% GAP-modified powders, the peak temperature of GAP–N₃ decomposition and its main chain are 249.0 °C and 347.5 °C, respectively, which are 1.8 °C and 5.4 °C lower than the corresponding decomposition peak temperature of pure GAP. This is consistent with the thermal decomposition results of 10% GAP-modified powders. Besides, the onsets of thermal decomposition temperature for 10.0%, 20.0% GAP-modified powders, and pure NC powders are found to be only slightly different. It shows that the pure NC powders' sensitivity and hazard properties are close to those of the GAP-modified powders.

3.5 Impact Sensitivity

The impact sensitivity of GAP-modified nitrocellulose spherical powders was characterized by drop weight tests, the results are listed in Table 2.

It can be seen that the sensitivity of GAP-modified nitrocellulose spherical powders is reduced significantly as the mass ratio of GAP increases due to plastification of the nitrocellulose powders. Therefore, it appears that the energetic binder GAP can be used for reducing the sensitivity of nitrocellulose spherical powders, and might be useful to improve the safety of propellant preparation processes.

3.6 Fluidity and Impact Sensitivity of the Propellant Slurries

After mixing in a vertical mixer with 50 rpm at 48 °C for 30 min, a small amount of propellant slurry was taken into a Teflon beaker to watch the fluidity (Figure 6). For the GAP-modified NC balls the composition of the propellants were treated in the same way. The results showed that the viscosity of the propellant slurry *a* with pure NC balls was low, but its plasticizing and curing quality was poor. As the mixing process continues, the viscosities of propellant slurries *b* with 10% GAP/NC balls and *c* with 20% GAP/NC balls increased, but their plasticizing qualities were good. Obviously, NC was pre-plasticized by GAP, the growth of a solid-liquid interface was enhanced and the plasticizing of the GAP-modified NC balls was improved. Thus, the quality of the propellant slurries was improved too. In addition, the curing process was monitored by measuring the increasing viscosity of the propellant slurry at 60 °C with a rotational rheometer. As expected, the curing process of the propellant slurries *b* and *c* was much faster than that of *a* (Figure 7). All three curves showed only a moderate increase at the beginning and after a certain time of curing suddenly a sharp viscosity increase. Though the viscosity increase of the propellant slurries with GAP-modified NC balls occurred much faster, the pot life was still sufficient for many applications.

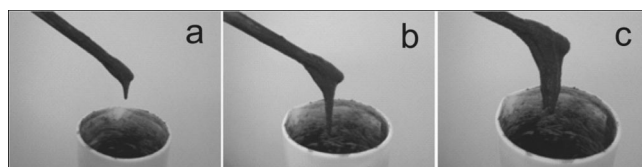


Figure 6. Fluidity of the propellant slurries (containing *a*: NC fluid balls with 0.0% GAP; *b*: NC fluid balls modified with 10.0% GAP; *c*: NC fluid balls modified with 20.0% GAP).

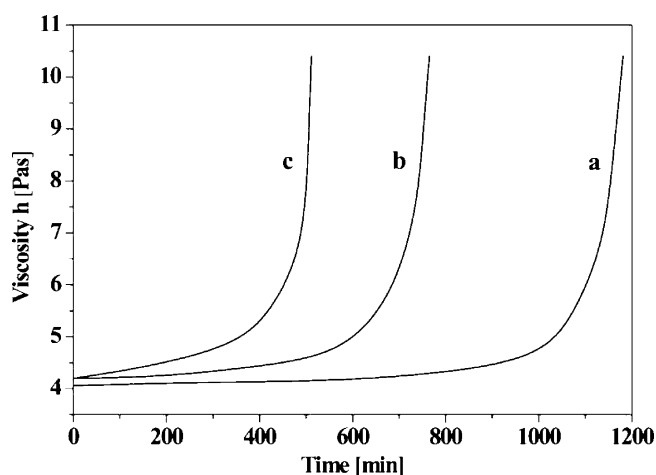


Figure 7. Viscosity increase of different propellant slurries during curing at 60° (*a*: NC fluid balls with 0.0% GAP; *b*: NC fluid balls modified with 10.0% GAP; *c*: NC fluid balls modified with 20.0% GAP).

The impact sensitivities of the propellants were characterized by drop weight tests, the results are listed in Table 4. It shows that the sensitivities of propellants *b* and *c* with 10% and 20% GAP in the NC balls are lower than that of propellant *a*, which indicates that the mechanical sensitivity of CDB propellants could be reduced by using GAP-modified NC balls.

3.7 Effects of GAP-Modified Nitrocellulose Spherical Powders on the Mechanical Characteristics of the Propellant

The mechanical characteristic of cross-linked modified double base propellants with GAP-modified nitrocellulose spherical powders are listed in Table 5, the microstructures of samples *a* with 0%, sample *b* with 10% and sample

Table 4. Sensitivities to impact of propellant slurries^{a)}.

Sample number	<i>a</i>	<i>b</i>	<i>c</i>
Sensitivity to impact [J]	6.85	8.06	10.08

a) Samples: *a*=NC fluid balls with 0.0% GAP; *b*=NC fluid balls modified with 10.0% GAP; *c*=NC fluid balls modified with 20.0% GAP.

Table 5. Effects of GAP-modified nitrocellulose spherical powders on the mechanical properties of the appropriate XLDB propellants^{a)}.

Sample	−40 °C			+20 °C			+50 °C		
	σ_m [MPa]	ε_m [%]	<i>E</i> [MPa]	σ_m [MPa]	ε_m [%]	<i>E</i> [MPa]	σ_m [MPa]	ε_m [%]	<i>E</i> [MPa]
<i>a</i>	0.71	25.2	5.02	0.36	33.0	2.42	0.31	32.4	2.07
<i>b</i>	0.92	28.8	6.13	0.44	43.4	2.98	0.36	40.9	2.11
<i>c</i>	1.10	29.1	7.24	0.51	51.8	3.28	0.41	51.5	2.34

a) Sample *a*=propellant containing pure NC powders. Sample *b*=propellant containing 10% GAP-modified nitrocellulose spherical powders. Sample *c*=propellant containing 20% GAP-modified nitrocellulose spherical powders. σ_m as a maximum tensile strength, ε_m as an elongation at maximum tensile strength, cross head speed: 100 mm min^{−1}.

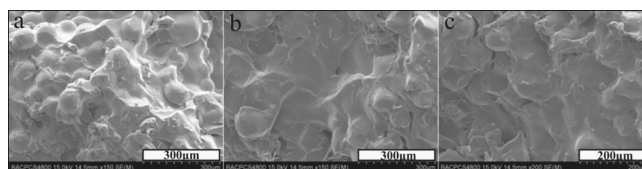


Figure 8. Microstructures of fracture surface of propellant samples containing GAP-modified NC powders with different amounts of GAP (*a*: NC fluid balls with 0.0% GAP; *b*: NC fluid balls modified with 10.0% GAP; *c*: NC fluid balls modified with 20.0% GAP).

c with 20% GAP are shown in Figure 8. From Table 5, it can be seen that the maximum tensile strengths (σ_m) of samples *b* and *c* are larger than those of sample *a* at temperatures of −40 °C, +20 °C, and +50 °C. The elongations at maximum tensile strengths (ε_m) of samples *b* and *c* did not change significantly at −40 °C (25.2% < ε_m < 29.1%). But ε_m has greatly improved at temperatures of +20 °C and +50 °C. It was also found that pure NC powders of sample *a* were not plasticized completely (photograph *a* of Figure 8), and their contour profiles were relatively clear. The GAP-modified nitrocellulose powders of samples *b* and *c* were plasticized better than that of sample *a* (photographs *a* and *c* of Figure 8), which was demonstrated by their contour profiles being blurred. This kind of structure may greatly increase ε_m and σ_m of the propellant. The reason might be that the GAP-modified nitrocellulose powders could be plasticized sufficiently due to the pre-plasticizing effect of GAP. If the GAP/NC balls are better plasticized the remaining hydroxyl (−OH) groups from NC in the modified powders form a higher cross-linking density by reacting with the isocyanate groups (−NCO) from N100. The higher amount of GAP accelerates the plasticizing speed of modified nitrocellulose powders, but it shortens the “pot life” of the propellant slurry. Thus only an appropriate amount of 10% to 20% GAP in modified powders will be useful to improve the mechanical characteristics of the propellant.

4 Conclusions

SEM showed that GAP-modified nitrocellulose powders had high roundness rate, no bonding between the particles,

and excellent fluidity. FT-IR spectroscopic characterization indicated that GAP and nitrocellulose were composited together in the NC powders. Increasing the amount of GAP in the modified nitrocellulose powders is useful for improving its plasticizing ability.

TG-DTG measurements showed that the thermal decomposition reactions of GAP shifted to lower temperatures in the thermal decomposition process of modified nitrocellulose powders with 10.0% GAP and 20.0% GAP. The first thermal decomposition peak temperatures of NC/GAP composites are 3.2 °C and 4.3 °C lower than the corresponding peak temperatures of GAP compositions with pure NC and the second decomposition peaks are 3.2 °C and 4.3 °C lower than the main chain decomposition peaks of pure GAP respectively.

The impact sensitivities of GAP-modified NC spherical powders are reduced by GAP. The modified NC powders can improve the quality of propellant slurries and the mechanical properties of the propellants giving maximum tensile strengths (σ_m) between 0.36 MPa < σ_m < 1.10 MPa and elongations at maximum tensile strength (ε_m) between 28.8% < ε_m < 51.8% in the temperature range from -40 °C to +50 °C.

Symbols and Abbreviations

d_{50}	Median diameter [μm]
σ_m	Maximum tensile strength [MPa]
ε_m	Elongation at maximum tensile strength [%]
E	Elastic module
DTG	Differential thermogravimetry
DINA	Dinitroxyethylnitramine
GAP	Glycidyl azide polymer
XLDB	Cross-linked modified double base
P(EO-THF)	Copolyether of ethylene oxide and tetrahydrofuran
NC	Nitrocellulose
NG	Nitroglycerin
RDX	Cyclotrimethylenetrinitramine
CARBON	Carbon black
N100	Triisocyanate
TPB	Triphenylbismuthine
C ₂	Centralite II
SEM	Scanning electron microscopy
TGA	Thermal gravimetric analysis

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