


Article

Thermal Analysis and Stability of Boron/Potassium Nitrate Pyrotechnic Composition at 180 °C

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Featured Application: This research aims to obtain the decomposition process of boron/potassium nitrate pyrotechnic composition, and verify its high temperature stability to meet the charge requirements of the separation device such as the cutter for the lunar probe.

Abstract: Aerospace missions require that pyrotechnic compositions are able to withstand 180 °C. Therefore, this paper studies the thermal stability and output performance of boron/potassium nitrate (abbreviated BPN) used in pyrotechnic devices. Firstly, differential scanning calorimetry (DSC) and thermogravimetric (TG) tests are used to analyze the thermal reaction process of KNO₃, boron, and BPN to qualitatively judge their thermal stability. Then, apparent morphology analysis, component analysis, and the p-t curve test, which is the closed bomb test to measure the output power of the pyrotechnic composition, are carried out with BPN samples before and after the high-temperature test to verify BPN stability at 180 °C. The weight change of boron powder caused by chemical reactions occurs above 500 °C. When the temperature is lower than the peak exothermic temperature of decomposition, no obvious chemical reaction occurs with KNO₃, and only physical changes (crystal transformation and melting) occur. Combined with a verification test at 180 °C for two days, it is concluded that boron and KNO₃ components are stable at 180 °C. With an increase in boron content, the thermal stability of BPN is improved, with the best performance achieved when the ratio is 25:75 (B:KNO₃). BPN samples without binder have the best thermal stability. In a test at 180 °C for five days, the binder affects the weight loss and p-t curve of BPN, and BPN with fluororubber binder is better than BPN with unsaturated polyester binder.

Keywords: boron/potassium nitrate; thermal analysis; high temperature stability; energetic material

1. Introduction

Boron/potassium nitrate (B/KNO₃; abbreviated BPN) can be used for the output charge of an engine igniter, a fire transfer charge, a small thrust dynamite output charge, a high temperature projectile charge, etc. BPN has the significant characteristics of high combustion heat and low moisture absorption per unit weight. [1] BPN is also listed as a linear reference for the security of pyrotechnic agents [2].

At present, aerospace missions require that BPN composition is thermally stable. In some special launch missions [3], such as of deep space spacecraft, pyrotechnic devices must withstand a large temperature range, usually from −100 to +130 °C [4,5]. Considering that there is a certain safety

margin for temperature, it is of great significance to test and evaluate the thermal stability and output performance of BPN after exposure to the temperature of 180°C, providing a basis for its application in the aerospace field. This research aims to obtain the decomposition process of boron/potassium nitrate pyrotechnic composition, and verify its high temperature stability to meet the charge requirements of the separation device such as the cutter for the lunar probe.

K.R. Rani Krishnan et al. studied the effect on the ignition behavior of potassium boron nitrate pyrotechnic powder of adding RDX and HMX components which are two commonly used high energy explosives. In their paper, thermogravimetric (TG) and differential scanning calorimetry (DSC) analyses which use Perkin Elmer TGA 7 and DSC 7 in flowing argon medium (30 mL min) at 5 °C/min, were carried out on the mixture of potassium boron nitrate and explosive components [6], which provided some inspiration for the present paper. R. Turcotte et al. researched the thermal analysis of black gunpowder in which KNO₃ had a pre-ignition reaction under the action of sulfur and charcoal. The pre-ignition reaction occurred at a lower temperature than the decomposition temperature of KNO₃ itself, and the decomposition temperature of KNO₃ drifted under a complex system [7,8]. ES. Freeman, Seyed Ghorban Hosseini et al. [9,10] studied the thermal decomposition process of KNO₃ by the TG-TDA method, and obtained the transformation, melting and decomposition process of KNO₃. A. Eslami et al. [11] studied the thermal reaction characteristics of B+KNO₃, B+Ba(NO₃)₂, B+PbO₂ and other mixed systems, and the reaction process of a B+KNO₃ mixed system was analyzed in stages.

The stability of chemical agents refers to the ability of a sample to maintain its physical, chemical and explosive properties from changing beyond the permitted range under certain circumstances [12]. Stability analysis of physical properties mainly encompasses apparent morphology, color, weight, crystal shape, phase change (melting point, boiling point, volatilization), moisture absorption, particle size, charge density, crystal crack, volatilization, oil permeability, migration, and dielectric constant. Chemical stability encompasses purity, composition, valence state, oxidation state, slow/rapid thermal decomposition, weight loss, and composition of explosive products. The stability of explosion performance encompasses explosion point, detonation heat, detonation velocity, combustion velocity, p-t curve (which is the closed bomb test to measure the output power of the pyrotechnic composition), detonation pressure, detonation capacity, flame sensitivity, hot wire sensitivity, impact sensitivity, friction sensitivity, static inductance, initiation sensitivity, and shock wave sensitivity. In addition to stability, compatibility is also a consideration. This is particularly true of chemical compatibility, including the internal compatibility between the pyrotechnic charge and contact metal (bridge wire, tube shell, cover sheet, motion mechanism, etc.).

At present, there are few reports on the thermal reaction process and high temperature stability. Existing studies of BPN mainly focus on the influence of the preparation process [13,14], particle size [15], ratio [16] and other factors related to ignition performance.

2. Materials and Methods

2.1. Materials

Laboratory reagent grade boron and KNO₃ powder were all purchased for tests. The particle size of powdered boron was approximately 1 μm, and KNO₃ was 99.5% pure. As shown in Table 1, using boron powder and KNO₃ as raw materials, BPN samples with different proportions were prepared.

Table 1. Samples of pure components and mixtures.

No.	Component	Composition (%)	Binder
1	Boron	100	–
2	KNO ₃	100	–
3	1#BPN	15:85	–
4	2#BPN	25:75	–
5	3#BPN	33:67	–
6	4#BPN	25:75	Fluorine rubber
7	5#BPN	25:75	Unsaturated polyester

2.2. Methods

The thermal stability of BPN after exposure at 180 °C was studied by methods such as thermogravimetric analysis and differential scanning calorimetry, and analysis of appearance morphology, weight loss, content, and output performance. The process scheme is shown in Figure 1.

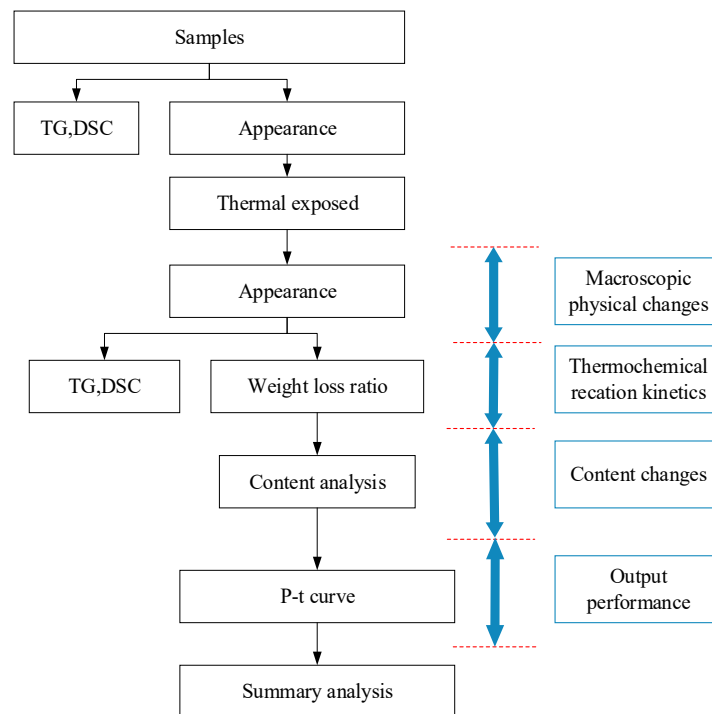


Figure 1. Experimental process scheme.

TG and DSC test data were applied to analyze the thermal reaction process of KNO₃, boron powder and BPN with different ratios, to qualitatively judge high-temperature stability. The apparent morphology analysis, thermogravimetric (TG) analysis, differential scanning calorimetry (DSC) analysis, and output power test were carried out on BPN samples after the high-temperature test to verify the stability of BPN under a high temperature of 180 °C, thereby providing a basis for its application in the aerospace field. A summary of the analysis method is presented in Table 2.

Table 2. Analysis methods for BPN thermal analysis and high temperature stability analysis.

Analysis Method	Content
Thermogravimetry	Measure the weight of loose sample before and after high temperature test with precision balance.
Appearance	Scanning microscope, optical digital microscope used to observe the color, particle size, etc.
DSC analysis	Analyze the initial decomposition temperature and peak decomposition temperature of samples.
Content analysis	Ion exchange method for determination of KNO ₃ content Mannitol complex titration method for determination of boron.
output performance	P-t curve pressure peak and rise time to evaluate the power capacity of samples.

3. Results and Discussion

3.1. Thermal Analysis of Single Component

The TG process of boron was studied with a nitrogen atmosphere. As shown in Figure 2, a weight loss of approximately 3.3% is observed from heating to 117.5 °C, which reflects the loss of moisture and volatiles in boron powder. Until 500 °C, the quality of boron powder samples increases gradually, and the speed increases after 650 °C. The total mass increases to approximately 118% at 1000 °C. It is speculated that the reaction in this stage is the slow reaction of boron powder and nitrogen, and the final product is BN. The reaction equation is

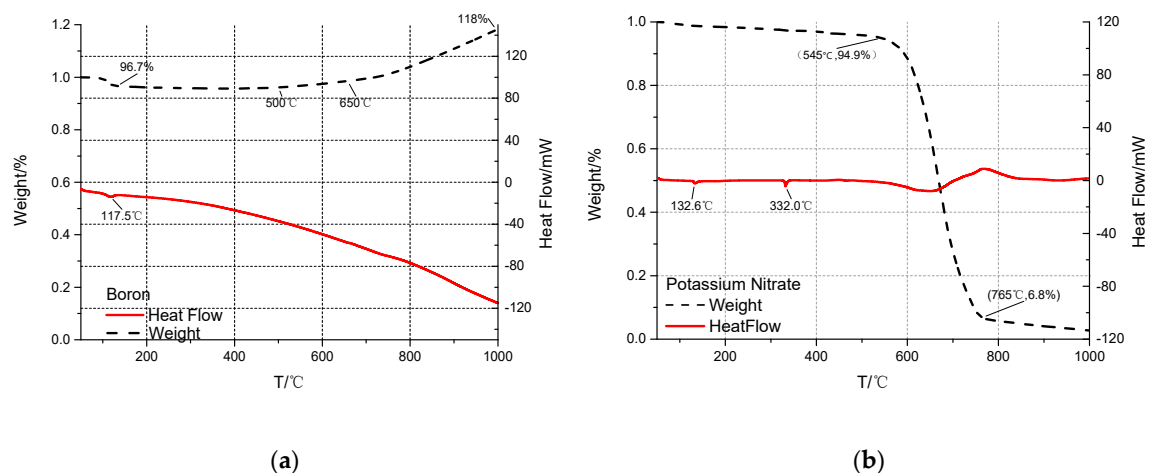


Figure 2. (a) Thermogravimetric-Differential Scanning Calorimetry (TG-DSC) curve of boron powder; (b) TG-DSC curve of KNO₃ (in nitrogen atmosphere).

The DSC curve of KNO₃ has two endothermic peaks. The first endothermic peak (peak temperature 132.6 °C) corresponds to the conversion temperature of KNO₃, which is the phase transition of KNO₃ from rhombic to trigonal. The second endothermic peak (peak temperature 332.0 °C) corresponds to the melting point of KNO₃. [9] The DSC curve shows that the decomposition of KNO₃ occurs at more than 500 °C. Combined with the TG curve, the mass change of KNO₃ is 5.1% at 545 °C, indicating that KNO₃ is melting but is not decomposed or gasified from the melting point (332.0 °C) to the temperature of 500 °C. Therefore, the physical stability of KNO₃ changes within the temperature range of 332–500 °C, but the chemical stability is not affected.

A weight loss of KNO₃ of 88.1% mainly occurred between 545 °C and 765 °C. According to E.S. Freeman [17], the decomposition of KNO₃ took place at this stage, generating nitrite, followed by the

decomposition reaction of potassium nitrite, and the product K_2O was reported to evaporate (m.p. $380\text{ }^\circ\text{C}$) [10]. The equation of weightlessness in this stage is [8]



Compared with the operating condition of $180\text{ }^\circ\text{C}$, the melting temperature of KNO_3 at $336.5\text{ }^\circ\text{C}$ is about $156\text{ }^\circ\text{C}$ higher. No obvious chemical reaction occurs before the peak temperature of decomposition (i.e., more than $545\text{ }^\circ\text{C}$). It can be qualitatively concluded that the boron powder and KNO_3 components are stable at a high temperature of $180\text{ }^\circ\text{C}$. It should be noted that this result is obtained according to the reaction temperature of DSC, and the stability of samples at constant temperature needs to be verified.

3.2. Thermal Analysis of BPN with Different Ratios

TG-DSC analysis of BPN samples with different ratios (heating rate $5\text{ }^\circ\text{C}/\text{min}$) were carried out, and the curves are shown in Figure 3. For BPN samples with different ratios, the thermal reaction process is similar to that of KNO_3 before $500\text{ }^\circ\text{C}$.

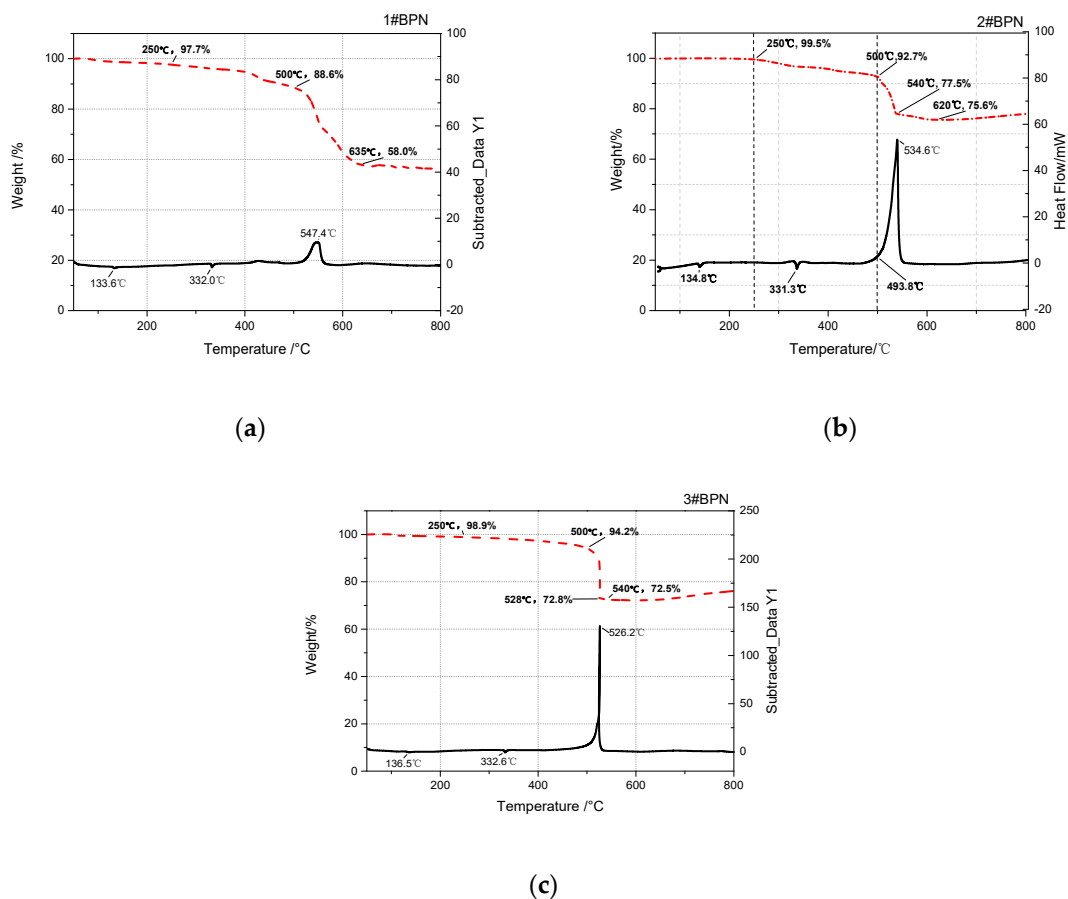


Figure 3. (a) TG–DSC curve of 1#BPN (15:85); (b) TG–DSC curve of 2#BPN (25:75); (c) TG–DSC curve of 3#BPN (33:67) (heating rate $5\text{ }^\circ\text{C}/\text{min}$).

As shown in Figure 3, the first endothermic peak (approx. $134\text{ }^\circ\text{C}$) corresponds to the conversion temperature of KNO_3 , and the second endothermic peak (approx. $332\text{ }^\circ\text{C}$) corresponds to the melting point of KNO_3 . Before $500\text{ }^\circ\text{C}$, the mass change of sample 1# and 2# is about 9%, while that of sample 3# is less than 5%. Between $500\text{ }^\circ\text{C}$ and $650\text{ }^\circ\text{C}$, there is a drastic decomposition reaction, and the

sample weight drops sharply. The temperature of the decomposition reaction is about 200 °C higher than the melting point. The main reaction at this stage is



The weight ratio results of BPN samples with different proportions at typical temperatures are shown in Table 3. At 200 °C, the mass variation of BPN samples with three ratios are in the range 0.9%–1.7%, and at 500 °C, 5.8%–11.4%. At 650 °C, the 2# (25:75) BPN sample (zero oxygen balance ratio) has the least weight loss. Based on TG analysis of KNO₃, the initial reaction temperature of BPN (500 °C) is about 50 °C lower than that of KNO₃ (545 °C). However, when comparing the endothermic peak temperature of DSC curves of samples, the exothermic peak temperature showed a trend of rising with the increase of boron content, as shown in Table 4.

Table 3. Weight statistics of potassium boron nitrate samples at different temperatures.

Samples	Weight Ratio @ 100 °C (%)	Weight Ratio @ 200 °C (%)	Weight Ratio @ 400 °C (%)	Weight Ratio @ 500 °C (%)	Weight Ratio @ 650 °C (%)
1#BPN (15:85)	99.3	98.3	94.8	88.6	57.5
2#BPN (25:75)	99.9	98.5	93.2	92.7	75.7
3#BPN (33:67)	100.0	99.1	97.4	94.2	72.5

Table 4. Peak temperature of DSC with different ratios of BPN.

Samples	First Endothermic Peak (°C)	Second Endothermic Peak (°C)	Exothermic Peak (°C)
1#BPN (15:85)	133.6	332.0	547.4
2#BPN (25:75)	134.8	331.3	534.6
3#BPN (33:67)	136.5	332.6	526.2

The results show that with an increase of boron content, the exothermic peak is advanced, but the thermal stability of BPN samples are improved, with the 2#BPN sample (25:75) showing the best performance. Analysis at 180 °C shows that the DSC decomposition temperature of BPN is above 500 °C, and TG weight loss ranges from 5.8% to 11.4% at 500 °C. It can be qualitatively judged that BPN is stable at 180 °C.

3.3. Thermal Exposure Test at 180 °C and Analysis

After drying at 60 °C for 4 h, the samples of BPN, boron powder and KNO₃ were placed in a temperature environment testing instrument for a thermal exposure test. The temperature settings were 180 °C for two and five days. The apparent morphology, DSC parameters, weight loss ratios and output performance of samples before and after the test were compared and analyzed.

Apparent Morphology

The apparent morphologies of KNO₃, boron and 4#BPN samples before and after different temperature storage are shown in Figures 4–6.

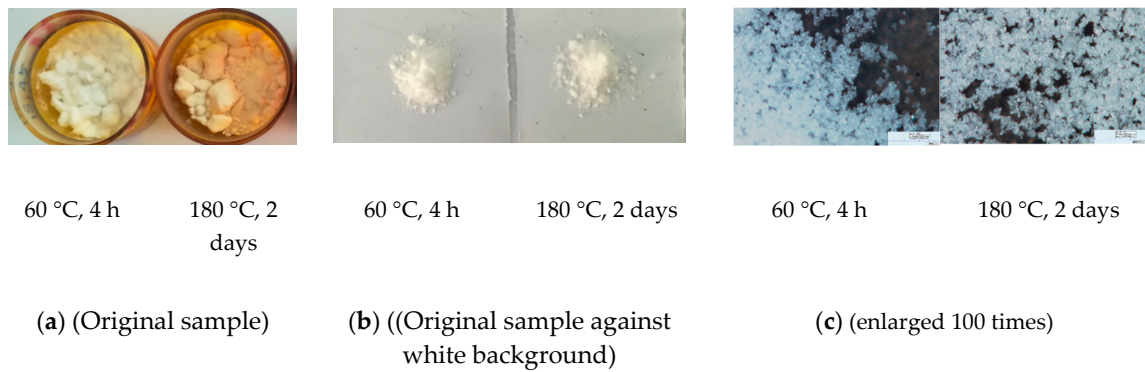


Figure 4. The apparent morphology of KNO_3 compared before and after different temperature tests.

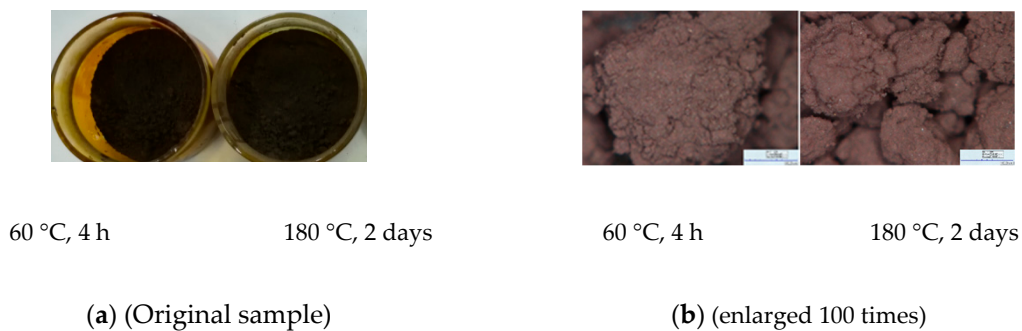


Figure 5. The apparent morphology of boron compared before and after different temperature tests.

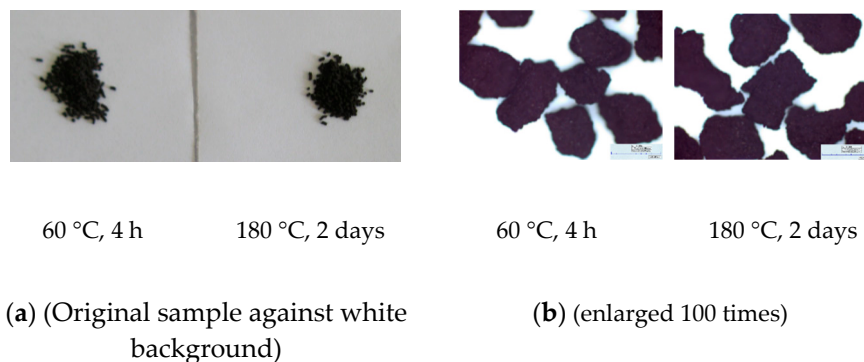


Figure 6. The apparent morphology of 4#BPN compared before and after different temperature tests.

After observing the boron powder, KNO_3 and 4#BPN samples before and after high temperature, it is found that the color of the KNO_3 sample changes slightly from white to yellow, and the particle size and shape do not change. The particle size, shape and color of boron powder and 4#BPN are not observed to change. In addition, there is no change in appearance quality, such as expansion of fractures, loose pits, shrinkage or melting residue.

DSC test

The DSC curves and DSC parameter comparison of 4#BPN sample are given in Figure 7 and Table 5 respectively.

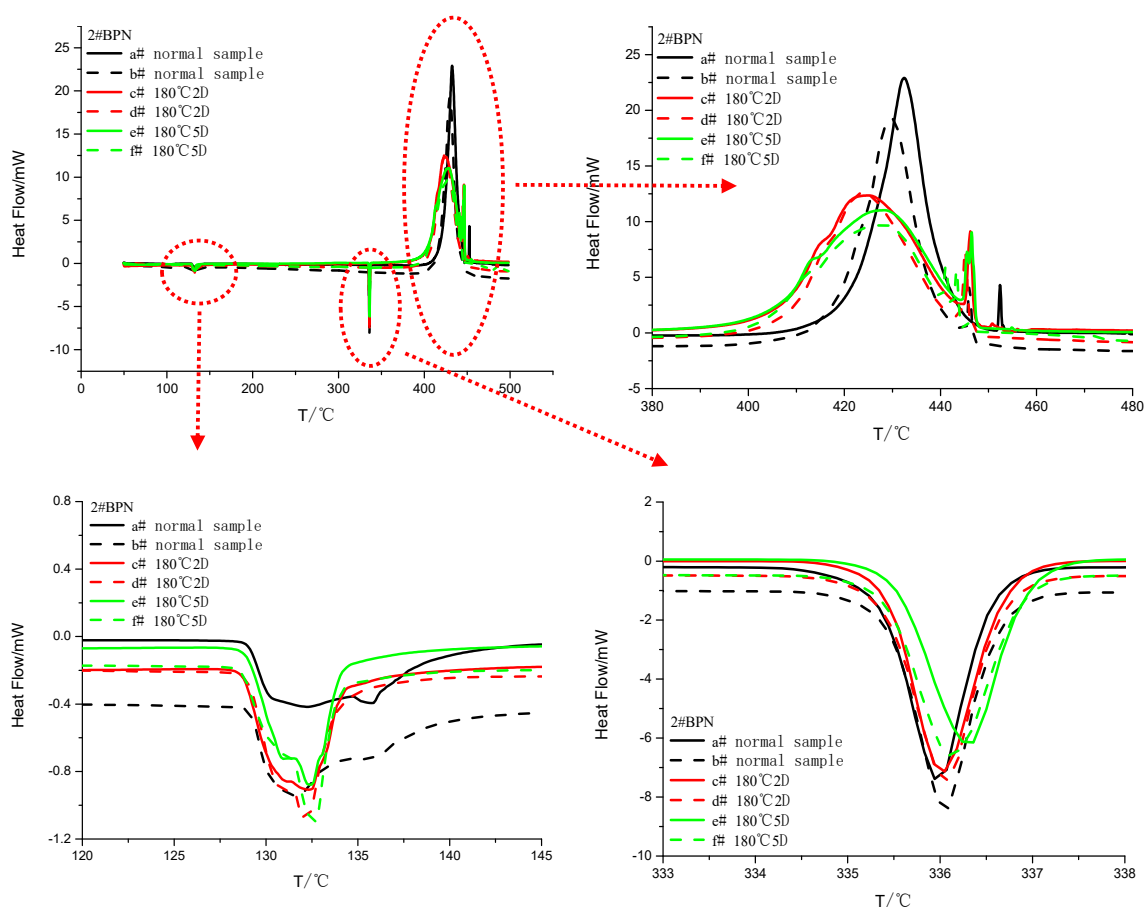


Figure 7. DSC curves of 4#BPN 60 °C, 4 h samples and 180 °C samples (heating rate 5 °C/min).

Table 5. DSC parameter comparison of 4#BPN sample (°C) (heating rate 5 °C/min).

Parametric	60 °C, 4 h	180 °C, 2 days	180 °C, 5 days
Initial temperature of first endothermic peak (°C)	129.04	129.45	128.84
First endothermic peak (°C)	131.15	131.35	131.55
Initial temperature of second endothermic peak (°C)	334.95	335.12	335.35
Second endothermic peak (°C)	336.02	336.06	336.23
Initial temperature of exothermic peak (°C)	405.34	406.37	404.33
Exothermic peak (°C)	431.28	424.77	428.51
Decomposition heat E (J·g ⁻¹)	3447	3454	3367
Energy loss rate ¹ (%)	0	1.01	3.26

¹ Energy loss rate: $(E_{60^{\circ}\text{C}4\text{h}} - E_{180^{\circ}\text{C}, \text{x d}}) / E_{60^{\circ}\text{C}4\text{h}}$.

According to the DSC analysis of the variation of the peak temperature, the temperature difference between the first endothermic peak and the second endothermic peak is very small, comparing the samples at 180 °C for two days, 180 °C for five days, and 60 °C for 4 h; $\Delta T < 1^{\circ}\text{C}$. The exothermic peak temperature variations of the samples at two days and five days are -6.51°C and -2.77°C , respectively, but the failure rates of the agents are 1.01% and 3.26%, respectively. This indicates that the exothermic peak temperature of the sample is advanced, but the energy is not consumed. It can be preliminarily concluded that BPN containing KNO_3 and boron powder has good thermal stability at 180 °C for two days and five days.

- Weight loss ratio

The weight loss ratios of BPN samples in 180 °C test are given in Table 6. Furthermore, the component variation of BPN samples within 180 °C test are given in Table 7.

Table 6. Weight loss ratios of BPN samples in 180 °C test.

Sample ¹	Weight Loss Ratio after 60 °C, 4 h (%)	Weight Loss Ratio after 180 °C, 2days (%)	Weight Loss Ratio after 180 °C, 5days (%)
1#BPN (15:85)	0.097	0.12	0.11
4#BPN (25:75)	0.372	1.04	0.98
5#BPN (25:75)	0.095	1.19	1.59
KNO ₃	0.0083	0.044	–
Boron	1.549	5.26	–

¹ Binders of 4#BPN, 5#BPN are fluorine rubber, and unsaturated polyester, respectively.

Table 7. Component variation of BPN samples in 180 °C test [18].

Sample	Component	60 °C, 4 h (%)	180 °C, 2 days (%)	180 °C, 5 days (%)
1#BPN (15:85)	KNO ₃ (%)	85.80	85.66	85.90
	Boron (%)	14.87	14.88	14.85
4#BPN (25:75)	KNO ₃ (%)	73.32	73.79	74.36
	Boron (%)	23.04	27.38	26.24

As shown in Table 6, from the comparison of the weight loss ratios of BPN samples in the 180 °C test for different times, after five days, the weight loss ratio of 1# BPN without binder is approximately 0.1%, that of 4#BPN containing fluororubber binder is approximately 1%, and that of 5# BPN containing unsaturated polyester is approximately 2%. Combined with the analysis of component content, the change of KNO₃ and boron in 1#BPN without binder in the 5-day 180 °C test is 0.5%, i.e., almost no change. Thus, it can be judged to some extent that the stability of BPN in which the main components are KNO₃ and boron is well under 180 °C for two days and five days. The reason for the component variation of 4#BPN and 5#BPN is the binder (i.e., 4#BPN contains 3% fluororubber and 5#BPN contains 5.6% unsaturated polyester).

- P-t curve

P-t curve data of BPN samples in 180 °C test is given in Table 8.

Table 8. P-t curve of BPN samples in 180 °C test [19,20].

Sample	Component	Peak Pressure (MPa)	Rise Time (ms)	Rate of Peak Pressure Change (%)
1#BPN (15:85)	60 °C, 4 h	2.11	3.09	−0.95
	180 °C, 5 days	2.09	2.94	
4#BPN (25:85)	60 °C, 4 h	2.57	1.82	−10.89
	180 °C, 5 days	2.29	1.33	
5#BPN (25:75)	60 °C, 4 h	3.34	4.09	−25.75
	180 °C, 5 days	2.48	4.59	

It can be seen from the data in Table 8 that the peak pressure of the three kinds of BPN decreases with increasing time of the 180 °C temperature test. The thermal analysis of the single component shows that boron and KNO₃ are relatively stable at 180 °C, and the peak pressure attenuation of the 1#BPN sample without binder is 0.95% (i.e., <1%), which also supports this argument. It is also known that the binder has a great impact on the high temperature stability of BPN. The high temperature stability of the fluororubber binder is better than that of unsaturated polyester.

4. Conclusions

In conclusion, we studied the thermal decomposition process of KNO₃, boron and BPN based on TG and DSC test data. The weight change of boron powder caused by chemical reaction occurred above 500 °C. When the temperature was lower than the peak exothermic temperature of decomposition, no

obvious chemical reaction occurred with KNO_3 , and only physical changes (crystal transformation and melting) occurred. Combined with a verification test at $180\text{ }^\circ\text{C}$ for two days, it is concluded that boron and KNO_3 components are stable at $180\text{ }^\circ\text{C}$. With the increase of boron content, the thermal stability of BPN sample improved, and the best performance was achieved when the ratio was 25:75 (B: KNO_3).

BPN samples without binder have the best thermal stability. In the 5 day $180\text{ }^\circ\text{C}$ test, the binder affected the weight loss and p-t curve of BPN. Finally, BPN with fluororubber binder is better than BPN with unsaturated polyester binder.

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Conflicts of Interest: The authors declare no conflict of interest.

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