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Thermal behaviors of a novel nitrogen-rich energetic compound

Hydrazinium 3,5-dinitroamino-1,2,4-triazole

Yan-Feng Li[1] · Lian-Jie Zhai[2] · Kang-Zhen Xu[1] · Bo-Zhou Wang[2] ·
Ji-Rong Song[1] · Feng-Qi Zhao[2]

Abstract A new nitrogen-rich energetic material, hydrazinium 3,5-dinitroamino-1,2,4-triazole (HDNAT), was synthesized. Thermal behavior and non-isothermal decomposition kinetics of HDNAT were studied with DSC and TG/DTG methods. The non-isothermal decomposition kinetic equation is $\frac{d\alpha}{dT} = \frac{10^{19.37}}{\beta} 6(1-\alpha)^{2/3}[1-(1-\alpha)^{1/3}]^{1/2}$ $\exp(-188.6 \times 10^3/RT)$. Self-accelerating decomposition temperature and critical temperature of thermal explosion of HDNAT are 168.1 and 178.1 °C, respectively. Specific heat capacity of HDNAT was determined with a micro-DSC method and the molar heat capacity is 258.96 J mol^{-1} K^{-1} at 298.15 K. Adiabatic time-to-explosion of HDNAT is about 100 s. The impact sensitivity, friction sensitivity, detonation velocity and detonation pressure of HDNAT are >13.1 J, 84 %, 9.0 km s^{-1} and 36.0 GPa, respectively.

Keywords 3,5-Dinitroamino-1,2,4-triazole · Hydrazinium salt · Non-isothermal decomposition kinetics · Specific heat capacity · Adiabatic time-to-explosion

✉ Kang-Zhen Xu
  xukz@nwu.edu.cn

✉ Bo-Zhou Wang
  wbz00@163.com

1   College of Chemical Engineering, Northwest University,
    Xi'an 710069, People's Republic of China

2   Xi'an Modern Chemistry Research Institute, Xi'an 710065,
    People's Republic of China

Introduction

In recent years, high energy density materials (HEDMs) have attracted considerable interests because of their excellent detonation properties and low sensitivities [1, 2]. Nitrogen-rich energetic materials are an important category of advanced HEDMs. Nitrogen-rich heterocycle compounds (triazole, furazan, tetrazole and tetrazine) usually have high energy content and positive enthalpy of formation because of a number of N–N, N=N, C–N, C=N bonds in molecules [1–4]. The large conjugated π bond is always formed in heterocyclic ring, which contributes to improve the stability of compound. Many literatures have reported that a majority of nitrogen-rich compounds are insensitive to impact, friction and static electricity, and possess excellent thermal stability. Nitrogen-rich compounds are easier to achieve oxygen balance and exhibit larger density [4, 5]. Therefore, nitrogen-rich compounds used as propellants, gas generating composition and insensitive explosives have many potential applications for military purposes or civilian fields [6–12].

As a kind of interesting nitrogen-rich energetic materials, some triazole compounds are extensively applied to the field of energetic materials [13–15]. A lot of studies have been carried out on triazole energetic compounds, such as 1,2,4-triazole, 1,2,3-triazole and their derivatives [16–24]. Nitramino group is also an important functional group of energetic materials. In 1989, Yu et al. reported an new energetic triazine compound, 2,4-dinitramino-1,5-dinitro-1,3,5-triazine [25]. The compound presents high nitrogen content (42.7 %), large density (1.89 g cm^{-3}) and positive oxygen balance (+2.7 %). However, the compound is unstable. Its two –NH–NO$_2$ groups are very active, so the compound easily decomposed to form 2,4-dinitramino-1,3,5-triazine or 2-nitramino-4-imino-1,3,5-triazine with

different conditions [26]. Because of the influence of two nitramino groups, 2,4-dinitramino-1,5-dinitro-1,3,5-triazine possesses strong acidity. Based on the theory, our group expected to prepare some organic salts of 2,4-dinitramino-1,5-dinitro-1,3,5-triazine to enhance the stability of compound. However, an unexpected reaction happened. We did not obtain hydrazinium 2,4-dinitramino-1,5-dinitro-1,2,4-triazine, but got a 1,2,4-triazole hydrazonium salt, hydrazinium 3,5-dinitroamino-1,2,4-triazole (HDNAT) [27]. In strong reducing condition of hydrazine hydrate solution, triazine ring changed into a 1,2,4-triazole ring with the losing of some groups (Scheme 1).

The synthesis and some properties of HDNAT have been reported by our group [27]. In this paper, we mainly report the non-isothermal decomposition kinetics, specific heat capacity and adiabatic time-to-explosion of HDNAT and then further evaluate its thermal stability.

Experimental

Sample

Hydrazinium 3,5-dinitroamino-1,2,4-triazole was prepared by our group according to Ref. [27], which is white powder after vacuum drying with a yield of 92.8 %. ^{1}H NMR (DMSO-d6, 500 MHz): 7.9228, 13.4263 ppm. ^{13}C NMR (DMSO-d6, 500 MHz): 150.937 ppm. IR (KBr, cm^{-1}), υ: 3319, 3180, 1586, 1542, 1367, 1252, 983, 719. Anal. calcd for $C_2H_7N_9O_4$(%): C 10.86, H 3.17, N 57.01; found C 11.02, H 3.26, N 57.28.

Experimental equipments and conditions

DSC test was performed using a DSC200F3 apparatus (NETZSCH, Germany) under a nitrogen atmosphere at a flow rate of 100 mL min^{-1}. The crucible is a standard aluminum product, and the condition of crucible is open. The heating rates are 5.0, 7.5, 10.0, 12.5 and 15.0 °C min^{-1} from ambient temperature to 350 °C, respectively. TG/DTG test was performed using a TG209F3 apparatus (NETZSCH, Germany) under a nitrogen atmosphere at a flow rate of 100 mL min^{-1}. The crucible is a standard aluminum product. The heating rate

is 10.0 °C min^{-1} from ambient temperature to 400 °C. Specific heat capacity was determined using a Micro-DSCIII apparatus (SETARAM, France). The heating rate is 0.15 °C min^{-1} from 10 °C to 80 °C. The sample mass was 40.50 mg. The impact and friction sensitivities were determined by using a ZBL-B impact sensitivity instrument (NACHEN, China) and a MGY-2 friction sensitivity instrument (NACHEN, China), respectively. The mass of drop hammer is 2.0 kg. The swing angle and gauge pressure are 50° and 0.6 MPa. The mass for each test is 30 mg.

Results and discussion

Thermal behavior

Typical DSC and TG/DTG curves (Figs. 1, 2) indicate that HDNAT has no endothermic melting process, and its thermal behavior is only an intense exothermic decomposition process, occurring at 160-235 °C with a mass loss of about 76.0 %. The extrapolated onset temperature and peak temperature at the heating rate of 10 °C min^{-1} are 184.9 and 192.2 °C, respectively. The enthalpy of decomposition is -2171 J g^{-1} (average value). So the decomposition temperature of HDNAT is high, and decomposition heat is large.

Non-isothermal decomposition kinetics

A multiple of heating method was employed to obtain the kinetic parameters [the apparent activation energy (E) and pre-exponential constant (A)] of the exothermic decomposition process for HDNAT (Fig. 3). The determined values of the beginning temperature (T_0), extrapolated onset temperature (T_e) and peak temperature (T_p) at the different heating rates are listed in Table 1. The calculated values of kinetic parameters by Kissinger and Ozawa methods (E and A) are also listed in Table 1 [28, 29]. The apparent activation energy obtained by Kissinger method is consistent with that by Ozawa method. The linear correlation coefficients (r) are all close to 1. So, the result is credible.

T (decomposition temperature) versus α (conversion degree) curves at different heating rates are shown in Fig. 4. By substituting corresponding data into Ozawa equation, the values of E for any given value of α are
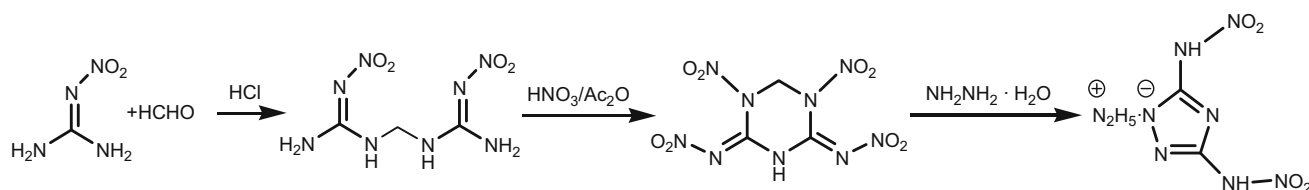
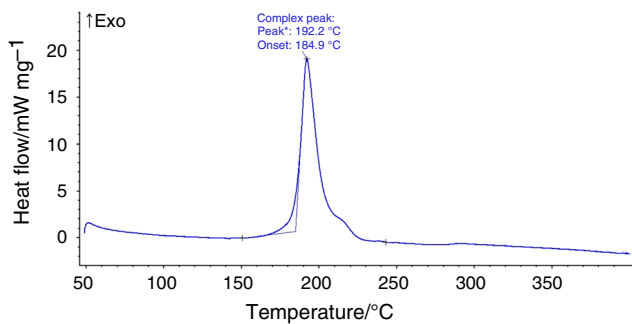**Scheme 1** Synthetic route of HDNAT
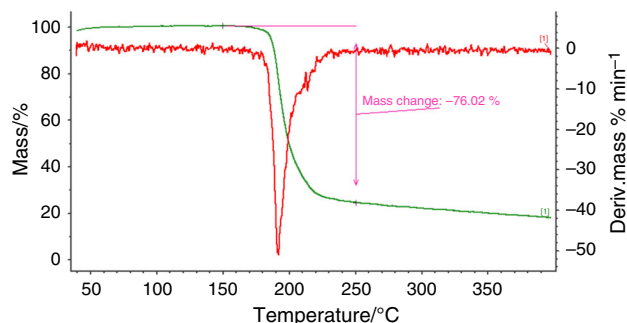
Fig. 1 DSC curve of HDNAT at 10 °C min^{-1}

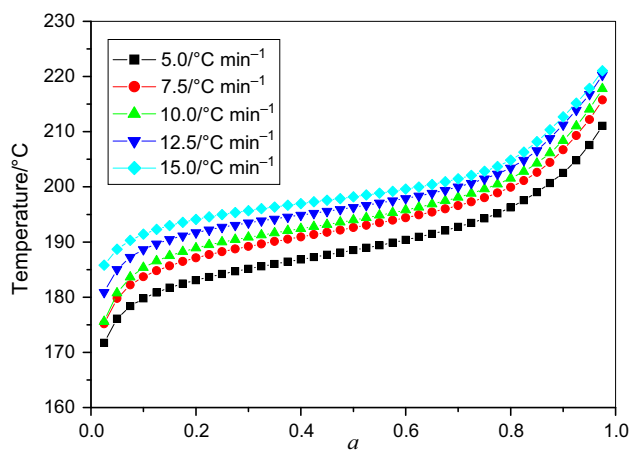Fig. 2 TG/DTG curves of HDNAT at 10 °C min^{-1}

Fig. 4 T versus α curves for HDNAT at different heating rates

obtained and shown in Fig. 5. The values of E steadily distribute from 170 to 220 kJ mol^{-1} in the α range of 0.2–0.8, and the average value of E is 187.65 kJ mol^{-1}, which is in agreement with that obtained by Kissinger method and Ozawa method from only peak temperature values. So, the values were used to check the validity of E by other methods.

Table 1 Values of T_0, T_e, T_p, ΔH_d and kinetic parameters of the decomposition process for HDNAT determined by DSC curves

β/K min^{-1}	T_o/°C	T_e/°C	T_p/°C	ΔH_d/J g^{-1}	E_k/kJ mol^{-1}	log$A$/s^{-1}	r_k	E_O/kJ mol^{-1}	r_O
5.0	156.47	177.54	186.86	−2171	188.6	19.37	0.985	186.7	0.986
7.5	162.21	181.61	191.24						
10.0	164.20	184.90	192.15						
12.5	165.79	187.82	194.83						
15.0	174.64	190.22	197.24						

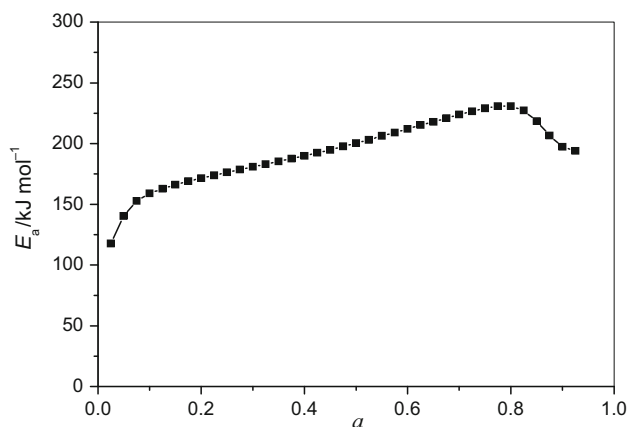Fig. 3 DSC curves of HDNAT at different heat rates
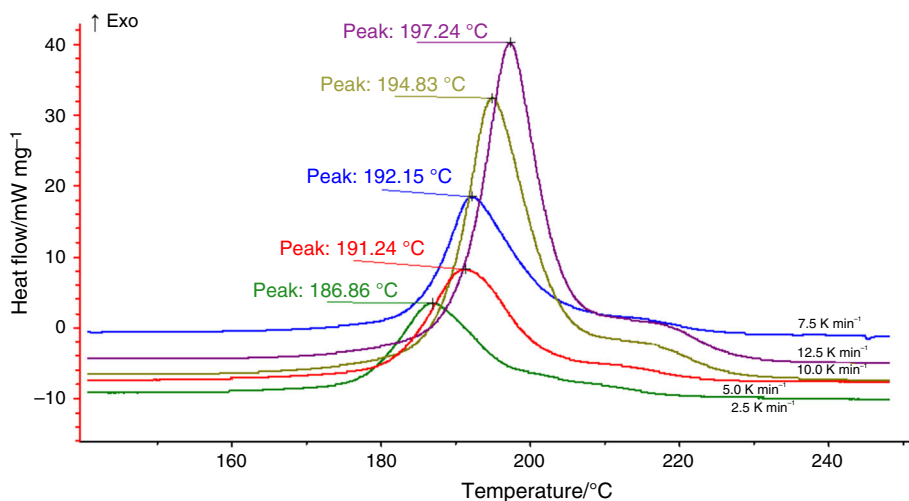
Fig. 5 E versus α curve for HDNAT by Ozawa method

Five integral equations (the general integral equation, the universal integral equation, MacCallum–Tanner equation, Šatava–Šesták equation and Agrawal equation) were cited to obtain the values of E, A and the most probable kinetic model function [f(α)] from each DSC curve [30–33]. Forty-one types of kinetic model functions taken from Ref. [30] and experimental data form each DSC curve were put into the above five integral equations for calculating by our special software, respectively. The kinetic parameters, linear correlation coefficient (r), standard mean square deviation (Q) and believable factor (d, where d = (1 − r)Q) are obtained and listed in Table 2. We can see that the values of E and logA obtained by the five equations agree with each other, and the mean value is much close to that by Kissinger method, Ozawa method and equal conversion degree method. So, the most probable kinetic model function is classified as:

$$f(\alpha) = 6(1-\alpha)^{2/3}[1-(1-\alpha)^{1/3}]^{1/2} \tag{1}$$

(No. 5, three-dimensional diffusion, 3D, n = 1/2), according to the unanimity rule of calculation results for each model equation [30]. The kinetic equation of the decomposition process for HDNAT is:

$$\frac{d\alpha}{dT} = \frac{10^{19.37}}{\beta}6(1-\alpha)^{2/3}[1-(1-\alpha)^{1/3}]^{1/2}\exp(-188.6 \\ \times 10^3/RT) \tag{2}$$

The self-accelerating decomposition temperature (T_{SADT}) and critical temperature of thermal explosion (T_b) are two important parameters required to ensure safe storage and process operations for energetic materials and then to evaluate the thermal stability. T_{SADT} and T_b can be obtained by Eqs. (3) and (4) [30, 34], and T_{SADT} and T_b for HDNAT are 168.1 and 178.1 °C, respectively.

$$T_{SADT} = T_{e0} = T_{ei} - n\beta_i - m\beta_i^2 \quad i = 1-5 \tag{3}$$

$$T_b = \frac{E_O - \sqrt{E_O^2 - 4E_O R T_{e0}}}{2R} \tag{4}$$

where E_O is the apparent activation energy obtained by Ozawa method.

The entropy of activation ($\Delta S^{\neq}$), enthalpy of activation ($\Delta H^{\neq}$) and free energy of activation ($\Delta G^{\neq}$) of the thermal decomposition process for HDNAT corresponding to $T = T_{eo}$, $A = A_k$ and $E = E_k$ obtained by Eqs. (5)–(7) are 122.6 J mol^{-1} K^{-1}, 188.5 kJ mol^{-1} and 134.4 kJ mol^{-1} [30, 31], respectively.

$$A = \frac{k_B T}{h}\exp\left(\frac{\Delta S^{\neq}}{R}\right) \tag{5}$$

$$A\exp\left(-\frac{E}{RT}\right) = \frac{k_B T}{h}\exp\left(\frac{\Delta S^{\neq}}{R}\right)\exp\left(-\frac{\Delta H^{\neq}}{RT}\right) \tag{6}$$

$$\Delta G^{\neq} = \Delta H^{\neq} - T\Delta S^{\neq} \tag{7}$$

where k_B is the Boltzman constant and h is the Plank constant.

Specific heat capacity

The determination result of specific heat capacity of HDNAT is shown in Fig. 6. In determined temperature range, Specific heat capacity of HDNAT presents a linear relationship with temperature. The fitted specific heat capacity equation is:

$$C_p(J\ g^{-1}\ K^{-1}) = 0.1847 + 3.3106 \\ \times 10^{-3}T\ (285.0\ K < T < 350.0\ K) \tag{8}$$

The specific heat capacity and molar heat capacity of HDNAT at 298.15 K are 1.1718 J g^{-1} K^{-1} and 258.96 J mol^{-1} K^{-1}, respectively.

Estimation of adiabatic time-to-explosion

The adiabatic time-to-explosion is also an important parameter for evaluating the thermal stability of energetic materials and can be calculated by Eqs. (9)–(10) [30, 35–39].

$$C_p\frac{dT}{dt} = QA\exp(-E/RT)f(\alpha) \tag{9}$$

$$\alpha = \int_{T_0}^{T}\frac{C_p}{Q}dT \tag{10}$$

where C_p is the specific heat capacity (J mol^{-1} K^{-1}), T is the absolute temperature (K), t is the adiabatic time-to-explosion (s), Q is the exothermic values (J mol^{-1}), A is the pre-exponential factor (s^{-1}), E is the apparent activation energy of the thermal decomposition reaction (J mol^{-1}), R is the gas constant (J mol^{-1} K^{-1}), f(α) is the

Table 2 Calculated values of kinetic parameters of decomposition reaction

| β/K min^{-1}	Equation	E/kJ mol^{-1}	$\log A$/s^{-1}	r	Q	d
5.0	The general integral equation	163.49	16.07	0.9972	4.19×10^{-3}	1.16×10^{-5}
	The universal integral equation	161.70	14.60	0.9972	4.18×10^{-3}	1.17×10^{-5}
	MacCallum–Tanner equation	163.69	16.03	0.9975	7.89×10^{-4}	1.98×10^{-6}
	Šatava–Šesták equation	162.73	15.97	0.9975	7.89×10^{-4}	1.98×10^{-6}
	Agrawal equation	163.50	16.06	0.9972	4.19×10^{-3}	1.16×10^{-5}
7.5	The general integral equation	164.27	16.16	0.9977	3.42×10^{-3}	7.71×10^{-6}
	The universal integral equation	162.57	14.71	0.9977	3.41×10^{-3}	7.79×10^{-6}
	MacCallum–Tanner equation	164.53	16.14	0.9979	6.43×10^{-4}	1.32×10^{-6}
	Šatava–Šesták equation	163.53	16.06	0.9980	6.43×10^{-4}	1.32×10^{-6}
	Agrawal equation	164.27	16.16	0.9977	3.42×10^{-3}	7.71×10^{-6}
10.0	The general integral equation	178.48	17.85	0.9977	3.58×10^{-3}	8.37×10^{-6}
	The universal integral equation	176.83	16.37	0.9976	3.56×10^{-3}	8.46×10^{-6}
	MacCallum–Tanner equation	178.87	17.85	0.9979	6.73×10^{-4}	1.44×10^{-6}
	Šatava–Šesták equation	177.07	17.69	0.9979	6.73×10^{-4}	1.44×10^{-6}
	Agrawal equation	178.48	17.85	0.9977	3.58×10^{-3}	8.37×10^{-6}
12.5	The general integral equation	201.31	20.44	0.9979	3.31×10^{-3}	7.08×10^{-6}
	The universal integral equation	199.72	18.91	0.9978	3.30×10^{-3}	7.15×10^{-6}
	MacCallum–Tanner equation	201.91	20.47	0.9980	6.22×10^{-4}	1.23×10^{-6}
	Šatava–Šesták equation	198.82	20.18	0.9980	6.22×10^{-4}	1.23×10^{-6}
	Agrawal equation	201.31	20.45	0.9979	3.31×10^{-3}	7.08×10^{-6}
15.0	The general integral equation	228.34	23.47	0.9985	2.31×10^{-3}	3.41×10^{-6}
	The universal integral equation	226.80	21.89	0.9985	2.30×10^{-3}	3.44×10^{-6}
	MacCallum–Tanner equation	229.17	23.55	0.9986	4.34×10^{-4}	6.00×10^{-7}
	Šatava–Šesták equation	224.56	23.11	0.9986	4.34×10^{-4}	6.00×10^{-7}
	Agrawal equation	228.34	23.47	0.9985	2.31×10^{-3}	3.41×10^{-6}
Mean		186.57	18.46			
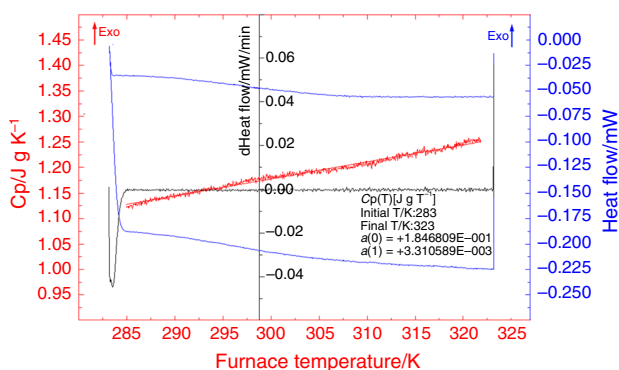
Fig. 6 Determination results of the continuous specific heat capacity of HDNAT

most probable kinetic model function and α is the conversion degree.

After integrating the Eq. (9), the adiabatic time-to-explosion equation can be obtained as:

$$t = \int_0^t dt = \int_{T_0}^T \frac{C_p \exp(E/RT)}{QAf(\alpha)} dT \qquad (11)$$

where the limit of temperature integration is from T_{00} to T_b. T_{00} can be obtained by Eq. (12) [30].

$$T_{00} = T_{0i} - n\beta_i - m\beta_i^2 \quad i = 1-5 \qquad (12)$$

In fact, the conversion degree (α) of energetic materials from the beginning thermal decomposition to thermal explosion in the adiabatic condition is very small, and it is very difficult to obtain the most probable kinetic model function [$f(\alpha)$] at the process. So, we separately used power low model [Eq. (13)], reaction-order model [Eq. (14)], Avrami–Erofeev model [Eq. (15)] and the above-obtained kinetic model function to estimate the adiabatic time-to-explosion and supposed different rate orders (n) [30, 31]. The calculation results are listed in Table 3.

$$f(\alpha) = n\alpha^{(n-1)/n} \qquad (13)$$

$$f(\alpha) = (1-\alpha)^n \qquad (14)$$

$$f(\alpha) = n(1-\alpha)[-\ln(1-\alpha)]^{(n-1)/n} \qquad (15)$$

From Table 3, the calculated results exhibit some deviation and the decomposition model has large influence

Table 3 Calculation results of adiabatic time-to-explosion

Equation	Rate order	Model	Time/s
Eq. (13)	$n = 1$	$f(\alpha) = 1$	25.8
	$n = 2$	$f(\alpha) = 2\alpha^{1/2}$	98.9
	$n = 3$	$f(\alpha) = 3\alpha^{2/3}$	130.1
	$n = 4$	$f(\alpha) = 4\alpha^{3/4}$	137.1
Eq. (14)	$n = 0$	$f(\alpha) = 1$	25.8
	$n = 1$	$f(\alpha) = 1 - \alpha$	26.2
	$n = 2$	$f(\alpha) = (1 - \alpha)^2$	26.7
Eq. (15)	$n = 1$	$f(\alpha) = 1 - \alpha$	26.2
	$n = 2$	$f(\alpha) = 2(1 - \alpha)[-\ln(1 - \alpha)]^{1/2}$	100.2
	$n = 3$	$f(\alpha) = 3(1 - \alpha)[-\ln(1 - \alpha)]^{2/3}$	131.6
	$n = 4$	$f(\alpha) = 4(1 - \alpha)[-\ln(1 - \alpha)]^{3/4}$	138.5
Obtained equation		$f(\alpha) = 6(1 - \alpha)^{2/3}[1 - (1 - \alpha)^{1/3}]^{1/2}$	57.6

on the estimating result of adiabatic time-to-explosion. But we believe the adiabatic time-to-explosion of HDNAT should be about 100 s. It is a relatively long time, and the result can be proved credible according to the change of DSC curves in the decomposition process.

Detonation properties of HDNAT

The impact and friction sensitivities of HDNAT are >13.1 J and 84 %, respectively, so HDNAT is relatively sensitive, especially to friction. Moreover, the impact sensitivity is lower than that of RDX (>7.4 J) [37]. The loading density of HDNAT can reach 1.80 g cm^{-3}. The detonation velocity and detonation pressure of HDNAT calculated based on the empirical K–J equations are 9.0 km s^{-1} and 36.0 GPa, respectively, which all are larger than these of RDX (8.7 km s^{-1} and 33.8 GPa) [36].

Conclusions

The thermal behavior of HDNAT presents only an intense exothermic decomposition process. The non-isothermal kinetic equation of the decomposition process was obtained. The self-accelerating decomposition temperature and critical temperature of thermal explosion of HDNAT are 168.1 and 178.1 °C, respectively.

Specific heat capacity equation of HDNAT is $C_p(\mathrm{J\ g^{-1}\ K^{-1}}) = 0.1847 + 3.3106 \times 10^{-3}\ T$ (283.0 K < T<353.0 K), and the molar heat capacity is 258.96 J mol^{-1} K^{-1} at 298.15 K. Adiabatic time-to-explosion of HDNAT was estimated to be about 20 s. The impact sensitivity, friction sensitivity, detonation velocity

and detonation pressure of HDNAT are >13.1 J, 84 %, 9.0 km s^{-1} and 36.0 GPa, respectively.

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