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Thermal safety study on the synthesis of HMX by nitrourea method

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ABSTRACT

HMX is one of the best high-energy explosives in military applications. In this paper, the reaction calorimeter (RC1^e) was used to monitor the reaction process of HMX synthesis by the nitrourea method. The research results show that the heat release rate is large on the reaction feeding stage, the feed rate should be controlled. The thermal parameters, such as adiabatic temperature rise($\Delta T_{ad,r}$) and maximum temperature of the synthesis reaction (MTSR), were calculated for assessing the thermal hazard of HMX synthesis. Although the urea nitrification stage is safer, the operating conditions of other reaction processes also need to be redesigned to improve the process safety level. These studies have potential applications in inherently safer operating measures and safer storage and transportation.

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1. Introduction

Nowadays, cyclotetramethylenetetranitramine(HMX) is one of the most powerful military nitramine explosives. It is the main charge explosive of warheads and the energetic component of composite propellants (Agrawal and Hodgson, 2007; Urbanski, 1967; Zhang, 1995). However, its application is limited owing to high cost. The conventional methods to synthesize HMX involve the nitrolysis of hexamine, and they are not economical or environmental friendly. It is important to find new ways to synthesize HMX to avoid the use of expensive raw materials. The study found HMX can be produced from DPT by direct nitrolysis, and DPT can be obtained with high yield from via nitrourea and/or dinitrourea (Lobanova et al., 2000, 2010; Il'Yasov et al., 2002, 2013). HMX is synthesized through the use of urea, nitric acid, formaldehyde, ammonia, etc., which are cheap, easily available and environmentally friendly starting material. And this process avoids the use of a large excess of acetic acid and acetic anhydride.

The synthesis reaction of energetic materials is mostly a nitrification reaction. In theory, the nitrification reaction has the characteristics of large heat release and high risk. When the heat released from the synthesis reaction exceeds the heat transferred by the cooling system, the excess heat is further accumulated for the increase of the system temperature, which is likely to induce the secondary decomposition of the reaction system (Vernières-Hassimi and Leveneur, 2015; Zhang et al., 2015). Therefore, it is of great significance to study the exothermic characteristics of the energetic materials during the synthesis process. Potential thermal hazards should be identified to further prevent accidents (Liu et al., 2012). To avoid runaway conditions, it is necessary to understand the heat release laws of the target reactions and potential side reactions, as well as the thermal stability and physical properties of the reactants and intermediates (Zang et al., 2013). Therefore, the exothermic characteristics of all stages of HMX synthesis should be identified to determine potential thermal hazards, guard against accidents. But the thermal hazard evaluation of HMX synthesis by nitrourea method has scanty literature information, which is vital for an inherently safer chemical process design.

The reaction calorimeter(RC1^e) plays a vital role in chemical production. It can monitor the parameter changes in the chemical reaction process online, and track the temperature, heat, and heat transfer data during the reaction (Duh et al., 1996; Zhang et al., 2016).

A large number of experiments have explored the thermal decomposition behavior and thermodynamics of various organic synthesis reactions (Duh, 2016; Hsueh et al., 2016). Various contents such as the risk rating according to the reaction exothermic, the synthesis reaction by the calorimetric test, and the thermal warning detection system were studied (Stoessel, 2008; Wozik and Westerterp, 2002; Valdes et al., 2015). In this paper, the typical exothermic law of the reaction process of synthesizing HMX by nitrourea method was studied through RC1^e. Then, use the data obtained by the calorimetric experiment to study on thermal safety of the process of synthesis of the HMX reaction of the nitrourea method. It is of great significance to the production of the chemical industry.

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**Fig. 1.** The diagram of Reaction calorimeter device.

2. Experiment

2.1. Samples

The properties of the chemicals used in the experiment,98 mass% nitric acid, fuming sulfuric acid, urea, 35~37 mass% formaldehyde solution, 25~28 mass% ammonia, ammonium nitrate, AR, was supplied by Tianjin Chemical Reagent Co. Ltd.
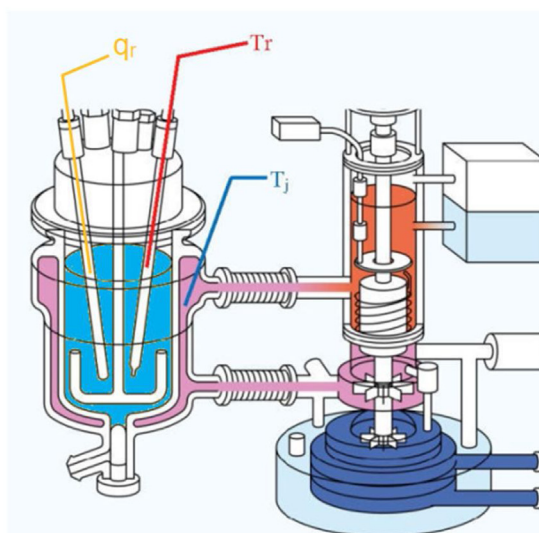
2.2. Reaction calorimeter (RC1^e)

The RC1^e was developed by Metter Toledo (Zurich, Switzerland) for chemical synthesis, reaction hazard analysis, and process optimization. The isothermal program simulation experiment was conducted by the RC1^e automatic synthesis reaction system. The reaction calorimeter can monitor the thermal parameter changes during the chemical reaction on-line, and track the temperature, heat, and heat transfer data during the reaction. Fig. 1 shows diagrams of the Reaction calorimeter device, reactor temperature (T_r), the cooling jacket temperature (Tj) and heat release rate (q_r).

The synthesis reaction of HMX reaction by the nitrourea method contained five steps. The first stage,32 g fuming sulfuric acid added to 32 g 98 mass% nitric acid, then the reaction temperature was controlled at -5.0°C and the mixture was stirred evenly at 300 rpm to form an acidic solution, 9 g urea was added to the reactor at a dosing rate of 0.3 g/min in isothermal mode, The reaction was continued for 60 min and the exotherm of this process was measured. The HMX synthesis reaction first stage can be expressed as Eq. (1):

$$O=C\overset{NH_2}{\underset{NH_2}{}} +2HNO_3 \xrightarrow{H_2SO_4} O=C\overset{N-NO_2}{\underset{N-NO_2}{}} +2H_2O$$

(1)
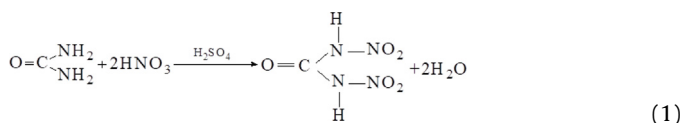
In the second stage, after the first stage reaction was completed, the reaction temperature was controlled at 0°C 54 g deionized water was added to the reactor at a dosing rate of 1.35 g/min in isothermal mode and the mixture was stirred evenly at 300 rpm. The reaction was continued for 20 min and the exotherm of this process was measured. The HMX synthesis reaction second stage can be expressed as Eq. (2):

$$O=C\overset{N-NO_2}{\underset{N-NO_2}{}} \xrightarrow[H_2O]{H^+} 2NH_2NO_2 + CO_2$$

(2)
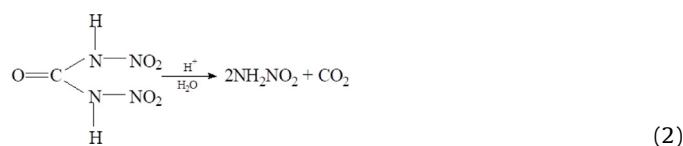
In the third stage, after the second stage reaction test is completed, 50 % of the reaction solution is left to continue the reaction. The reaction temperature was controlled at 10~12°C 45 g formaldehyde solution was added to the reactor at a dosing rate of 1.35 g/min in isothermal mode and the mixture was stirred evenly at 300 rpm. The reaction was continued 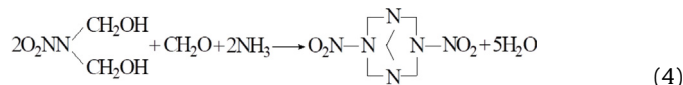for 60 min and the exotherm of this process was measured. The HMX synthesis reaction third stage can be expressed as Eq. (3):

$$NH_2NO_2 + 2CH_2O \longrightarrow O_2N-N\overset{CH_2OH}{\underset{CH_2OH}{}}$$

(3)
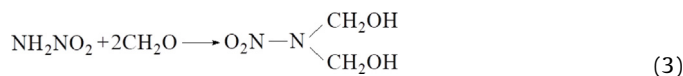
In the fourth stage, 50 % of the reaction solution after completion of the third stage reaction was again taken. The reaction temperature was controlled at 10~12°C ammonia was added to the reactor to make the reaction solution pH = 1.5, then, after raising the temperature of the reaction solution to 25°C, continue to add ammonia until pH = 7. The reaction process is isothermal mode, and the mixture was stirred evenly at 300 rpm. The reaction was continued for 30 min and the exotherm of this process was measured. The HMX synthesis reaction fourth stage can be expressed as Eq. (4):

$$2O_2NN\overset{CH_2OH}{\underset{CH_2OH}{}} +CH_2O+2NH_3 \longrightarrow O_2N-N\overset{N}{\underset{N}{<}} N-NO_2+5H_2O$$

(4)

Finally stage, 70 g fuming sulfuric acid was added to the reactor, then the reaction temperature was controlled at 34°C, 7 g DPT and10.3 g ammonium nitrate was added to the reactor at a dosing rate of 0.3 g/min and the mixture was stirred evenly at 300 rpm in isothermal mode, The reaction was continued for 60 min and
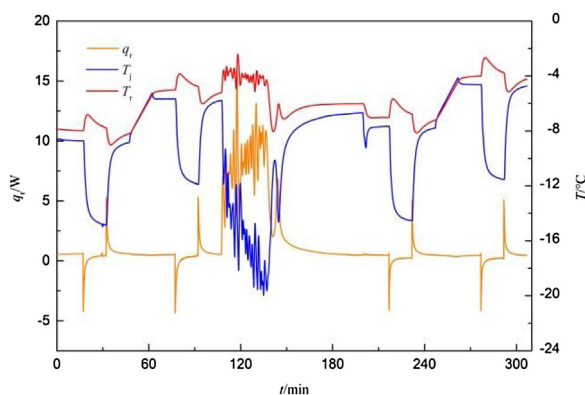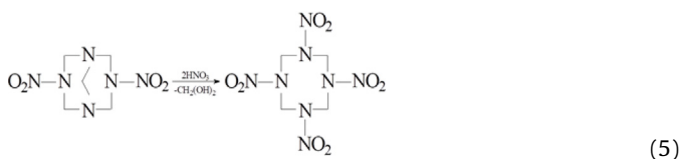
Fig. 2. The exothermic curve of urea nitrification process.

Fig. 3. The exothermic curve of Dinitrourea hydrolysis process.

the exotherm of this process was measured. The HMX synthesis reaction finally stage can be expressed as Eq. (5):

$$(5)$$

The reaction calorimeter (RC1^e) can measure the exothermic curve of each step reaction, and use the software of the instrument, Iterral5.4, to integrate the area of the exothermic rate curve of each step of the reaction process to obtain the accumulated heat release. The experimental conditions were unchanged, and 3 experiments were performed in each stage, and the measured data were averaged.

3. Results and discussion

3.1. Step 1 urea nitrification

The exothermic curve of the reaction process is shown in Fig. 2. Fig. 2 shows the T_r, Tj, and q$_r$. As can be seen in Fig. 2, with the addition of urea into the reactor, the heat release rate immediately increased at the beginning of synthesis, 0∼107 min is the instrument calibration stage before the reaction; 107∼198 min is the reaction process stage. From the figures, it is found that there is a fluctuation in the water jacket temperature (Tj) concerning reactor temperature (T_r) in the reaction process stage. This phenomenon occurs because heat is released during the reaction, which causes the temperature of the reaction system to rise. To ensure that the reaction is carried out at the temperature required for the process, RC1^e will remove heat in time by reducing the cooling jacket temperature. This phenomenon can be seen in the exothermic curve of the five step synthesis process of HMX. 198∼306 min is the instrument calibration stage after the reaction. At 141 min, the exothermic rate increased sharply and then decreased. This phenomenon indicates that the urea nitrification reaction has a mature stage. The urea nitration reaction immediately happened. Because of the function of the cooling control system, the reactor was cooled promptly after the synthesis reaction started, the heat released by the reaction does not cause the reactor temperature to rise. At the ripening stage of the completion of the reaction, the heat is gradually released. After 152 min, the exothermic rate curve is almost 0 W, and the jacket temperature is equal to the reactor temperature, which indicates that there is almost no heat change in the late stage of the reaction, and the nitrification reaction has ended after the maturity period.
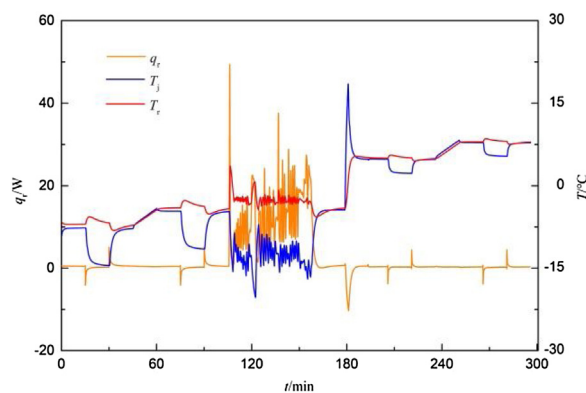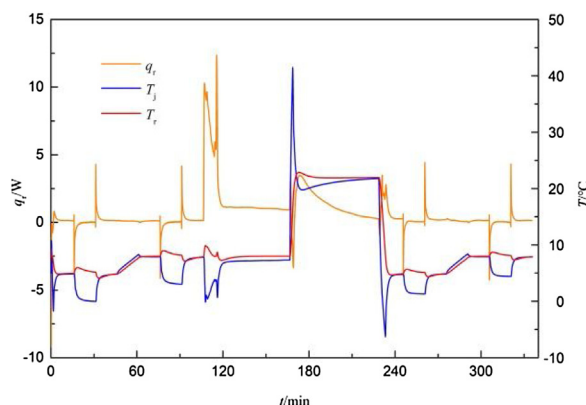

Fig. 4. The exothermic curve of condensation reaction(1) process.

By integrating the heat release rate curve in the figure, the total heat release amount of the nitrification process is 16.77 kJ, and the heat release amount is 1.887 kJ when the urea addition amount is converted into 1 g.

3.2. Step 2 dinitrourea hydrolysis

The exothermic curve of the reaction process is shown in Fig. 3. Fig. 3 shows the reactor temperature (T_r), jacket temperature (Tj) and heat release rate (q$_r$). With the addition of deionized water into the reactor, the synthesis reaction immediately happened and the heat release rate increased at the beginning of synthesis. When the addition of deionized water is completed, the rate of heat release is rapidly reduced. This phenomenon indicates that the hydrolysis reaction proceeds relatively quickly, and there is almost no heat accumulation during the reaction. After the addition is completed, the heat release rate becomes almost 0 W almost immediately. and the jacket temperature is equal to the reactor temperature, and the hydrolysis reaction has substantially ended during the addition process.

By integrating the heat release rate curve in the figure, the total heat release amount of the hydrolysis process is 40.753 kJ, and the heat release amount is 0.769 kJ when the deionized water addition amount is converted into 1 g.

3.3. Step 3 condensation reaction(1)

The exothermic curve of the reaction process is shown in Fig. 4. Fig. 4 shows the reactor temperature (T_r), jacket temperature (Tj) and heat release rate (q$_r$). 0∼107 min is the instrument calibration stage before the reaction; 107∼167 min is the reaction process stage; 167∼272 min is the instrument calibration stage
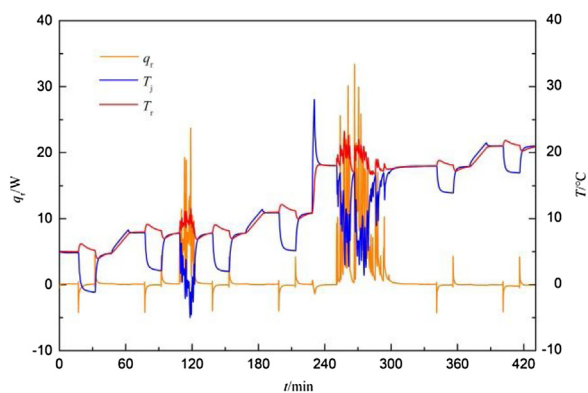
Fig. 5. The exothermic curve of condensation reaction(2) process.

Fig. 6. The exothermic curve of DPT nitrification process.
Heat curve of urea nitrification process Heat curve of Dinitrourea hydrolysis process.
Heat curve of condensation reaction(1) process Heat curve of condensation reaction(2-1) process.
Heat curve of condensation reaction(2-2) process Heat curve of DPT nitrification process.

after the reaction. The decrease in the exotherm rate at 167 min was due to the temperature rise of the reactor to 22 °C. The sudden increase in the exotherm rate at 229 min was due to the cooling of the reactor to 5 °C. It can be seen from the curve of the reaction stage that when the formaldehyde was added, the reaction starts instantaneously, the exothermic rate increases, and the number of exotherm increases. When the addition of the formaldehyde is completed, the exothermic rate rapidly decreases.

By integrating the heat release rate curve in the figure, the total heat release amount of the Condensation reaction(1) is 4.314 kJ, and the heat release amount is 0.097 kJ when the formaldehyde addition amount is converted into 1 g.

3.4. Step 4 condensation reaction(2)

The exothermic curve of the reaction process is shown in Fig. 5. Fig. 5 shows the reactor temperature (T_r), jacket temperature (T_j) and heat release rate (q_r). 0∼107 min is the instrument calibration stage before the reaction; 107∼133 min is the first stage to add ammonia; 133∼251 min is the instrument calibration stage after the reaction. 251∼296 min is the second stage of adding ammonia. 326∼431 min is the instrument calibration stage after the reaction. It can be seen from the curve of the reaction stage that when the ammonia was added, the reaction starts instantaneously, the exothermic rate increases, and the number of exotherm increases. When the addition of the ammonia is completed, the exothermic rate rapidly decreases. By observing changes in the reactor, it can be found that no white solids were produced during the first stage of ammonia addition during the reaction. The main occurrence of this stage is the neutralization reaction of ammonia and acid; During the second stage of ammonia addition, a white solid gradually appeared, which is the main product of this stage, DPT. At this stage, the main reaction occurred was the condensation reaction.

By integrating the heat release rate curve in the figure, The exotherms of the two-stage feed of ammonia were 5.715 kJ and 19.582 kJ, respectively. When the amount of ammonia added was converted to 1 g, the exotherms were 0.780 kJ and 1.133 kJ, respectively.

3.5. Step 5 DPT nitrification process

The exothermic curve of the reaction process is shown in Fig. 6. Fig. 6 shows the reactor temperature (T_r), jacket temperature (T_j) and heat release rate (q_r). 0∼107 min is the instrument calibration stage before the reaction; During the heating period of 107∼109 min, the decrease of exothermic rate was caused by temperature rise; 120∼172 min was the reaction stage, and 174∼279 min was the post-reaction calibration stage. It can be seen from the curve of the reaction stage that when the ammonium nitrate and
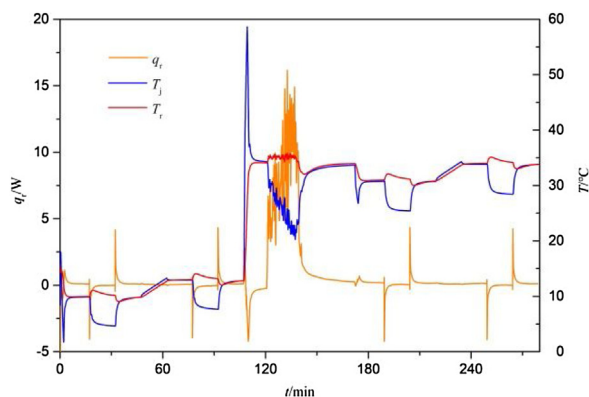
DPT were added, the reaction starts immediately and the exothermic rate increases. When the addition is completed, the exothermic rate gradually decreases. This phenomenon indicates that the reaction proceeds slowly, and the reaction stage system has a small amount of heat accumulation.

By integrating the heat release rate curve in the figure, the total heat release amount of the DPT nitrification process is 10.081 kJ, and the heat release amount is 1.440 kJ when the DPT addition amount is converted into 1 g.

3.6. Reaction process safety

To assess the thermal hazard of chemical reactions, it is necessary not only to analyze the temperature rise and heat accumulation of the synthesis reaction but also to analyze the possible thermal risks of the target synthesis reaction.

The heat accumulated by the system at any one time is equal to the amount of heat that can be released after all the reactants that have been added have reacted, minus the measured exotherm at the corresponding time. Import heat

$$A = \frac{\Delta H}{M_{tol}} \times m_{tol}; \tag{6}$$

Release heat

$$B = \int_{t_0}^{t} q_r dt; \tag{7}$$

Cumulative heat

$$C = A - B; \tag{8}$$

In the formula,

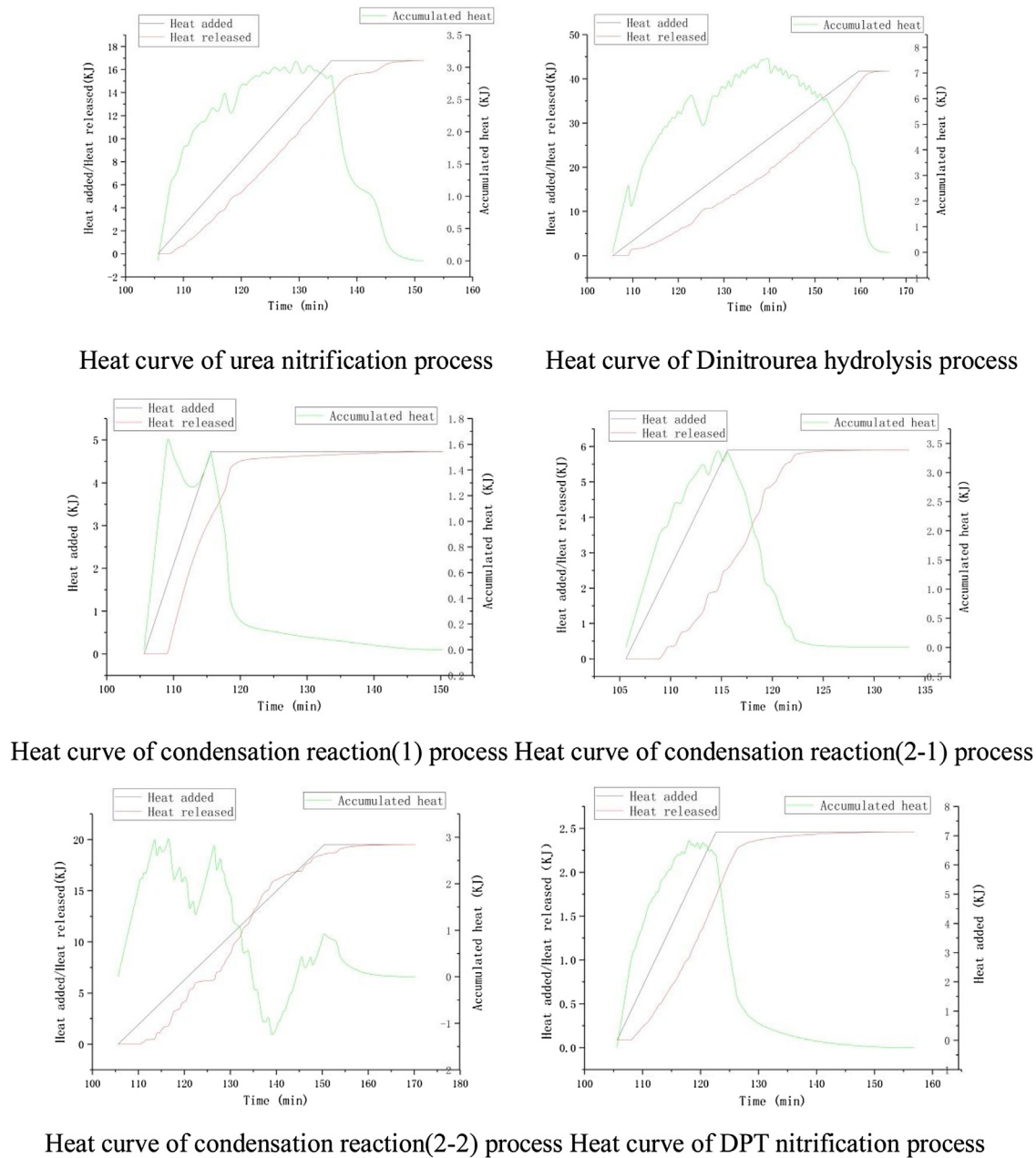$$\Delta H = \int_{t_0}^{t_{end}} q_r dt; \tag{9}$$

the total heat release of the reaction, kJ; M_{tol} is the total mass of the reactants to be added, kg; m_{tol} is the mass of the reactants added to the reactor at time t, kg. The heat accumulation curve is shown in Fig. 7

The heat accumulated at time t can cause the adiabatic temperature rise of the system $\Delta T_{ad, r}$ is

$$\Delta T_{ad,r} = \frac{C}{M_t \times C_p}; \tag{10}$$

M_t is the total mass of the reaction system at time t, kg; C_p is the specific heat capacity of the reaction system at time t, kJ·kg^{-1}·K^{-1}.

Heat curve of urea nitrification process   Heat curve of Dinitrourea hydrolysis process

Heat curve of condensation reaction(1) process Heat curve of condensation reaction(2-1) process

Heat curve of condensation reaction(2-2) process Heat curve of DPT nitrification process

Fig. 7. Heat accumulation curve.
T_{cf} curve of urea nitrification process T_{cf} curve of Dinitrourea hydrolysis process.
T_{cf} curve of condensation reaction(1) process T_{cf} curve of condensation reaction(2-1) process.
T_{cf} curve of condensation reaction(2-2) process T_{cf} curve of DPT nitrification process.

The maximum temperature T_{cf} at which the reaction heat is raised by the reaction at the time t is

$$T_{cf} = T_p + \Delta T_{ad,r}; \qquad (11)$$

T_P is the temperature of the reaction system before the cooling failure. When the isothermal model is adopted, it is the set reaction temperature. The maximum temperature of the synthesis reaction, MTSR, is the maximum temperature at which the heat accumulated in the system after the reaction is out of control (after cooling failure) can cause the reaction system to reach under adiabatic conditions.

$$MTSR = \max(T_{cf}); \qquad (12)$$

The T_{cf} curve at each stage of the reaction is shown in Fig. 8, following Eqs. (10) and (11), and the thermal parameters of synthesis reaction are listed in Table 1.

MTT(maximum temperature for technical reasons) is mainly selected based on the boiling point of the solvent and the reactor discharge pressure. Because this reaction system is open, the boiling point is mainly considered. For the urea nitrification process in acid medium, MTT selected was 83.0 °C, the boiling point of hydrogen nitrate. The criticality class is shown as follows: Tp < MTSR < MTT. For the Dinitrourea hydrolysis process in acid medium, MTT selected was 100.0 °C, the boiling point of water. The criticality class is shown as follows: Tp < MTSR < MTT. For the condensation reaction(1) process in acid medium, MTT selected was -19.5 °C, the boiling point of formaldehyde. The criticality class is shown as follows: Tp < MTT < MTSR. For the condensation reaction(2) pro-
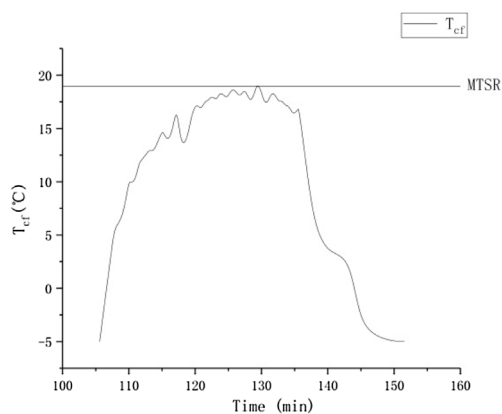
T_{cf} curve of urea nitrification process
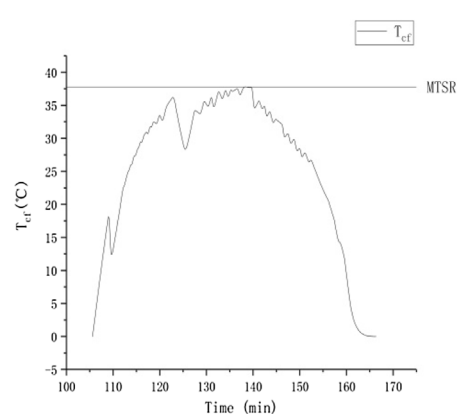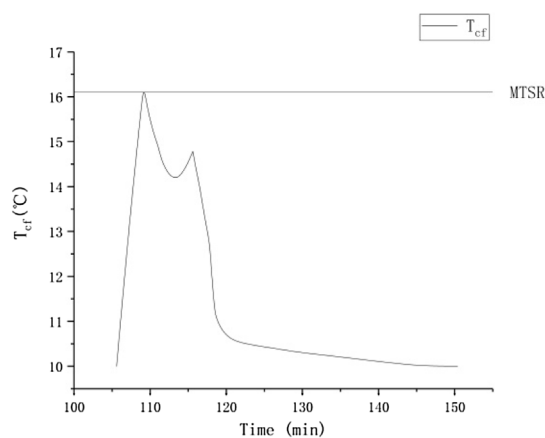
T_{cf} curve of Dinitrourea hydrolysis process

T_{cf} curve of condensation reaction(1) process
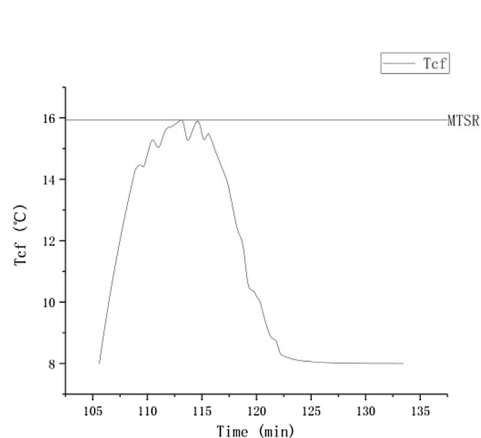
T_{cf} curve of condensation reaction(2-1) process
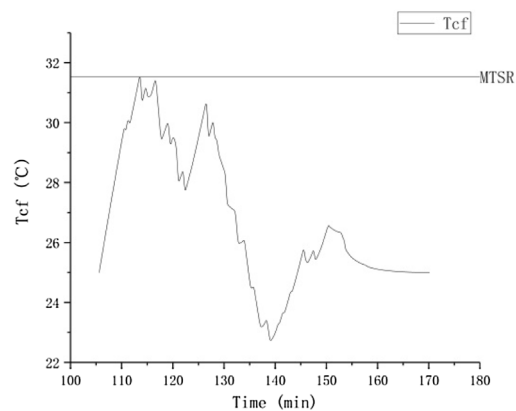
T_{cf} curve of condensation reaction(2-2) process
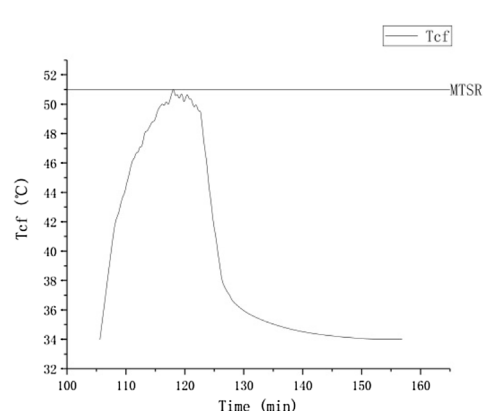
T_{cf} curve of DPT nitrification process

Fig. 8. The T_{cf} accumulation curve.

cess in acid medium, MTT selected was 37.7 °C, the boiling point of ammonium hydroxide. Tp < MTSR < MTT. For the DPT nitrification process in acid medium, MTT selected was 83.0 °C the boiling point of hydrogen nitrate. The criticality class is shown as follows: Tp < MTSR < MTT. Once the target reaction is out of control, the thermal accumulation of reaction products leads to a secondary decomposition reaction, and the final temperature may reach MTT. In this situation, only a pre-set quench or adequate emergency venting can be used to substantially lessen the risk of a runaway reaction. Therefore, to reduce the severity of the synthesis reaction and the

Table 1
Thermal parameters of a synthesis reaction. (Data from the experiment).

Reaction stage	ΔrH/kJ	ΔTad/°C	Tp/°C	MTSR/°C	MTT/°C
urea nitrification process	16.77	21.24	−5	16.24	83.0
Dinitrourea hydrolysis process	41.72	18.12	0	23.12	100.0
condensation reaction(1) process	4.73	4.78	10-12	14.78	−19.5
condensation reaction(2-1) process	5.90	7.89	10-12	15.89	37.7
condensation reaction(2-2) process	19.51	6.40	25	31.40	
DPT nitrification process	7.13	16.70	34	50.70	83.0

possibility of inducing secondary decomposition, it is necessary to redesign the process with a proactive approach. For the synthesis under acid condition, because the nitrification product and mixed acid solution produced in the first step has a higher viscosity, the situation such as an inhomogeneous mixture of reactants may occur to cause more accumulations in the reactor. It indicates that catastrophic accidents cannot be prevented in the event of a cooling failure. Since the system of the reaction liquid is complicated and a large amount of nitric acid is present, the stability of the system is relatively poor. In this situation, the operating conditions of the process for preparing HMX need to be redesigned, including changing the stirring speed, the impeller, and the dosing rate, to effectively reduce the heat accumulation. Accelerate the mixing speed to speed up heat exchange. When the reaction solution is thicker, increasing the size of the impeller can avoid heat buildup. Lowering the feed rate can slow down the heat release, to substantially decrease the risk of HMX synthesis.

4. Conclusions

(1) HMX synthesis reaction was conducted by RC1^e techniques to obtain the fundamental data as a basis for evaluating the reaction criticality classes, optimizing the operating conditions, and determining the appropriate temperature in preparation, manufacturing, storage, and systematically transportation.

(2) According to the RC1^e measurement, the calculated $\Delta T_{ad,r}$ of various stages of the synthesis reaction was 21.24 °C, 18.12 °C, 4.78 °C, 7.89 °C, 6.40 °C and 16.70 °C, respectively. Compared to the urea nitrification process, other synthetic stages were more likely to trigger secondary decomposition owing to the higher adiabatic temperature rise of synthesis if the cooling system failed and the feed valves were not cut off in time.

(3) Comparatively speaking, the urea nitrification process was safer than the other synthetic stages according to the reaction condition of HMX synthesis, this phenomenon is due to the low reaction temperature and the strong ability to remove heat. Nevertheless, further researches regarding the process optimization of HMX synthesis are needed to establish an inherently safer operating condition on a commercial scale.

Declaration of Competing Interest

The authors declare that there is no conflict of interest.

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