Thermal decomposition kinetics of ammonium dinitramide–guanylurea dinitramide mixture analyzed by isoconversional methods

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ABSTRACT

The thermal decomposition kinetics of a mixture of high-energy oxidizers ammonium dinitramide (ADN) and guanylurea dinitramide (GUDN) have been studied by nonisothermal differential scanning calorimetry (DSC). Friedman's and Vyazovkin's advanced isoconversional (AIC-V) methods were used to investigate the dependence of activation energy (E_a) on conversion (α). A strong dependence of E_a on α was observed, indicating a complex decomposition process. E_a is ~165–175 kJ mol^{-1} at the start of the reaction, then reaches a maximum of ~196–199 kJ mol^{-1} at α = ~0.4, followed by a strong decrease to ~135 kJ mol^{-1} near the end. E_a has also been determined for the mixture and the individual components using Kissinger's method. Our results suggest that the decomposition of ADN greatly influences the observed E_a dependence on α. This is further supported by Fourier transform infrared (FTIR) results and the enthalpy values of exothermic decomposition.

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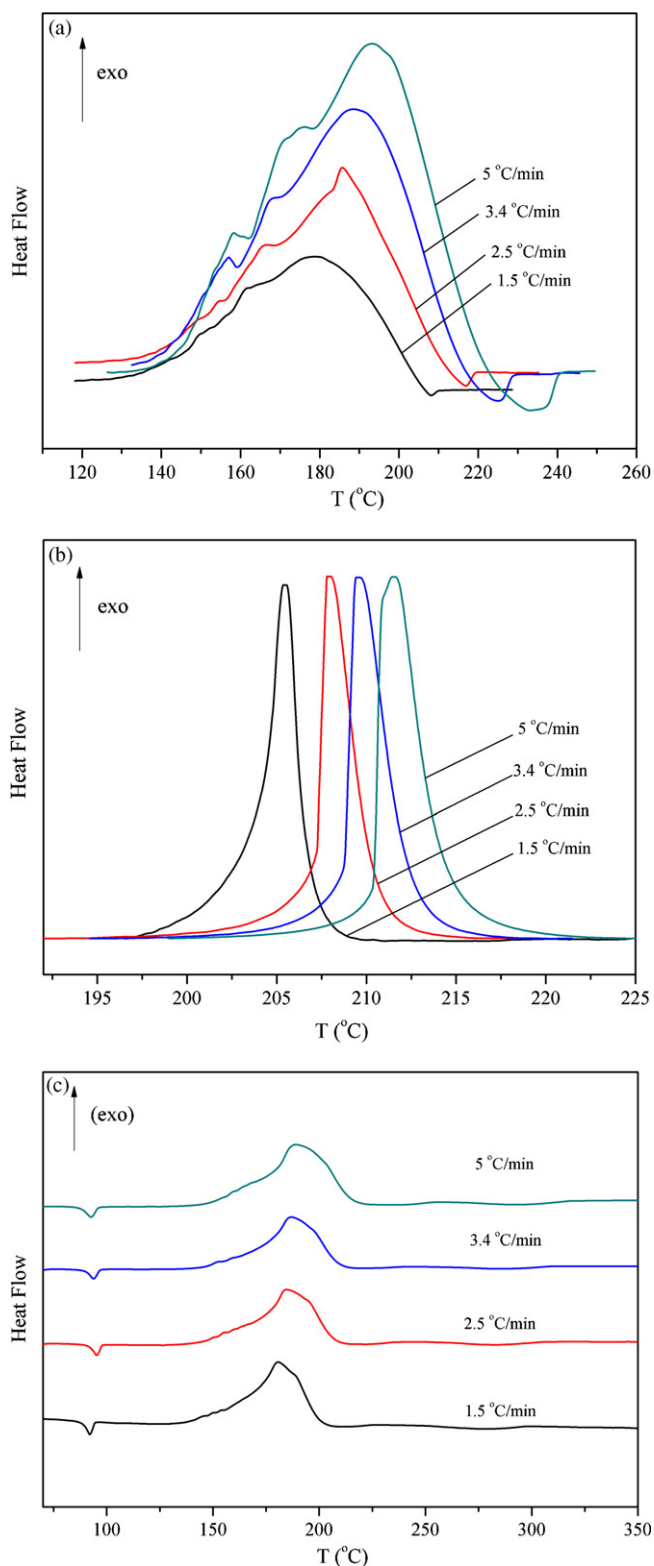
1. Introduction

Energetic materials are used extensively in composite propellants and explosives. Many new high-energy oxidizers were synthesized and characterized for military and space applications. A number of various dinitramide salts have been synthesized and characterized in the recent years [1]. Among the many dinitramide salts that have been reported, the outstanding ones that have attracted extensive interest are ammonium dinitramide (ADN) and guanylurea dinitramide (GUDN). A monograph highlighting the advances made on different dinitramides has been recently published by the authors [2]. ADN is emerging as a promising candidate to replace conventional ammonium perchlorate in solid propellants [3,4]. The chlorine-free and non-toxic combustion products and the high specific impulse (I_{sp}) of ADN based propellants are very attractive and are being studied all over the world for their potential use in modern composite solid propellants. On the other hand, GUDN or FOX-12 is a nitrogen rich dinitramide salt which is intended for use as gas generating compositions in air bags [5]. Its excellent stability and very low mechanical sensitivity finds potential use in insensitive munitions explosives [6–8]. The chemical structure of ADN and GUDN is shown in Scheme 1.

It has been shown that high performance insensitive munitions compositions can be prepared employing GUDN with various high-energy oxidizers [8]. Melt–cast charges of ADN along with high-energy oxidizers such as cyclotrimethylenetrinitramine (RDX), cyclotetramethylenetetranitramine (HMX) and hexanitrohexazaisowurtzitane (HNIW or CL-20) are reported [9]. Considering the insensitive nature of ADN and GUDN, melt–cast charges employing these two oxidizers could give high-energy formulations with reduced sensitivity and high thermal stability.

The kinetics of the thermal decomposition of ADN and its mixtures has been widely studied by many model-fitting and model-free methods [10–16]. Only limited studies have been carried out on the thermal decomposition characteristics of GUDN [17,18]. These studies derive only E_a and the pre-exponential factor ln(A) by model-fitting methods and none of them address the dependence of the E_a on α. Model-fitting methods derive the overall kinetics based on a single heating rate and provide only a single E_a value for the whole process. However, the thermal decomposition processes of many energetic materials involve multi-step kinetics. As such, model-fitting methods cannot be used for reliable kinetic analysis of the material decomposition over the experimental range of temperatures. On the other hand, isoconversional methods provide a comprehensive description of the decomposition process of a heterogeneous solid-state reaction. Based on the results of the International Confederation for Thermal Analysis and Calorimetry (ICTAC) kinetics project [19], the multi-heating rate methods together with isoconversional methods are the most reliable tech-

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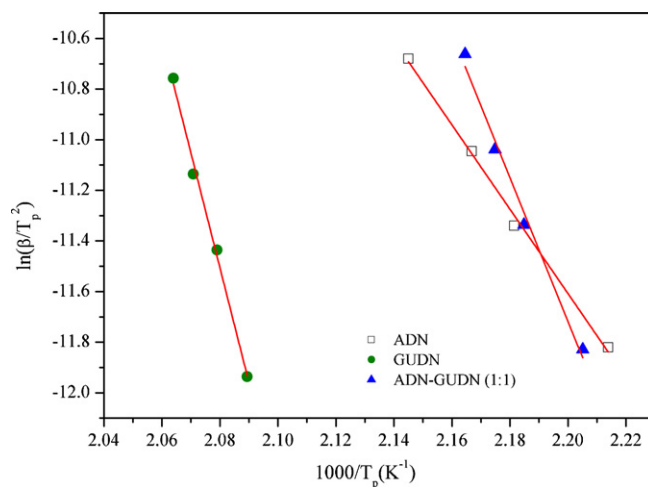**Scheme 1.** Chemical structures of high-energy oxidizers used.

niques for the analysis of thermal data. Isoconversional methods [20] are increasingly being used for obtaining reliable and consistent kinetic data for polymers [21,22], energetic materials [23,24] and other materials [25].

In this paper, a detailed study on the mixture of these two novel oxidizers was carried out to determine the kinetics and the associated energetics of the thermal decomposition. The isoconversional methods of Friedman and AIC-V were used to evaluate the E_a values at different stages of α during the thermal decomposition of ADN–GUDN mixture. The results are compared to the E_a values calculated using the Kissinger method for the mixture and for its individual components. The role of individual components on the observed apparent E_a values of the mixture was further discussed based on the decomposition enthalpy values and as well as from Fourier transform infrared (FTIR) spectroscopic results. The results from the kinetic analyses of ADN–GUDN mixture will provide more insights into the combustion characteristics, performance and safety data of the chosen composition.

2. Experimental

2.1. Materials

ADN was prepared in the laboratory according to a reported procedure by the reaction of ammonium sulfamate ($NH_2SO_3NH_4$) with fuming HNO_3/conc. H_2SO_4 mixture [26]. The prepared ADN was twice recrystallized from ethylacetate, dried under vacuum and stored in a desiccator.

GUDN was prepared from the double decomposition reaction of ADN with guanylurea sulfate. Guanylurea sulfate (30.2 g, 0.1 mol) was dissolved in 50 ml of water with slight warming and stirring. When the solution is clear, it was taken out and cooled to room temperature. ADN (24.8 g, 0.2 mol) dissolved in 10 ml of water was added to the above solution. The precipitated fine white crystals were filtered, washed several times with cold water and finally dried under vacuum. The material was used without further purification.

ADN–GUDN mixture (1:1) was prepared by mixing equal amounts of ADN and GUDN (by weight) in an agate mortar under inert atmosphere. The thoroughly mixed ADN–GUDN was further dried under vacuum and stored in a desiccator.

2.2. Methods

Differential scanning calorimetry (DSC) experiments were carried out using a Setaram Labsys DSC-1600 differential scanning calorimeter. Nonisothermal runs were performed at constant heating rates of 1.5, 2.5, 3.4 and 5 °C min⁻¹ in the temperature range from 30 to 300 °C. Samples were placed in open aluminium pans (100 μl) and heated in a flowing nitrogen (50 ml min⁻¹) atmosphere. A sample mass of 4.5–5 mg were used for the nonisothermal experiments. Specific standards recommended by the supplier have been employed for the heat and temperature calibrations.

FTIR measurements were done using a Shimadzu FTIR-8300 spectrophotometer using KBr pellets.

Table 1
Temperature at the maximum of the DSC exothermic peak (T_p) for the different systems analyzed.

β (°C min⁻¹)	T_p (°C)		
	ADN	GUDN	ADN–GUDN (1:1)
1.5	178.7	205.6	180.5
2.5	185.4	208.0	184.7
3.4	188.5	209.9	186.8
5.0	193.2	211.5	189.0

The data analysis and kinetic calculations from the DSC measurements were done by using either Microsoft® EXCEL, Microcal ORIGIN® or Mathworks MATLAB® software.

3. Results and discussion

Fig. 1 shows the thermograms obtained from DSC for ADN, GUDN and ADN-GUDN (1:1) at heating rates of 1.5, 2.5, 3.4 and 5 °C min⁻¹. The corresponding temperature values at the peak of the exothermic curve (T_p) are reported in Table 1. The data from Table 1 have been used for the determination of activation energy by using a simple model based on Kissinger method.

As seen in Fig. 1, all the systems undergo exothermic decomposition. ADN displayed a broad exothermic peak in the temperature range of 140–240 °C, while GUDN displayed a narrow exothermic peak in the temperature range of 195–220 °C. It is also seen that the onset temperature of ADN decomposition is much lower that of the onset temperature of GUDN decomposition. The DSC thermograms of the ADN–GUDN mixture showed an exothermic peak in the temperature range of 150–210 °C, and a small endotherm near 92 °C which is attributed to the melting of ADN. A closer look at the DSC thermograms of the mixture revealed that the decomposition temperature regime is much closer to that of ADN. As seen in Table 1, the T_p values for ADN and ADN–GUDN mixture are very close, while the values for GUDN are 22–25 °C higher than that of mixture.

3.1. Kinetic computations

The basic equation for kinetic analysis is shown in Eq. (1).

$$\beta\frac{d\alpha}{dT} = f(\alpha)A \, \exp\left(-\frac{E_a}{RT}\right) \tag{1}$$

where T is the temperature, β is the linear heating rate, A is the pre-exponential factor, R is the gas constant, $f(\alpha)$ is the differential function of conversion, and α is the degree of conversion which is a normalized measure of reaction progress ranging from 0 to 1 as a function of time or temperature.

For our kinetic analysis, we employed the Kissinger method as well as isoconversional methods. The Kissinger method is used for the comparison of the E_a of ADN and GUDN with that of the ADN–GUDN mixture. The Friedman's and AIC-V method were employed on nonisothermal decomposition data of ADN–GUDN mixture at different heating rates. Isoconversional methods assume that at a constant extent of conversion, the reaction rate is dependant only on the temperature. This assumption is very useful as it allows equations to be derived that can calculate E_a without any prior knowledge of the analytical form of the conversion function $f(\alpha)$. As a result, the kinetic information calculated using isoconversional methods can provide more reliable insights into the kinetics and mechanism of complex reaction processes.

Fig. 2. Kissinger plots of $\ln(\beta/T_p^2)$ vs $1000/T_p$ for ADN, GUDN and ADN–GUDN (1:1).

is evaluated from the T_p values at different heating rates β using Eq. (2).

$$\ln\left(\frac{\beta_i}{T_{p,i}^2}\right) = Const - \frac{E_a}{RT_{p,i}} \qquad (2)$$

where subscript i denotes different heating rates. Unlike the iso-conversional methods, the Kissinger method takes a simplified approach that yields only a single E_a value for the whole process. The activation energy has been calculated from the linear plots of $\ln(\beta/T_p^2)$ vs $1000/T_p$ and the plots are shown in Fig. 2.

The results on the activation energies for ADN, GUDN and ADN–GUDN (1:1) calculated from Fig. 2 are reported in Table 2 along with the correlation coefficients (R^2). In all cases, we observed excellent fitting goodness with R^2 values above 0.99.

As can be seen from Table 2, the E_a of ADN and GUDN are 138.5 and 377 kJ mol^{-1} respectively. The E_a value of 235.8 kJ mol^{-1} for ADN–GUDN mixture lies in-between that of the rest. The observed E_a of the mixture results from the added contribution of the exothermic decomposition of ADN and GUDN taking place simultaneously in the temperature range of 150–210 °C.

Ostmark et al. [17] reported an E_a value of 277 kJ mol^{-1} for GUDN from the DSC measurements using ASTM E698-79. The authors have attributed the large E_a to its high degree of thermal stability. Zhao et al. [18] reported a value of 237.7 kJ mol^{-1} for GUDN by Kissinger method with a poor correlation coefficient of 0.9531. They used the kinetic reaction model $f(\alpha) = 2\alpha^{1/2}$ to describe the decomposition. Due to the difference in experimental conditions and processing of data, a comparison of the E_a values for GUDN reported in the literature with that of our results could not be made. In our studies, the larger E_a for GUDN can also be attributed to its high thermal stability and its extensive stabilization by hydrogen bonding.

Simple models such as the Kissinger method, allow studying the decomposition process from a broad perspective. It provides with very little specific information on the various thermal events happening during the decomposition reactions such as variation of E_a

Fig. 1. DSC thermograms at different heating rates using normalized heat flow axes, obtained for (a) ADN, (b) GUDN and (c) ADN–GUDN (1:1).

3.2. Kissinger method

The Kissinger method [27] has been used to estimate the E_a of ADN, GUDN and ADN–GUDN mixture. Assuming that the condition $d^2\alpha/dT^2 = 0$ is satisfied at the peak of each heat flow profile, the E_a

Table 2
Activation energies obtained with Kissinger method.

	E_a (kJ mol^{-1})	R^2
ADN	138.5	0.997
GUDN	377.0	0.996
ADN–GUDN (1:1)	235.8	0.990
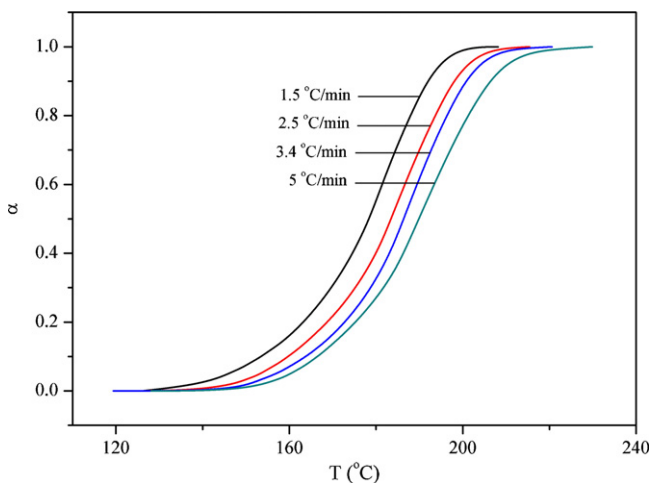
Fig. 3. Degree of conversion (α) as a function of temperature (T) at different heating rates for ADN–GUDN (1:1).

with α. It also fails to establish whether the process undergoes a single or multi-step kinetics. In this context, isoconversional methods provide more details as they determine the E_a dependence with respect to α.

3.3. Isoconversional analysis of ADN–GUDN decomposition

Isoconversional methods have been successfully used for the kinetic analysis of ADN by several authors [10,11,16].

The heat flow values recorded from the nonisothermal DSC experiments can be converted to α based on Eq. (3).

$$\alpha = \frac{\text{AUC}_0^T}{\text{AUC}_0^\infty} \tag{3}$$

where AUC_0^T is the sample peak area from 0 to T and AUC_0^∞ is the total sample peak area. Fig. 3 shows the variation of α with temperature (T) during the thermal decomposition of ADN–GUDN mixture at various heating rates. The exothermic peak areas in Fig. 1(c) were used to obtain the plots of α with T shown in Fig. 3.

It is seen in Fig. 3 that the run at 1.5 °C min^{-1} covers a temperature range of 130–205 °C, whereas the run at 5 °C min^{-1} covers about 145–220 °C min^{-1}.

3.3.1. Friedman's method

The method suggested by Friedman [28] shown in Eq. (4) is obtained by simple rearrangement of Eq. (1).

$$\ln\left(\frac{d\alpha}{dt}\right)_{\alpha,i} = \ln[A_\alpha f(\alpha)] - \frac{E_{a\alpha}}{RT_{\alpha,i}} \tag{4}$$

where the subscripts i and α denotes the different heating rates used and the conversion values. The value of $d\alpha/dt$ in Eq. (4) is obtained numerically using a spacing of $\Delta\alpha = 0.02$ and linear interpolation of the experimental data. For a given α, the data points of $\ln(d\alpha/dt)_{\alpha,i}$ vs $1/T_{\alpha,i}$ at different heating rates can be fitted to a straight line whose slope gives the activation energy. The dependence of E_a on α using Friedman's method is shown in Fig. 4.

3.3.2. Advanced isoconversional method of Vyazovkin

The advanced isoconversional method developed by Vyazovkin [29] is an integral method whose basic formula is shown in Eq. (5).

$$(g(\alpha))_\alpha = A_\alpha J[E_{a\alpha}, T_\alpha] \tag{5}$$
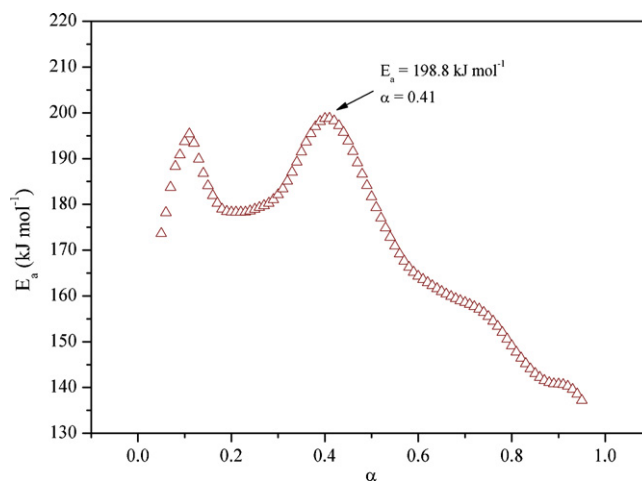
Fig. 4. Dependence of E_a with α according to Friedman's method for the decomposition of ADN–GUDN (1:1).

$$\Omega = \sum_{i=1}^{n} \sum_{j \neq i}^{n} \frac{J(E_{a\alpha}, T_{\alpha,i})}{J(E_{a\alpha}, T_{\alpha,j})} \tag{6}$$

$$J[E_{a\alpha}, T_{\alpha,i}] = \int_{T_{\alpha-\Delta\alpha,i}}^{T_{\alpha,i}} \exp\left[\frac{-E_{a\alpha}}{RT_i}\right] dT_i \tag{7}$$

where $g(\alpha)$ is the integral reaction model, the indexes i and j in Eq. (6) denote different heating rates, n is the total number of heating rates, and J is the temperature integral. Eq. (6) is formulated such that it is possible for the integration in Eq. (7) to be performed over small steps of $\Delta\alpha$. This reduces the systematic error observed in other integral methods which are essentially integrated using $\Delta\alpha = \alpha$. The use of $\Delta\alpha$ in the integral also makes the AIC-V method mathematically equivalent to Friedman's method as $\Delta\alpha$ approaches 0. However, for a fairer comparison with our numerical implementation of Friedman's method, $\Delta\alpha$ was chosen to be 0.02 in our calculations. The integral in Eq. (7) is solved numerically using the in-built function 'quadv' in MATLAB. The activation energy for any particular α is evaluated by minimizing Ω in MATLAB using the in-built function 'fminbnd'. The plot of the dependence of E_a on α is shown in Fig. 5.
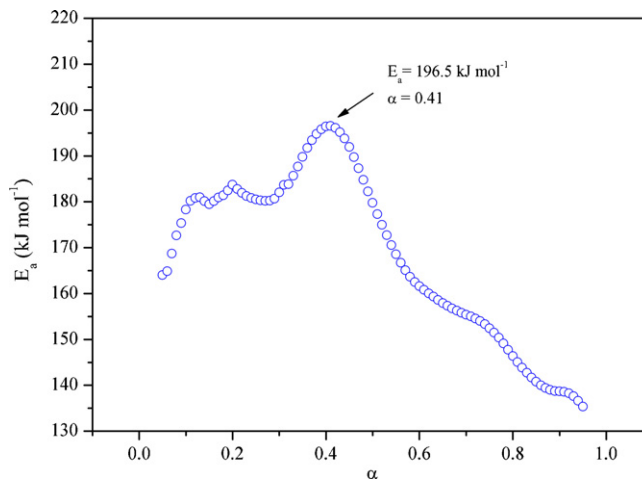

Fig. 5. Dependence of E_a with α according to AIC-V method for the decomposition of ADN–GUDN (1:1).

3.3.3. Comparison of results from isoconversional methods

The dependence of E_a on α presented in Figs. 4 and 5 indicates changes in the decomposition mechanism in the investigated range of T. The two isoconversional methods produce similar curves but with some inconsistency between $\alpha = 0$ to 0.25. This could be due to experimental noise from the uneven heat flow profile observed immediately after the start of decomposition as observed for ADN (Fig. 1a) as well as the ADN–GUDN (1:1) mixture (Fig. 1c). The peak E_a values by both methods lie very close at $\sim$196–199 kJ mol^{-1} at the same conversion level of 0.4. The average values (for α in the range of 0.05–0.9) of the E_a obtained by Friedman's and AIC-V method are 171.2 and 168.3 kJ mol^{-1} respectively. The E_a calculated by the Kissinger method for the mixture 235.8 kJ mol^{-1} (cf. Table 2) however, is $\sim$40 kJ mol^{-1} higher than that of the peak E_a value from the isoconversional methods. The reason could be because Kissinger method relies solely on the relative positions of T_p rather than considering the whole heat flow profile.

For the same range of heating rates employed in the study, the results obtained by Friedman's method show that E_a increases for $\alpha < 0.1$, then decreases for $0.1 < \alpha < 0.2$, then increases for $0.2 < \alpha < 0.4$, and then finally decreases steadily for $\alpha > 0.4$. The results obtained using the AIC-V method noticeably differs from Friedman's method in the range of $0.1 < \alpha < 0.3$ where the E_a values are almost constant instead. Otherwise, the behavior of E_a is very similar. The initial spiked E_a values shown in Fig. 4 at $0.1 < \alpha < 0.2$ could be attributed to the susceptibility of Friedman's method to experimental noise. Both Figs. 4 and 5 clearly shows a large variation of E_a with α, therefore indicating a complex decomposition mechanism (due to competitive and as well as reactions complicated by diffusion) in the range of α values studied. The behaviour is also an evidence of multi-step processes [10,11] where it involves the changing of reaction mechanisms at different conversion during the decomposition of ADN–GUDN mixture.

Vyazovkin and Wight [30] have given a detailed account on the dependence of E_a with α by providing a comprehensive account from studying the shapes of the dependence curves. In this work the increasing trend in E_a with α in the conversion regions 0–0.1 and 0.2–0.4 could be attributed to the competing reactions. On the other hand the rapid decrease in E_a from $\alpha \sim 0.4$ till the end of the reaction may be an indication of the mass transfer controlled diffusion mechanism [30]. Vyazovkin and Wight [10] have observed a similar behaviour for the decomposition of ADN and the decomposition process has been described with a multi-step mechanism. Recently, we used isoconversional methods and observed a similar trend for the decomposition of ADN prills [16].

The decomposition of ADN interacts with GUDN over a wide temperature range and the overall E_a value of the mixture is accounted from the combined contributions of the individual components. A comparison of the enthalpy values from the decomposition of ADN, GUDN and its mixture at 3.4 °C is made. The enthalpy of ADN decomposition is 2275.9 J g^{-1} (exo), while that of GUDN is 716.7 J g^{-1} (exo). So the 1:1 mixture of these two should result in a net enthalpy of 1496.3 J g^{-1} for the exothermic decomposition. However, the observed value for the mixture is much higher at 2036.1 J g^{-1} (exo) instead. This suggests that various oxidation reactions are occurring among the decomposition products of ADN and GUDN which may have altered the reaction path.

To further prove the significant role of ADN in the decomposition of ADN-GUDN mixture, FTIR spectroscopic measurements were performed on the mixture and as well as on the small amount of residue obtained after the decomposition. The results are presented in Fig. 6.

As seen in Fig. 6, the mixture exhibits characteristic peaks due to –C=O at 1689.6 cm^{-1} (from guanylurea), –C=N at 1743.5 cm^{-1} (from guanylurea) and the –NO$_2$ vibrations can be seen from 1020
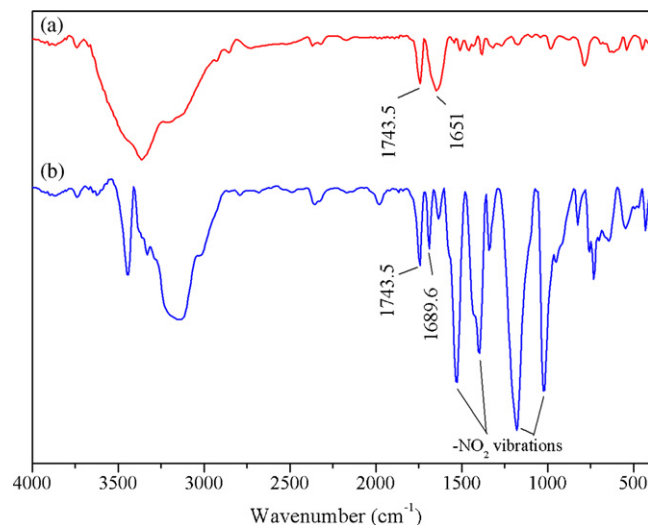

Fig. 6. Comparison of FTIR spectra of (a) residue obtained from DSC after the thermal decomposition of mixture and (b) ADN-GUDN (1:1).

to 1520 cm^{-1} (from the dinitramide anions of ADN and GUDN). The IR spectra of the residue obtained does not exhibit the characteristics peaks of –NO$_2$ vibrations, but shows the frequencies of –C=N and –C=O at 1743.5 and 1651 cm^{-1} respectively. This further confirms that ADN and the dinitramide anion from GUDN has decomposed completely in the mixture, suggesting the scission of N–N bonds, whereas the small amount of residue observed mainly originates from the guanylurea moiety. The chemical composition of the residue has not been characterized in our studies, and is beyond the scope of the present work. The proximity of the exothermic peaks for ADN decomposition with that for the ADN–GUDN mixture (cf. Table 1) further supports the observation for the significant role of ADN in the decomposition characteristics of the mixture. Further investigations would be required to better understand the complexities of the decomposition of GUDN and ADN–GUDN mixture.

4. Conclusions

The apparent activation energy for the thermal decomposition of ADN–GUDN mixture has been shown to vary with α based on isoconversional methods. Results on DSC experiments for the ADN–GUDN mixture yield E_a in the range of 165–200 kJ mol^{-1} for α from 0 to 0.4 followed by a decrease from 200 to 135 kJ mol^{-1} between α from 0.4 to 1 as analyzed by Friedman's and AIC-V methods. A comparison has been made between the peak E_a calculated from the isoconversional methods and the E_a values calculated using the Kissinger method. From our results, the large variation of E_a with α for ADN–GUDN mixtures indicates a complex decomposition mechanism that possibly includes competitive and diffusion controlled reactions. Comparison of the enthalpy values and the FTIR results suggest that in the ADN–GUDN mixture, the decomposition of ADN and the dinitramide anion in GUDN plays a prominent role for the observed E_a dependence on α.

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