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# Thermogravimetric Analysis of Rubber Glove Pyrolysis by Different Iso-conversional Methods

Jinxing Wang[1] · Haibo Zhao[1]

**Abstract** Pyrolysis kinetics of rubber glove with the heating rates ($\beta$) of 5, 10, 20 and 40 °C min$^{-1}$ were investigated by thermogravimetric analyzer from 100 to 1000 °C in nitrogen atmosphere. The results indicate that, during pyrolysis processes of rubber glove the peak of differential themogravimetry curves move toward higher temperature with the increase of heating rate and the change of temperature is approximately 40 °C from 5 to 40 °C min$^{-1}$. Meanwhile, three iso-conversional methods (Kissinger's method, Doyle's approximation and Flynn–Wall–Ozawa method) are used for analysing the non-isothermal pyrolysis kinetic of rubber glove. It is found that the difference of the apparent activation energies obtained by the three iso-conversional methods is no more than 21 kJ mol$^{-1}$ and the overall trends are very similar. The range of apparent activation energies for rubber glove pyrolysis is between 103.88 and 184.33 kJ mol$^{-1}$.

**Keywords** Rubber · Pyrolysis mechanism · Pyrolysis kinetic · TG · Iso-conversional method

## Introduction

Energy and waste management are two key issues of concern with the escalating industrial and economic growth [1]. The problem of solid waste disposal including medical wastes and other toxic hazardous wastes is growing throughout the world [2]. Especially, the hospital wastes will pose a great health risk to the public because of their infectivity if they are not properly disposed [3].

Rubber glove is the necessary consumable in every hospital. In order to prevent rubber's aging, the most common method is the chemical modification of natural rubber. It has been found that rare-earth ions could exert better protective effect on the process of aging of rubber [4]. Therefore, the rubber waste usually contains high concentrations of heavy metals and has become an important source of pollution. Disposal of rubber gloves (RG) is of growing interest due to more and more strict legislation in many countries. Traditionally the destruction of these wastes via landfill deposition and incineration were regarded as the preferred treatment options. However, land filling performs a potential danger because of the possibility of accidental fires with high emissions of hazardous gases [5]. What is worse, RG could not readily degrade and land filling of RG may pollute soils and groundwater resources. Compared with the landfill method, incineration can circumvent these disadvantages and reduce toxic effects of infectious materials, but one of the shortcomings of the incineration technology is the production of significant amounts of solid residue such as fly ash. Toxic chemicals like heavy metals are rich in fly ash [6]. Besides, Combustion of rubber glove will produce pollutants, including polycylic aromatic hydrocarbons (PAHs), benzene, styrene, phenols, and butadiene, which are harmful to human health [7]. These have put undue demands on conventional disposal options of RG such as landfills and incineration to lower these detriment. To reuse these wastes and minimize emissions of toxic substances, pyrolysis has been proposed as a promising process for treating rubber waste. In the pyrolysis process (heated under oxygen-deficient condition), the organic components of the material are

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decomposed, generating liquid and gaseous products with higher heating values (HHVs) [8]. The inorganic ingredients (fillers, metals, etc.) remain practically unaltered and free of the binding organic matter, and therefore metals could be separated and the remaining solid may be reused (additive, fillers, pigment, etc.) or as a last resort, it would be a minimum waste to be landfilled [9].

Thermogravimetric analyzer (TG) is an excellent tool for studying the pyrolysis kinetics. It is widely used because of its experimental simplicity and the wealth of information obtained from a simple thermogram [10]. Besides, thermal analysis techniques like TG provide rapid, quantitative methods for the examination of processes and allow for the estimation of effective kinetic parameters for various decomposition reactions [11]. The knowledge of the kinetics is essential for the subsequent design of industrial systems. Regarding on this subject, many methods have been introduced to different researches [3, 10–14]. For example, Singh et al. [15] used a model based on the first-order reactions to compare the kinetic parameters of pyrolysed waste materials obtained for TG-MS and TG-FTIR. Park et al. [11] used the single reaction model to analyze the activation energy and pre-exponential factor for the thermal decomposition of refuse derived fuel. Aboulkas et al. [14] used TG to investigate the thermal degradation processes for a series of mixtures of oil shale/plastic and performed a kinetic analysis by Doyle's approximation. Naskar et al. [10] analyzed the degradation kinetics of bitumen/waste plastics blend by using Kissinger and Flynn–Wall–Ozawa methods, which do not require knowledge of the reaction mechanism. Among them, isoconversional methods have been widely accepted to study reaction kinetics, including combustion and pyrolysis, because of its operational simplicity. So far, the iso-conversional methods of analyzing reaction kinetics are still not coincident, although the equations of these iso-conversional methods are semblable. These iso-conversional methods mainly include Kissinger's method [12, 13], Doyle's approximation [11, 14] and Flynn–Wall–Ozawa method [10].

The main purpose of this work is to compare the kinetic parameters obtained by three iso-conversional methods, Kissinger's method, Doyle's approximation and Flynn–Wall–Ozawa method. In the present work, pyrolysis kinetics of rubber is studied by thermogravimetric analysis and these apparent activation energies obtained by three iso-conversional methods were compared. It is anticipated that this study will be useful in disposing rubber waste.

## Experiments

In the present study, RG is provided by Health Center of HUST. The elemental composition, proximate analysis and calorific value of RG are shown in Table 1. The elemental composition and proximate analysis are based on ASTM D5373 criterion and GB212-91/GB212-84 criterion, respectively. The subscript "ad" means air dried basis. Therefore, $M_{ad}$, $V_{ad}$, $A_{ad}$ and $FC_{ad}$ mean the content of moisture, volatile, ash and fixed carbon in the samples on air dried basis. $C_{ad}$, $H_{ad}$, $O_{ad}$, $N_{ad}$ and $S_{ad}$ mean the content of C, H, O, N and S element in the samples on air dried basis. Calorific value is also measured on dry basis. These samples are dried in oven at 105 °C for 24 h, then RG is milled and sieved to obtain pure fractions of uniform particle from 0.1 to 0.2 mm. After above treatment, the samples are mixed and stored in a desiccator for test.

Themogravimetry (TG) experiments are carried out on a simultaneous thermal analysis (WCT-1D). The non-isothermal pyrolysis of the samples is performed in the furnace of the thermobalance under controlled temperature to obtain the corresponding TG and differential themogravimetry (DTG) pyrolysis curves. The TG baselines are corrected by subtraction of predetermined baselines which are determined under identical conditions except for the absence of a sample.

Each sample was heated to 100 °C over 10 min and was maintained at 100 °C for 5 min for sample drying. Then, the sample was heated to 1000 °C for pyrolysis by different heating rates ($\beta$), i.e., 5, 10, 20, 40 °C min$^{-1}$. In order to eliminate the effects caused by the mass and heat transfer limitations, a small quantity of samples (10 mg) are loaded into an $Al_2O_3$ crucible for each run under non-isothermal condition. In addition, ambient atmosphere during temperature-programmed pyrolysis is provided by a continuous nitrogen flow of 60 ml min$^{-1}$.

## Results and Discussion

### Kinetic Method for the Results of Experiments

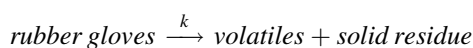The pyrolysis process of RG can be represented by the following reaction:

$$rubber\ gloves \xrightarrow{\ k\ } volatiles + solid\ residue$$

**Table 1** Data from elemental composition, proximate analysis and calorific value of rubber gloves

| Sample | Proximate analysis (wt%) | | | | Ultimate analysis (wt%) | | | | | Calorific value (MJ kg$^{-1}$) |
|---|---|---|---|---|---|---|---|---|---|---|
| | $M_{ad}$ | $V_{ad}$ | $A_{ad}$ | $FC_{ad}$ | $C_{ad}$ | $H_{ad}$ | $O_{ad}$ | $N_{ad}$ | $S_{ad}$ | |
| RG | 0.52 | 69.16 | 19.33 | 10.99 | 67.09 | 4.52 | 13.39 | 0.28 | 0.82 | 30.32 |

For pyrolysis process, the basic equation for kinetic analysis is shown in Eq. (1):

$$\frac{d\alpha}{dT} = \frac{A}{\beta} e^{(-E/RT)} f(\alpha) \tag{1}$$

In Eq. (1) $A$ (s$^{-1}$) and $E$ (kJ mol$^{-1}$) are Arrhenius parameters (the pre-exponential factor and the apparent activation energy, respectively), $f(\alpha)$ is the reaction model, $R$ (kJ mol$^{-1}$ K$^{-1}$) is the universal gas constant, $T$ (K) is absolute temperature, $t$ (s) is time, $\beta$ (=d$T$/d$t$) is the heating rate and $\alpha$ is the degree of conversion.

For solid waste [3, 4, 15], the assumed pyrolysis reaction mechanism is usually $(1-\alpha)^n$, where $n$ is the reaction order in the non-isothermal experiment. Usually the reaction order is assumed to be 1 [16, 17]. The degree of conversion of the reaction process can be expressed as [18, 19]:

$$\alpha = \frac{m_i - m_t}{m_i - m_\infty} \tag{2}$$

where $m_i$ is the initial mass of the sample, $m_t$ is the mass of the sample at time $t$, $m_\infty$ is the residual mass of the sample at the end of the reaction.

By the kinetic method it is usually expected to derive an appropriate kinetic description of the process in terms of the $E$. In this work, apparent activation energy from dynamic data is obtained by three iso-conversional methods (Kissinger's method, Doyle's approximation and Flynn–Wall–Ozawa method), without knowledge of reaction mechanism. These iso-conversional methods involve carrying out a series of experiments at different heating rates. More detailed explanation can be found in the literatures [10–14].

Firstly, Kissinger's method [12, 13] is based on Eq. (3):

$$\ln\left(\frac{\beta}{T_\alpha^2}\right) = \ln\left[\frac{AR}{E_\alpha g(\alpha)}\right] - \frac{E_\alpha}{RT_\alpha} \tag{3}$$

For Doyle's approximation, Eq. (4) is formulated [11, 14]:

$$\ln \beta = \ln\left[\frac{AE_\alpha}{Rg(\alpha)}\right] - 5.331 - 1.052\frac{E_\alpha}{RT_\alpha} \tag{4}$$

Flynn–Wall–Ozawa method [10, 20] is based on Eq. (5):

$$\log \beta = \log\left[\frac{AE}{g(\alpha)R}\right] - 2.315 - \frac{0.457E_\alpha}{RT_\alpha} \tag{5}$$

In these equations, $g(\alpha)$ is the integration function, which is shown as $\int_0^\alpha \frac{d\alpha}{(1-\alpha)^n}$, $T_\alpha$ and $E_\alpha$ represent the corresponding temperature and apparent activation energy when $\alpha$ is a designed value, respectively. In Eq. (3), because $\alpha$ is a constant, $\ln(\beta/T_\alpha^2)$ versus $-1/(RT_\alpha)$ obtained at

several heating rates yields a straight line whose slope allows evaluating of the apparent activation energy $E_\alpha$. Similarly, both the plot $\ln\beta$ versus $-1.052/(RT_\alpha)$ in Eq. (4) and the plot of $\log\beta$ versus $-0.457/(RT_\alpha)$ in Eq. (5) can obtain a straight line which can be used to evaluate the apparent activation energy.

## TG and DTG Curves Analysis

First of all, each sample is heated to prespecified final temperatures (900, 925, 950, 975, or 1000 °C) for pyrolysis by the heating rate of 10 °C min$^{-1}$ and is maintained at the final temperature for 3 h, respectively. The mass loss (TG) and temperature curves during the rubber glove pyrolysis are showed in Fig. 1. It can be seen that five mass loss (TG) curves are almost coincident before reaching the end temperature, which testifies reproducibility of the results. Moreover, it can be easily seen that the final temperature can impact on the degree of pyrolysis. The higher the final temperature is, the greater the degree of pyrolysis is. In the stage of heat preservation, the decline of TG curves is slow and even can be ignored. For pyrolysis characteristics of RG, therefore, more concerns should be focused on the stage of heating process.

The mass loss (TG) and the rate of mass loss (DTG) curves during the rubber pyrolysis at these heating rates are showed in Figs. 2 and 3, respectively. It can be seen that the main mass loss stages could be distinguished gradually with the increase of heating rate. Corresponding, the DTG curves have only one obvious mass loss peak. Furthermore, the DTG peaks move toward higher temperature and the absolute values of DTG peaks decrease with the increase of heating rate. From Table 1, it can be easily seen that RG has very high volatile (69.16 %), while the decline of TG curves approximately is 60 % at 2500 s when the TG curves appear turning point. That is, <90 % of organic
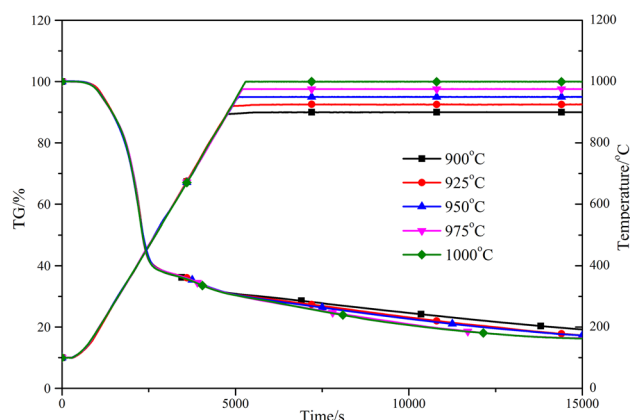


**Fig. 1** Different ultimate temperature for TG experiments at the heating rate of 10 °C min$^{-1}$
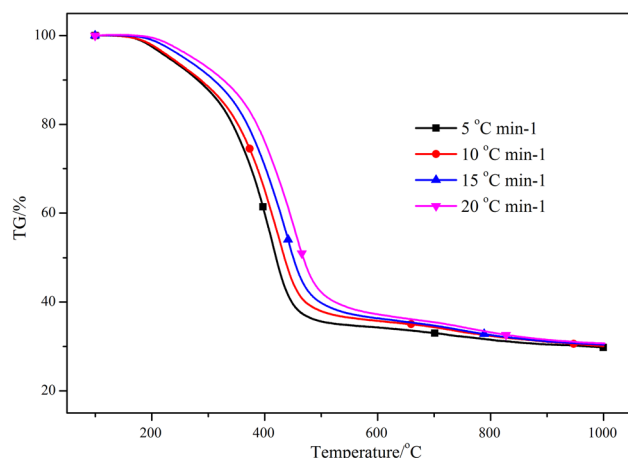
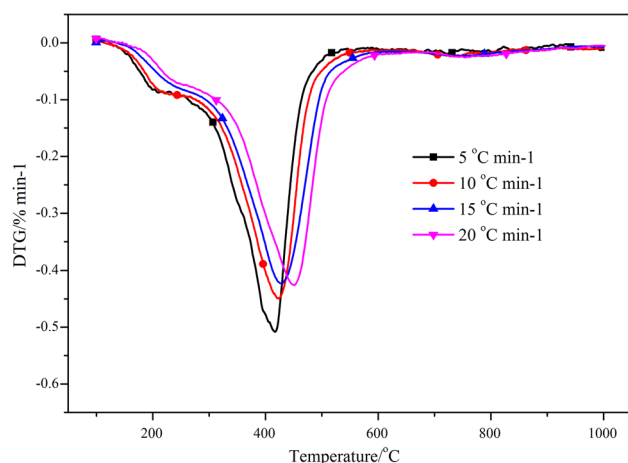**Fig. 2** TG curves of rubber gloves at different heating rates (β)



**Fig. 3** DTG curves of rubber gloves at different heating rates (β)

volatile components can release promptly. The most possible explanation is that the overwhelming majority of organic volatile components can release at 400 °C, and greater heating rates delays the release of part volatile and reduces the amount of volatile release at specific temperature.

In order to quantify the effect of heating rate, some pyrolysis characteristic parameters have been defined. $D_{max}$ is the peak of mass loss rate, $T_{max}$ is the temperature achieving to the DTG major peak, $T_{h1}$ and $T_{h2}$ are the

temperature when the mass loss rates first and second reach 50 % of $D_{max}$ during the heating process, respectively. Table 2 shows these pyrolysis characteristic parameters at different heating rates. It could be noted that the heating rate influences the release temperatures of volatile, as well as the mass loss rate at the same temperature. The $T_{h1}$ increase from 342.6 to 374.1 °C and the $T_{h2}$ increase from 443.9 to 488.4 °C with the increase of β. That is to say, the peak of DTG curves were shifted toward higher temperature with the increase of heating rate, and the change of temperature is approximately 40 °C from β of 5 to 40 °C min$^{-1}$. Correspondingly, the maximum mass loss rate decreases from 0.5079 to 0.4253 % min$^{-1}$. These results are in good agreement with those of the thermal degradation of polyropylene obtained by Dimitrios [21].

## Analysis of Kinetic Parameters

Although the pyrolysis of rubber need to last quite a long time (as shown in Fig. 1), TG in the stage of fast pyrolysis occupied a overwhelming proportion. Thus, the kinetic parameters in this paper are only applicable to the stage of fast pyrolysis. In Eq. (3), $m_\infty$ is the residual mass of the sample at 1000 °C.

According to Eq. (3), Eq. (4) and Eq. (5), the corresponding straight line should be obtained by means of the plot of $\ln(\beta/T_\alpha^2)$ versus $-1/(RT_\alpha)$, the plot of $\ln\beta$ versus $-1.052/(RT_\alpha)$ and the plot of $\log\beta$ versus $-0.457/(RT_\alpha)$, respectively. From the slope of line, we can estimate apparent activation energy ($E_\alpha$) of the dynamic pyrolysis process at various conversions α.

In the present study, four different heating rates (5, 10, 20 and 40 °C min$^{-1}$) are used to evaluate the relationship between $E_\alpha$ and α by the three iso-conversional methods. Nine different ratios of conversion (α): 10, 20, 30, 40, 50, 60, 70, 80, 90 %, are marked for the kinetic analysis corresponding to the RG pyrolysis. In order to intuitively show the results, the horizontal axis need to expand 1000 times. According to the three iso-conversional methods, straight lines are obtained and shown in Fig. 4. The intercept of linear fittings showed in Table 3 for each degree of conversion and the corresponding activation energy ($E_\alpha$) values and correlation coefficient ($R^2$) are also listed in this table. As shown in Table 3, the linear correlation

**Table 2** Pyrolysis characteristic parameters of samples at different rates

| Heating rate (°C min$^{-1}$) | $T_{h1}$ (°C) | $T_{max}$ (°C) | $D_{max}$ (% min$^{-1}$) | $T_{h2}$ (°C) |
|---|---|---|---|---|
| 5 | 342.6 | 416.9 | 0.5079 | 443.9 |
| 10 | 351.1 | 424.3 | 0.4498 | 458.5 |
| 20 | 356.9 | 427.2 | 0.4229 | 474.6 |
| 40 | 374.1 | 452.9 | 0.4253 | 488.4 |

(a) $\ln(\beta/T_\alpha^2)$ vs. -1000/(RT)



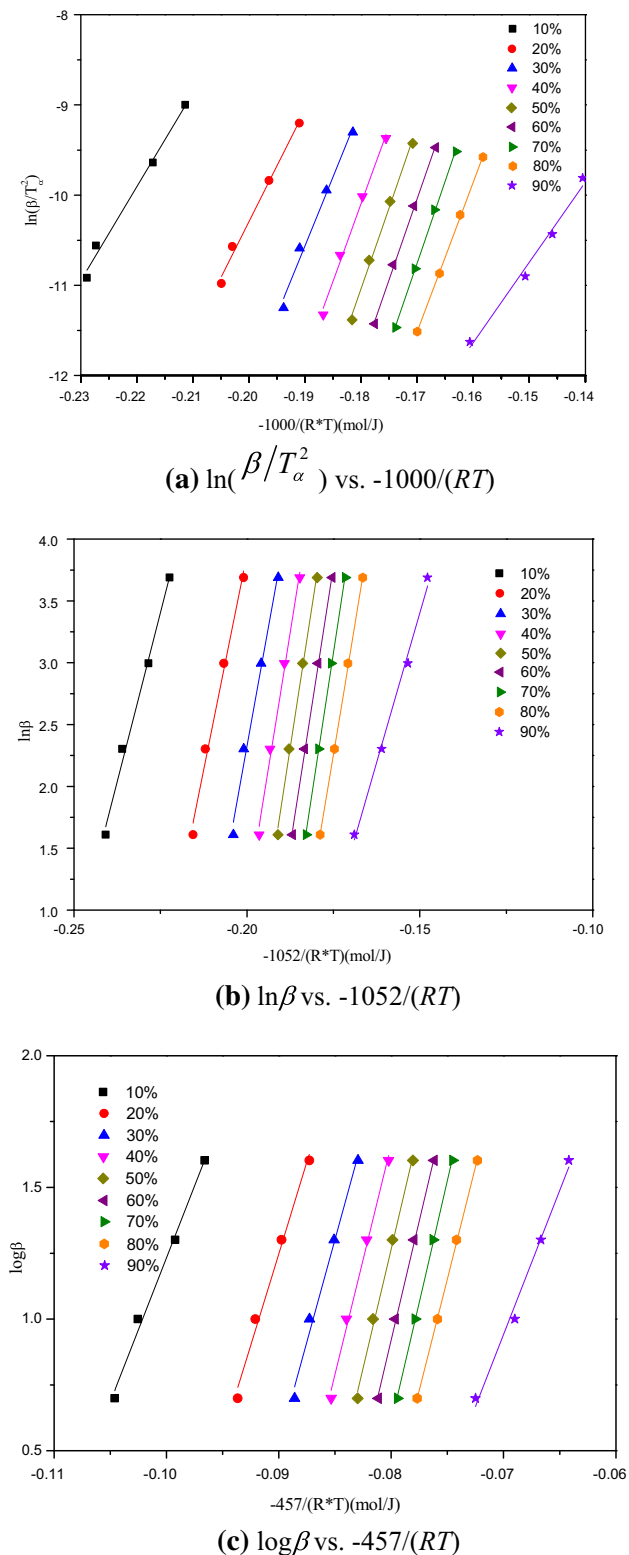(b) $\ln\beta$ vs. -1052/(RT)



(c) $\log\beta$ vs. -457/(RT)

**Fig. 4** Comparisons of Eqs. (3, 4 and 5) with 5, 10, 20 and 40 °C min$^{-1}$ for various RG conversion. **a** $\ln(\beta/T_\alpha^2)$ versus $-1000/(RT)$, **b** $\ln\beta$ versus $-1052/(RT)$, **c** $\log\beta$ versus $-457/(RT)$

coefficients for getting the activation energy are in the range of 0.98004–0.99954 and the fitting is good for every linear plot.

The consistent rule in the three methods is that the apparent activation energy ($E_\alpha$) gradually increases with the increase of conversion when α is <70 %. The possible reason is that at low conversions, the decomposition of RG proceeds to cut the side chain of polymer, and then the main chain decomposes with the increase of conversions [22]. Furthermore, the apparent activation energy ($E_\alpha$) decreases with the increase of conversion when α is more than 70 %, which manifests that temperature is likely to be the main factor constraining pyrolysis. These rules of apparent activation energy ($E_\alpha$) are similar to the pyrolysis of low density polyethylene (LDPE) and polypropylene (PP) [14], and the apparent activation energy also depends on the material types. Moreover, the difference of the apparent activation energies obtained by the three iso-conversional methods is no more than 21 kJ mol$^{-1}$ and the overall trends are very similar. The range of apparent activation energies for RG pyrolysis is between 103.88 and 184.33 kJ mol$^{-1}$. Therefore, the above results indicate that the three iso-conversional methods show a good match in spite of the existence of little difference. However, it should be pointed out that the kinetic parameters in this work are based on mass loss rate characterized only by temperature with different heating rates. The effect of sample quality, gas atmospheres and flow velocity on RG pyrolysis should be further studied in the following study.

## Conclusions

This work conducts pyrolysis experiments of rubber glove with different heating rates ($\beta$) of 5, 10, 20 and 40 °C min$^{-1}$ and pyrolysis kinetics analysis through three iso-conversional methods (Kissinger's method, Doyle's approximation and Flynn–Wall–Ozawa method). Following conclusions are made in this study.

1. During pyrolysis processes of rubber glove, the peak of DTG curves move toward the higher temperature with the increase of heating rate and the change of temperature is about 40 °C from 5 to 40 °C min$^{-1}$.
2. The consistent rule in the three methods is that the apparent activation energy ($E_\alpha$) gradually increase with the increase of conversion when α is <70 % and the rules reverse when α is more than 70 %.
3. The difference of the apparent activation energies obtained by the three iso-conversional methods is no more than 21 kJ mol$^{-1}$ and the overall trends are very

**Table 3** Apparent activation energies obtained by TG data at different rates by the three iso-conversional methods

| Iso-conversional method | $\alpha$ | Intercept | $E_\alpha$ (kJ mol$^{-1}$) | $R^2$ |
|---|---|---|---|---|
| Kissinger's method | 0.1 | 12.94901 | 103.88 | 0.98714 |
| | 0.2 | 14.41498 | 123.54 | 0.98837 |
| | 0.3 | 18.51131 | 153.01 | 0.98004 |
| | 0.4 | 20.93795 | 172.49 | 0.99023 |
| | 0.5 | 21.30375 | 179.68 | 0.99356 |
| | 0.6 | 20.36872 | 178.88 | 0.99004 |
| | 0.7 | 20.15549 | 181.82 | 0.99875 |
| | 0.8 | 16.70674 | 166.03 | 0.99943 |
| | 0.9 | 2.60031 | 88.97 | 0.98067 |
| Doyle's approximation | 0.1 | 28.0175 | 109.38 | 0.99022 |
| | 0.2 | 31.72232 | 139.25 | 0.98499 |
| | 0.3 | 33.44587 | 155.62 | 0.98282 |
| | 0.4 | 35.92704 | 174.33 | 0.99152 |
| | 0.5 | 36.37432 | 181.68 | 0.99442 |
| | 0.6 | 35.48193 | 181.16 | 0.99776 |
| | 0.7 | 35.30046 | 184.13 | 0.99892 |
| | 0.8 | 31.9013 | 169.41 | 0.99949 |
| | 0.9 | 17.94592 | 96.94 | 0.99184 |
| Flynn–Wall–Ozawa method | 0.1 | 12.1595 | 109.27 | 0.9892 |
| | 0.2 | 13.76884 | 139.12 | 0.98489 |
| | 0.3 | 14.51814 | 155.49 | 0.98296 |
| | 0.4 | 15.59919 | 174.23 | 0.99149 |
| | 0.5 | 15.79606 | 181.62 | 0.99416 |
| | 0.6 | 15.41379 | 181.17 | 0.99769 |
| | 0.7 | 15.34938 | 184.33 | 0.99898 |
| | 0.8 | 13.85401 | 169.35 | 0.99954 |
| | 0.9 | 8.60002 | 109.41 | 0.98611 |

similar. The range of apparent activation energies for RG pyrolysis is between 103.88 and 184.33 kJ mol$^{-1}$.

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