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Kinetic, thermodynamic and chemical reaction analyses of typical surgical face mask waste pyrolysis

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\section*{Article info}

\textbf{Keywords:}
Surgical face mask
Pyrolysis
Kinetics
Thermodynamics
Volatile products
TG-FTIR-MS

\textbf{Abstract}

The epidemic of Corona Virus Disease 2019 (COVID-19) has led to the generation of a large number of waste surgical masks. In recent years, pyrolysis is considered to be an environmental-friendly and efficient method to dispose such solid waste. In this work, the thermal degradation behaviors, kinetic parameters, thermodynamic parameters, pyrolytic products and chemical reactions of typical surgical face mask waste were studied using thermogravimetric analysis (TGA) and Fourier transform infrared (FTIR)-mass spectrometry (MS) analysis in inert atmosphere. It is concluded that the surgical face mask waste pyrolysis in nitrogen can be considered to be a one-step reaction. In addition, the mean $E$ (activation energy) value and the mean $A$ (pre-exponential factor) value are $237.19$ kJ/mol and $1.36 \times 10^{14}$ min$^{-1}$, respectively. $g(\alpha) = (-\ln(1-\alpha))^{2/3}$ (reaction model) may be utilized to characterize the surgical face mask waste pyrolysis in nitrogen. The above kinetic parameters are capable to estimate the surgical face mask waste pyrolysis behaviors in nitrogen. Thermodynamic parameters suggest that the surgical face mask waste pyrolysis can be considered to be an endothermic and non-spontaneous reaction. Inorganic substances, alkanes, alkenes, naphthenic hydrocarbons, aldehydes and ketones are the major volatile products. The amount of the aliphatic compounds is the highest. Specific chemical reactions generating these volatile products are proposed.

\section{Introduction}

The epidemic of Corona Virus Disease 2019 (COVID-19) has led to the generation of a large number of waste surgical masks and most of them are generated in daily life. Those waste surgical masks generated in daily life are classified as domestic waste and are less likely to carry virus such as novel coronavirus compared with those generated in hospitals. Those waste surgical masks are the object of the present study.

The waste surgical masks generated in daily life are dumped in specialized dustbins to avoid the secondary pollution caused by the potential contact of the waste medical surgical masks with other domestic wastes, then they are collected by the professionally trained staffs wearing the professional protective tools (such as the protective clothing, goggles, medical surgical masks, protective gloves, protective shoes, etc.). In the process of collection, these staffs will spray the disinfectant (such as 84 disinfectant, medical alcohol, etc.) into the specialized dustbins and the recycling bags containing the waste surgical masks. Finally, the recycling bags containing the waste surgical masks are delivered to the specialized trucks, which will transport the waste surgical masks to the dispose plant. During the transport process, the specialized trucks and the relevant transport roads will be sprayed with disinfectant, too.

It is a great challenge for the dispose plant to dispose these waste surgical masks. Traditional methods for solid waste disposal such as landfill and incineration will greatly pollute soil, water and air \cite{1,2}. In recent years, pyrolysis has proved successful for the disposal of solid waste. It can effectively recycle valuable chemical feedstock or fuel from the solid waste in an environmental-friendly way \cite{3-5}. It has been widely reported that by appropriately operating the pyrolysis process parameters, the pyrolytic product yield can be optimized \cite{6,7}. Thus, it is necessary and important to study the pyrolysis characteristics, kinetics, thermodynamics, pyrolytic products and chemical reactions of the waste surgical masks.

Previous research was mainly devoted to the pyrolysis of the major component of the surgical masks namely polypropylene (PP). As to the pyrolysis kinetics of PP, Kim et al. \cite{8} conducted the kinetic analysis of the PP pyrolysis at isothermal conditions (678–738 K) in nitrogen. In the temperature range of 678–693 K, the obtained average activation energy...
(E) and pre-exponential factor (A) values were 133 kJ/mol and 8.82 × 10^9 min^-1, respectively. Besides, the determined reaction model (g(α)) was 3(1-α)^2/3 (α stands for the conversion rate). However, in the temperature range of 723–738 K, the obtained average E and A values were 99 kJ/mol and 1.79 × 10^10 min^-1, respectively. The determined g(α) was 2(1-α)/(ln(1-α))^{1/2}. Using the α data under non-isothermal conditions (2, 10, 20, 50 K/min in the nitrogen atmosphere, Aboukas et al. [9] performed the kinetic analysis of the PP pyrolysis via three typical model-free methods (Friedman (FR) method [10,11], Kissinger-Akahira-Sunose (KAS) method [12–14], Flynn-Wall-Ozawa (FWO) method [15–18]) and two model-fitting methods (Goats-Redfern (CR) method [19,20], Criado method [21]). The obtained average value of E was in the range of 179–188 kJ/mol. The estimated g(α) was 1-(1-α)^1/3. The mean E value for the PP pyrolysis under nitrogen was also calculated employing KAS method (206 kJ/mol) [22], Freeman-Carroll integral method (167–181 kJ/mol) [23] and FWO method (222.3 kJ/mol) [24].

In the medical surgical mask, the filter layer is comprised of three layers. The outer layer is called the waterproof layer, which is comprised of health-level spunbonded non-woven fabric. The interlayer is called filter layer or core layer, which is the most important layer for the medical surgical mask. The filter layer is comprised of the efficiently filterable melt-blown non-woven fabric. The inner layer is called moisture-absorption layer, which is comprised of health-level spunbonded non-woven fabric. It should be noted that the component of the three layer is different (the structure and the degree of polymerization of PP contained in the three layers are different due to the performance requirement). In addition, cross-linking agent is also used in the medical surgical mask besides PP. In the present study, realistic medical surgical mask waste was adopted for thermogravimetric experiments. The pyrolysis behaviors of the realistic medical surgical mask waste are inevitably different from those of pure PP.

In addition, even though the pyrolysis kinetic parameters (E, A and g(α)) of the pure PP were estimated in the previous studies, the mean E and A values are not consistent among the previous research, and the g(α) is even totally different from each other among the previous research. More importantly, the applicability and feasibility of the estimated kinetic parameters in the previous research were not verified. It should also be noted that the thermodynamic parameters of the pure PP pyrolysis, which are always obtained based on the E and A values, receive little attention to date to the best knowledge of authors.

As to the volatile products of the PP pyrolysis, Kuipers et al. [25,26] analyzed the volatile products of the PP pyrolysis in a bench-scale rotating cone reactor in nitrogen using gas chromatography (GC) at 898 K, 923 K and 1073 K. They found that the volatile products mainly contained methane, ethene, propene, butene, butadiene and other unknown alkanes. Ballice et al. [24] also analyzed the volatile products of the PP pyrolysis in a plasma reactor in nitrogen using GC at 1073 K. It should be noted that the previous studies mainly focused on the volatile products of the PP pyrolysis at specific temperatures. The real-time volatile products throughout the entire PP pyrolysis process, which are of great importance for the analysis of the chemical reactions occurring in the PP pyrolysis process, were not studied.

In this study, the practical surgical face mask waste pyrolysis behaviors were investigated using thermogravimetric analysis (TGA) in nitrogen. The E, A and g(α) were calculated using three typical model-free methods as well as a representative model-fitting method. Furthermore, these three kinetic parameters’ accuracy was analyzed. The enthalpy change (ΔH), the Gibbs free energy (ΔG) as well as the entropy (ΔS) were calculated and analyzed. The pyrolytic products as well as the chemical reactions during the pyrolysis process were analyzed using real-time TGA-Fourier transform infrared (FTIR)-MS in helium.

2. Experiment conditions and kinetic theory

2.1. Experiment conditions

2.1.1. Material

The disinfected surgical mask wastes, which were provided via Shengming Medical Technology company limited (Yiwu City, Jiangsu Province, China), was employed for the TGA and online TGA-FTIR-MS tests. It should be noted that the surgical mask contains face mask and rope. The studied component in the current study is the face mask (not rope), whose major composition is PP, as shown in Fig. 1. The chemical structure of the PP adopted in the present study is presented in Fig. 2. The proximate and ultimate analyses of the specimen are presented in Table 1.

2.1.2. TGA tests

A TGA with the type of SDT A851E was used to monitor the surgical mask waste degradation under nitrogen at the slow/conventional heating rates [31] (shown in Table 2) of 15, 20, 25 and 30 K/min, which is conducive to obtaining a large number of pyrolysis products and facilitating the subsequent analysis [31–33]. The nitrogen flux was set to be 100 mL/min. 323–883 K was set to be the temperature region. The specimen weight was about 3.6 mg.

2.1.3. Online TGA-FTIR-MS tests

A real-time TGA coupled with FTIR (Agilent 7890B-5977B) and MS facilities (Nicolet i550) were used to analyze the compositions of the pyrolytic products of the surgical mask waste under helium. The FTIR spectra are generated according to that different functional groups absorb different infrared with different wavenumbers and the absorbance intensity is in proportional with the gas concentration. Mass spectrometer is mainly composed of ion source, mass analyzer and detector. The gas products volatilized from the thermogravimetric analyzer are first broken into fragments ions under the electron ionization (EI) in the ion source, and then the fragments ions are arranged by the mass analyzer in the order of mass-to-charge ratio (m/Q) to form mass spectra. The helium flux was also 100 mL/min. The program temperature was set to increase from 323 K to 883 K following 20 K/min. The specimen weight was about 3.6 mg.

2.2. Theory for kinetic calculation

The solid degradation is generally characterized by Eq. (1), t, T are reaction time and temperature, respectively, a is the conversion rate and its calculation formula is shown in Eq. (2). k(T) stands for the dependence of reaction rate on reaction temperature and its specific calculation expression is shown in Eq. (3) (where E, A, R stand for the activation energy, pre-exponential factor and universal gas constant, respectively).

\[ f(a) \] represents the differential form of the reaction model [35,36]. In Eq. (1),...
Table 1
Proximate and ultimate analyses of the specimen.

| Proximate analysis (wt%) | Ultimate analysis (wt%) |
|-------------------------|-------------------------|
| volatile matter         | C | H | N | S | Other elements |
| ash                     | 98.87 | 0.92 | 0.21 | 85.16 | 0.04 | 0.24 | 0.32 |

Note: a: dry basis; b: by difference.

Table 2
Commonly used pyrolysis parameters in industrial pyrolysis process [31–34].

| Pyrolysis technologies | Heating rate (K/min) | Temperature range (K) |
|------------------------|----------------------|-----------------------|
| Slow/conventional pyrolysis | 6-60 | 550-950 |
| Fast pyrolysis         | 600-12,000         | 850-1250 |
| Flash pyrolysis        | >60,000            | 1050-1350 |

(2), $m_0$ represents the initial mass of the solid before pyrolysis. $m$ represents the residual mass of the solid at one certain moment in the pyrolysis process. $m_w$ represents the final mass of the solid after pyrolysis. Combining Eq. (1) and Eq. (3), Eq. (4) can be obtained (therein, linear heating rate $\beta = \frac{dT}{dt}$, $da/dT$ is the reaction rate). By integration of Eq. (4), Eq. (5) can be obtained. Therein, $g(a)$ represents the integral form of the reaction model. $T_g$ is the temperature corresponding to $a$ during the reaction progress at different heating rates, $y = E/RT; y_o = E/RT_o$ in the present study. 34 types of $g(a)$ expressions were used, as listed in Table 3. It should be noted that no analytical solution occurs for $p(y)$ in Eq. (5), $p(y)$ were generally approximated for the kinetic calculation. In this work, four typical approximate solutions of $p(y)$ and corresponding kinetics calculation methods were adopted, as illustrated in sections 0–2.2.4.

\[
\frac{da}{dt} = k(T)f(a)
\]

\[
\alpha = \frac{m_0 - m}{m_0 - m_w}
\]

\[
k(T) = \exp \left( \frac{-E}{RT} \right)
\]

\[
\frac{da}{dT} = \frac{A}{\beta} \exp \left( -\frac{E}{RT} \right) f(a)
\]

\[
g(a) = \int_a^1 f(a) \exp \left( -\frac{E}{RT} \right) dT
\]

\[
p(y) = \exp \left( -1.052 \frac{E}{RT_o} - 5.331 \right)
\]

2.2.1. FWO method

The approximate formula for $p(y)$ utilized for the FWO method is shown in Eq. (6). By combining Eq. (6) with Eq. (5), the FWO method can be obtained and presented as Eq. (7) ($T_o, E_o, A_o$ are the temperature, activation energy and pre-exponential factor corresponding to one certain conversion rate $a$, respectively). The $E_o$ value could be obtained via the correlation of $\ln y$ with $1/T$ in the case of the same $a$ under multiple heating rates.

| No. | $g(a)$ | $f(a)$ | Rate-determining mechanism |
|-----|--------|--------|----------------------------|
| 1   | $1 - (1 - a)^{1/4}$ | $4(1 - a)^{3/4}$ | Chemical reaction |
| 2   | $(1 - a)^{-1/2} - 1$ | $2(1 - a)^{3/2}$ | Chemical reaction |
| 3   | $(1 - a)^{-1} - 1$ | $(1 - a)^2$ | Chemical reaction |
| 4   | $(1 - a)^{-2} - 1$ | $2(1 - a)^3$ | Chemical reaction |
| 5   | $(1 - a)^{-3} - 1$ | $3(1 - a)^4$ | Chemical reaction |
| 6   | $1 - (1 - a)\alpha$ | $2(1 - a)\alpha$ | Chemical reaction |
| 7   | $1 - (1 - a)^2$ | $3(1 - a)^2$ | Chemical reaction |
| 8   | $1 - (1 - a)^3$ | $3(1 - a)^3$ | Chemical reaction |
| 9   | $a^{3/2}$ | $2(1 - a)\alpha$ | Nucleation |
| 10  | $a^{3/2}$ | $2(1 - a)\alpha$ | Nucleation |
| 11  | $a^{3/2}$ | $2(1 - a)\alpha$ | Nucleation |
| 12  | $a^{3/2}$ | $2(1 - a)\alpha$ | Nucleation |
| 13  | $\ln(1 - a)$ | $2(1 - a)\alpha$ | Nucleation |
| 14  | $-\ln(1 - a)$ | $2(1 - a)\alpha$ | Assumed random nucleation and its subsequent growth |
| 15  | $[- \ln(1 - a)]^{3/2}$ | $3/2(1 - a)[- \ln(1 - a)]^{3/2}$ | Assumed random nucleation and its subsequent growth |
| 16  | $[- \ln(1 - a)]^{3/2}$ | $2(1 - a)[- \ln(1 - a)]^{1/2}$ | Assumed random nucleation and its subsequent growth |
| 17  | $[- \ln(1 - a)]^{3/2}$ | $3(1 - a)\alpha$ | Assumed random nucleation and its subsequent growth |
| 18  | $[- \ln(1 - a)]^{3/2}$ | $4(1 - a)[\ln(1 - a)]^{3/2}$ | Assumed random nucleation and its subsequent growth |
| 19  | $[- \ln(1 - a)]^{3/2}$ | $1/2(1 - a)[\ln(1 - a)]^{1/2}$ | Assumed random nucleation and its subsequent growth |
| 20  | $[- \ln(1 - a)]^{3/2}$ | $1/3(1 - a)[\ln(1 - a)]^{2/3}$ | Assumed random nucleation and its subsequent growth |
| 21  | $[- \ln(1 - a)]^{3/2}$ | $1/4(1 - a)[\ln(1 - a)]^{3/4}$ | Assumed random nucleation and its subsequent growth |

(continued on next page)
The approximate formula for $p(y)$ utilized for the KAS method is shown in Eq. (8). Combining this formula with Eq. (5), the KAS method can be obtained and presented as Eq. (9). The $E_y$ value could be obtained via the correlation of $\ln(\beta/T^2)$ with $1/T$ in the case of the same $\alpha$ under multiple heating rates.

$$p(y) = T_a^{0.92} \exp \left( \frac{-E_y}{RT_a} \right)$$

(10)

$$\ln \left( \frac{\beta}{T_a^2} \right) = \ln \left( \frac{A_y E_y}{g(\alpha) R} \right) - 0.312 - \frac{1.0008 E_y}{RT_a}$$

(11)

2.2.4. CR method

The above three methods are all model-free methods, that is, the values of $E$ can be obtained without knowing the specific reaction model. CR method belongs to mode-fitting methods. For model-fitting methods, the $E$ and $A$ values could be obtained on the basis of known reaction model. The approximate formula of $p(y)$ proposed by Murray and White [12] is the first two terms of Eq. (12). By combining this formula with the Eq. (5), the CR method can be obtained, as shown in Eq. (13). In general, for most cases of solid pyrolysis, $E/RT > 1$, then $1-2RT/E=1$, then, Eq. (13) can be simplified to Eq. (14). The $E_0$ and $A_0$ values could be estimated via the correlation of $\ln(g(\alpha)/T^2)$ with $1/T$ at different temperatures (corresponding to conversion rates) at single heating rate by assuming $g(\alpha)$ in advance.

$$p(y) = \exp \left( -\frac{y}{\gamma} \right) \times \left( 1 + \frac{2!}{\gamma^2} + \frac{3!}{(\gamma)^3} + \frac{4!}{(\gamma)^4} + \ldots \right)$$

(12)

$$\ln \left( \frac{g(\alpha)}{T_a^2} \right) = \ln \left( \frac{A_0 R}{E_0 \beta} \right) \left( 1 - \frac{2RT_a}{E_0} \right) - \frac{E_0}{RT_a}$$

(13)

$$\ln \left( \frac{g(\alpha)}{T_a^2} \right) = \ln \left( \frac{A_0 R}{E_0 \beta} \right) - \frac{E_0}{RT_a}$$

(14)

2.2.5. Thermodynamic theory

The enthalpy change expressed as $\Delta H$, Gibbs free energy expressed as $\Delta G$ and entropy expressed as $\Delta S$ are three major thermodynamic parameters. They are often utilized for evaluating the reaction feasibility and the energy change of the reaction system. The specific calculation formulas of these three thermodynamic parameters are shown as follows [39,41–43]:

$$\Delta H = E_y - RT$$

(15)

$$\Delta G = E_y + RT_a \ln \frac{k_b T_m}{h}$$

(16)

$$\Delta S = \frac{\Delta H - \Delta G}{T_m}$$

(17)

Therein, $T$ and $T_m$ are the corresponding temperature for each conversion rate and the peak temperature in the reaction rate curves, respectively. $k_b$ stands for the Boltzmann constant (1.381 × 10⁻²³ J/K⁻¹) and $h$ stands for the Planck constant (6.626 × 10⁻³⁴ J/s).

3. Results and discussion

3.1. Thermogravimetric data

The changes of $\alpha$ and $\alpha$ ds/dt with $T$ under 15, 20, 25 and 30 K/min are shown in Fig. 3. The maximum $\alpha$ ds/dt, the temperature for the maximum $\alpha$ ds/dt, and the average $\alpha$ ds/dt for the entire pyrolysis process are presented in Table 4. It is indicated that the surgical face mask waste pyrolysis mainly occurs between 600 and 775 K. With the increase of $\beta$, the shapes of the $\alpha$ and $\alpha$ ds/dt curves vary little, but the shift of the curves to the larger temperature zone occurs. The reason for this phenomenon is called “temperature hysteresis”, that is, the thermal transmission rate within the experimental sample cannot catch up with the
heating rate of the external program, and with the rise of heating rate, the influence of this phenomenon becomes more obvious. In addition, the increase of heating rate of the external program, and with the rise of heating rate, the increase of \( \beta \) has little effect upon the maximum and average \( \alpha/dT \), which is probably due to that the \( \alpha/dT \) value decreases with the increase of the \( \beta \) value and increases with the increase of the \( T \) value. As shown in Fig. 3, at the same \( \alpha \), the \( T \) value increases with the increase of the \( \beta \) value. At this time, the influences of \( \beta \) and \( T \) on the value of \( \alpha/dT \) may cancel out from each other. Besides, the temperature for the maximum \( \alpha/dT \) gradually increases with the \( \beta \).

### 3.2. Kinetic analysis

#### 3.2.1. Model-free methods

Activation energy, which is commonly expressed as \( E \), is an important parameter in the kinetics study. It refers to the minimum energy required for the reactant molecules to change from a normal state to an active state which is prone to the physical/chemical changes [6]. In this study, three typical model-free methods (KAS, FWO, Starink methods) are utilized to estimate the values of \( E \) under different \( \alpha \). The specific \( E \) values are presented in Table 5, and the changes of \( E \) with \( \alpha \) are shown in Fig. 4. It can be seen from Fig. 4 and Table 5 that in the pyrolysis process of the surgical face mask waste, the value of \( E \) hardly changes with \( \alpha \). Therefore, the surgical face mask waste pyrolysis in nitrogen could be titularly considered to be a single-step reaction. The average value of \( E \) for the entire degradation process can be estimated as 237.19 kJ/mol.

#### 3.2.2. Model-fitting method

According to section 3.2.1, the surgical face mask waste pyrolysis in nitrogen could be titularly considered to be a single-step reaction. In this section, the thermogravimetric data under 15, 20, 25 and 30 K/min and the CR method (a representative model-fitting method) were used to estimate the average \( E \) value for the whole pyrolysis process of the surgical face mask waste in nitrogen with known reaction models \( g(\alpha) \) (as listed in Table 3). As shown in Table 5, the values of \( E \) with the correlation coefficient \( R^2 \) are within the range of 0.979 to 0.9973, indicating that the accuracy of the calculation results by the CR method may be quite high. Therefore, \( g(\alpha) \) may be utilized to describe the surgical face mask waste pyrolysis in nitrogen. Then, the average value of \( A \) can be calculated to be 1.36 \times 10^{14} \text{ min}^{-1}.

“Kinetic Compensation Effect” commonly appears between \( E \) and \( A \) [10]. The corresponding function can be expressed as Eq. (18). In Eq. (18), \( a \) as well as \( b \) stand for the constants and \( a = \ln K_{iso}, b = 1/(RT_{iso}) \). \( K_{iso} \) stands for the isokinetic rate. \( T_{iso} \) stands for the isokinetic temperature. Provided that the values of \( T_{iso} \) are within the pyrolysis temperature range of the surgical face mask waste, the CR method and the adopted 34 types of reaction models can be considered to be suitable to calculate the kinetic parameters.

Fig. 5 shows the variations of ln\( A \) with \( E \). The values of \( a, b \) and 95% confidence interval (CI) for \( a, 95\% \text{CI for } b \) and \( T_{iso} \), are presented in Table 7. It indicates that the \( T_{iso} \) values at 15, 20, 25 and 30 K/min are 729.63 K, 738.50 K, 738.32 K and 740.82 K respectively, which are all within the pyrolysis temperature range of the surgical face mask waste (600–750 K). The \( R^2 \) value is quite close to 1 (\( R^2 = 0.999 \)). Therefore, it is suitable to use the 34 types of reaction models and CR method for the kinetic analysis. The correlation between \( E \) and \( A \) could be characterized to be ln\( A = 0.1632E - 1.1959 \).

\[
\text{ln} A = a + b E
\] (18)

#### 3.2.3. Verification of the feasibility of kinetic parameters

On the basis of the determined \( g(\alpha), E \) and \( A \), Eq. (19) which was obtained via Eqs. (5) and (6) is used to predict the \( a \) and \( \alpha/dT \) under 15, 20, 25 and 30 K/min. Fig. 6 shows the comparison between the predicted and experimental \( \alpha/dT \) values at 15, 20, 25 and 30 K/min. It can be seen from Fig. 6 that the prediction \( a \) and \( \alpha/dT \) values at 15, 20, 25 and 30 K/min.
| $\alpha$ | $E$ (KJ/mol) | $\ln(A)$ (min $^{-1}$) | $R^2$ | $E$ (KJ/mol) | $\ln(A)$ (min $^{-1}$) | $R^2$ | $E$ (KJ/mol) | $\ln(A)$ (min $^{-1}$) | $R^2$ | $E$ (KJ/mol) | $\ln(A)$ (min $^{-1}$) | $R^2$ |
|--------|-------------|-----------------|------|-------------|-----------------|------|-------------|-----------------|------|-------------|-----------------|------|
| 1      | 49.45       | 47.06           | 0.996 | 48.26       | 47.33           | 0.996 | 49.02       | 47.19           | 0.996 | 51.64       | 47.53           | 0.996 |
| 2      | 129.66      | 19.78           | 0.997 | 129.41      | 19.74           | 0.997 | 124.25      | 19.09           | 0.997 | 138.19      | 21.57           | 0.997 |
| 3      | 530.68      | 85.57           | 0.998 | 530.25      | 84.72           | 0.998 | 510.44      | 83.18           | 0.998 | 564.03      | 82.04           | 0.998 |
| 4      | 546.63      | 86.79           | 0.999 | 546.10      | 85.88           | 0.999 | 525.75      | 82.78           | 0.999 | 580.85      | 81.72           | 0.999 |
| 5      | 681.42      | 109.71          | 0.998 | 679.97      | 108.38          | 0.994 | 655.04      | 104.54          | 0.961 | 722.86      | 115.47          | 0.935 |
| 6      | 441.74      | 68.83           | 0.996 | 441.71      | 68.22           | 0.992 | 425.00      | 65.70           | 0.996 | 470.06      | 73.06           | 0.996 |
| 7      | 456.78      | 71.42           | 0.997 | 456.70      | 70.77           | 0.994 | 439.46      | 68.16           | 0.990 | 485.98      | 75.76           | 0.998 |
| 8      | 398.16      | 61.32           | 0.994 | 398.25      | 60.81           | 0.998 | 383.08      | 58.55           | 0.997 | 423.92      | 65.25           | 0.998 |
| 9      | 135.59      | 20.62           | 0.990 | 135.30      | 20.56           | 0.998 | 129.94      | 19.88           | 0.994 | 144.45      | 22.45           | 0.998 |
| 10     | 135.59      | 20.62           | 0.990 | 135.30      | 20.56           | 0.998 | 129.94      | 19.88           | 0.994 | 144.45      | 22.45           | 0.998 |

**Table 6**

Estimated $\ln A$, $E$ results through CR approach.
under each heating rate are in good agreement with the experiment ones. The small differences between the experimental and simulated results may be due to that the pyrolysis process of the surgical face mask waste cannot be strictly considered as a single-step reaction and errors occur in the estimation process of the kinetic parameters owing the adoption of the approximate solutions of \( p(y) \). The obtained reaction model \( g(\alpha) \), activation energy \( E \) and pre-exponential factor \( A \) in the present study may be utilized to estimate the degradation behaviors of the surgical face mask waste under nitrogen under wider heating rates.

\[
\alpha = 1 - \exp\left\{ - \frac{AE}{bR} \exp\left( -\frac{1.052E}{bR} - 5.331 \right) \right\}
\]

(19)

3.3. Thermodynamics parameter analysis

Fig. 7 depicts the changes of \( \Delta H, \Delta G \) and \( \Delta S \) with \( \alpha \) under 15, 20, 25 and 30 K/min. It can be seen from Fig. 7 that in the entire pyrolysis process, the \( \Delta H \) value varies from 211.08 kJ/mol to 248.25 kJ/mol. The \( \Delta G \) value basically remains unchanged. The \( \Delta S \) value varies from 33.12 J/(mol-K) to 81.2 J/(mol-K). The average \( \Delta H, \Delta G, \Delta S \) values corresponding to the whole decomposition process are 233.14 kJ/mol, 187.37 kJ/mol and 61.35 J/(mol-K), respectively.

Previous studies have shown that in one certain reaction, \( \Delta H \) reflects the energy differences between reactants and products [44]. If the \( \Delta H \) value is positive, it indicates that the energy of reactants is less than that of products, and the reaction is an exothermic reaction. As shown in Fig. 7(a), the \( \Delta H \) value is positive at each conversion rate, so it could be inferred that the surgical face mask waste pyrolysis belongs to an endothermic reaction. In addition, at the same conversion rate, the heating rate almost exerts no effect upon the \( \Delta H \) value. It was pointed out that if the difference between the average \( \Delta H \) value and the average \( E \) value does not exceed 7 kJ/mol, the thermal degradation would probably occur [45]. In this study, the difference between the mean \( \Delta H \) value and the mean \( E \) value (calculated via the three model-free methods) is 4.05 kJ/mol, so it can be speculated that the surgical face mask waste pyrolysis is prone to occur. In addition, the change of the \( \Delta H \) value with the conversion rate \( \alpha \) is much similar to that of \( E \).

\( \Delta G \) reflects the probability and difficulty level for the occurrence of one certain reaction [46]. The larger the \( \Delta G \) value is, the less probably the corresponding reaction will occur. In addition, if the \( \Delta G \) value is positive, the corresponding reaction may not occur spontaneously. As can be seen from Fig. 7(b), the \( \Delta G \) value for the pyrolysis process of the surgical face mask waste is positive at each conversion rate, so it may be concluded that the surgical face mask waste pyrolysis is a non-spontaneous reaction and merely occurs if external energy is provided (for example, heating). In addition, as shown in Fig. 7(b), at the identical \( \alpha \), with the increase of heating rate, the \( \Delta G \) value slightly declines. It may be concluded that with the rise of \( \beta \), the movement rate of the reactant molecules became faster. The probability of the collision between the reactant molecules increased, and the thermal degradation of the surgical face mask waste became easier. In the whole pyrolysis process, the \( \Delta G \) value remained almost unchanged, indicating that the feasibility of the surgical face mask waste pyrolysis changed little with \( \alpha \).

\( \Delta S \) is generally used to reflect the disorder degree of one certain reaction system [46]. If the \( \Delta S \) value is positive, it indicates that the disorder degree of the corresponding reaction system increases after the reaction occurs, and the reactants' disorder degree is smaller compared with the products. As can be seen from Fig. 7(c), in the pyrolysis process of the surgical face mask waste, the \( \Delta S \) values for the entire pyrolysis process are all positive, and the \( \Delta S \) value generally increases as the pyrolysis progresses, which may suggest that once the surgical face mask waste pyrolysis occurred, the disorder degree of the reaction system increased. And as the reaction continuously progressed, the disorder degree of the products gradually increased. This is consistent with the phenomenon that large quantities of volatile products formed continuously during the surgical face mask waste pyrolysis process, as illustrated in section 3.4. In addition, at the same conversion rate \( \alpha \), the \( \beta \) exerts little effect upon the \( \Delta S \) value.

3.4. Online TG-FTIR-MS analysis

Fig. 8 shows the absorbance intensity of the generated volatile products versus time during the surgical face mask waste degradation under helium. It is shown in Fig. 8 that a peak at 19.35 min occurs in the absorbance intensity curve, which indicates that large quantities of volatile products formed at this moment. The FTIR spectra for these products under the peak moment are shown in Fig. 9. The possible functional groups are listed in Table 8.

As shown in Fig. 9 and Table 8, the absorption peaks 3725 cm\(^{-1}\) corresponds to the stretching vibration of O-H, which indicates that water vapor/ethanol were generated in the pyrolysis of the surgical mask waste. The absorption peaks 3081, 2966, 2933 and 2885 cm\(^{-1}\) correspond to the stretching vibration of C-H and the absorption peaks 1462, 1379, 695, 991, 893 and 950 cm\(^{-1}\) correspond to the deformation vibration of C-H, which indicate that aliphatic compounds were generated in the pyrolysis of the surgical mask waste. The absorption peaks 2381, 2360 and 2306 cm\(^{-1}\) for the C=S stretching vibration and the absorption peak 672 cm\(^{-1}\) for the C=S the deformation vibration indicate that carbon dioxide was generated in the pyrolysis of the surgical mask waste. The absorption peaks 2181 and 2106 cm\(^{-1}\) for the

Table 7

| \( \beta \) (K/min) | \( a \) (min\(^{-1}\)) | 95% CI for \( a \) | \( b \) (mol/kJ) | 95% CI for \( b \) | \( k_{\exp} \) | \( T_{\exp} \) (K) | \( R^2 \) |
|-----------------|-----------------|----------------|----------------|----------------|----------|----------------|-------|
| 15              | -1.5983         | (-2.3160, -0.8096) | 0.1649         | (0.1631, 0.1665) | 0.2105   | 729.627        | 0.99  |
| 20              | -1.2949         | (-2.0523, -0.5375) | 0.1629         | (0.1611, 0.1645) | 0.2739   | 738.497        | 0.99  |
| 25              | -1.1121         | (1.8670, -0.3563)  | 0.1629         | (0.1611, 0.1646) | 0.3285   | 738.316        | 0.99  |
| 30              | -0.8249         | (-1.5832, -0.0665) | 0.1624         | (0.1607, 0.1639) | 0.4383   | 740.817        | 0.99  |
| Combination of all heating rates | -1.1960         | (-1.5654, -0.8265) | 0.1632         | (0.1624, 0.1640) | 0.3024   | 736.868        | 0.99  |
Fig. 6. Contrast between experiment and prediction $\alpha$ and $d\alpha/dT$. 
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The stretching vibration indicate the generation of carbon monoxide in the pyrolysis of the surgical mask waste. In addition, the absorption peak 2741 cm$^{-1}$ for the C–H stretching vibration and the absorption peaks 1764, 1745, 1731, 1650 cm$^{-1}$ for the C=O stretching vibration indicate that aldehydes/ketones were generated in the pyrolysis of the surgical mask waste. The above-mentioned volatile products as a function of time are presented in Fig. 10.

According to the Bouguer-Lambert-Beers law expressed as Eq. (20), the absorbance intensity of the volatile product is in a positive proportion to its concentration. Thus, as shown in Fig. 10, it can be concluded that the concentration of the volatile products in descending order is aliphatic compounds > carbon dioxide > aldehydes or ketones > carbon monoxide > water vapor or alcohols. It should be noted that the concentration of the aliphatic compounds is much higher than that of other volatile products, which may suggest that recycling waste medical

Fig. 7. The variations of $\Delta H$, $\Delta G$, $\Delta S$ with conversion rate ($\alpha$).

Fig. 8. Absorbance intensity of volatile products versus time.

Fig. 9. FTIR spectra for pyrolytic products at peak moment.
The types of functional groups contained in the volatile products [46].

| No. | Absorption peaks (cm⁻¹) | Function groups | Vibration | Corresponding products |
|-----|------------------------|----------------|-----------|------------------------|
| 1   | 3725                   | O–H            | Stretching | O–H for water vapor/alcohols |
| 2   | 2081, 2966, 2933, 2885 | C–H            | Stretching | C–H for aliphatic compounds |
| 3   | 1462, 1379, 695, 991, 893, 950 | C–H   | Deformation | vibration |
| 4   | 2381, 2360, 2306       | C=O           | Stretching | C=O for carbon dioxide |
| 5   | 672                    | C=O           | Deformation | vibration |
| 6   | 2181, 2106             | C=O           | Stretching | C=O for carbon monoxide |
| 7   | 2741                   | C–H           | Stretching | C–H for aldehydes |
| 8   | 1764, 1745, 1731, 1650 | C=O           | Stretching | C=O for aldehydes/ketones |

Table 9

Pyrolytic products determined via MS.

| No. | Possible products | Formula | Molecular weight | Family |
|-----|------------------|---------|------------------|--------|
| 1   | methane          | CH₄     | 16               | Alkanes |
| 2   | water            | H₂O     | 18               | Inorganic substances |
| 3   | ethylene         | C₂H₄    | 28               | Alkenes |
| 4   | carbon monoxide  | CO      | 28               | Inorganic substances |
| 5   | propylene        | C₃H₆    | 42               | Alkenes |
| 6   | carbon dioxide   | CO₂     | 44               | Inorganic substances |
| 7   | acetaldehyde     | C₄H₄O   | 44               | Aldehydes |
| 8   | butene           | C₄H₈    | 56               | Alkenes |
| 9   | acetone          | C₃H₆O   | 58               | Ketones |
| 10  | 1,3-pentadiene   | C₅H₈    | 68               | Alkenes |
| 11  | 2-amylene        | C₅H₁₀   | 70               | Alkenes |
| 12  | 1,2-dimethyl cyclopropane | C₅H₁₂ | 70 | Naphthenic hydrocarbons |
| 13  | methacrylaldehyde | C₄H₆O   | 70               | Aldehydes |
| 14  | pentane          | C₅H₁₂   | 72               | Alkenes |
| 15  | 2,4-hexadiene    | C₆H₁₀   | 82               | Alkenes |
| 16  | 2-methyl-1,3-pentadiene | C₇H₁₀ | 82 | Alkenes |
| 17  | 2-hexene         | C₆H₁₂   | 84               | Alkenes |

Fig. 10(c), the 1,3-end-hydrogen transfer and β-breaking also occurred in the generated secondary free radicals in reaction (a). Ethyl radicals were consequently generated. Part of the ethyl radicals lost the hydrogen radicals to form ethylene. Part of the ethyl radicals captured the hydrogen radicals to form ethane, which may be oxidized by the hydroxyl radicals to form acetaldehyde. As shown in Fig. 11(d), the 1,4-end-hydrogen transfer and β-breaking occurred in the generated secondary free radicals in reaction (a). Propyl and olefin radicals were generated. Part of the propyl radicals lost the hydrogen radicals to form propylene. Part of the propyl groups captured the hydrogen radicals to form propane which may be oxidized by the hydroxyl radical to form acetone. The breakage of the hydrogen radicals and β-breaking occurred to the olefin radicals, which generated 1–3-pentadiene. As shown in Fig. 11(e), the random chain scission, the 1,2-end-hydrogen transfer and the capture of the hydrogen radicals occurred in sequence in the generated secondary free radicals in reaction (a), which generated butane. As shown in Fig. 11(f), the 1,5-end-hydrogen transfer and β-breaking occurred in the generated secondary free radicals in reaction (a), which generated 2-amyloid and olefin radicals. Part of the 2-amyl radicals lost the hydrogen radicals to form 2-amylene and 1,2-dimethylcyclopropane. Part of the 2-amyl groups captured the hydrogen radicals to form pentane. The breakage of the hydrogen radicals and β-breaking occurred in the olefin radicals, which generated 2-ethyl-1,3-pentadiene. As shown in Fig. 11(g), the 1,5-end-hydrogen transfer and β-breaking occurred in the generated primary free radicals, which generated isobutyl radicals. The isobutyl radicals lost the hydrogen radicals to form isoprene which was oxidized by the hydroxyl radicals to form methacrylaldehyde. As shown in Fig. 11(h), the random chain scission, the end-hydrogen transfer and the capture of the hydrogen radicals occurred in the generated secondary free radicals in reaction (a), which generated 2-hexene and 2,4-hexadiene. As to water vapor, it may be generated by the water evaporation inside the specimen. As to the carbon monoxide, it may be generated by the decarboxylation of aldehydes or ketones. As to the carbon dioxide, it may be generated by the water evaporation inside the specimen. As to the carbon monoxide, it may be generated by the decarboxylation of aldehydes or ketones. As to the carbon dioxide, it may be generated by the decarboxylation of aldehydes or ketones.

4. Conclusions

In this work, the degradation behaviors of the surgical face mask waste in nitrogen were investigated using thermogravimetry at 15, 20, 25 and 30 K/min. Three types of model-free methods as well as one...
model-fitting method were utilized for the kinetic and thermodynamic analyses. In addition, the pyrolytic products as well as the potential chemical reactions were studied using real-time TG-FTIR-MS analyses in the work reported in this paper.

Fig. 11. Potential chemical reactions during the pyrolytic process.

Acknowledgements

The authors appreciate the support from National Natural Science Foundation of China (No. 51806106), China Postdoctoral Science Foundation (No. 2020M680069) and Jiangsu Planned Projects for Postdoctoral Research Funds (No. 2020Z141).

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