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Pyrolysis characteristics and kinetic analysis of waste cooking oil

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The pyrolysis characteristics of waste cooking oil, pretreated by removing impurities and water, were studied by a thermal analysis technology at heating rates of 5ºC, 10 ºC and 20 ºC /min. The pyrolysis mechanism function f(α) and kinetic parameters of waste cooking oil pyrolysis were determined by the Coats-Redfern method through TG curves. The results showed that the pyrolysis of waste cooking oil could be divided into three stages: volatilization of light components, pyrolysis of heavy components and crack and polymerization of the residue. A three-dimensional diffusion model of the Jander function 3D(n=2) was conformed to the pyrolysis process of waste cooking oil with the mechanism function f(α)=6(1-α)2/3[1-(1-α)1/3]1/2. The activation energy for the main pyrolysis stage of waste cooking oil pyrolysis increased with the increase in heating rate. The triglycerides in waste cooking oil firstly cracked into fatty acids, acroleins and fatty aldehydes. Then, at a temperature about 400-500 ºC, the products of the first step decomposed to small molecules.

Key words: Waste cooking oil; Kinetic analysis; Activation energy; Triglycerides; Coats-Redfern method

INTRODUCTION

In the past several hundred years, fossil fuel has been an important source of energy relied on for survival and development of human society. With the increase in global energy demand and the growing concern of the environment, it is extremely urgent to look for alternative low-carbon, clean and renewable energy resources. The utilization of wastes is an important way to reduce greenhouse gas emissions. According to the Chinese waste cooking oil analysis report, there were above 4.5 million tons waste cooking oil generated in China, but only 8 wt. % of them were used in industrial production. Therefore, a reasonable treatment of waste cooking oil is of great significance to realize the goal of low-carbon and clean utilization of energy resources.

Waste cooking oil is mainly derived from animal fat and vegetable oil which are commonly used in food and beverage industry. It contains toxins. Once it flows to rivers, it will cause pollution of water bodies, which will lead to environmental pollution; if you consume waste cooking oil, it will destroy the white blood cells and digestive tract mucosa, causing food poisoning, and even cancer. But because of its high viscosity and high acid value, it is difficult to be directly used [1]. Waste cooking oil is a kind of biomass oil; it cannot be ignored that waste cooking oil, as a new type of energy, gives similar products as do pyrolysis of fossil fuels. Biomass oil as a substitute for fossil fuels has a certain research foundation, so waste cooking oil has a great application prospect.

The methods of utilizing waste cooking oil are as follows: soap and detergent production by saponification reaction, feed grade mixed oil after processing, biodiesel production by trans-esterification with alcohols, as well as biofuels and biogas production by pyrolysis [1, 2]. A lot of studies have been focused on the pyrolysis of waste oil or co-pyrolysis of waste oil and other biomass to produce biodiesel and biogas. The factors of influence include temperature, retention time, heating rate etc. [3-7]. Techniques of thermal and kinetic analysis have been widely applied to reveal the pyrolysis mechanism of biomass [8-10]. Thermal cracking, despite its shortcomings (such as special distillation equipment, various reaction plans and products), is one of the best technologies compared to others. Therefore, this paper proposed waste oil research by pyrolysis. The common methods used for kinetic analysis of biomass pyrolysis include integral methods, differential methods, distributed activation energy method, etc. [11]. The Coats-Redfern method, that is a kind of integral method, has been extensively used to determine the kinetic parameters for biomass pyrolysis due to its simplicity and accuracy.

In this work, the characteristics of waste cooking oil pyrolysis were studied by thermogravimetry (TG) coupled with Fourier transform infrared spectroscopy (FTIR). The pyrolysis kinetic parameters of waste cooking oil were calculated by the Coats-Redfern method through TG curves at heating rates of 5°C, 10°C and 20°C/min,

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respectively. The gasification mechanism of waste oil was determined by analysis.

EXPERIMENTAL

Materials and methods

The waste cooking oil used in this study was obtained from the sewer of the city Kunming, China and pretreated for removing impurities and water. Elemental analysis and proximate analysis were carried out on a Euro-EA 3000. The fatty acid composition of waste cooking oil was analyzed by gas chromatography coupled with mass spectrometry (GC-MS).

The pyrolysis characteristics of waste cooking oil were studied using a TG apparatus (NETZSCH STA 449F3) at the heating rates of 5 °C, 10 °C and 20 °C/min, respectively. Approximately 10 mg of a sample were put into the crucible and heated from ambient temperature to 800 °C with different heating rates in a high-purity nitrogen (99.999%) flow of 50 mL/min. The sample was naturally cooled to ambient temperature after the experiment.

The Perkin Elmer Frontier Fourier transform infrared spectrometer was used to analyze the gas products. The import/export of FTIR was connected with the thermal analyzer. The temperature of the gas pool was 280 °C. The range of infrared spectrum wave numbers was 4000-450 cm^{-1}, and heating rate was 20 °C /min.

Kinetic analysis

The experimental data for kinetic analysis of waste cooking oil pyrolysis were obtained by TG at different heating rates. The degree of conversion for the waste cooking oil pyrolysis is defined as:

$$\alpha = \frac{1-f_t}{1-f_0} \qquad (1)$$

where f_t is the residual rate at time t, f_0 is the final residual rate.

The reaction rate can be described by a kinetic equation under non-isothermal conditions with a linear regime of temperature increase in time [12] as follows:

$$\frac{d\alpha}{dT} = \frac{A}{\beta}\exp\left(-\frac{E}{RT}\right) f(\alpha) \qquad (2)$$

Where α is the degree of conversion, β is the heating rate, E is the activation energy of the reaction, R is the universal gas constant with a value of 8.314J•mol^{-1}•K^{-1}, $f(\alpha)$ is differential mechanism function.

Integral for the equation (2) is expressed as:

$$G(\alpha) = \frac{A}{\beta}\int_{T_0}^{T}\exp\left(-\frac{E}{RT}\right)dT \qquad (3)$$

$G(\alpha)$ is defined as:

$$G(\alpha) = \int_0^{\alpha}\frac{1}{f(\alpha)}d\alpha \qquad (4)$$

Combining equations (6) and (7), equation (3) can be presented as equation (5).

$$\frac{A}{\beta}\int_{T_0}^{T}\exp\left(-\frac{E}{RT}\right)dT = \frac{AE}{\beta R}P(\text{u}) \qquad (5)$$

$$P(u) = \int_0^{u}\frac{-e^{-u}}{u^2}du = \frac{e^{-u}}{u^2}\left(1-\frac{2!}{u}+\frac{3!}{u^2}-\frac{4!}{u^3}+\cdots\right) \qquad (6)$$

$$u = -\frac{E}{RT} \qquad (7)$$

Then make the first approximation to equation (6) and simultaneous equations (3), (5) and (6). The approximate formula of Coats-Redfern can be obtained as:

$$G(\alpha) = \frac{A}{\beta}\frac{RT^2}{E}\left(1-\frac{2RT}{E}\right)\exp\left(-\frac{E}{RT}\right) \qquad (8)$$

From the logarithms of both sides of equation (8), equation (9) is obtained:

$$\ln\left[\frac{G(\alpha)}{T^2}\right] = \ln\left[\frac{AR}{\beta E}\left(1-\frac{2RT}{E}\right)\right]-\frac{E}{RT} \qquad (9)$$

For most reactions, the activation energy $E>>RT$. The equation (9) can be simplified as

$$\ln\left[\frac{G(\alpha)}{T^2}\right] = \ln\left(\frac{AR}{\beta E}\right)-\frac{E}{RT} \qquad (10)$$

So for a constant heating rate, the linear relationship between $\ln(G(\alpha)/T^2)$ and $1/T$ can be obtained. The E can be determined from the slope while A can be calculated from the intercept.

RESULTS AND DISCUSSION

Analysis of the characteristics of waste oil

The results of the ultimate analysis of waste cooking oil are listed in Table 1, showing that the main elements of waste cooking oil are carbon (C), hydrogen (H) and oxygen (O). The fatty acid composition of waste cooking oil was analyzed by GC-MS (see Table 2), showing that the carbon chain length of the fatty acids of waste cooking oil in the range of C16~C18, is consistent with the literature data [13].

Table 1. Ultimate analysis of waste cooking oil

Sample	C	H	O^a	N	S	M
Waste cooking oil (wt. %)	77.81	12.33	8.93	0.86	0.07	0.54

M: Moisture content; ^{a}: The oxygen (O) content is determined by difference.

Table 2. GC-MS composition of waste cooking oil.

Composition	C16:0	C18:0	C18:1	C18:2	Others[a]
Content (wt. %)	16.89	7.55	37.45	25.53	12.58

[a]: Calculated by difference.

TG analysis of waste cooking oil pyrolysis

The TG curves of waste cooking oil pyrolysis at different heating rates are shown in Fig. 1. There were three weight loss stages for the process of waste cooking oil pyrolysis. The first stage was the volatilization of components with low boiling points, such as moisture, aldehydes, ketones, glycerin and lower fatty acid produced by the decay of oil and fat within the temperature range from ambient temperature to about 200°C [14]. The weight loss rate of the first stage was about 1.6 wt. %. The pyrolysis of heavy components (second stage) took place from about 200°C to 507°C with a weight loss rate above 95 wt. %. The third stage was the crack and polymerization of the residue over 507°C with a weight loss rate of <1wt. %. As shown in Fig. 2, the main weight loss stage for waste cooking oil pyrolysis moved to high temperature zone on rising the temperature. The reason was that the sample was heated more uniformly at the low heating rate, which was conducive to the diffusion of pyrolysis gas, so that the weight loss was more likely to be detected by the balance. The residual rate decreased on rising the temperature, because the heating time of the sample was longer at low heating rate, which promoted the polymerization of macromolecules.

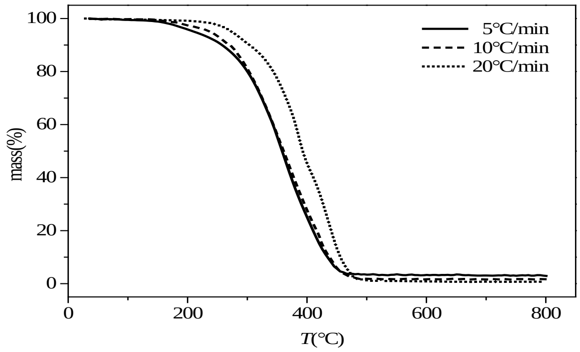


Fig. 1. TG curves of waste cooking oil pyrolysis at different heating rates.

Kinetic calculation of waste cooking oil pyrolysis

The degrees of conversion as a function of temperature for waste cooking oil pyrolysis at different heating rates are shown in Fig. 2. On rising the temperature, the degree of conversion increased and reached a plateau. Similarly to the TG curves, the α-T curves moved to the high temperature zone with the increase in heating rate.

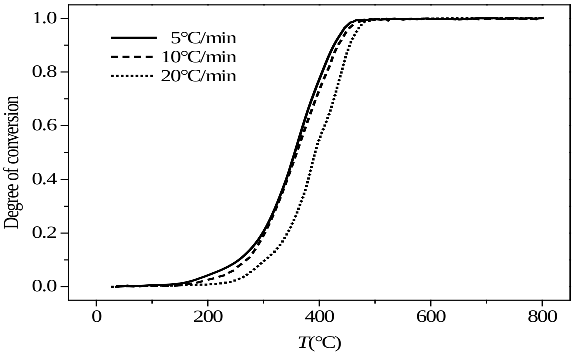


Fig. 2. Degree of conversion as a function of temperature for waste cooking oil pyrolysis.

According to the TG and α-T curves, the second stage of waste cooking oil pyrolysis for different heating rates was chosen for calculation of kinetics because of the significant weight loss rate. Based on the maximum correlation coefficient of $\ln[G(\alpha)/T^2]$ and $1/T$, Jander function 3D (n=2) was determined to be the mechanism function of the waste cooking oil pyrolysis from the eighteen mechanism functions listed in Table 3.

The differential and integral forms of Jander function 3D (n=2) are shown as equations (11) and (12), respectively.

$$f(\alpha) = 6(1-\alpha)^{2/3}\left[1-(1-\alpha)^{1/3}\right]^{1/2} \quad (11)$$

$$G(\alpha) = \left[1-(1-\alpha)^{1/3}\right]^{2} \quad (12)$$

Using equations (10) and (12), the Coats-Redfern method is expressed as:

$$\ln\left[\frac{\left[1-(1-\alpha)^{1/3}\right]^{2}}{T^2}\right] = \ln\left(\frac{AR}{\beta E}\right) - \frac{E}{RT} \quad (13)$$

The relationship curves of $\ln\{[1-(1-\alpha)^{1/3}]^2\}/T^2$, and $1/T$ at different heating rates are shown in Fig. 3. The activation energy E and the frequency factor A can be calculated through slope and intercept, respectively, were obtained after fitting linear lines by the least square method. The kinetic parameters of waste cooking oil pyrolysis at different heating rates are shown in Table 4.

Table 3. Expressions of some mechanism functions.

No.	Function	*f*(*α*)	*G*(*α*)	R^2
1	Mample rule, n=1	1-*α*	-ln(1-*α*)	0.9869
2	Jander 2D, n=0.5	$4(1-\alpha)^{1/2}\left[1-(1-\alpha)^{1/2}\right]^{1/2}$	$\left[1-(1-\alpha)^{1/2}\right]^{1/2}$	0.9871
3	Jander 3D, n=0.5	$6(1-\alpha)^{2/3}\left[1-(1-\alpha)^{1/3}\right]^{1/2}$	$\left[1-(1-\alpha)^{1/3}\right]^{1/2}$	0.9918
4	Jander 2D, n=2	$(1-\alpha)^{1/2}\left[1-(1-\alpha)^{1/2}\right]^{-1}$	$\left[1-(1-\alpha)^{1/2}\right]^{2}$	0.9946
5	Jander 3D, n=2	$3/2(1-\alpha)^{2/3}\left[1-(1-\alpha)^{1/3}\right]^{-1}$	$\left[1-(1-\alpha)^{1/3}\right]^{2}$	0.9953
6	G-B	$3/2\left[(1-\alpha)^{-1/3}-1\right]^{-1}$	$1-2/3\alpha-(1-\alpha)^{2/3}$	0.9935
7	Anti Jander 3D	$3/2(1+\alpha)^{2/3}\left[(1+\alpha)^{1/3}-1\right]^{-1}$	$\left[(1+\alpha)^{1/3}-1\right]^{2}$	0.9774
8	Z-L-T 3D	$3/2(1-\alpha)^{4/3}\bullet\left[(1-\alpha)^{-1/3}-1\right]^{-1}$	$\left[(1-\alpha)^{-1/3}-1\right]^{2}$	0.9660
9	Avrami-Erofeev, n=1/4	$4(1-\alpha)\left[-\ln(1-\alpha)\right]^{3/4}$	$[-\ln(1-\alpha)]^{1/4}$	0.9470
10	Avrami-Erofeev, n=1/3	$3(1-\alpha)\left[-\ln(1-\alpha)\right]^{2/3}$	$[-\ln(1-\alpha)]^{1/3}$	0.9750
11	Avrami-Erofeev, n=2/5	$5/2(1-\alpha)\left[-\ln(1-\alpha)\right]^{3/5}$	$[-\ln(1-\alpha)]^{2/5}$	0.9800
12	Avrami-Erofeev, n=1/2	$2(1-\alpha)\left[-\ln(1-\alpha)\right]^{1/2}$	$[-\ln(1-\alpha)]^{1/2}$	0.9832
13	Avrami-Erofeev, n=2/3	$3/2(1-\alpha)\left[-\ln(1-\alpha)\right]^{1/3}$	$[-\ln(1-\alpha)]^{2/3}$	0.9854
14	Avrami-Erofeev, n=3/4	$4/3(1-\alpha)\left[-\ln(1-\alpha)\right]^{1/4}$	$[-\ln(1-\alpha)]^{3/4}$	0.9859
15	Avrami-Erofeev, n=3/2	$2/3(1-\alpha)\left[-\ln(1-\alpha)\right]^{-1/2}$	$[-\ln(1-\alpha)]^{3/2}$	0.9877
16	Avrami-Erofeev, n=2	$1/2(1-\alpha)\left[-\ln(1-\alpha)\right]^{-1}$	$[-\ln(1-\alpha)]^{2}$	0.9880
17	Avrami-Erofeev, n=3	$1/3(1-\alpha)\left[-\ln(1-\alpha)\right]^{-2}$	$[-\ln(1-\alpha)]^{3}$	0.9884
18	Avrami-Erofeev, n=4	$1/4(1-\alpha)\left[-\ln(1-\alpha)\right]^{-3}$	$[-\ln(1-\alpha)]^{4}$	0.9885

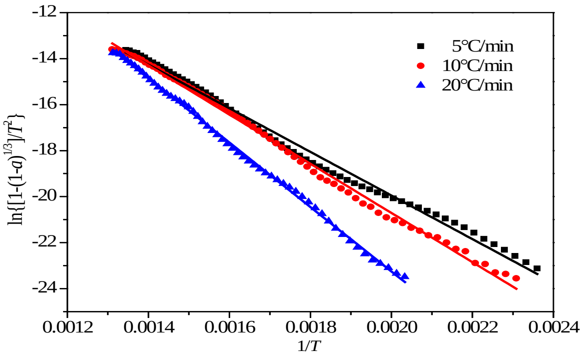


Fig. 3. Relationship curves of ln{[1-(1-α)1/3]2}/T2 and 1/T at different heating rates.

As can be seen, the linear correlation coefficients are over 0.99, which indicated that the model of Jander function 3D (*n*=2) conformed to the process of waste cooking oil pyrolysis. The activation energy *E* and the frequency factor *A* increased with the increase in heating rate, the side chains rupture of aromatic compounds, the C-C bonds cleavage of oxy-compounds in the intermediate products and the decomposition of long-chain hydrocarbons [17, 18].

The bands at 2400-2200 cm^{-1} and 726-586 cm^{-1} indicate the existence of CO_2 resulting from the decarboxylation of fatty acids. The bands in the range of 1880-1650 cm^{-1}, due to C=O stretching vibrations, indicated that the generated lipids, aldehydes and ketones contained C=O bonds [16,19]. The characteristic peaks in the range of 1650-1320 cm^{-1} may be caused by the stretching vibrations of aromatic ring C-C and in-plane bending of carboxylic acid COOH, indicated the production of carboxylic acids and hydrocarbons.

Table 4. Kinetic parameters of waste cooking oil pyrolysis.

Heating rate *β* (℃•min^{-1})	Temperature range (℃)	*n*	*E* (kJ•mol^{-1})	*A* (min^{-1})	R^2
5	160-470	2	78.81	1.75×10^4	0.9919
10	180-490	2	88.89	2.09×10^5	0.9963
20	215-507	2	114.61	2.18×10^8	0.9977

The bands at 1300-990 cm^{-1} may be caused by stretching vibrations of C-O-C, linear stretching vibrations of C-C or in-plane bending vibrations of C-H in the aromatic ring, which indicates that ethers, hydrocarbons or phenolic exhalations may be generated in the process of waste cooking oil pyrolysis [20]. From the literature [21], the odd 1-alkenes and n-alkanes are generated by decarboxylation of free radical RCOO cracked from the triglycerides, followed by disproportionation and ethylene elimination. Another free radical RCH2O transformed to the even alkanes and alkenes by loss a ketene and following disproportion and ethylene elimination. The important production of H_2 mainly results from the crack of hydrocarbons, dehydrogenation of olefins, and the proton extraction from cycloolefins and aromatic hydrocarbons [22] which cannot be detected by FTIR.

The FTIR spectra of waste cooking oil pyrolysis at different temperatures are shown in Fig. 5. The evolution of gas is very weak before 200℃, showing that the primary compounds of waste cooking oil have not pyrolysed at this phase. The characteristic peaks of pyrolysis products become noticeable, indicating waste cooking oil pyrolysis. The primary pyrolysis products contain lipids, hydrocarbons, aldehydes, ketones, acids, etc.

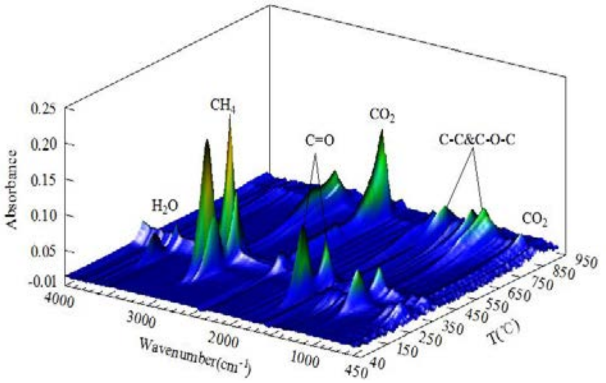
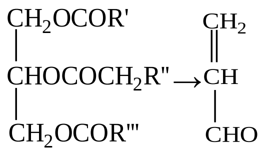


Fig. 4. FTIR spectra of waste cooking oil pyrolysis(20℃).

The pyrolysis products after 450℃ are mainly small molecules such as CO_2, CH_4, ketones, aldehydes, ethers, hydrocarbons, etc. The waste cooking oil is mainly derived from vegetable oil and animal fat, which means that the main components of waste cooking oil are all kinds of fatty acid glycerides. However, a part of the fatty acid glycerides would generate acids, aldehydes, ketones, glycerin, hydrocarbons, and bifunctional oxides *via* oxidation, hydrolysis and pyrolysis in the process of frying and long-term exposure to air and sunshine [23,24]. Although the composition of waste cooking oil is complicated, the studies of fatty acid glycerides pyrolysis made great contribution to reveal the mechanism of waste cooking oil pyrolysis. Chang and Wan provided a complete scheme of triglyceride pyrolysis, which was summarized as two steps [25,26]. Firstly, the triglycerides cracked into fatty acids, acrolein and fatty aldehydes at temperatures lower than 300℃ expressed as equation (14). Secondly, the products of the first step cracked into short-chain hydrocarbons at a higher temperature about 400-500℃. The mechanism can be expressed as reactions (14) - (19). The TG-FTIR analysis shows that this theory conforms to the pyrolysis process of waste cooking oil.



$$\begin{array}{l} CH_2OCOR' \\ | \\ CHOCOCH_2R'' \\ | \\ CH_2OCOR''' \end{array} \longrightarrow \begin{array}{l} CH_2 \\ \| \\ CH \\ | \\ CHO \end{array}$$

$$+R'COOH+R'''COOH+R''CH=CO \qquad (14)$$

$$RCOOH \rightarrow CO_2 + RH \qquad (15)$$

$$2RCOOH \rightarrow CO_2 + H_2O + RCOR \qquad (16)$$

$$2RCH=CO \rightarrow 2CO + RHC=CHR \qquad (17)$$

$$CH_2=CHCHO \rightarrow CO + C_2H_4 \qquad (18)$$

$$2RCOCH_2R \rightarrow 2R_2 + CO=C_2H_4 \qquad (19)$$

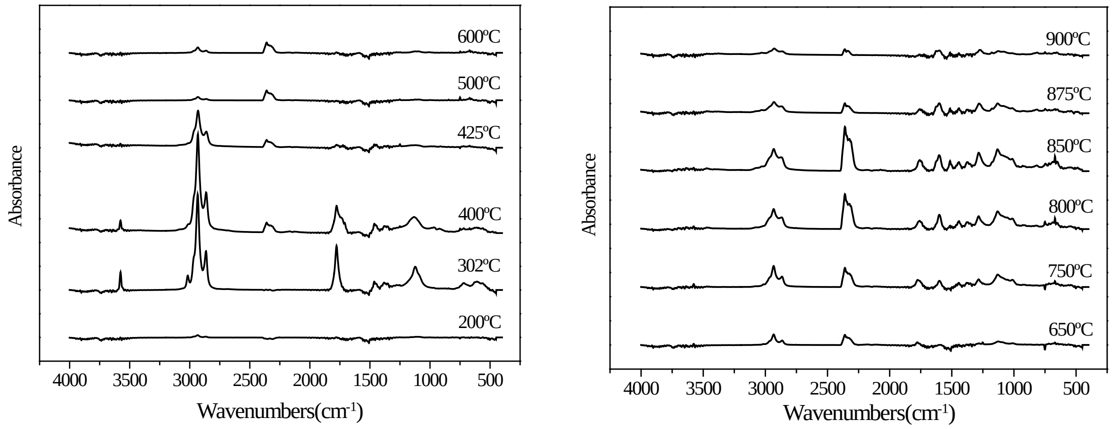


Fig. 5. FTIR spectra of waste cooking oil pyrolysis at different temperatures.

CONCLUSIONS

The pyrolysis of waste cooking oil was investigated. The process of waste cooking oil pyrolysis could be divided into three stages: (i) volatilization of light components from ambient temperature to about 200℃, (ii) pyrolysis of heavy components from about 200℃ to 507℃ and (iii) crack and condensation of the residue above 507℃. The weight loss rates for these three stages were about 2 wt. %, 95 wt. % and 1wt. %, respectively.

The mechanism function $f(\alpha)$ and the kinetic parameters for the main stage of waste cooking oil pyrolysis were determined by the Coats-Redfern method. The Jander function 3D(n=2) was chosen to determine the mechanism of waste cooking oil pyrolysis with the mechanism function $f(\alpha)=6(1-\alpha)^{2/3}[1-(1-\alpha)^{1/3}]^{1/2}$. The activation energies were 78.81kJ/mol at 160-470℃at 5℃/min, 88.89kJ/mol at 180-490℃ at 10℃/min and 114.61kJ/mol at 215-507℃ and 20℃/min, respectively, which showed that activation energy increased with the increase in heating rate.

There were two steps of the pyrolysis of triglycerides in waste cooking oil. Firstly, the triglycerides cracked into fatty acids, acrolein and fatty aldehyde at temperatures lower than 300℃. Secondly, the products of the first step decomposed into short-chain hydrocarbons, aldehydes, ketones, H_2O, CO, CO_2, H_2, etc., at temperatures about 400-500℃.

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