

Article

Kinetic Study of the Pyrolysis of Waste Printed Circuit Boards Subject to Conventional and Microwave Heating

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Received: 2 July 2012; in revised form: 8 August 2012 / Accepted: 24 August 2012 /

Published: 31 August 2012

Abstract: This paper describes a kinetic study of the decomposition of waste printed circuit boards (WPCB) under conventional and microwave-induced pyrolysis conditions. We discuss the heating rates and the influence of the pyrolysis on the thermal decomposition kinetics of WPCB. We find that the thermal degradation of WPCB in a controlled conventional thermogravimetric analyzer (TGA) occurred in the temperature range of 300 °C–600 °C, where the main pyrolysis of organic matter takes place along with an expulsion of volumetric volatiles. The corresponding activation energy is decreased from 267 kJ/mol to 168 kJ/mol with increased heating rates from 20 °C/min to 50 °C/min. Similarly, the process of microwave-induced pyrolysis of WPCB material manifests in only one stage, judging by experiments with a microwave power of 700 W. Here, the activation energy is determined to be only 49 kJ/mol, much lower than that found in a conventional TGA subject to a similar heating rate. The low activation energy found in microwave-induced pyrolysis suggests that the adoption of microwave technology for the disposal of WPCB material and even for waste electronic and electrical equipment (WEEE) could be an attractive option.

Keywords: kinetic study; microwave-induced pyrolysis; waste printed circuit boards (WPCB); activation energy

Nomenclature:

A	Pre-exponential factor, s^{-1}
E	Activation energy, kJ/mol
f	Function of conversion
k	Thermal decomposition rate constant, s^{-1}
n	Reaction order
R	Gas constant = 8.314 kJ/(kmol K)
t	Pyrolysis time, s
T	Absolute temperature, K
T_0	The temperature at which the pyrolysis of WPCB begin, °C
T_{1m}	The temperature at which the DTG curve of WPCB reaches the first peak, °C
T_{2m}	The temperature at which the DTG curve of WPCB reaches the second peak, °C
W	Weight of sample at time t, mg and/or g
W_0	Initial weight of sample, mg and/or g
W_∞	Final weight of sample, mg and/or g
x	Conversion of WPCB, defined as: $x = w_0 - w / (w_0 - w_\infty)$
Greek letters	Descriptions
β	Heating rate, °C min^{-1}

1. Introduction

Due to the rapid development of technology, the lifespan of electrical and electronic equipment (EEE) is very short, resulting in increasing quantities of waste electronic and electrical equipment (WEEE). It is estimated that some 40–60 million tons of WEEE are generated worldwide every year, which poses grave risks to human health and the environment [1,2]. However, in spite of its hazardous properties, WEEE is also considered a precious resource, so the development of effective WEEE recycling techniques is of increasing interest. As one of the most important branches of the WEEE stream, waste printed circuit boards (WPCB) are generally considered to be representative of WEEE, and have received increasing attention from the public and researchers because they contain a wealth of nonferrous metals in addition to a variety of toxic materials [3,4]. Thus, the recycling of WPCB is important not only as regards environmental protection and the recovery of valuable materials, but also for providing guidance to WEEE recycling.

Pyrolysis can be described as the thermal decomposition of organic components in an oxygen-free atmosphere to yield char, oil, and gas, and it holds promise as an approach to optimal waste upgrading, especially for organic polymers. Moreover, operational conditions in pyrolytic processes can be optimized to improve the yield or the quality of desirable products by means of innovative heating methods or catalysts. Microwave pyrolysis (MWP) is one focus of current research and has drawn the widespread interest of foreign and domestic researchers [5–8]. The applications of MWP include the pyrolysis of plastic waste [5,6], the pyrolysis of biomass waste [7–13], sewage sludge [14,15], and waste automotive engine oil [16–18], among others.

Although several groups have studied the pyrolysis of WPCB, their focus has mainly been on the pyrolytic kinetics [19–22] and the formation and fate of brominated compound [22–24] in conventional heating schemes (such as electric heating), also known as conventional pyrolysis. Due to the presence of metals, which are considered incompatible with microwave heating, the microwave-induced pyrolysis of WPCB has barely been studied. Under microwave irradiation, a discharge will be triggered when there are metal tips or sharp corners in the material being treated. This is a result of field enhancement and field emission of electrons when the induced electric potential exceeds the coulomb potential. As a result of these discharges, metal tips can be melted, implying that a discharge can produce local temperatures of 1000 °C–2000 °C [25–27]. Our research group has studied the heating effects associated with microwave-metal discharges and found that the energy conversion ratio from electrical energy to heat can reach as high as 30% [28]. Based on the unique material characteristics of WPCBs and the discharge properties of metals, we have previously established that microwave-induced pyrolysis holds promise as a way to process WPCBs [26–30].

Kinetics studies of microwave-induced pyrolysis of WPCBs have rarely been performed. However, an understanding of the pyrolytic kinetics is important for reactor design, process optimization, and the general improvement of microwave heating applications. This calls for the development of thermal decomposition kinetic models that can accurately predict the pyrolysis processes that occur in WPCBs under microwave irradiation. The prevailing thermal gravimetric analysis (TGA) technique is able to predict such thermal decomposition processes quite accurately, and is considered to be representative of conventional pyrolysis modeling. In this work, the pyrolytic kinetics of WPCBs subject to both conventional heating and microwave heating are studied. We present and compare kinetic parameters of interest that include the activation energy, a pre-exponential factor, and the reaction order in the chemical reaction-controlled region. These data may be of use in recycling process design and in energy recovery schemes for WPCB reuse in pyrolytic processes.

2. Materials and Methods

2.1. Materials and Sample Preparation

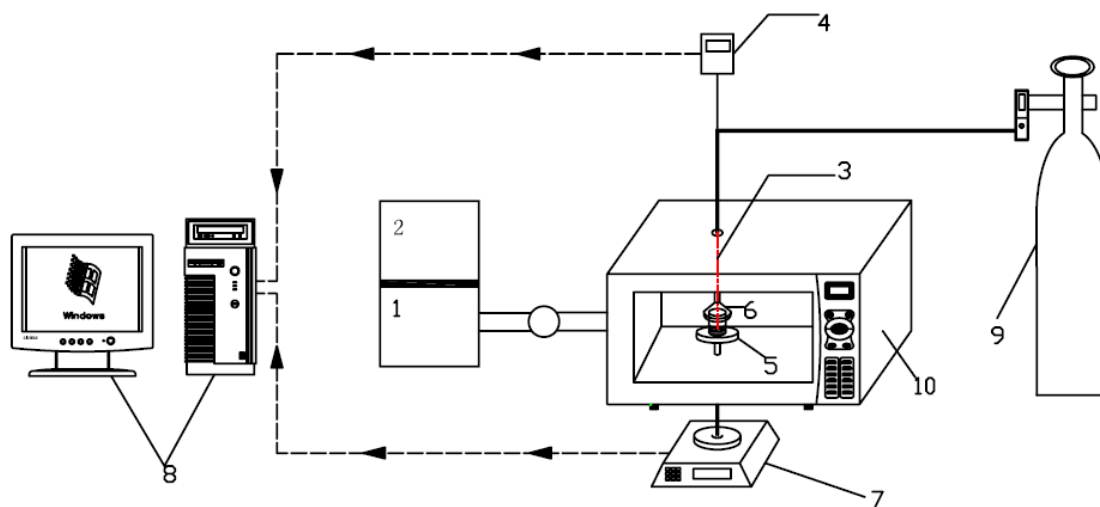
Waste samples of printed circuit boards, obtained from a recycling plant in Jinan, China, served as the raw material for our studies. The WPCBs were first shredded into fragments of about 1 cm × 1 cm (referred to as “big particles”), and then a representative fragment was sampled and crushed into a fine particle state (approximately 40–60 mesh) in the laboratory. The proximate analysis of WPCBs was carried out by referring to the coal proximate analysis method. The weight percentages of moisture, ash, volatiles and fixed carbon were 0.48%, 73.62%, 25.12 % and 0.78%, respectively.

2.2. Apparatus and Procedures

The pyrolysis experiments were carried out in a thermogravimetric analyzer (Mettler-Toledo, TGA/DSC/1600HT) for the conventional heating data, and in a modified household microwave oven (Galanz P7021TP-6) for the microwave-induced pyrolysis experiments. For the conventional TGA of WPCB, about 15.0 mg of fine particle material was placed in a crucible pan and heated in an inert atmosphere of nitrogen (N₂ flow rate of 20 mL/min) over a temperature range of 50–900 °C at heating

rates of 10, 20, 30 and 50 °C/min. The temperature and weight of the material was monitored with a computer. For the microwave-induced pyrolysis of WPCB, 40 g of “big particle” material was placed in a quartz tube (60 mm length, 55 mm i.d.) which was itself surrounded by a quartz container and purged with nitrogen (99.999% pure) at a flow rate of 100 mL/min. During microwave irradiation, the temperature was measured by two ways: one is an online measurement by placing the thermocouple 5 mm directly above the sample to monitoring the temperature of volatility; the other is an offline measurement by setting different irradiation time (e.g., 40 s, 60 s, 90 s, 120 s, 150 s, 3 min, 5 min,...) and inserting the thermocouple into the center of the sample immediately after turning off microwaves. The thermocouple we used was connected with a filter capacity to reduce the disturbance from microwaves and improve the reliability of the temperature measurement. The offline temperature measurement was used to correct the online measurement results by averaging at certain time points. Concurrently, the sample mass when the thermocouple centered 5 mm directly above was recorded via an electronic balance system underneath. Figure 1 shows a diagram of the experimental setup.

Figure 1. Experimental set-up for microwave pyrolysis of WPCB: (1) microwave generation system; (2) microwave control system; (3) thermocouple; (4) temperature monitoring system; (5) PTFE tray system; (6) quartz container; (7) weighing system; (8) signal acquisition and processing system; (9) nitrogen and (10) microwave oven.



2.3. Pyrolysis Kinetics

Pyrolysis of WPCB is generally a complex process. It is difficult to discover a full kinetic analysis of complex systems, but some kind of “effective” or “average” kinetic description is still needed. In this work, the overall rate of the pyrolytic reactions of WPCB is described by the following equation:

$$\frac{dx}{dt} = kf(x) \quad (1)$$

where $f(x)$ is a function depending on the decomposition mechanism, which can be in the form of an n th order of reaction: $f(x) = (1 - x)^n$.

Constant k obeys the Arrhenius correlation:

$$k = A \exp\left(-\frac{E}{RT}\right) \quad (2)$$

Rearranging and integrating, we have:

$$\int_0^x \frac{dx}{(1-x)^n} = \frac{A}{\beta} \int_{T_0}^T \exp(-E/RT) dT \quad (3)$$

Based on Equation (3), Coats-Redfern's method was derived for numerical determination of the kinetic parameters, as Equations (4) and (5):

when $n \neq 1$:

$$\ln \left\{ \frac{1 - (1-x)^{1-n}}{T^2(1-n)} \right\} = \ln \left[\frac{AR}{\beta E} \left(1 - \frac{2RT}{E}\right) \right] - \frac{E}{RT} \quad (4)$$

when $n = 1$:

$$\ln \left\{ \frac{-\ln(1-x)}{T^2} \right\} = \ln \left[\frac{AR}{\beta E} \left(1 - \frac{2RT}{E}\right) \right] - \frac{E}{RT} \quad (5)$$

When $2RT/E$ is small enough to be ignored, Equations (4) and (5) can be simplified as Equations (6) and (7):

when $n \neq 1$:

$$\ln \left\{ \frac{1 - (1-x)^{1-n}}{T^2(1-n)} \right\} = \ln \left(\frac{AR}{\beta E} \right) - \frac{E}{RT} \quad (6)$$

when $n = 1$:

$$\ln \left\{ \frac{-\ln(1-x)}{T^2} \right\} = \ln \left(\frac{AR}{\beta E} \right) - \frac{E}{RT} \quad (7)$$

With regards the scenario that RT/E is not negligible, the Equations (4) and (5) can be transformed into Equations (8) and (9):

when $n \neq 1$:

$$\ln \left\{ \frac{1 - (1-x)^{1-n}}{T^2(1-n) \left(1 - \frac{2RT}{E}\right)} \right\} = \ln \frac{AR}{\beta E} - \frac{E}{RT} \quad (8)$$

when $n = 1$:

$$\ln \left\{ \frac{-\ln(1-x)}{T^2 \left(1 - \frac{2RT}{E}\right)} \right\} = \ln \frac{AR}{\beta E} - \frac{E}{RT} \quad (9)$$

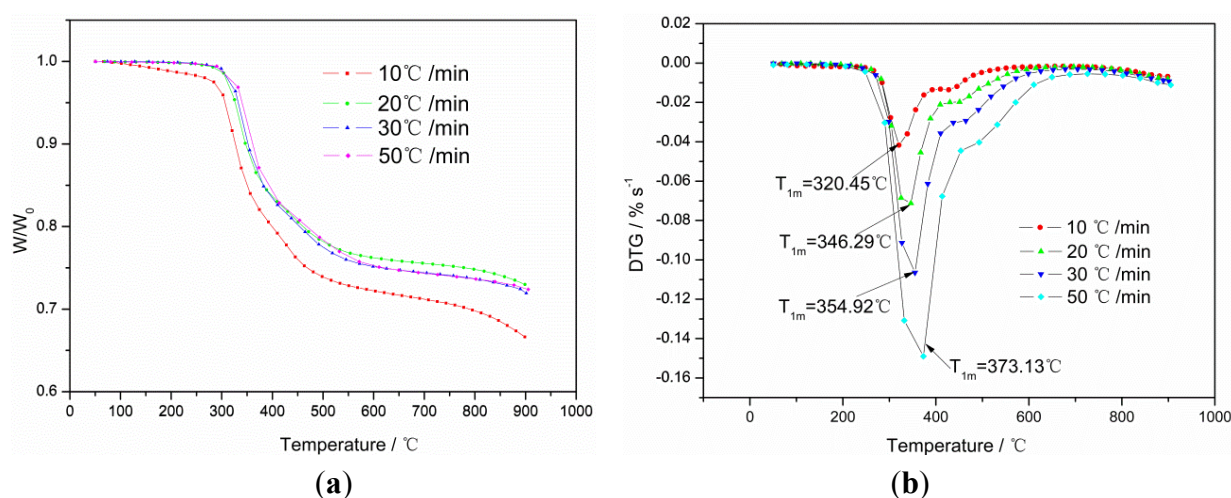
Iterative method and the least squares method should be combined to determine the value of n , A and E .

3. Results and Discussion

3.1. Conventional Pyrolysis of WPCBs Using TGA

The TG and DTG curves from the conventional heating experiments are shown in Figure 2. As evidenced by the DTG curve, there may be two overlapped degradation stages due to the presence of a turning point around 430 °C. However, there were no obvious divide between these two stages, thus it can be considered that one degradation stage was found during the pyrolysis process, characterized by an obvious weight loss peak. The main decomposition occurred at 300 °C–600 °C with an accompanying conversion of about 90%. The main thermal degradation temperature in this paper were a little high when compared to the work of Chen *et al.* [19] and Chiang *et al.* [31] which could be caused by high heating rates and different compositions. Thus, the general weight loss in the WPCBs can be characterized by the following stages: the weight of the sample barely changed when its temperature remained below 300 °C; thereafter the sample underwent a rapid weight loss in the temperature range of 300 °C–600 °C. This weight loss process comprised the pyrolysis phase, where about 25% of the original weight was lost. Subsequently, the weight of the sample was largely maintained with a very slow weight loss process.

Figure 2. Thermogravimetry curves (a) and derivative thermogravimetry curves (b) at different heating rates.



With increased heating rates, the characteristic temperatures of T_0 , T_{1m} , T_{2m} (description in nomenclature, T_{2m} was not marked in Figure 2 due to the vague division between the second peak and the first one) were increased, the DTG curve was shifted towards higher temperature regions, and the value of dx/dt and the maximum decomposition rates were also improved. We can conclude that the pyrolysis reaction was postponed to higher temperature range with increasing heating rates, because the time interval in which the sample is exposed to a given temperature decreases as the heating rate increases. This result is in accordance with the work of other researchers. The final residual weight rate was strongly influenced by variations in the heating rate. The residual weight rate decreased as a result of mass-loss during thermal decomposition, indicating volatile matter was ejected. With low heating rates, the time interval in which the sample is exposed to a given temperature is long, so that the

sample has ample time to decompose, improving the reaction depth. As many metal components including the metals with low melting or boiling points, such as Hg, Sn, Pb, Zn, *etc.*, were contained in WPCBs, long residence time may intensify the volatility or emission of heavy metal components. Moreover, compared to the proximate analysis results of WPCBs, WPCBs can obtain a complete pyrolysis with the heating rates from 20 °C/min to 50 °C/min. Thus, the following kinetic study of the pyrolysis process is based mainly on the heating rates of 20, 30 and 50 °C/min.

As most materials decompose (about 90% conversion occurred) in the temperature range of 300 °C–600 °C, which comprises the main reaction region, the kinetic study of the thermal decomposition in this temperature range is the main focus of our work. The Coats-Redfern method was used to determine the kinetic parameters for heating rates of 20, 30, and 50 °C/min and their values are shown in Table 1.

Table 1. Kinetic parameters for conventional pyrolysis of WPCB at three heating rates.

β (°C/min)	n	E	lnA	R ²
20	8.97	266.52	53.51	0.996
30	7.36	219.62	43.69	0.997
50	5.46	168.78	33.22	0.997

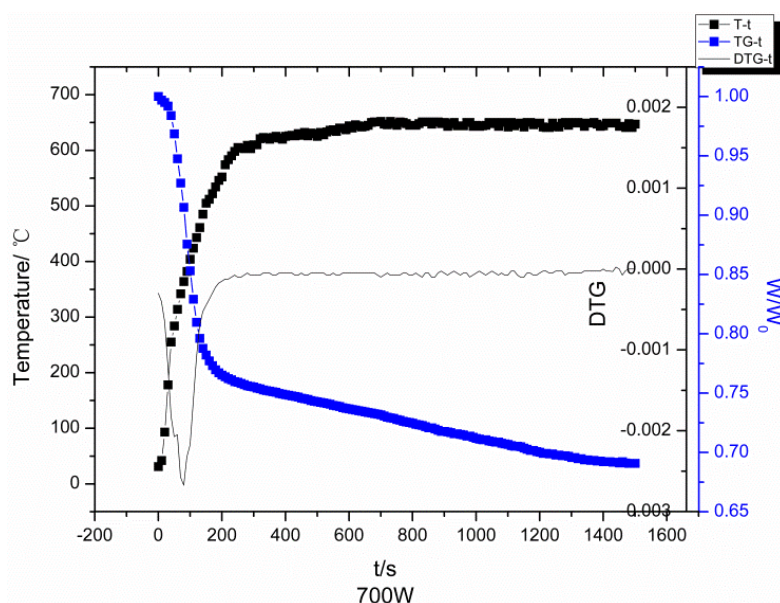
The activation energy was found to be 168–267 kJ/mol, decreasing with an increase in heating rate. Our results indicate a significant difference in activation energy for different heating rates. The variation of the activation energy with heating rate gives rise to a variation in the pre-exponential factor. This can be explained with a kinetic compensation effect that can be expressed as a linear relationship between the logarithm of the pre-exponential factor and the activation energy ($\ln(A) = kE + b$). This effect is mainly due to mathematical, physio-chemical, and experimental causes, which is widely researched [32,33]. Although this coupling of the kinetic parameters can result in similar values of the kinetic constant, the lower activation energy indicates the energy required to activate molecules to start the chemical reaction is lower because chemical reaction occurs on the condition that the molecular energy is equal to or higher than the activation energy. And more molecules will be activated at a given temperature, if the activation energy is lower. Although it is hard to observe the differences of the start of decomposition at different heating rates in Figure 2, which are mainly due to reaction shifts to a high temperature range with the increased heating rate, the reaction rate at a given temperature was obviously improved with the increase in heating rate, as illustrated in Figure 2b. The accelerated reaction rate can be attributable to the strengthened activation of molecules due to lower activation energy. The reaction order was also decreased with an increase in heating rate which demonstrated the thermal decomposition process was very complex when the heating rate was low. And a high heating is beneficial to eliminate unnecessary reactions to make the decomposition process simple.

3.2. Microwave-induced Pyrolysis of WPCB

The heating rate of WPCB material subject to microwave irradiation is determined by many factors, such as the sample's wave-absorption capacity and the heating effect from microwave-metal discharges. Thus, in contrast to the heating rates in conventional heating schemes, it is much easier to control the microwave power in experiments involving microwave-induced pyrolysis of WPCBs.

Figure 3 presents the temperature, residual weight rate and DTG of WPCBs as a function of time when exposed to an incident microwave power of 700 W at 2.45 GHz. The temperature of the sample clearly skyrockets at the beginning of these experiments, and then is followed by an approximately linear increase with a measured heating rate of about 140 °C/min in the range of 250 °C–500 °C, followed by an increase in temperature to above 600 °C at a lower rate of about 60 °C/min, and finally it stabilizes at a temperature of about 650 °C. Correspondingly, the decomposition of WPCBs can be divided into two stages: the sample was rapidly pyrolyzed with a rapid rate of weight loss at temperatures below 500 °C, after which the samples experienced a slow weight loss process.

Figure 3. T-t, TG-t, and DTG-t curves of WPCBs in the microwave pyrolysis process.

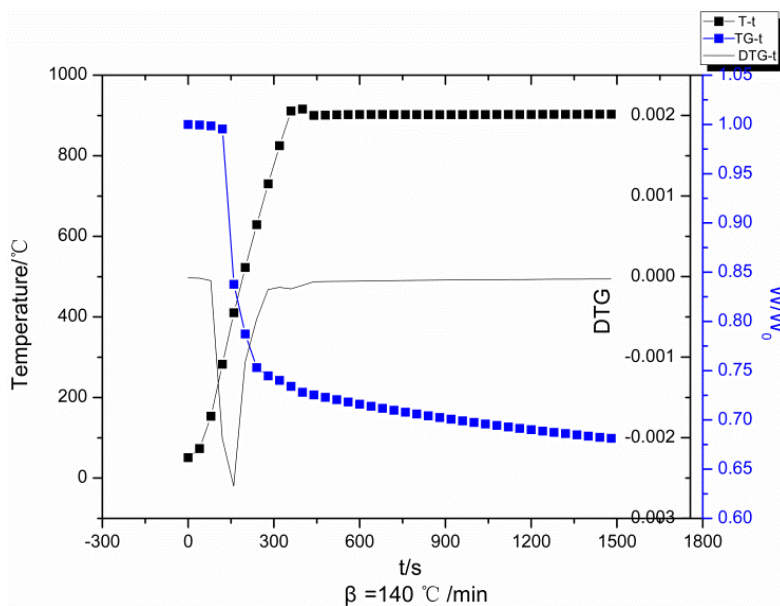


For a better comparison, a simulation of microwave-induced pyrolysis of WPCB material was carried out by modeling a conventional TGA with a heating rate of 140 °C/min from 50 °C to 900 °C, and subsequent temperature maintenance at 900 °C for 20 min. In fact, it is hard to simulate the heating process of WPCB under microwave irradiation completely. Thus, a simulation of the main pyrolysis phase was simulated. Figure 4 presents the temperature, residual weight rate and DTG of WPCB as a function of time.

Compared with Figure 3, which shows real data from microwave experiments, the overall profiles are similar, except for the lack of an induction phase in the microwave-induced pyrolysis data, since metal-microwave sparks occurred immediately at the onset of microwave irradiation, leading to high local temperatures and a partial decomposition of WPCBs which results in significant weight loss, even though the average overall temperature may be low. As there is only one obvious peak in the data for the pyrolysis stage where the main reaction happened in both pyrolysis processes, our thermal kinetic study will be focused on this range.

It should be noted that the value of the pre-exponential factor A and the activation energy E is a number that is conditioned upon a constant heating rate during the entire pyrolysis process. For the microwave-induced pyrolysis experiments, the temperature curves have an approximately linear phase in the temperature range of 250 °C–500 °C.

Figure 4. T-t, TG-t, and DTG-t curves of WPCBs in simulation of the conventional heating process with a heating rate of 140 °C/min.



Thus, the heating rate during this phase can be considered constant. The following kinetic parameters were calculated based on the data related to this phase. For the simulated TGA, the main pyrolysis phase starts at 280 °C, and this linear phase lies in the temperature range of 280 °C–530 °C. The values of the calculated kinetics parameters are shown in Table 2.

Table 2. Kinetic parameters for pyrolysis of WPCB under both conventional TGA and microwave heating scheme.

Pyrolysis method	n	E	lnA	R ²
Microwave power	2.41	48.68	9.50	0.992
Simulated TGA	6.90	106.20	22.10	0.990

Two main differences can be easily observed between the microwave pyrolysis process and the conventional process: one is the sequence of events at the beginning of the pyrolysis reaction; the other one is the activation energy of the main pyrolysis phase. As to the differences in the pyrolysis start, WPCB was pyrolyzed with weight loss at the onset of microwave irradiation, compared with an induction phase of about 100 s in the simulated pyrolysis process. The promotion effect on reaction start caused by microwave irradiation is mainly due to metal-microwave discharge as mentioned above. With regard to the activation energy, it should be same under the same heating rates if the kinetic processes in both cases are same. The fact that the activation energy in microwave heating is remarkably lower than that in conventional heating indicates that the kinetics in microwave heating is different from that in conventional heating schemes. With respect to the application of microwave heating to chemical reactions, both “thermal effect” and “non-thermal effect” are put forward and discussed. Here, the kinetics for microwave pyrolysis process can be attributable to the microwave internal heating style, wherein samples are heated uniformly, leading to reductions in temperature gradients in the sample and a reduced heat loss via conduction: more heat in the sample facilitates

chemical reactions [33]. Moreover, the microwave-metal discharge plasma may be also beneficial in driving chemical reactions, as a result of “non-thermal” effects or a thermal catalyst-type effect. Due to the remarkably reduced activation energy caused by microwave heating style, certain special effects (*i.e.*, non-thermal effects) can be concluded to contribute to the reaction kinetics.

In conclusion, the pyrolysis kinetics of WPCB subject to microwave heating are different from those in conventional heating, which makes the research on microwave-induced pyrolysis of waste very worthwhile. The results will help us further our research on microwave pyrolysis of electronic waste and other similar waste. And, more work is required to investigate the non-thermal effect of microwave heating on activation energy in the future.

4. Conclusions

This work describes a kinetic study of the thermal decomposition of WPCBs subject to both conventional TGA electric heating and a microwave thermogravimetric system, although the experimental conditions such as particle size, sample mass, flow rate of purge gas, *etc.*, were different in the microwave pyrolysis *vs.* conventional TGA experiments. Our results indicate that the activation energy is decreased significantly with increasing heating rates for the thermal decomposition of WPCB material. The pre-exponential factor changed with activation energy mainly as a result of a kinetic compensation effect. When WPCB material was inundated with 2.45 GHz microwaves, a rapid heating process took place, with a volumetric expulsion of volatile matter that occurs immediately after the sample exposure to microwaves. Compared with a simulated conventional TGA incorporating a similar heating rate, the activation energy in microwave-induced pyrolysis is much smaller. This can be attributed to the internal-type heating style and a catalyst effect caused by the presence of microwave heating or microwave-metal discharges. A high disposal efficiency and low activation energy indicated by the microwave-induced pyrolysis of WPCBs makes the adoption of microwave technology an attractive approach for the disposal of WPCBs and even WEEE materials.

Acknowledgments

The authors thank the support of Program for New Century Excellent Talents in University (NCET-10-0529).

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