

## On the pyrolysis kinetics of scrap automotive tires

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### Abstract

Pyrolysis kinetics of scrap tires of passenger car and truck have been investigated thermogravimetrically under heating rates of 5, 10, 20 and 30 K/min and temperature range 373–1273 K in nitrogen. The results show that the initial reaction temperatures are 482–521 K for the tire of passenger car and 458–511 K for truck tire. Both tires exhibit similar behaviors that the initial reaction temperature decreases, but reaction range and reaction rate increase when heating rate is increased. The overall rate equation for each tire can be modeled satisfactorily by a simple one equation from which the kinetic parameters such as the activation energy ( $E$ ), the pre-exponential factor ( $A$ ), and the reaction order ( $n$ ) of unreacted material based on Arrhenius form are determined using Friedman's method. The results show that two tires behave similarly and the average kinetic parameters of two tires are  $E = 147.95 \pm 0.21$  kJ/mol,  $A = (6.295 \pm 1.275) \times 10^{10} \text{ min}^{-1}$ , and  $n = 1.81 \pm 0.18$ . The predicted rate equations compare fairly well with the measured data. © 2001 Elsevier Science B.V. All rights reserved.

**Keywords:** Pyrolysis kinetics; Scrap tires; Thermogravimetric analysis; Arrhenius equation

### 1. Introduction

Scrap tires accumulated in stockpiles or uncontrolled dumps can cause serious problems to the environment, public health and safety. The piled tires provide breeding sites for mosquitoes that can spread serious diseases and are vulnerable to cause fire hazards. It is estimated that over 100,000 t scrap tires were generated annually in Taiwan [1]. Since rubber materials are essentially non-biodegradable, they are not suitable either for compost or land-filling. Accordingly, thermal treatment by pyrolysis or incineration of these materials is attractive.

A properly functioned thermal-treatment system cannot only resolve the disposal problems, but also make energy or fuel recovery from the wastes [2,3]. However, in order to

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provide a more detailed thermal and flow analyses and more accurate performance evaluation of an incinerator, the rate of thermal decomposition of solid wastes must be known [4–6]; and that eventually relies on the kinetic studies of solid wastes under pyrolysis or burning condition.

This work is concerned with the pyrolysis kinetics of scrap tires of passenger cars and trucks. Automotive tires contains mainly rubber-related materials such as natural rubber (NB), butadiene rubber (BR) or styrene butadiene rubber (SBR). Brazier and Nickel [7] used differential scanning calorimetry and derivative thermogravimetric analysis to study the peak temperatures and product yields of natural rubber, polybutadiene rubber, and styrene–butadiene rubbers of various blends in nitrogen at a heating rate of 10 K/min. Thermal decomposition of commercial sample of butyl rubber was studied to determine the activation energy and reaction order in nitrogen [8]. Thermal analysis of poly-chloroprene rubber was investigated at various heating rates to determine the apparent activation energy and correlation of its mechanical aging with the formation of volatile products [9,10]. Pyrolysis mechanism of natural rubber was investigated by Groves and Lehrle [11] who found that the monomer and principal dimers were formed mainly as primary products, though the formation of the dimer also involved monomer recombination processes. The effect of operating parameters on the yields of liquid oil compositions and the quality of both liquid and solid products were studied by Wei et al. [12] in a fluidized bed. Thermal degradation kinetics of BR, SBR, and polybutadiene rubbers were investigated by Lin et al. [13–15] in which two or three reactions were involved for the mixed rubbers. The kinetic studies by Chen et al. [16–18] show that one reaction is involved when SBR or epoxy resin is decomposed in an inert gas, but two reactions are involved when oxygen is present in the carrier gas. Since thermal decomposition is not only an independent process, but also a first step in the gasification or combustion process. In addition, the rate of heat supply, total energy and operation time are important design parameters for a pyrolysis system to recover the gaseous and carbon products. Consequently, the pyrolysis kinetics of scrap tires deserves more investigations.

In this paper, pyrolysis kinetics of scrap tires of passenger car and truck under non-isothermal conditions are investigated by thermogravimetric measurement. The experiments were carried out for various heating rates (5, 10, 20 and 30 K/min) with nitrogen as the carrier gas. The apparent kinetic parameters such as the activation energy, the pre-exponential factor, and the reaction order of unreacted material, and thus, the pyrolysis rate equation for each tire are determined.

## **2. Experiments and data analysis**

### *2.1. Test material*

The tire samples were supplied by a local tire recycling company. Fibers and steel wires were removed automatically while the tires were shredded and then ground to powders. Tire powders were brought back for laboratory analysis. The powders were screened by 42 mesh (or 355  $\mu\text{m}$ ) for tires of passenger car and by 9 mesh (or 2 mm) for truck tire. The initial weight of sample powders was 8.42–15.38 mg. The basic properties of screened powders

Table 1  
Proximate analysis and lower heating value of scrap tires

	Passenger car	Truck
Moisture (wt.%)	0.54	0.87
Combustible (wt.%)	93.73	94.59
Ash (wt.%)	5.73	4.54
LHV (kJ/g)	32.755	31.554

Table 2  
Elemental composition of scrap tires (wt.%)

	Passenger car	Truck
C	81.16	85.19
H	7.22	7.42
O	2.07	1.72
N	0.47	0.31
S	1.64	1.52
Ash	7.44	3.84

obtained by proximate analysis and heating value test are listed in Table 1, which shows that combustible compositions are dominant. The lower heating values (LHVs) of two tires are 31.6–32.8 kJ/g. The elemental analysis listed in Table 2 shows that both tires mainly contain carbon (81.2–85.2%), the rest are hydrogen (7.22–7.42%), oxygen (1.72–2.07%), sulfur (1.52–1.64%), and nitrogen (0.31–0.47%).

## 2.2. Test apparatus

The thermogravimetric system (Shimadzu TGA-50) for decomposition studies is shown schematically in Fig. 1. It consists of an electrically heated reaction tube of 18 mm in inner diameter and 165 mm in length. About 9 mg of the sample was placed in a quartz cell of 6 mm in diameter. The cell was then put on a platinum crucible, hung by a 155 mm long suspension wire, and connected to the thermobalance. The temperature of reaction tube is 373–1273 K and is controlled by a type-K chromel–alumel thermocouple wire placed about 1–2 mm below the platinum crucible. The heating rate of the reaction tube can be preset manually in the range of 0–99 K/min. In this study, heating rates  $\beta = 5, 10, 20$  and 30 K/min were chosen. The carrier gas passed through the reaction tube from top to bottom and volatile products were collected by a water trapper.

Note that in order to obtain the intrinsic reaction rate, the effects of heat transfer and temperature gradient in the sample should be eliminated. The heat transfer effect can be reduced by purging carrier gas velocity as high as possible [19]. As such, flow rate of nitrogen gas was supplied at 50 ml/min for all the tests. In addition, two methods might be used to reduce any error in temperature measurement due to temperature gradient: (i) bring the thermocouple in contact with the sample or (ii) reduce the sample size. As pointed out by Antal et al. [20], the first method is not usually possible with a thermogravimetric apparatus

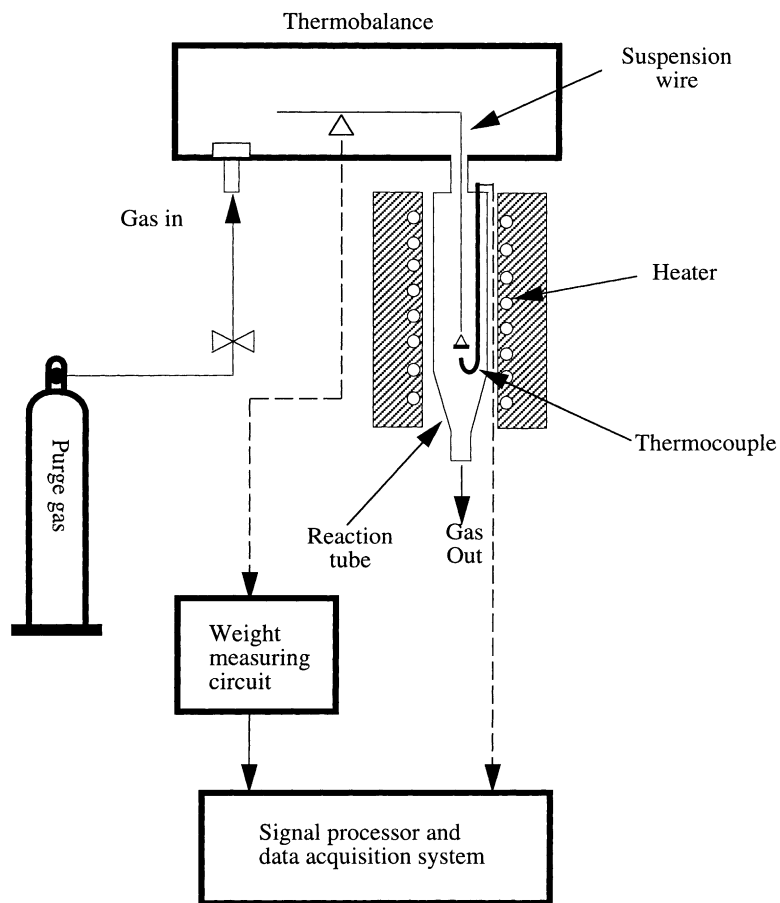


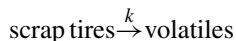
Fig. 1. Schematic of thermogravimetric apparatus.

(including the present one), and the difficulties in using a sample size below 0.1 mg would be greatly aggravated. It was found in this study that there was no essential difference in the temperature measurements between the samples of 3 and 10 mg, and a sample weight of ~10 mg was used for all the tests.

Variation of the sample mass with reaction temperature was detected by the photo-electric element and weight-measuring circuits in the thermobalance. The signals were then transmitted to the personal computer through an analog-to-digital converter for subsequent data storage, analysis and plotting. The experiments started after all the control units had been set, and the change of the sample mass (the TG curve) and the derivative of the mass change (the DTG curve) with respect to reaction temperature were recorded every 6 s. The thermobalance measured mass to 0.001 mg, with an accuracy of  $\pm 1\%$ . When the experiment was finished, the furnace's heat supply was turned off, but carrier gas was kept flowing until the reactor cooled down below 100°C.

### 2.3. Analysis of kinetic parameters from TG data

When polymers are subject to heating or burning conditions, complicated reaction mechanism such as random-chain scission, end-chain scission, chain stripping, cross-linking and coke formation would take place. To focus on the determination of apparent kinetic parameters useful for engineering purposes, a simple reaction is assumed as following:



Further, the rate equation of conversion factor  $\alpha$  is expressed in Arrhenius relation in the form of [21]

$$\frac{d\alpha}{dt} = A \exp\left(-\frac{E}{RT}\right) f(\alpha) \quad (1a)$$

$$\alpha = \frac{W_0 - W}{W_0 - W_f} \quad (1b)$$

where  $t$  is the time (min),  $A$  the pre-exponential factor ( $\text{min}^{-1}$ ),  $E$  the activation energy (kJ/mol),  $T$  the reaction temperature (K),  $R$  the universal gas constant ( $=8.313 \text{ J/mol K}$ ),  $W$  (mg) the mass of the sample at time  $t$ , and  $W_0$  (mg) and  $W_f$  (mg) are the initial and final (or residual) mass of the sample, respectively. The functional form of  $f(\cdot)$  in Eq. (1a) is assumed to be the  $n$ th order of the unreacted material according to

$$f(\alpha) = (1 - \alpha)^n \quad (2)$$

That is, the rate equation for conversion factor in terms of the reaction rate constant  $k$  is

$$\frac{d\alpha}{dt} = k(1 - \alpha)^n \quad (3)$$

$$k = A \exp\left(-\frac{E}{RT}\right) \quad (4a)$$

or

$$\ln k = \ln A - \frac{E}{RT} \quad (4b)$$

The kinetic parameters in the above equation such as  $A$ ,  $n$ ,  $E$ , and  $k$  can be determined once the TG or DTG data are obtained. In this study, the Friedman's method [22] was employed to evaluate these parameters, and is described briefly below.

The natural logarithm of Eq. (1a) is taken to obtain

$$\ln\left(\frac{d\alpha}{dt}\right) = -\frac{E}{RT} + n \ln[Af(\alpha)] \quad (5)$$

For a fixed  $\alpha$ , the second term in the right-hand side of Eq. (5) is constant. Hence, the plot of  $\ln(d\alpha/dt)$  versus  $1/T$  from the data at various heating conditions with  $\alpha$  as the parameter can be correlated by the least-square method to yield a straight line, for which the slope is  $-E/R$

and the intercept is  $\ln[Af(\alpha)]$ . Thus, the apparent activation energy can be obtained from the averaged value of the activation energy for different conversion factors  $\alpha$ . Substituting this averaged value  $E$  back into Eq. (5) in conjunction with the data of  $\alpha$  and  $d\alpha/dt$ , the second term in the right-hand side of Eq. (5) can be obtained. Combining Eq. (2) then yields

$$\ln[Af(\alpha)] = \ln A + n \ln(1 - \alpha) \quad (6)$$

The averaged values of  $\ln[Af(\alpha)]$  obtained for various heating conditions are plotted against  $\ln(1 - \alpha)$  to yield a least-square straight line, for which the slope is  $n$  and the intercept is  $\ln A$ . This completes the determination of all kinetic parameters. Note that the Arrhenius form has been widely used for describing the one-step reaction rate [21]. When two or more reaction steps are involved, the reaction rate constant  $k$  in the simple one equation is replaced by  $k_i$  in the  $i$ th step such that Eq. (1a) and Friedman's method can still be applied [16,17,23,24].

### 3. Results and discussion

#### 3.1. Effect of heating rate

Figs. 2 and 3 display the variations of fractional mass (TG curves) and derivative of mass-change (DTG curves) with respect to reaction temperature  $T$  at various heating rates for tires of passenger cars and trucks, respectively. Both figures show that each TG curve is quite smooth. Further, the reaction zones shift to the high temperature region and peak heights increase when the heating rate increases. Both DTG curves in Figs. 2 and 3 show one dominant peak. A second lower-value peak appears at higher temperature and is more obvious for truck tire at high heating rate. However, the smoothness of both TG curves suggests that degradations of two tires may be accounted for or approximated by one lumped reaction even if both tires contain various similar compounds such as NR and BR (to be seen later).

The reaction characteristics of scrap tires in this study are summarized in Table 3, which shows that initial reaction temperature  $T_i$  decreases but final reaction temperature  $T_f$  increases when heating rate is increased. Thus, the reaction range  $\Delta T (=T_f - T_i)$  increases

Table 3  
Reaction characteristics of scrap tires

$\beta$ (K/min)	$T_i$ (K)	$T_m$ (K)	$T_f$ (K)	$W_f/W_0$ (%)
Passenger car				
5	520.6	694.1	774.6	43.9
10	492.0	669.8	775.9	39.9
20	485.8	687.1	795.4	38.1
30	481.8	697.6	821.3	37.1
Truck				
5	511.0	665.6	766.8	38.9
10	480.9	672.6	784.7	38.0
20	478.5	687.7	806.1	38.4
30	458.6	702.7	826.2	37.6

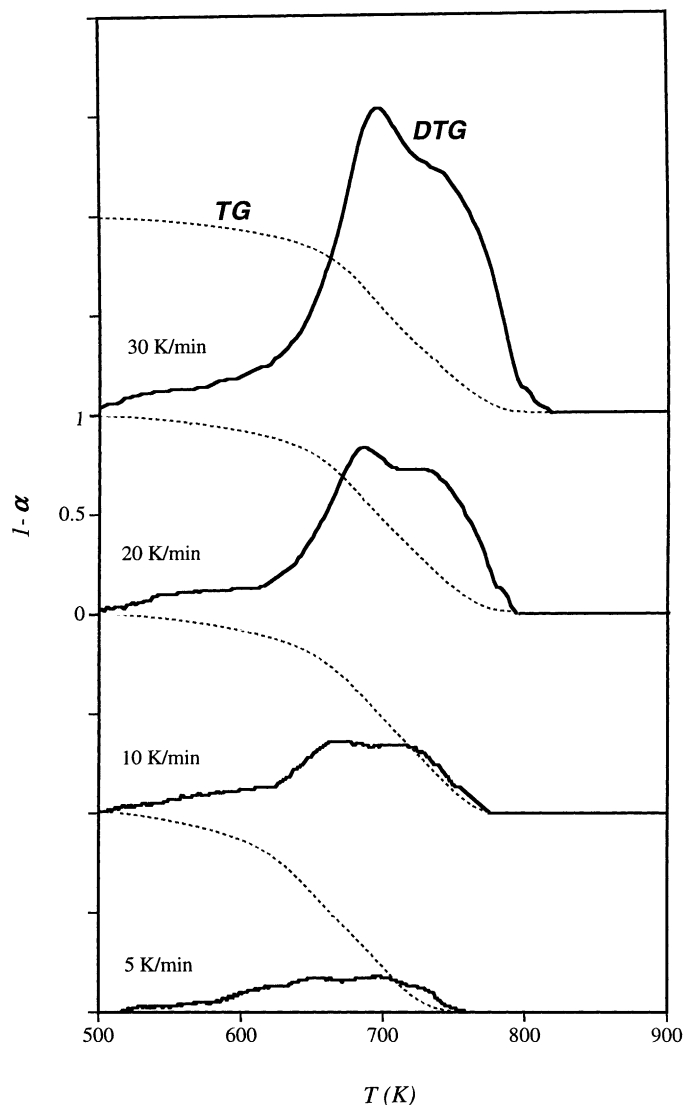


Fig. 2. TG (dashed) and DTG (solid) curves for various heating rates in nitrogen for tire of passenger car.

with increasing heating rate. Table 3 also shows that the initial reaction temperature is about 482–521 K (209–248°C) and 458–511 K (185–238°C) for tires of passenger car and truck, respectively, and the respective peak temperature  $T_m$  at which conversion rate is maximum (i.e.  $d\alpha/dT = 0$ ) is about 669–698 K (396–425°C) and 665–703 K (392–430°C). Note that the decomposition temperature is about 470–670 K for processing oils, 600–680 K for NR, and 680–750 K for BR [7,10,14,23]. The residual  $W_f/W_0$  listed in Table 3 is 37.1–43.9%

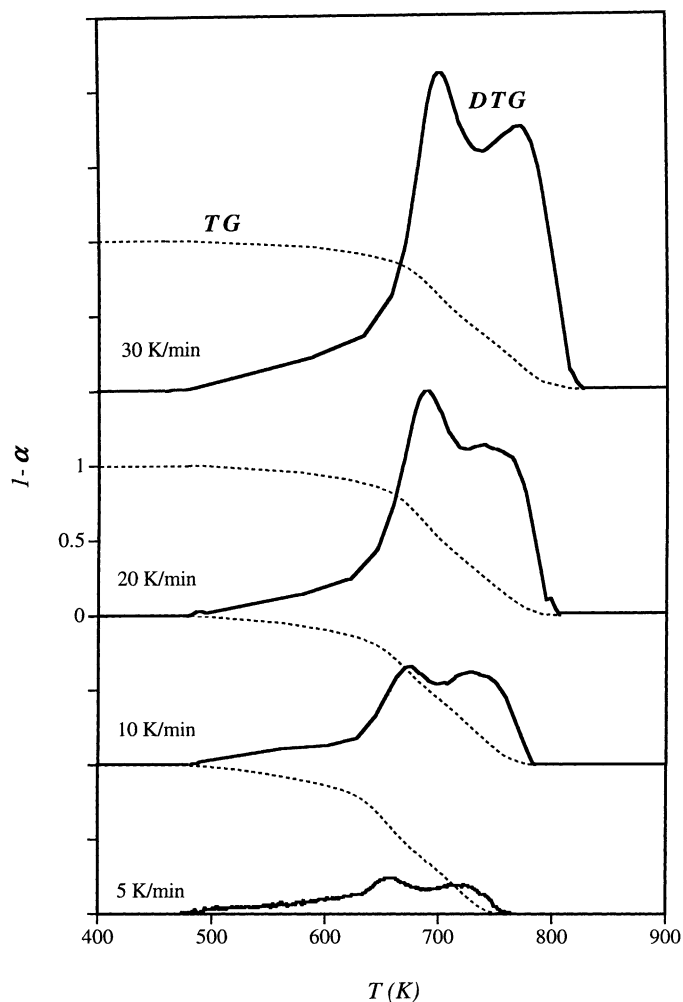


Fig. 3. TG (dashed) and DTG (solid) curves for various heating rates in nitrogen for truck tire.

for tire of passenger car and is 37.6–38.9% for truck tire, varying slightly with heating rate. The residual contains mainly carbon black.

Fig. 4 plots the activation energy versus conversion factor for both cases. It is seen from Fig. 4 that the activation energy of tire of passenger car is about 117.24 kJ/mol for  $0 \leq \alpha \leq 0.2$ , and is about 183.3 kJ/mol for  $0.2 < \alpha \leq 1.0$ . The activation energy of truck tire is 83.47 kJ/mol for  $0 \leq \alpha \leq 0.2$ , and is about 151.57 kJ/mol for  $0.2 < \alpha \leq 1.0$ . The plots in Fig. 4 also suggest that one simple reaction may suffice to describe the reaction rate for each tire in this study. Note that the initial conversion of  $0 \leq \alpha \leq 0.2$  corresponds to the dehydration and decomposition of processing oils, and is followed up by the decomposition of NR and/or SBR for  $0.2 < \alpha \leq 1.0$  [7,10,15,16].



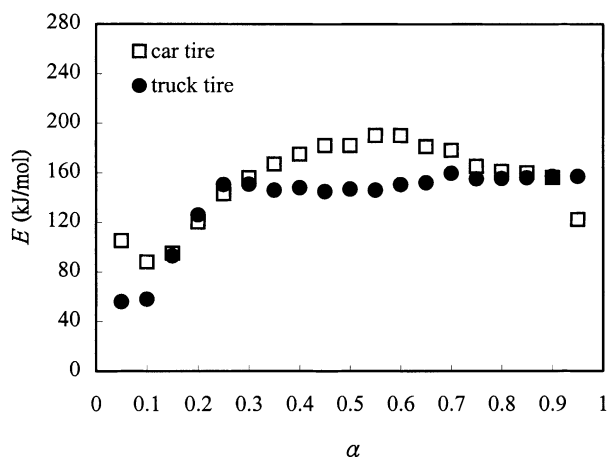


Fig. 4. Activation energy versus conversion factor.

The kinetic parameters derived from all TG and DTG data using Friedman's method in the temperature range 373–1273 K are listed in Table 4, which shows that the activation energy  $E = 147.64$  kJ/mol, pre-exponential factor  $A = 7.57 \times 10^{10} \text{ min}^{-1}$ , and reaction order  $n = 1.98$  for the tire of passenger car; and  $E = 148.06$  kJ/mol,  $A = 5.02 \times 10^{10} \text{ min}^{-1}$ , and  $n = 1.63$  for truck tire. It appears that two types of tire behave similarly. The average values from two tires listed in Table 4 are  $E = 147.95 \pm 0.21$  kJ/mol,  $A = (6.295 \pm 1.275) \times 10^{10} \text{ min}^{-1}$ , and  $n = 1.81 \pm 0.18$ . It is worth mentioning that the pyrolysis kinetic parameters may vary

Table 4  
Comparisons of kinetic parameters of scrap tires<sup>a</sup>

Type	$E$ (kJ/mol)	$A$ ( $\text{min}^{-1}$ )	$n$
Car <sup>b</sup>	147.64 (1.0)	$7.57 \times 10^{10}$	1.98
Truck <sup>b</sup>	148.06 (1.0)	$5.02 \times 10^{10}$	1.63
Sidewall <sup>c</sup>	203.9 (0.695)	$2.08 \times 10^{15}$	1.0
	195.1 (0.225)	$1.44 \times 10^3$	1.0
	42.1 (0.077)	$1.92 \times 10^{18}$	1.0
Tread <sup>c</sup>	127.3 (0.636)	$3.78 \times 10^{16}$	1.0
	209.0 (0.213)	934	1.0
	28.7 (0.15)	$3.27 \times 10^6$	1.0
Tire <sup>d</sup>	125.58 (0.20)	$2.68 \times 10^{11}$	1.0
	178.74 (0.28)	$6.78 \times 10^{13}$	1.0
	244.04 (0.52)	$2.85 \times 10^{17}$	1.0

<sup>a</sup> The values in parenthesis are the corresponding weighting factor in each reaction.

<sup>b</sup> This work.

<sup>c</sup> From [23].

<sup>d</sup> From [24].

with the composition and the type of rubber. Comparisons of pyrolysis kinetic parameters with other studies are given in Table 4. Notice that three stages of reaction are considered with the assumed first-order rate in Kim et al. [23] and Teng et al. [24] and kinetic parameters in each stage are listed in Table 4 with corresponding weighting factor (or percentage of volatile by weight) noted in the parenthesis. As mentioned previously, due to the smoothness of each TG curve in this study, a simple one reaction was assumed such that the weighting factor is unity in this study. It is seen from Table 4 that the activation energies of this work lie in 127.3–203.9 kJ/mol of Kim et al. [23] for the first two reactions accounting for a subtotal of weighting factors 0.85 and 0.92, respectively, for the sidewall and the tread of scrap tire. The activation energies of this work also lie in 125.58–244.04 kJ/mol of Teng et al. [24]. Although a simple one reaction was assumed, but reaction order  $n$  and thus  $A$  were determined directly from TG data in this study, instead of assuming  $n = 1$  in the studies of Kim et al. [23] and Teng et al. [24].

### 3.2. Pyrolysis rate equation

When the values of the kinetic parameters listed in Table 4 are substituted into Eq. (3), the rate equations of scrap tires in nitrogen are as follows.

For tire of passenger car:

$$\frac{d\alpha}{dt} = 7.57 \times 10^{10} \exp\left(-\frac{17,760}{T}\right) (1 - \alpha)^{1.98} \quad (7a)$$

or

$$\ln k = 25.05 - \frac{17,760}{T} \quad (7b)$$

For truck tire:

$$\frac{d\alpha}{dt} = 5.02 \times 10^{10} \exp\left(-\frac{17,811}{T}\right) (1 - \alpha)^{1.63} \quad (8a)$$

or

$$\ln k = 24.64 - \frac{17,811}{T} \quad (8b)$$

The Arrhenius plot of  $\ln k$  versus  $1000/T$  together with the data are shown in Figs. 5 and 6, respectively, for tires of passenger car and truck. It is seen that the derived equations based on one simple reaction fit the data fairly well; the correlation coefficient  $\gamma^2$  is 0.924 for tire of passenger car and 0.905 for truck tire. Note that the reaction temperature is related to the heating rate  $\beta$  by

$$\frac{dT}{dt} = \beta \quad (9)$$

Hence, the conversion factor  $\alpha$  or the fractional mass  $1 - \alpha$  can be recovered by integrating Eqs. (7a)–(9) or Eqs. (8a) and (9) simultaneously. Figs. 7 and 8 show the TG data and computed values obtained by using the Runge–Kutta numerical integration scheme for both

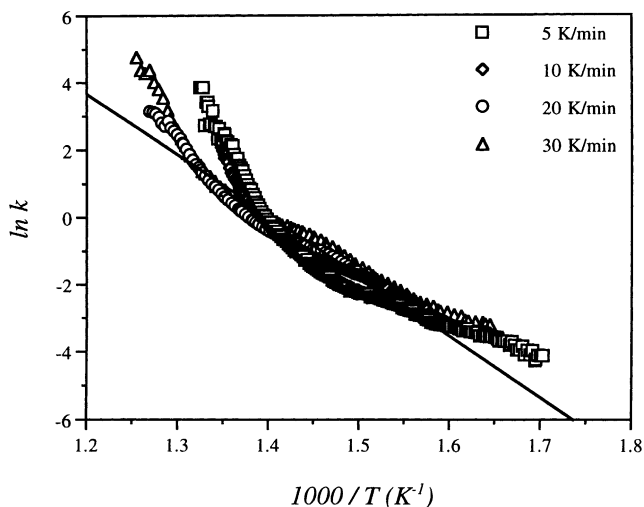


Fig. 5. Arrhenius plot of tire of passenger car in nitrogen. Symbols are the data, and solid line is the rate equation ( $\gamma^2 = 0.924$ ).

tires. It is seen that computed values agree fairly well with the TG data for small heating rates. However, there are some overestimates at the beginning of the reaction ( $\alpha \leq 0.25$ ). This may be due to the fact that activation energy in the initial conversion is actually lower than the average activation energy (see Fig. 4). As seen above, the pyrolysis time and reaction temperature can be determined once the reaction rate equation and/or heat supply to the pyrolysis system are prescribed.

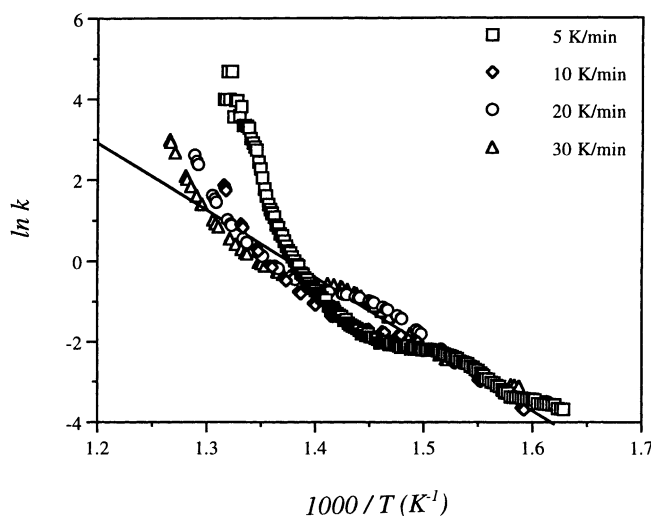


Fig. 6. Arrhenius plot of truck tire in nitrogen. Symbols are the data, and solid line is the rate equation ( $\gamma^2 = 0.905$ ).

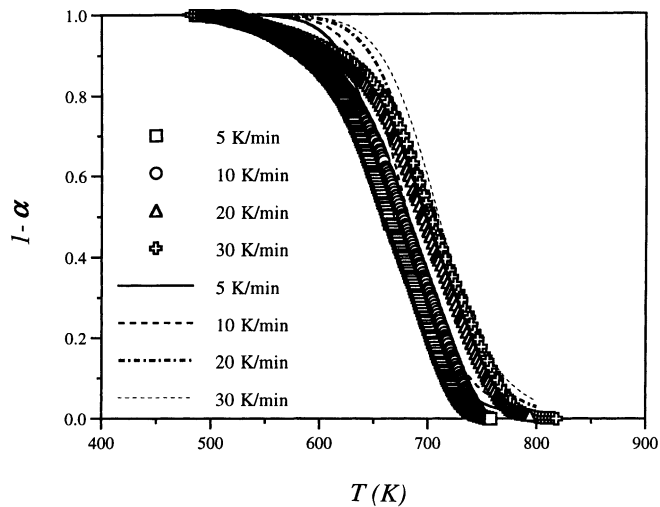


Fig. 7. Comparison of TG data (symbols) and computed values (solid lines) from numerical integration of rate equation for tire of passenger car.

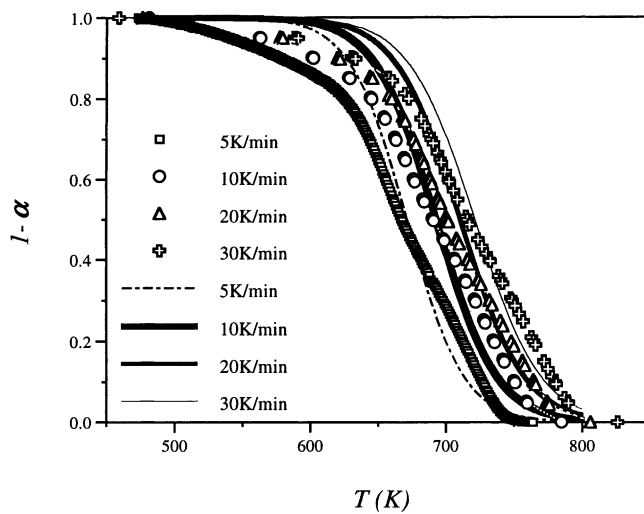


Fig. 8. Comparison of TG data (symbols) and computed values (solid lines) from numerical integration of rate equation for truck tire.

#### 4. Conclusions

Pyrolysis kinetics of scrap tires of passenger car and truck in nitrogen have been investigated thermogravimetrically for heating rates of 5, 10, 20 and 30 K/min and temperature

range 373–1273 K (100–900°C) with nitrogen as the carrier gas. The results show that the initial reaction temperature is 482–521 K (209–748°C) for the tire of passenger car and is 458–511 K (185–238°C) for truck tire. The initial reaction temperature decreases and reaction range increases when heating rate is increased for both tires. The reaction begins with the dehydration and decomposition of processing oils, followed by decomposition of natural rubber and/or butadiene rubber.

The rate equation for each tire can be modeled satisfactorily by a simple one reaction. The kinetic parameters for each tire based on Arrhenius equation are determined for the activation energy, the pre-exponential factor and reaction order using Friedman's method. Reasonable agreements are achieved between the measured data and predicted values. Two tires behave similarly in pyrolysis decomposition. The average kinetic parameters of two tires are  $E = 147.95 \pm 0.21$  kJ/mol,  $A = (6.295 \pm 1.275) \times 10^{10} \text{ min}^{-1}$ , and  $n = 1.81 \pm 0.18$ . The results should be useful for the rational design and operation of pyrolysis or incineration system in which waste tires are involved.

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