

# Kinetic Study of Thai-lignite Char Gasification Using the Random Pore Model

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## Abstract

Products from coal gasification are considered as promising fuels for Thailand in this decade. The coal gasification process consists of two main steps; i) pyrolysis step, and ii) char gasification step (which is a controlling step). To have a better understanding of the overall process and have a proper reactor design for Thai-lignite gasification, the kinetics of the char gasification step is needed to be clarified, while the effects of the pyrolysis step on char gasification reactivity are also needed to be studied. Two Thai-lignite chars, char A and char B, were produced from two different compositions of Thai lignite by a horizontal furnace at different pyrolysis conditions, in order to investigate effects of the pyrolysis step on char gasification reactivity. It was found that the reactivity of char gasification increased with increasing pyrolysis heating rate (5 °C/min, 10 °C/min and flash pyrolysis) and decreasing pyrolysis temperature. These were due to generation of surface area and no formation of crystal structures during pyrolysis conditions. Char A and char B were then gasified by using a thermogravimetric analyzer under atmospheric air and three different heating rates for kinetic study. The kinetic parameters were calculated by using the random pore model and the homogeneous model. It was observed that the results obtained by using the random pore model corresponded well to the experimental results for both char A and char B. On the other hand, the values of homogeneous model deviated from the experimental results with progress of reaction, especially in the latter period of reaction. The results also indicated that the structure change of char particles was one of the important parameters for coal gasification. In this study, activation energies of char A and char B were found at 136.5 and 150.4 kJ/mol, respectively.

**Keywords:** Thai-lignite, Kinetics, Char gasification, Random pore model, TGA

## 1. Introduction

Since the conventional fuel demand has already reached its critical situation, an alternative fuel production has therefore gained much attention in recent years. The products from a coal gasification process are considered promising fuels for this moment and the near future, since there are quite a number of coal reserves in Thailand (approximately 870.05 million tons from various parts in Thailand such

as Mae Moh, Krabi, Saba Yoi, and Sin Pun [1]) and the amount of natural gas and oil reserves for energy production in Thailand are much lower than the amount of coal reserves [2]. Coal gasification is a process for converting carbon and volatile matters in coal, by using a gasifying agent, into a fuel gas which consists of "syngas" (hydrogen and carbon monoxide) and methane. Reaction can occur under controlled conditions of temperature and pressure (at a given

sufficient time) [3-6]. The coal gasification process can be divided into two main steps; i) pyrolysis step, and ii) char gasification step [6-10]. However, the char gasification step is a controlling step for overall conversion process because its reaction rate is much slower than the pyrolysis step. As a result, kinetics of the char gasification step can provide a basic knowledge for a better understanding and a proper reactor design for the coal gasification process.

Many models have been used to analyze the kinetics of gasification; such as a homogeneous model, a core model and a random pore model. Recently, the random pore model has been applied together with a temperature programmed reaction technique (TPR) to calculate the kinetic parameters of char gasification reaction in atmospheric air [11, 12]. It is found that the kinetic parameters (activation energy, frequency factor and parameters of structural change) very well represent the experimental data, based on the equation of the random pore model [12, 13]. The random pore model is a kinetic model proposed by Bhatia and his group, in which the physical structure changes during gasification reaction, is also considered [14]. The technique that provides more kinetic information of the gas-solid reaction when a number of experiments are the same as an isothermal experiment is called the temperature programmed-reaction. The TPR technique is a method that the weight loss of a solid sample is measured during the heating of the solid sample to a desired temperature, at constant heating rate under conditions of reactive gas.

Although coal gasification is one promising process for energy production at the moment, but there are only a few works on coal gasification in Thailand, especially in the area relating to the kinetics of the char gasification step (controlling step of overall conversion process) for Thai lignite. In this paper, the calculations of kinetic parameters (activation energy, frequency factor and parameter of structural change) based on the random pore

model together with the TPR technique are the main focus of study, including the comparison between results obtained by using the random pore model and the homogeneous model. In addition, char samples acquired from different pyrolysis conditions were also gasified by a thermogravimetric analyzer to clarify the effects of pyrolysis heating rate on the reactivities of char gasification.

## 2. Materials and Methods

### 2.1 Sample preparations

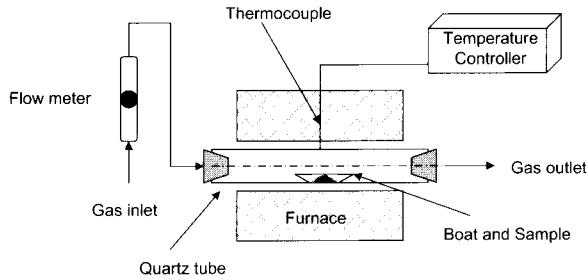
Two different compositions of Thai-lignite from Mae Moh Mine (as called Thai-lignite A and Thai-lignite B) were used. Both Thai lignite were ground and sieved to particle size less than 75  $\mu\text{m}$  and then dried in vacuum oven at 70  $^{\circ}\text{C}$  for 24 hr. Table 1 shows the proximate analysis and ultimate analysis of these samples.

Thai-lignite A and B were pyrolyzed by using a horizontal furnace at desired temperature of 900  $^{\circ}\text{C}$  and heating rate of 10  $^{\circ}\text{C}/\text{min}$  in nitrogen atmosphere and cooled afterward to ambient temperature in order to produce char A and B for the char gasification process.

A schematic diagram of the horizontal furnace for pyrolysis is illustrated in Figure 1. Sample holder containing the raw lignite was placed into the center of a quartz tube and  $\text{N}_2$  gas was continuously fed through the quartz tube. After steady conditions, for approximately 30 min, the quartz tube was heated to a desired temperature by the horizontal furnace at a heating rate of 10  $^{\circ}\text{C}/\text{min}$  and held for 10 min at the desired temperature. The temperature of the reactor was measured and controlled by a thermocouple (K-type) and a temperature controller (SHIMADEN, FP21), respectively. After the reaction reached its completion, the char product was cooled to the ambient temperature in the atmospheric nitrogen gas, and then kept in a desiccator waiting for the char gasification process in the next step.

**Table 1:** Proximate analysis and ultimate analysis of samples.

	Proximate analysis [% dry-basis]			Ultimate analysis [% wt.]				
	VM	FC	Ash	C	H	N	S	O <sub>diff.</sub>
Lignite A	48.6	33.7	17.7	65.0	5.1	2.3	1.4	26.3
Lignite B	40.7	24.8	34.5	67.7	5.9	2.4	3.1	21.0
Char A	9.1	58.7	32.2	-	-	-	-	-
Char B	12.2	33.0	54.8	-	-	-	-	-



**Figure 1:** A schematic diagram of the horizontal furnace reactor.

Char A and char B were consequently gasified by the Thermogravimetric analyzer, TGA (PerkinElmer, Pyris), at a temperature range of 300-550 °C and three different heating rates (2, 5 and 10 °C/min) in atmospheric air. For each run, 5 mg of char were placed into a crucible, and then heated under the controlled heating rate to the desired temperature. The reaction temperature was directly measured by a thermocouple. Weight loss of sample was continuously recorded as a function of temperature (TPR data). Char conversion ( $X$ , dry, ash-free.) as a function of temperature ( $T$ ) can be obtained by equation (1).

$$X = 1 - \frac{W}{W_0} \quad [\text{daf.}] \quad (1)$$

where  $W$  and  $W_0$  are weights of the remaining solid at time  $t$  and initial weight of char at  $t = 0$ , respectively.

The powder X-ray diffraction method (XRD) with a diffractometer (Bruker AXS Model D8 Discover) was used to analyze the crystalline properties of samples. The X-ray patterns were recorded by using Cu K $\alpha$  radiation. The power supply was 40 kV and 40 mA. The peaks were obtained by step at  $2\theta$ , scan speed at 0.3 sec/step and increment of 0.025 degree. Peaks of crystal structures were compared with those of the reference materials.

## 2.2 Theoretical background

### 2.2.1 The Homogeneous Model

This model reduces heterogeneous gas-solid reactions which occurred in the coal gasification process to homogeneous reactions by assuming that the gas is reacting with char in all possible places, both outside and inside the

particle surface. From this basis assumption, the reaction rate can be represented by equation (2).

$$\frac{dX}{dt} = k_0 e^{-E/RT} (1-X) \quad (2)$$

As the TPR technique usually employs heating of samples at a constant rate ( $a$ ), the temperature ( $T$ ) is then related to time ( $t$ ) by equation (3).

$$T = T_0 + at \quad (3)$$

where  $T_0$  is the temperature at which heating is started. Equation (2) and (3) can be integrated and linearized to give equation (4) [12].

$$f_v = \ln \left[ \frac{1}{T^2} \ln \left( \frac{1}{1-X} \right) \right] = \ln \left( \frac{k_0 R}{aE} \right) - \frac{E}{RT} \quad (4)$$

Activation energy,  $E$ , and frequency factor,  $k_0$ , can be acquired by plotting the relationship of in the left hand side and the reaction temperature.

### 2.2.2 The Random Pore Model

The Random pore model does consider physical structural changes during the gasification reaction. Bhatia *et. al.* [14] found that a random pore model could be applied to the coal gasification reaction. Bhatia and his group considered the random growing and overlapping of pores' surfaces, which can increase and reduce the area available for reaction, simultaneously. The original model was applied to the TPR data of char gasification by Miura [12], as illustrated in equation (5).

$$\frac{dX}{dt} = k_0 e^{-E/RT} (1-X) [1 - \Psi \ln(1-X)]^{1/2} \quad (5)$$

where  $\Psi$  is a parameter related to the pore structure of the unreacted sample ( $X = 0$ ) as shown in equation (6).

$$\Psi = \frac{4\pi L_0}{\rho_c S^2} \quad (6)$$

where,  $S$ ,  $L$  and  $\rho$  represent the pore surface area, pore length, and true density, respectively.

However, Miura [12] suggested that the value of  $\Psi$  should be calculated from curve fitting between  $X$  and  $T$  from the experimental data. Actually, the relationship between apparent rate and temperature is one kind of Arrhenius plot which the activation energy is obtained from the slope of the Arrhenius plot. The equation for the Arrhenius plot of each reaction as shown in equation (7) and (8) can be determined by linearization of equation (5).

$$F(X) = \ln \frac{(dX/dt)}{1-X} = G(X) - \frac{E}{RT} \quad (7)$$

$$G(X) = k_0 [1 - \psi \ln(1 - X)]^{1/2} \quad (8)$$

As seen in equation (8), frequency factor,  $k_0$ , and parameter of structural change,  $\Psi$ , are represented as a function of  $G(X)$ . Thus, equation (8) is linearized for calculation of  $k_0$  and  $\Psi$ , which is shown in equation (9).

$$G^2(X) = k_0^2 - k_0^2 \psi \ln(1 - X) \quad (9)$$

Moreover, a standard error of calculation,  $\sigma$ , is calculated from the square root of the mean square deviation shown as equation (10).

$$\sigma = \sqrt{\frac{e_1^2 + \dots + e_n^2}{n}} \quad (10)$$

Where the  $e_i$  is an error between the experimental data and the calculated data at point  $i$  and  $n$  is a total number of point in each experiment.

### 3. Results and Discussion

#### 3.1 Effects of pyrolysis conditions on reactivity of char gasification

Normally, the char gasification step is a controlling step for the overall conversion process; however, reactivity of char gasification is still affected by a pyrolysis process which is a first step of gasification process. Therefore, the effect of pyrolysis heating rate on the reactivity of char gasification can give a better understanding of fundamental behaviors of the Thai-lignite gasification process. Processes of char preparation and char gasification have already been illustrated in the Materials and

Methods section. Figure 2 shows the loss of char conversions as a function of temperature. The char samples from rapid pyrolysis show the highest reactivity of char gasification, and followed by char samples from pyrolysis at heating rate 50 °C/min and 10 °C/min, respectively. It can be noted that the reactivity of char gasification increases with increases of the pyrolysis heating rate. These results can be explained by an increase in the surface area of each char sample, which was presented in terms of adsorption volume at evaluated pressure, when the pyrolysis heating rate increases. A BET analyzer (Belsorp II, BEL JAPAN, INC.) was therefore employed to analyze the characteristics of materials by using carbon dioxide as adsorbate gas. Table 2 shows mean pore diameter, total pore volume and surface areas of Thai-lignite A, char samples from pyrolysis at 10 °C/min and rapid heating rate.

As seen in Table 2, it is found that the mean pore diameter of samples decreased with increases in pyrolysis heating rate, whereas the surface area exhibited the opposite result. From the results of BET analysis, it can be seen that the pyrolysis at higher heating rate gave rise to development of surface area in char samples, as it produced smaller pore diameter. According to previous research [7, 8, 13], it has been reported that the development in surface area of char sample obtained from higher pyrolysis heating rate is probably due to extremely volatile explosions from inside of char particles during the pyrolysis process at higher heating rates. In case of pyrolysis at lower heating rates, bubbles of volatile matters would be formed and slowly released (no explosion) from inside of particles simultaneously, resulting in a bigger pore diameter and lower pore surface area. From the above findings, it can be suggested that the reactivity of char gasification increased with increases in the pyrolysis heating rate, due to the development of active surface area at higher pyrolysis heating rates.

In order to study the effect of pyrolysis temperature on char gasification reactivity, Thai-lignite B was pyrolyzed by the horizontal furnace at different pyrolysis temperatures (700 and 900 °C) to produce char samples. Afterward, char samples were gasified by TGA under atmospheric air and a heating rate of 10 °C/min. Figure 3 shows the reactivity of char gasification in atmospheric air at

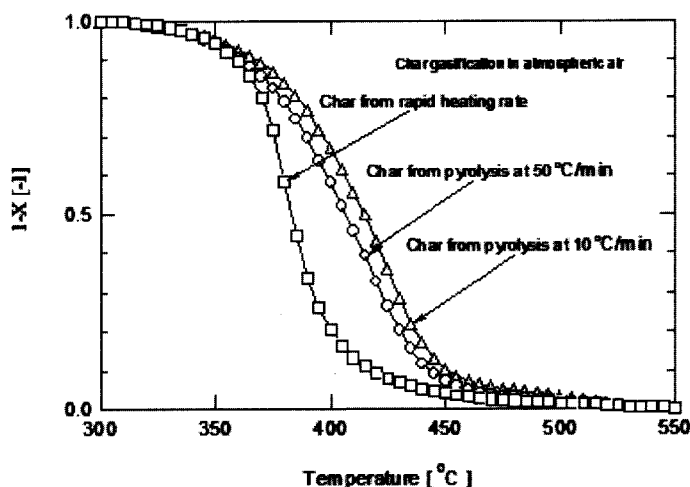
gasification heating rate of 10 °C/min as a function of temperature. It is clearly observed that the gasification reactivity of char samples from pyrolysis at 700 °C was higher than that of char samples from pyrolysis at 900 °C in a range of temperature from 300 to 500 °C. Therefore, it can be concluded that the gasification reactivity decreases with increases in the pyrolysis temperature.

The results from these experiments are found to correspond to the works of many researchers [5, 7, 15, 16]. It has been reported that the reactivity of char gasification decreases with severity of pyrolysis conditions. Furthermore, those researchers also suggested that some kinds of inherent mineral matters can act as catalysts (Ca, Na, K, etc.) and the loss of their activities is caused by sintering and formation of intercalated compounds or through vaporization. In order to elucidate this finding, the X-ray diffraction method (XRD) was carried out in this study to investigate structures of char

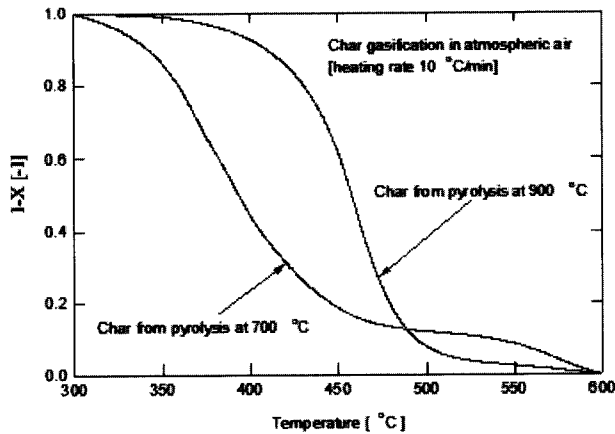
samples. Peaks of crystal structures were obtained by comparing with structures of referent materials as maintained in the XRD library, and based on the elements in Thai-lignite [17]. Figure 4 shows the XRD patterns of char samples from pyrolysis at temperatures 700 and 900°C. Main peaks of crystal structures, which can be detected by the XRD, consist of quartz (SiO<sub>2</sub>), calcium oxide (CaO) and calcium sulfide (CaS). It is observed that most of crystal-structure peaks of char sample from pyrolysis at 900 °C are higher than those of char sample from pyrolysis at 700 °C, except the peak of calcium oxide. It is well recognized that the calcium oxide is a primary calcium species which can act as catalyst for the gasification process [7, 15]. Thus, the lower gasification reactivity of char samples is probably due to the formation of inert crystal structures and the consumption of calcium oxide at higher pyrolysis temperature.

**Table 2:** Physical properties of char samples from different pyrolysis heating rate (using BET Plot-adsorbate CO<sub>2</sub> and adsorption temperature 25 °C).

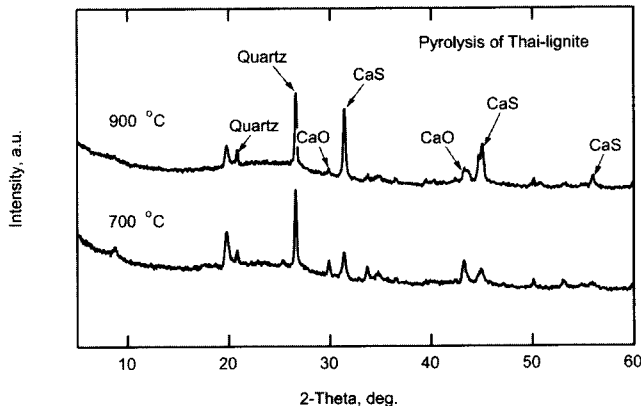
Samples	Mean pore diameter [nm]	Total pore volume [cm <sup>3</sup> g <sup>-1</sup> ]	Surface area [m <sup>2</sup> g <sup>-1</sup> ]
Raw Thai-lignite A	0.9295	1.76E-02	75.7
Char A (pyrolysis at 10 °C/min)	1.1847	6.62E-02	223.5
Char A (rapid pyrolysis)	0.8465	6.66E-02	314.7



**Figure 2:** Relationship between char conversion, char A was prepared by pyrolysis at 900 °C and three different heating rates (10, 50 °C/min and rapid heating rate). Chars were gasified by TGA at a heating rate of 2 °C/min under atmospheric air.



**Figure 3:** Reactivities of char gasification in atmospheric air and a heating rate of 10 °C/min. Chars were produced from two different pyrolysis temperatures, 700 °C and 900 °C.



**Figure 4:** X-ray diffract patterns for chars from two different pyrolysis temperatures (700 and 900 °C).

### 3.2 Calculation of the kinetic parameters by the random pore model

Figure 5 shows the temperature programmed-reaction data (TPR data) for char A and char B, respectively. The TPR data is a relationship between char conversion and its reaction temperature. Residual char fraction,  $1-X$ , and its reaction temperature are presented in  $y$  axis and  $x$  axis, respectively. For both char samples, it is found that the residual char fraction continuously decreased from 300 °C to 350 °C, and then decreased more rapidly over the range of temperature from 350 to 450 °C. For temperatures over 450 °C, the residual char fraction is found to decrease with a slow rate until the completion of reaction at 550 °C. It is also observed that the curve of the change in the

residual char fraction shifts to higher reaction temperature when increasing the heating rate.

The Arrhenius plot is a relationship between the apparent rate,  $(dX/dt)/(1-X)$ , and its reaction temperature,  $1/T$ , and therefore, it can be used to calculate the apparent activation energy,  $E$ , as illustrated in Figure 6. The straight lines of each  $X$  in the Arrhenius's plots (for char A and char B) were obtained by calculation of the  $(dX/dt)/(1-X)$  at three different heating rates; 2, 5, and 10 °C/min, as the TPR experiments must be conducted at these three different heating rates. It can be seen in the figure that the parallel lines at the different  $X$  values provide the same slope, which is considered as the activation energy of each reaction. From these Arrhenius's plots, the activation energies are observed at 136.5 and 150.4 kJ/mol for char A

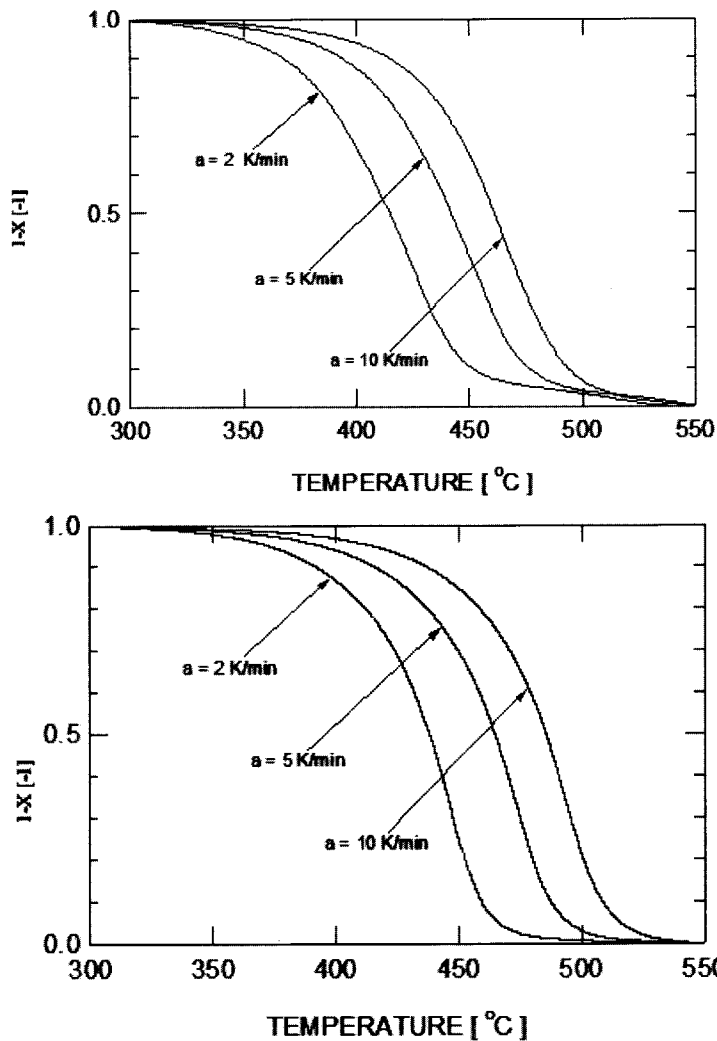
and char B, respectively. These values of activation energy are found to be close in magnitude to those obtained by other research works [18].

By utilizing equation (9), the relationship between  $G(X)$  and  $\ln(1-X)$  can be performed to acquire the  $k_0$  and  $\Psi$  as shown in Figure 7. The values of  $k_0$  can be obtained from the slope of

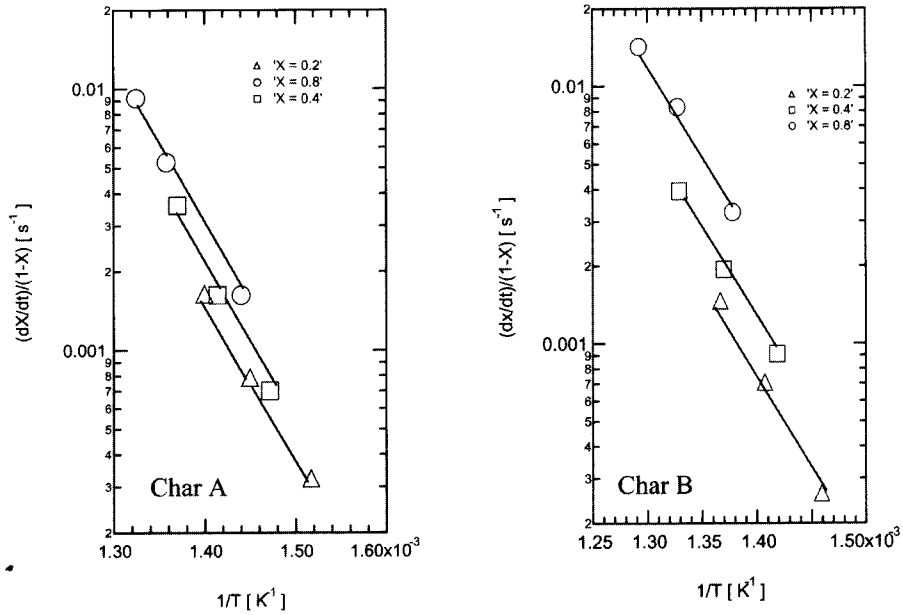
the relationship between  $G(X)$  and  $\ln(1-X)$ , while the values of  $\Psi$  can be drawn from the intercept point at y axis (at  $\ln(1-X) = 0$ ). The values of all kinetic parameters are listed in Table 3. Rate equations of char gasification with air for char A and char B are also presented in equations (11) and (12), based on the random pore model, respectively.

Char A: 
$$\frac{dX}{dt} = 18 \cdot 10^6 e^{-136500/RT} (1-X)[1 - 0.61 \ln(1-X)]^{1/2} \quad (11)$$

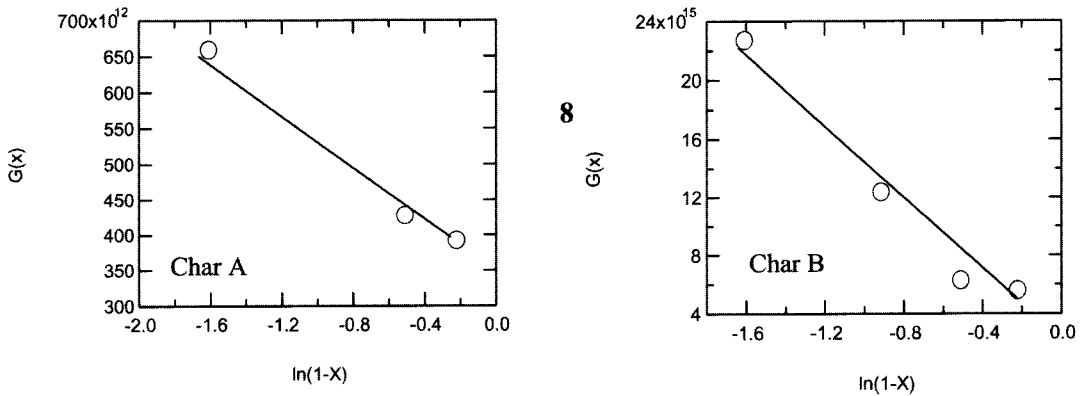
Char B: 
$$\frac{dX}{dt} = 70.1 \cdot 10^6 e^{-150400/RT} (1-X)[1 - 2.2 \ln(1-X)]^{1/2} \quad (12)$$



**Figure 5:** TPR data of char A and char B gasification in atmospheric air at heating rate 2, 5 and 10 °C/min by the Thermogravimetric Analysis (TGA).



**Figure 6:** The Arrhenius's plots of  $(dx/dt)/(1-X)$  vs.  $1/T$  for the gasification of char A and char B under the condition of atmospheric air by TGA.

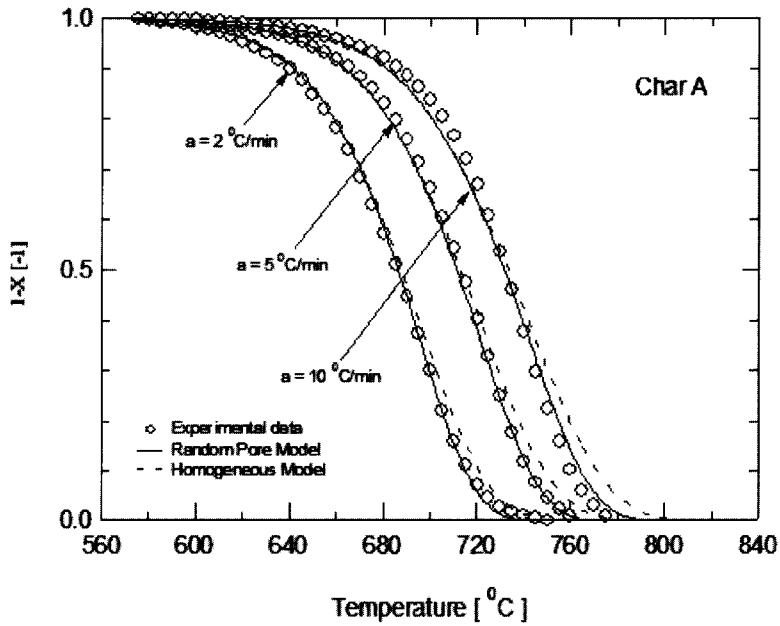


**Figure 7:** The relationship between  $G(X)$  and  $\ln(1-X)$  for char A and char B gasification with atmospheric air by TGA.

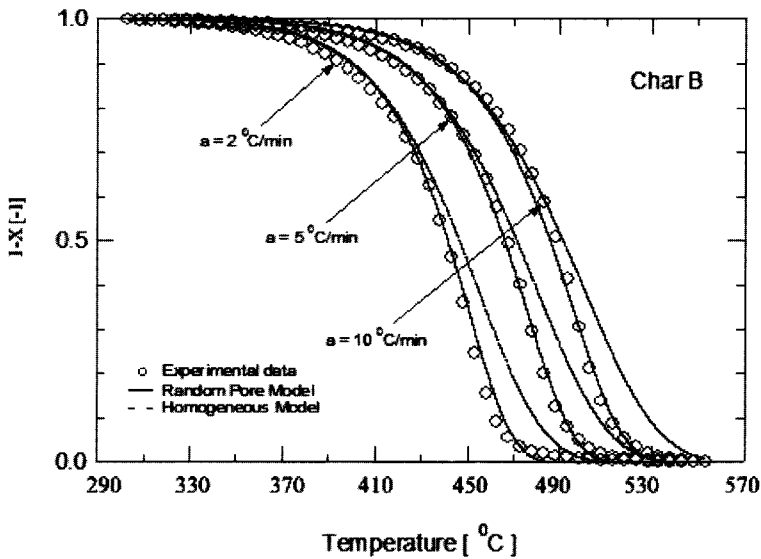
**Table 3:** Kinetic parameters of char A and char B gasification using air (gasifying agent) and TGA. Calculating by homogeneous model (HM) and random pore model (RPM).

Samples	Activation energy ( $E$ , kJ/mol)	Overall frequency factor ( $k_0$ , $s^{-1}$ )	Parameter of pore structure (-)	Error (%)
Char A [ RPM ]	136.5	$18 \times 10^6$	0.61	2.087
Char A [ HM ]	136.5	$1.12 \times 10^9$	-	2.978
Char B [ RPM ]	150.4	$70.1 \times 10^6$	2.20	1.520
Char B [ HM ]	150.4	$4.5 \times 10^9$	-	5.325





**Figure 8:** Comparison between experimental data and gasification model, RPM and HM, for char A gasification in atmospheric air at three heating rates by TGA.



**Figure 9:** Comparison between experimental data and gasification model, RPM and HM, for char B gasification in atmospheric air at three heating rates by TGA.

Figures 8 and 9 illustrate the comparison between experimental results and the results from calculation. It is found that the results obtained from the calculation fit very well with

the results obtained from experiments for both char samples. As a result of that, the behaviors of char gasification of the Thai lignite can be well represented by the definition of the random

pore model. By the random pore model, the reactivity of char gasification will be influenced by changes of pore structure during progress of reaction as represented by a parameter of pore structure,  $\Psi$ . The lower value of  $\Psi$  (more regular structure in sample) provides lower changing in pore structure during reaction progress. The standard errors of calculation are also shown in Table 3.

### 3.3 Comparison between the random pore model and the homogeneous model

In this study, the kinetic parameters were also investigated by employing the homogeneous model. The activation energies and the frequency factors of char gasification by using char A and char B are given in Table 3. The gasification rates of char A and char B, based on the homogeneous model, are presented in equation (13) and (14), respectively.

Char A:

$$\frac{dX}{dt} = 1.12 \cdot 10^9 e^{-136500/RT} (1-X) \quad (13)$$

Char B:

$$\frac{dX}{dt} = 4.50 \cdot 10^9 e^{-150400/RT} (1-X) \quad (14)$$

The values obtained from equation (13) and (14) are represented by the dashed lines in Figures 8 and 9. For char A at gasification heating rate 2 °C/min, it can be observed that, over a temperature range from 300 to 400 °C, the calculated values obtained from the homogeneous model correspond well to both of the experimental values and the calculated values obtained from the random pore model. After that, the curve of homogeneous model starts to deviate continuously from those obtained from experimental data and the random pore model until the completion of reaction at 550 °C. As can be seen in Figure 9, the reactivity of char B based on the homogeneous model also shows a similar behavior to the reactivity of char A. From the observation, it can be suggested that the behavior of char gasification at the early period of reaction can be represented by both the random pore model and the homogeneous model, whereas the gasification reactivity in the latter period of reaction is better represented by the random pore model. This is because the change in the pore

structure has more effect in this period of reaction. Moreover, it is also found that the differences in the reaction rate at  $X = 0$  of the random pore model and homogeneous model are 62 and 64 times different for char A and B, respectively. These different values correspond well with differences of their overall frequency factors.

Table 3 shows that the standard errors of calculated data based on the homogeneous model, are higher than those of data obtained from the random pore model at every condition. As the result, it can be confirmed that the random pore model can provide higher accuracy than the homogeneous model when considering the gasification reactivity of char A and char B gasification with air (from initial reaction to final reaction) under the period of chemical reaction controlled at temperatures lower than 1000 °C. In other words, the behavior of the gasification reactivity of char A and char B corresponds to the basic assumptions of the random pore model from initial reaction until final reaction.

## 4. Conclusions

As part of the effects of pyrolysis conditions on reactivity of char gasification, it can be suggested that the reactivity of char gasification increases with an increase in pyrolysis heating rates. This is due to the development of active surface area at higher pyrolysis heating rates. Also some kinds of inherent mineral matters acting as catalyst (Ca, Na, K, etc.) are found to lose their activities and decrease their dispersion at higher pyrolysis temperatures; these are due to the sintering and formation of crystal structures. Thus, the reactivity of char gasification is also found to decrease with increases in pyrolysis temperature.

For char A and char B at a gasification heating rate of 2 °C/min, it can be observed that the calculated values obtained from the homogeneous model correspond well to both of the experimental values and the calculated values obtained from the random pore model at only the early range of reaction temperatures. From this finding, it can be suggested that the behavior of char gasification at the early period of reaction can be represented by both the random pore model and the homogeneous model, whereas the gasification reactivity in the latter period of reaction is better represented by the

random pore model. This is due to the fact that the change in pore structure has more effect in the latter period of reaction.

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