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Research Article

EXPERIMENTAL STUDY ON THE WEIGHT LOSS DYNAMIC OF LONG FLAME COAL PYROLYSIS

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ABSTRACT

Long flame coal from the Shenfu-Dongsheng coal field in the Ordos Basin is pyrolyzed for weight loss experiments using HYLZ-2 cryogenic dry distillation furnace. The pyrolysis procedure is designed to contain the dry dewatering phase (20 to 245 °C), the transition phase of slight pyrolysis (245 to 460°C or other 485°C, and 510°C) both at 5°C/min constant heating rate, and the strong isothermal pyrolysis phase at those three temperatures to the end point. For the isothermal pyrolysis, the activation energy E =162 KJ/mol, and the pre-exponential factor ln A=20.333.For the constant heating rate pyrolysis, the activation energy E =27.86 KJ/mol, and the pre-exponential factor lnA=5.649.

Keywords: temperature time sampling roadmap; constant heating rate pyrolysis; isothermal pyrolysis; weight loss dynamics.

INTRODUCTION

Low-rank coal, including lignite and long flame coal, consists of the primary part of China's coal. With high moisture, high volatile fraction, and low thermal value of long flame coal gives rise to its modification in the macromolecular structure, thus affecting its direct applications [1-3]. Thus, the pyrolysis is used to improve the applicability of long flame coal [4-6]. The pyrolysis temperature of 300°C - 600°C is defined as low pyrolysis temperature. In this temperature range, the large molecular structure of long flame coal begins to disintegle and decompose, producing gas, tar, and semi-coke. The process and equipment of the semi-coke (blue carbon)production plant in northern Shaanxi Province of China are designed and operated based on low temperature pyrolysis. Because thermo-gravity analysis (TGA) techniques of isothermal pyrolysis and constant heating rate pyrolysis can be used to obtain a curve of mass change over time or/and temperature, they are an important means of studying the dynamics of low-rank coal pyrolysis [13-16]. These studies have revealed the effects of different particle size, end of temperature and heating rate, etc. The low rank coal tested are included ultra-finecoal, Dongshengcoal, mixed coal, Weizhoulong flame coal, Zhundongcoal, Xinjiang bituminous coal [7-12]. There is a study on the fixed bed reactor N_2 atmosphere, different pressure, and temperature of Tangshan bituminous coal. There is also the use of thermal gravity-infrared-mass spectrometry technology to study the pyrolysis properties of four different coal species in nitrogen atmosphere [13,14], and there are studies specifically comparing the single reaction model and distribution activity energy of coal pyrolysis [15,16]. Although TGA has the advantages of short operating cycles, accurate measurements, and a large amount of information obtained, the deficiency of solid intermediates between the initial sample and the termination sample is the only shortage. To this end, according to the process of semi-coke production plant in northern Shaanxi Province of China, a suitable reaction equipment and scientifically arranged temperature time sampling roadmap to prepare solids samples over time. The weight loss rate of those samples is used for dynamic study.

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EXPERIMENTAL DETAIL AND WEIGHT LOSS DATA

Laboratory instruments

HYLZ-2 cryogenic dry distillation furnace is selected as equipment with a standard stainless-steel retort.

Solid sampling

This experiment is designed to implement a temperature time sampling roadmap for 20 solid samples. The low ash, low sulfur, high volatile long flame coal from the Ordos Basin has been chosen as the coal sample. Two kinds of pyrolysis methods, constant heating rate pyrolysis and isothermal pyrolysis, have been used in the experimental operation. The temperature time sampling roadmap is consisted with three in turn phases and corresponding pyrolysis method:

The dry dewatering phase employed of constant heating rate pyrolysis from 20°C to 245°C at 5°C /minute heat rate.

The transition phase of slight pyrolysis employed of constant heating rate pyrolysis from 245°C to 460°C, or designed temperature of 485°C or 510°C, at 5°C /minute heat rate. The strong pyrolysis phase to the end point employed of isothermal pyrolysis at 460°C or designed temperature of 485°C or 510°C. During this phase, total 6 samples are collected at each isothermal temperature. They are collected at 6 different times, 0, 20, 60, 120, 200, and 320 minutes.

Weight loss data

The 3 kg of long flame coal produced in the Ordos Basin Shenfu-Dongsheng coal field is broken into a 1 mm sieve in a shredder, then baked in a 60°C oven for two hours, cooled in the air, put into a

plastic bag, then placed in a drying dish. At the beginning of each new experiment, 70.0 grams has been weighted and sealed into the standard stainless-steel retort as the starting sample, marked as #0. The 70.0 g starting sample is heated from 20°C to 245°C at 5°C /minute heat rate as a dry dehydration phase sample, marked as #00. The 70.0 g starting sample is subjected to a dry dehydration phase, then continue heating to the designed isothermal pyrolysis temperature as a slight pyration phase sample. The 1 before a dash is marked as the sample isothermal pyrolysis at 460°C. The 2 before a dash is marked as the sample isothermal pyrolysis at 485°C. The 3 before a dash is marked as the sample isothermal pyrolysis at 510°C.

DYNAMIC CALCULATIONS [17-20]

The reactions of gas-solid multi-phase chemical reactions are complex and are commonly used to represent the relationship between reaction rate and conversion rate:

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

Weight loss rate and conversion rate

Since the conversion rate is represented by the weight loss rate data, there is

$$\alpha = \frac{W_0 - W}{W_0 - W_f} \quad (2)$$

W_0: Sample original amount, 70.0 grams

W: Residual weight in standard stainless-steel retort for a pyrolysis experiment.

W_f: Final residual weight in standard stainless-steel retort for the maximum pyrolysis experiment, which is 48.9 grams with a maximum temperature of 510°C and a maximum temperature of 320 minutes.

Isothermal pyrolysis

When n is 1, the conversion rate is only related to the isothermal pyrolysis time as:

$$-ln(1-\alpha) = kt + C \quad (3)$$

According to equation(3), under isothermal pyrolysis condition, the -ln(1-a) is in a straight line with time t. The slope of the line is the velocity constant of isothermal pyrolysis k and the intercept is the integral constant C.

Arrhenius equation is an empirical relationship between the velocity constant of isothermal pyrolysis and temperatures as:

$$lnk_i = lnA - \frac{E}{RT_i} \quad (4)$$

According to equation(4), under isothermal pyrolysis condition, the velocity constant of isothermal pyrolysis is in a straight line with reciprocal of the temperature. The slope of the line can be used to solve the activation energy E and the intercept is the pre-exponential factor A.

Constant heating rate pyrolysis

Constant heating rate means that both temperature and time are variables, but temperature is rising at a constant rate, i.e.

$$\beta = \frac{dT}{dt} \quad (5)$$

When E being treated as a constant, Equation (1), (4), and (5) can be treated as:

$$\frac{d\alpha}{dT} = \frac{A(1-\alpha)^n}{\beta}\exp\left(-\frac{E}{RT}\right) \quad (6)$$

Set the initial conditions of a=0 when $T=T_0$,and get:

$$\int_0^\alpha (1-\alpha)^{-n} d\alpha = \frac{A}{\beta}\int_{T_0}^{T} exp\left(-\frac{E}{RT}\right) dT \quad (7)$$

According to Doyle approximate integrals and at n-1,

$$\ln[-\ln(1-\alpha)] = \ln\left(\frac{AE}{\beta R}\right) - 5.33 - \frac{E}{RT} \quad (8)$$

According to Equation 8, the ln[-ln(1-a)] vs the reciprocal of the temperature is a straight line. The slope and intercept of the line can be used to solve the activation energy E and the intercept is the pre-exponential factor A. To avoid the mathematic difficulty, let a=0.001 at the starting point (temperature T=20 °C, and time t=0 minute), and let a=0.999 at the final sampling point(temperature T=510 °C, and time t=320 minute).

Isothermal pyrolysis results

Table 1 listed 18 isothermal pyrolysis weight loss (WL) data and their conversion rate and calculations.

Table 1: 18 isothermal pyrolysis WL, conversion rate, and calculations

Item	WL/g	a	1-a	ln(1-a)
0#	0.01	0.00047	0.9995	-0.0005
00#	5.7	0.27	0.73	-0.3147
1-1	15.5	0.7346	0.2654	-1.3265
1-2	16	0.7583	0.2417	-1.42
1-3	17	0.8057	0.1943	-1.6383
1-4	17.6	0.8341	0.1659	-1.7965
1-5	18.1	0.8578	0.1422	-1.9507
1-6	18.6	0.8815	0.1185	-2.133
2-1	14.6	0.6924	0.3076	-1.179
2-2	16.3	0.7725	0.2275	-1.4807
2-3	17	0.8057	0.1943	-1.6383
2-4	18.4	0.872	0.128	-2.056
2-5	18.4	0.872	0.128	-2.056
2-6	18.8	0.891	0.109	-2.2164
3-1	15.4	0.7299	0.2701	-1.3088
3-2	17.2	0.8152	0.1848	-1.6883
3-3	18.4	0.872	0.128	-2.056
3-4	19.4	0.9194	0.0806	-2.5186
3-5	20.3	0.9621	0.0379	-3.2724
3-6	21.09	0.9953	0.0047	-5.3519

Based on Equation (3), the relationship between ln(1-a) and isothermal pyrolysis time t at three different temperatures are plotted in Figure 1.

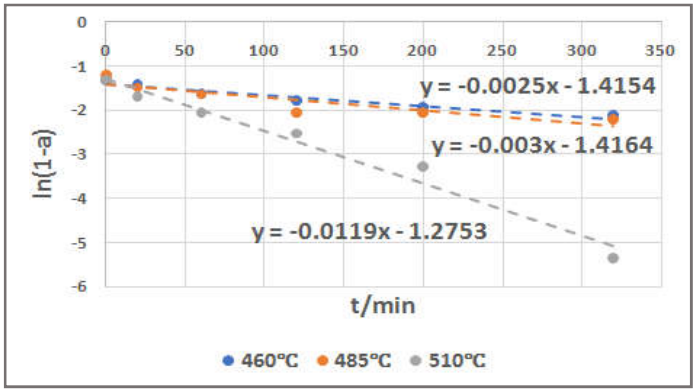

Figure 1: The relationship between ln(1-a) and isothermal pyrolysis time t at three different temperatures

Thus, the velocity constants k and integral constants C are obtained for three isothermal temperature, and listed in Table 2.

Table 2: The velocity constants k and integral constants Cat three isothermal temperatures

T/°C	k/min^{-1}	C
460	0.0025	-1.415
485	0.003	-1.315
510	0.0119	-1.275

Figure 2 is the plotted of the relationship between lnk and 1/T of the isothermal pyrolysis.

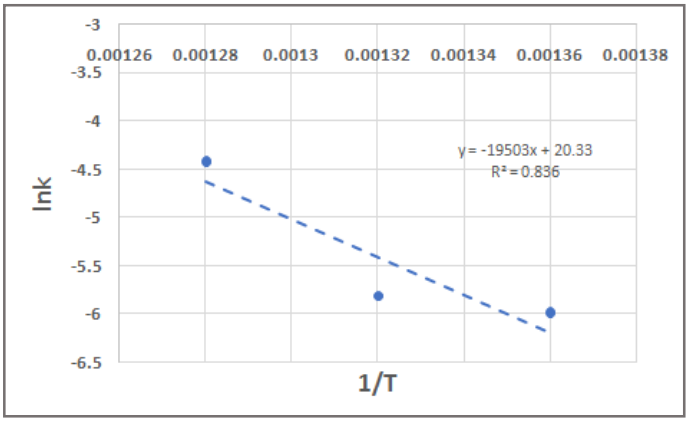

Figure 2: The relationship between lnk and 1/T of the isothermal pyrolysis

The slope of the line can be used to solve the activation energy E =162 KJ/mol, and the pre-exponential factor ln A=20.333.

Constant heating rate pyrolysis results

The constant heating rate, 5°C/min), pyrolysis involved the dry dewatering phase and the transition phase of slight pyrolysis. The relevant data of those two phases are listed in Table 3.

Table 3: The relevant data of those two constant heating rate pyrolysis

Item	1/T	ln(-ln(1-a))
0#	0.003413	-6.9073
00#	0.001931	-1.1555
1-1	0.001364	0.2825
2-1	0.001319	-0.1081
3-1	0.001277	0.2691

Based on the Equation (8), the relationship between ln(-ln(1-a)) and 1/T of the constant heating rate pyrolysis is plotted in Figure 3.

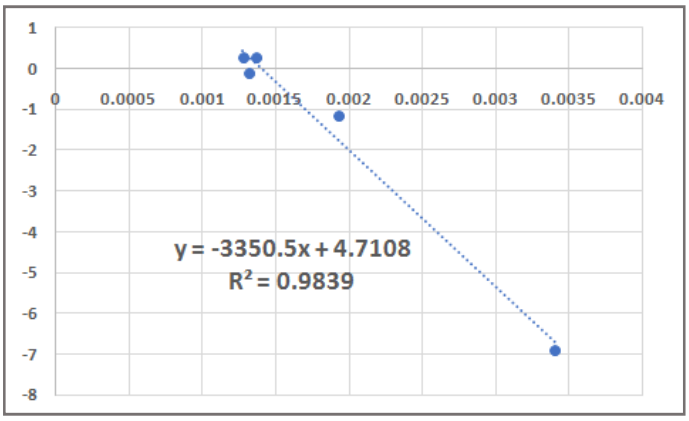

Figure 3: The relationship between ln(-ln(1-a)) and 1/T of the constant heating rate pyrolysis

The slope of the line can be used to solve the activation energy as:

$$-\frac{E}{R} = -3351 \quad (9)$$

Therefore, E =27.86 KJ/mol for constant heating rate pyrolysis.
The intercept can be presented as:

$$ln\left(\frac{AE}{\beta R}\right) - 5.33 = 4.71 \quad (10)$$

Put every constant into Equation (10), the pre-exponential factor ln A=5.649.

CONCLUSIONS

Long flame coal from the Shenfu-Dongsheng coal field in the Ordos Basin is pyrolyzed for weight loss experiments using HYLZ-2 cryogenic dry distillation furnace to collect the solid residual. The pyrolysis procedure is designed to contain the dry dewatering phase (20 to 245 °C), the transition phase of slight pyrolysis (245 to 460 °C or other 485 °C, and 510 °C) both at 5 °C/min constant heating rate, and the strong isothermal pyrolysis phase at those three temperatures to the end point. For the isothermal pyrolysis, the activation energy E =162 KJ/mol, and the pre-exponential factor ln A=20.333. For the constant heating rate pyrolysis, the activation energy E = 27.86 KJ/mol, and the pre-exponential factor ln A=5.649. These pyrolyzed solid residual is used to study the weight loss dynamics, can also be used to research other topics, such as volatilization dynamics, desulfurization dynamics.

Symbol description

A : pre-exponential factor, min-1
C : integrated constant
E : activation energy, KJ/mol
n : reaction order
R : gas constant, 8.314 J/(mol.K)
T : thermodynamic temperature, K
t : time, minute
α : conversion rate
β : constant heating rate, K/min

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