International Journal of Science and Research Development
Vol. 01, Issue 02, pp.039-042, May, 2022
Available online at http://www.scienceijsrd.com

ISSN: 2583-4231

Research Article

STUDY ON THE SAMPLING METHOD FOR ARTIFICIAL PYROLYSIS OF LOW-RANK COAL

[1]Xuemei Zhang, [1,*]Dong Li, [1]Qinghua Ma, [2]Xiaodong Zhao, [3]Yaling Wei and [4]Jingyuan Hao

[1]Energy and Chemical Industry Big Data Application Teaching and Research Center, Xi'an Siyuan University, Xi'an, Shaanxi, China
[2]Beijing Deqin Technology Development Co., Ltd., Beijing, China
[3]Ningxia Sanfang Coal Quality Testing Technology Co., Ltd., Yinchuan, Ningxia, China
[4]School of Chemical Engineering, Xi'an Jiaotong University, Xi'an, Shaanxi, China

Received 28th March 2022; **Accepted** 25th April 2022; **Published online** 30th May 2022

Abstract

Long flame coal from the Shenfu-Dongsheng coal field in the Ordos Basin is pyrolyzed with HYLZ-2 cryogenic dry distillation furnace. A unique temperature time sampling roadmap is designed to content the dry dewatering phase (20 to 245℃), the transition phase of slight pyrolysis (245 to 460℃) both at 5℃/min constant heating rate, and the strong pyrolysis phase (isothermal pyrolysis at 460℃) to the end point. Eight solid samples were analyzed for their characteristics (weight loss rate, volatile, sulfur, and random reflectivity R_{ran}, maximum reflectivity R_{max},). The results are listed in a table, shown in a graph, and semi-quantitatively analyzed. These solid sample scan also be used for other characteristic tests for thermal dynamics study.

Keywords: Stainless steel retort, Pyrolysis, Temperature time sampling roadmap, Characteristic analysis.

INTRODUCTION

Under isolated air conditions, low-rank coal is heated to a higher temperature and a series of physical changes and chemical reactions occur, and produces gas, tar, and semi-coke. This process is called artificial pyrolysis and is the initial step during the main processes of low-rank coal utilization including gasification, liquification, coking and combustion (Zhai Wei *et al.,* 2014; Mi Zhiping and Wang Ningbo, 2010; Wan Xiangming *et al.,* 2015). Good understanding the mechanism of pyrolysis is essential for controlling the releasing rate of gas and tar and enhance the conversion of solid residue, char. Some scholars have used thermal gravity analysis (TGA or DTG) to detect gas or tar composition and content over time, temperature change dynamics throughout the pyrolysis process of low-rank coal (Li Wenjun *et al.,* 2020; Zheng Mingdong *et al.,* 2006; Lu Tai *et al.,* 2005; Guo Longfei *et al.,* 2018). Also, scholars have studied changes in gas products using differential thermal infrared spectroscopy (TGA-FTIR) and differential thermal mass spectroscopy (TGA-MS) and established a coal pyrolysis model (FG-DVC)for the correlation between coal structure and reaction (Solomon *et al.,* 1992; Wu *et al.,* 2014). Since there are three products in any artificial pyrolysis process, gas, tar, and char, there has not been any method of solid sampling regarding the intermediate char products of the artificial pyrolysis. The classical chemical dynamics study method is to sample from a large reactor at different times, then analyze how the reaction concentration of these series of samples, and to calculates the reaction dynamics parameters, reaction rate constants, active energy, and pre-factors, over time. Compared with traditional method, the current thermal weight analysis method supplemented by infrared, mass spectroscopy, etc. to study the thermal dynamics of low-rank coal, has several shortcomings, such only two solid samples of the reaction before the experiment and the product after the completion of the experiment, while there are no other solid samples in the reaction process. In view of the above analysis, this paper would like to explore how to use suitable reaction equipment and scientifically arranged temperature time sampling to prepare a series of reaction solids samples over time.

Corresponding Author:** ***Dong Li,
Energy and Chemical Industry Big Data Application Teaching and Research Center, Xi'an Siyuan University, Xi'an, Shaanxi, China.

EXPERIMENTAL DETAILS

Laboratory instruments

To meet the requirements of maintaining residual solid samples in the reactor and collecting exhausted gas liquid samples, HYLZ-2 cryogenic dry distillation furnace is selected as equipment with aluminum retort been replaced by standard stainless-steel retort. HYLZ-2 cryogenic dry distillation furnace consists of three parts: heating devices, including thermocouples, thermocouple casings, heating devices, millivolt thermometers, and cold contact thermostats; stainless steel retort, including caps, bodies, export tubes, and temperature measuring tanks; and gas-liquid collection device, including a catheter, a conical bottle, and a cooling tank. This set of laboratory instruments has the advantages of equipment sculpting and easy operation, as well as national standards such as seal up inspection standards and temperature precise control requirements. Due to the limited volume of standard stainless-steel retort, so it is not possible to take multiple samples from the stainless-steel retort for one pyrolysis, instead only one solid sample could be collected for one pyrolysis. There's a need to creatively design a temperature time sampling roadmap to solve the challenge.

Temperature time sampling roadmap

Based on the scientifical definition, one reaction will be one same reaction if starting with the same reactant, going through the exact same reaction conditions (temperature, pressure, and time), and reaching the end point. From the starting point to the end point, several intermediate points were selected as the

imaginary "sampling points" to stop the pyrolysis and collected the solid char from the standard stainless-steel retort as the samples for kinetic study. According to the industry scale semi-coke (blue carbon) production plant in northern Shaanxi Province, there are only two kinds of methods of pyrolysis: constant heating rate pyrolysis and isothermal pyrolysis. The temperature time sampling roadmap is designed to content three phases: the dry dewatering phase, the transition phase of slight pyrolysis, and the strong pyrolysis phase to the end point. During the dry dewatering phase and the transition phase of slight pyrolysis, the constant heating rate pyrolysis have been employed. During the strong pyrolysis phase to the end point, the isothermal pyrolysis is employed.

EXPERIMENTAL OPERATIONS

Coal sample

To meet the design requirements of the exact same starting reactant, the low ash, low sulfur, high volatile long flame coal from the Ordos Basin has been chosen as the coal sample. It is broken into 1 mm over sieve in the shredder, and then baked in an oven at 60℃ for two hours, then cooled in the air, store into plastic bags and placed in a drying dish. It is remarked as #0 sample.

Dry dewatering phase

Take 70 grams from the dry dish and placed in the standard stainless-steel retort and heated HYLZ-2 cryogenic dry distillation furnace for 45minutes at a constant rate of 5℃/minute from 20℃ to 245℃. Define it as a #00 sample.

Transition phase of slight pyrolysis

In this phase, coal has entered the transition stage of desorption gas, softening, melting, slight pyrolysis. Heat HYLZ-2 cryogenic dry distillation furnace for another 43 minutes at a constant rate of 5℃/minute from 245℃ to 460℃. Define it as a 1-1 sample.

Strong pyrolysis phase to the end point

At 460℃ isothermal pyrolysis, not only the small molecular side chain, but also the large molecule side chain is broken. 1 before the dash indicates the strong pyrolysis isothermal temperature is 460℃, and the number after the dash indicates the pyrolysis time is counting. 1 means the isothermal heating time is zero minutes. 2 means the isothermal heating time is 20 minutes, and so on. This experiment prepared a total of 6 samples at 460℃. Table 1 listed the 6 isothermal pyrolysis sample heating time at 460℃.

Table 1. The heating time of 6 samplesat 460℃

Sample	1-1	1-2	1-3	1-4	1-5	1-6
Time/min	0	20	60	120	200	320

RESULTS AND DISCUSSION

To study the pyrolysis thermodynamics, temperature time sampling diagrams are used to ensure that a series of reaction solids samples change over time for the same reaction, as shown by eight experiments from room temperature to 460℃. Two categories of analysis were done for each of the eight solid samples. One is the analysis of weight loss rate, volatile, and sulfur. Another is the coal rock analysis.

Performance Analysis

The results of the performance analysis are listed in Table 2. The weight loss rate (WLR) is defined as:

$$WLR = \frac{present\ mass - start\ mass}{start\ mass} 100\% \quad (1)$$

The performance analysis of volatile and sulfur are tested according to national standard. The values taken by the sample are arithmetic averages.

Table 2. Performance analysis of volatile, sulfur, and WLR

Sample	Sulfur /%	Volatile /%	WLR /%
#0	0.1645	30.35	0.00
#00	0.1435	29.95	8.14
1-1	0.1365	22.25	22.14
1-2	0.1360	21.76	22.86
1-3	0.1335	19.53	24.29
1-4	0.1335	20.12	25.14
1-5	0.1345	18.80	25.86
1-6	0.1350	18.57	26.57

The measured WLR, volatile, and sulfur vs time are separately presented in Figure 1 to Figure 3. In addition to the horizontal coordinates for time and ordinates for the measured data, the starting temperature is room temperature, represented at the time equals zero. Furthermore, two vertical dashed lines are added to represent temperature. The first dotted line, which intersects the horizontal coordinates for a temperature of 245℃. The second dotted line, which intersects the horizontal coordinates for a temperature of 460℃. The temperature between the starting point to the first dotted line is increased from 20℃ to 245℃ represents dry dewatering phase. The temperature between the first dotted line to the second dotted line is increased from 245℃ to 460℃ represents the transition phase of slight pyrolysis.

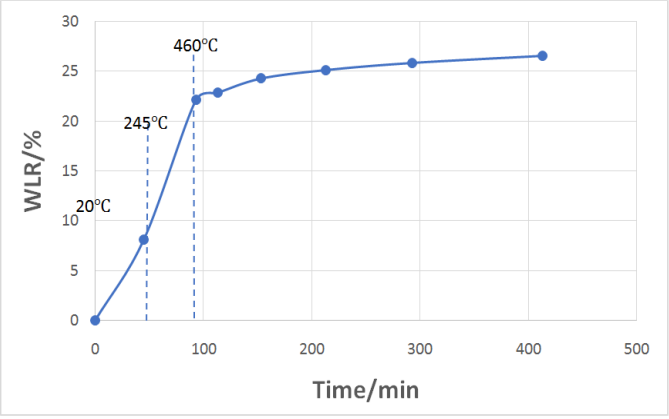

Figure 1. WLR vs time

Figure 1 shows how the WLR changes over time. From Figure 1, the maximum WLR occurs in the transition phase of slight pyrolysis, while the minimum WLR occurs in the strong pyrolysis phase to the end point.

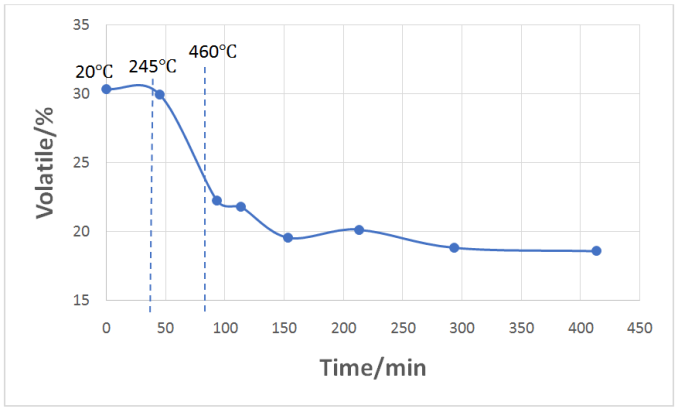

Figure 2. Volatile vs time

Figure 2 Shows that volatility decreases over time as the temperature increases. From Figure 2, the maximum reduction rate of volatile occurs in the transition phase of slight pyrolysis. During the strong pyrolysis phase to the end point, the rate of volatilization decrease is smaller than the previous phase.

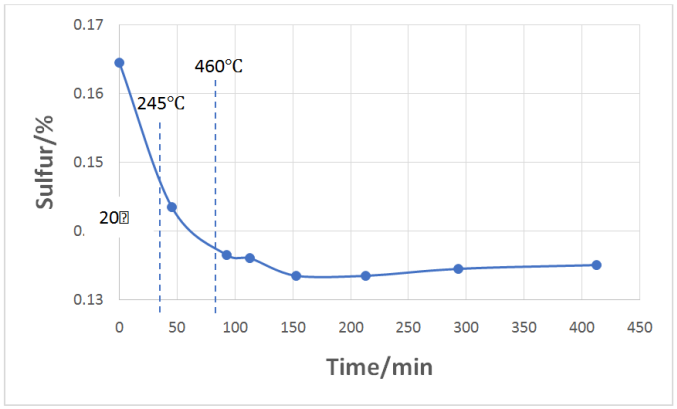

Figure 3. Sulfur vs time

Figure 3 shows the decrease in sulfur as the temperature increases over time. As can be seen from Figure 3, the maximum reduction in sulfur occurs during the dry dewatering phase. The sulfur reduction rate is also significant during the transition phase of slight pyrolysis. In the stage of strong pyrolysis phase to the end point, reduction in sulfur is not obvious.

Coal Rock Analysis

TheMSP-9000C coal rock instrument has been used to analyze each of the 8samples according to national standards (Liu *et al.,* 2019; Zhai Zhenyong *et al.,* 2018 & 2019; Liang Dingcheng *et al.,* 2016). Table 3 lists the random reflectance R_{ran}, and the maximum reflectance R_{max} of 8 samples.

Table 3. Coal rock analysis

Sample	R_{ran}	R_{max}
#0	0.541	0.576
#00	0.595	0.633
1-1	1.382	1.471
1-2	1.35	1.437
1-3	1.422	1.514
1-4	1.562	1.663
1-5	1.568	1.669
1-6	1.625	1.73

The measured the random reflectance R_{ran} and the maximum reflectance R_{max} vs time are separately presented in Figure 4and Figure 5.

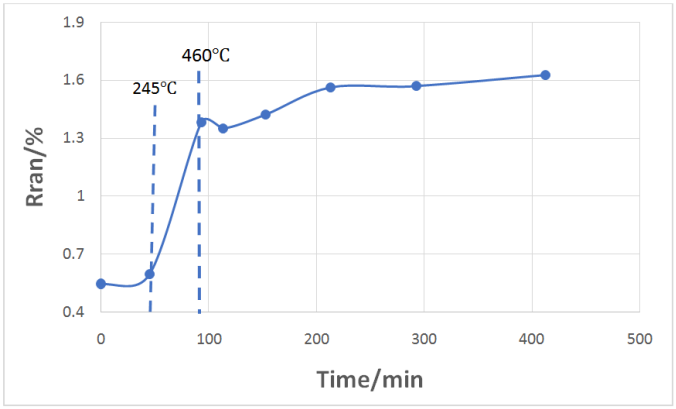

Figure 4. Random reflectance R_{ran} varies over time

The average random reflectance is increased 1.084 during the pyrolysis from 0.541 to 1.625.

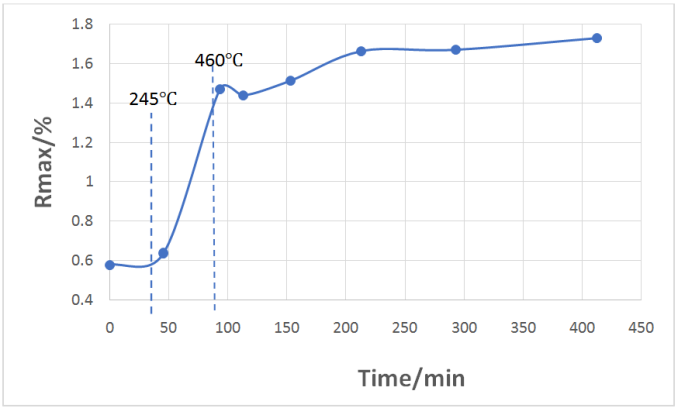

Figure 5. Maximum reflectance R_{max} varies over time

The average value of the maximum reflectance, R_{max}, is increased 1.154 during the pyrolysis from 0.576 to 1.73.

Conclusion

1. The selection of HYLZ-2 cryogenic dry distillation furnace and stainless-steel retort has the advantages of easy operation, air seal up inspection standards and temperature precise control requirements and other national standards. It is suitable to prepare a series solid sample.
2. For the special requirement that the entire process of stainless-steelretort, a unique temperature time sampling roadmap can be designed to content three phases: the dry dewatering phase, the transition phase of slight pyrolysis, and the strong pyrolysis phase to the end point. During the dry dewatering phase and the transition phase of slight pyrolysis, the constant heating rate of 5℃/min pyrolysis have been employed. During the strong pyrolysis phase to the end point, the isothermal pyrolysis at 460℃ is employed.
3. Eight solid samples were analyzed for characteristic (weight loss rate, volatile, and sulfur) and coal rock (random reflectance R_{ran} and maximum reflectance R_{max}) according to national standards. The results are analyzed semi-quantitatively using data sheet and graphs respectively.

4. The results of coal rock analysis confirm that the low-order coal under the action of thermal energy, starting from the removal of the small molecule side chain in the coal structure, gradually to the large molecule side chain began to break and remove (i.e., the active parts gradually decreased) of the quality process.

REFERENCES

GB/T 480-2000, aluminum screening low temperature dry distillation test method for coal.

Guo Longfei, left, Zhao Jun, etc. Study on the behavior and dynamics of super-fine coal thermolytics, *Clean Coal Technology,* 2018, 24 (22):40-45.

Li Wenjun, Chen Shanshan, Chen Yanpeng, etc. Study on thermally heavy coal thermolytic reaction dynamics. *China Coal,* 2020, 46 (3): 84-89.

Liang Dingcheng, Xie Qiang, Party Potash Tao, etc. Study on the microstructure and deformation of the temperature and thermal solution semi-coke in different coal steps. *Journal of China University of Mining,* 2016, 45(4):799-806.

Liu Penghua, Wang Shaoqing, Guo Qiang, etc. Different coal grade homogeneity mirror body AFM study, *Coal Technology,* 2019,38(12):88-91.

Lu Tai, Zhang Cuizhen, Wu Chao. Study on the effects of particle size and heating rate on coal thermal decomposition. *Coal Conversion,* 2005, 28(1):17-20

Mi Zhiping, Wang Ningbo. The current situation and development trend of coal cryogenic dry distillation technology, *Clean Coaltechnology*, 2010, 16(2):33-37.

Solomon PR, Serio MA, Suuberg EM. Coal pyrolysis: experiments, kinetic rates and mechanisms. *Prog Energy Combust Sci.,* 1992,18:133–220

Wan xiangming, Yan Guodong, Wang Yubin. Low-order coal low-temperature dry distillation technology development analysis, *Drying Technologyand Equipment,* 2015,13(2):1-6

Wu D, Liu G, Sun R. Investigation on structural and thermodynamic characteristics of per hydrous bituminous coal by Fourier transform infrared spectroscopy and thermogravimetry/mass spectrometry. *Energy Fuels,* 2014,28:3024–35.

Zhai Wei, Zhang Hongli, Wang Rong, etc. Study on the low-temperature dry distillation process of hypothermia coal in Shenfu. *Coal processing and comprehensive utilization,* 2014,(10):78-80.

Zhai Zhenyong, Xu Hao, Tang Dayong, etc. Study on the change law of pore structure in the process of thermal solution of coal in different coal steps, *Coal Science and Technology,* 2019, 47 (9): 74-79.

Zhang Zhigang. Study on the law of escape of coal-like pyrogenic gas products of different degrees of desal tension, *Coal Technology,* 2018, 37(7):293-295.

Zheng Mingdong, Bai Dayong. Dongsheng coal non-isothermal thermolytic properties and dynamic parameters aredetermined. *Coal Chemical,* 2006, 34 (2): 13-16.

****