

Article

Moderate-Temperature Pyrolysis Characteristics of Lump Coal Under Varying Coal Particle Sizes

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Abstract: Pyrolysis is an important methodology for achieving efficient and clean utilization of coal. Lump coal pyrolysis demonstrates distinct advantages over pulverized coal processing, particularly in enhanced gas yield and superior coke quality. As a critical parameter in lump coal pyrolysis, particle size significantly influences heat transfer and mass transfer during pyrolysis, yet its governing mechanisms remain insufficiently explored. This research systematically investigates pyrolysis characteristics of the low-rank coal from Ordos, Inner Mongolia, across graded particle sizes (2–5 mm, 5–10 mm, 10–20 mm, and 20–30 mm) through pyrolysis experiments. Real-time central temperature monitoring of coal bed coupled with advanced characterization techniques—including X-ray diffraction (XRD), Raman spectroscopy, Brunauer–Emmett–Teller (BET) analysis, scanning electron microscopy (SEM), gas chromatography (GC), and GC–mass spectrometry (GC-MS)—reveals particle-size-dependent pyrolysis mechanisms. Key findings demonstrate that the larger particles enhance bed-scale convective heat transfer, accelerating temperature propagation from reactor walls to the coal center. However, excessive sizes cause significant intra-particle thermal gradients, impeding core pyrolysis. The 10–20 mm group emerges as optimal—balancing these effects to achieve uniform thermal attainment, evidenced by 20.99 vol% peak hydrogen yield and maximum char graphitization. Tar yield first demonstrates a tendency to rise and then decline, peaking at 14.66 wt.% for 5–10 mm particles. This behavior reflects competing mechanisms: enlarging particle size can improve bed permeability (reducing tar residence time and secondary reactions), but it can also inhibit volatile release and intensify thermal cracking of tar in oversized coal blocks. The BET analysis result reveals elevated specific surface area and pore volume with increasing particle size, except for the 10–20 mm group, showing abrupt porosity reduction—attributed to pore collapse caused by intense polycondensation reactions. Contrasting previous studies predominantly focused on less than 2 mm pulverized coal, this research selects large-size (from 2 mm to 30 mm) lump coal to clarify the effect of particle size on coal pyrolysis, providing critical guidance for industrial-scale lump coal pyrolysis optimization.



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Keywords: low-rank coal pyrolysis; wide particle size; lump coal

1. Introduction

According to the Statistical Review of World Energy 2024, published by the Energy Institute, coal represents 26.5% of global energy consumption, making it the second-largest

source after oil, which accounts for 31.6%. Crucially, global proven coal reserves stand at approximately 1.07 trillion tons, significantly exceeding those of other fossil fuels—oil reserves are estimated at around 244 billion tons and natural gas at about 188 trillion cubic meters (equivalent to roughly 170 billion tons of oil equivalent). This abundance translates into a reserve-to-production (R/P) ratio of over 130 years for coal, far outstripping the roughly 50 years estimated for both oil and natural gas [1]. As one of the most consumed fossil fuels globally, and underpinned by its vast and long-lasting proven reserves, the utilization of coal frequently brings issues such as low energy efficiency and environmental pollution [2–4].

Promoting clean coal technologies is critical for enhancing energy efficiency and mitigating carbon emissions [5–8]. Conventional thermochemical processes such as coking (operating at 1000–1100 °C) and gasification (1000–1600 °C) enable coal conversion to metallurgical coke and syngas, respectively, yet face inherent limitations: coking requires specific caking coals and generates significant polycyclic aromatic hydrocarbons (PAHs), while gasification incurs substantial energy penalties due to extreme operational conditions and complex gas cleaning requirements [9–12]. In contrast, coal pyrolysis (500–900 °C) emerges as a versatile alternative, capable of thermally decomposing a wider range of coal ranks into three main products: high-calorific value gas, high-value-added tar, and high-quality char [13–17]. Critically, these outputs enable cascade utilization of coal resources: char serves as clean solid fuel for power generation or gasification feedstock; tar fractions refine into chemical precursors like benzene, toluene, and phenolic resins; while pyrolysis gas for fuels industrial heating or hydrogen production [18–20]. By simultaneously addressing aspects of resource utilization efficiency, product flexibility, and emission control, pyrolysis establishes itself as a promising pathway for fossil fuel utilization.

The characteristics of coal pyrolysis usually rely on various factors, including coal type, temperature, particle size, atmosphere, and reactor design [21–25]. Among them, particle size is highlighted as a critical factor, which often affects heat transfer, mass transfer, and other related characteristics. It holds the key to the design and industrialization of coal pyrolysis reactors. At present, many researchers have conducted extensive research on the effect of particle size on coal pyrolysis. Chen et al. [26] found that the pyrolysis reactivity of Shenmu coal lessened continuously with the lessening of the particle size in the range of 0–150 µm, and the release amount of CH₄, aliphatic hydrocarbons, and light aromatic hydrocarbons gradually reduced. Zhu et al. [27] reported that the appearance time of volatiles was delayed with the increase in particle size. A mechanism combining intra-particle and inter-particle mechanisms was proposed to explain the evolution process of most organic volatiles. Zhao et al. [28] displayed that within the particle size range of 0–180 µm, coal with smaller particle sizes has a higher pyrite content, which adversely affects coke quality. Tian et al. [29] found in the pyrolysis of Shandong bituminous coal in the particle size range of 0–1400 µm that the larger the particle size, the higher the mass loss and ash content, indicating its high pyrolysis reaction activity. Wu et al. [30] demonstrated that with the increase in the particle size of raw coal, the volatile removal process is limited by mass transfer and forms dispersed pores, which leads to an increase in the specific surface area and pore volume of the coke and the degree of order.

At present, mainstream research primarily targets pulverized coal, where the particle size of raw coal is usually between 0 and 2000 µm [26–33]. However, research on lump coal with large particle sizes is relatively scarce. In large-scale lump coal pyrolysis, the effect of particle size on the mass and heat transfer in the pyrolysis reaction is not clear. Additionally, the coal pyrolysis technology has specific advantages such as reduced dust content in volatiles and superior coal quality, which make it a promising approach among coal pyrolysis technologies. Therefore, in this research, we focus on the characteristics

of lump coal pyrolysis at various particle sizes. By monitoring the temperature rise in the center of the coal bed and the change in the pyrolysis gas production rate, the role of particle size in the heat transfer of the coal bed is methodically examined. In addition, the effect of particle size on the production and conversion of pyrolysis products is revealed by analyzing the pyrolysis products, including gas and tar. A series of analytical experiments (including ultimate analysis, Raman spectrum, XRD, BET, and SEM) on char are also implemented to demonstrate the effect of particle size on the pyrolysis performance of lump coal. This research is essentially aimed at providing a fairly solid theoretical basis for the industrial application of lump coal pyrolysis technology.

2. Experiment

2.1. Coal Samples Preparation

The workflow of this research is shown in Figure 1. In this research, subbituminous coal from Inner Mongolia, China, was utilized as feedstock. The proximate analysis and ultimate analysis results of the coal are presented in Table 1. The crushed raw coal is divided into the particle sizes required for testing through standard sieves with corresponding sizes, including 2~5 mm, 5~10 mm, 10~20 mm, and 20~30 mm. Different particle sizes of the coal mass are dried in an oven at 105 °C for 12 h and then cooled to room temperature in a drying oven before testing.

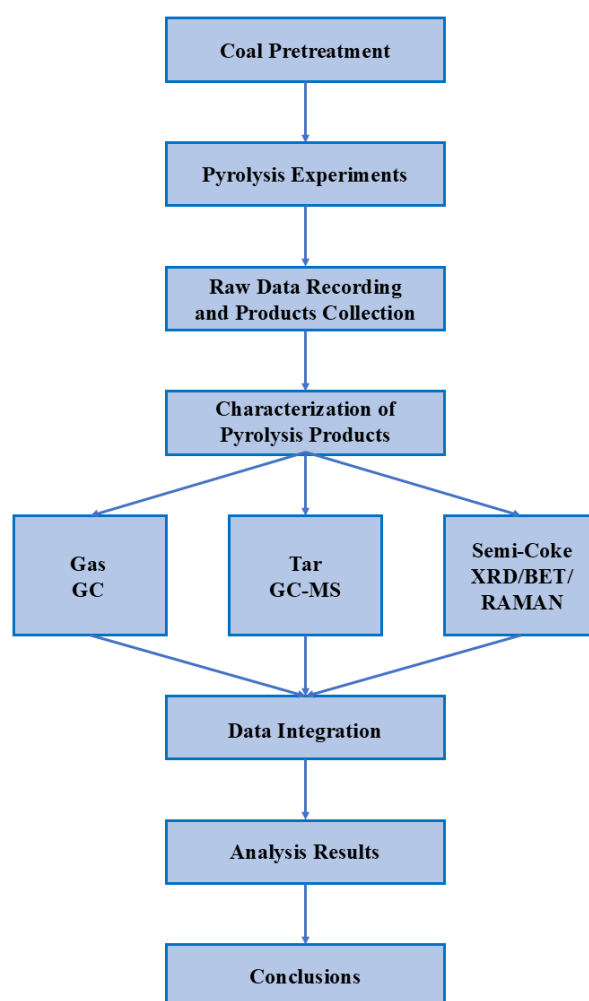


Figure 1. Workflow diagram integrating experimental procedures and results analysis.

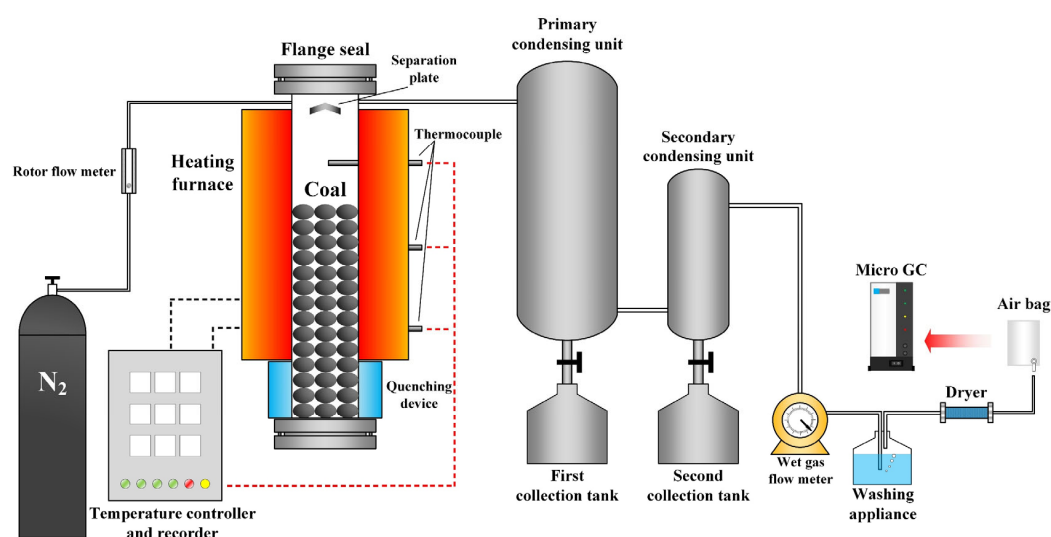
Table 1. Proximate and ultimate analysis of coal.

Proximate Analysis wt. %					Ultimate Analysis wt. %			
M_{ad}	A_d	V_d	FC_d^*	C_d	H_d	N_d	S_d	O_d^*
9.64	8.34	32.32	59.34	72.19	3.74	0.86	0.14	14.72

* By difference. Subscript ad denotes air-dried basis (used in M_{ad} for moisture content); subscript d indicates dry basis (A_d , ash yield; V_d , volatile matter; FC_d , fixed carbon).

2.2. Pyrolysis Experiment

Figure 2 illustrates the 30 kW pyrolysis experimental apparatus used in this study. The system consists of a feeding unit, a pyrolysis reaction unit, a condensation unit for tar capture, a tail gas treatment unit, and a temperature controller. The reactor dimensions are as follows: length 200 mm, width 120 mm, and height 800 mm. Thermocouples are placed at heights of 200 mm, 400 mm, and 600 mm inside the reactor to monitor the temperature distribution. It is also worth mentioning that the central position of the coal bed in this experiment is at a height of 400 mm. Therefore, the temperature changes inside the coal bed can be observed during the experimental process.

**Figure 2.** Schematic diagram of the 30 kW coal pyrolysis device.

Before starting the experiment, the flange seal is opened directly above the reactor, and the dried raw coal is added to the reactor. The separation plate near the reactor inlet allows the coal to be evenly and smoothly stacked inside the reactor. After the feeding is completed, the flange is closed and high-purity nitrogen is introduced into the reactor for a while to achieve an inert atmosphere. Before starting the heating process, the initial temperature cycle data should be recorded. Excessively high pyrolysis temperatures can lead to rapid heating of the coal bed, which complicates the observation of the influence of particle size changes on heat transfer within the coal bed. Conversely, excessively low pyrolysis temperatures would prolong the experiment duration undesirably. Taking these factors into account, the final pyrolysis temperature was set at 600 °C. The heating rate was maintained at 10 °C/min, with each experimental run lasting 200 min. After completing all the preparation steps, the heating switch is activated, and a programmed process of increasing the temperature in the reactor is started. After that, record the temperature data and wet flowmeter readings every 5 min, while also collecting the pyrolysis gas that has been purified. After observing that there is no bubble formation in the container containing the absorbing solvent, the pyrolysis reaction is considered to be complete. Then, we terminate the heating and wait for the natural cooling inside the reactor. Finally, we

remove the bottom baffle of the reactor and release the char resulting from pyrolysis. The tar–water mixture in the collection tank is stored in glass containers and separated by azeotropic distillation with acetone. The pyrolysis products were characterized accordingly, and their yields were also recorded. The coal pyrolysis experiments of various particle sizes were carried out on the basis of the above experimental design.

2.3. Calculations of the Pyrolysis Product Yield

The coke produced in the experiment is collected in an iron drum, and its actual mass can be evaluated by weighing. For calculating the yield of char, we can employ the following formula:

$$Y_s = M_s / M \quad (1)$$

where M_s represents the charred mass, and M denotes the mass of raw coal, which is controlled as 1 kg in this research.

By recording the difference in wet flow meter readings over the time interval between two records, the gas production rate for that short period can be obtained. Analysis of the composition of the gas samples during this period can provide the exact composition of the pyrolysis gas. With these two types of data, an approximate pyrolysis gas yield can be obtained for each period, and by accumulating these data, the total pyrolysis gas yield can be confirmed. To this end, one can write the following:

$$M_g = M_{g1} + M_{g2} + \dots + M_{gn-1} + M_{gn} \quad (2)$$

$$M_{gn} = V_{gn} \times \varphi_{ni} \times m_i \quad (3)$$

$$Y_g = M_g / M \quad (4)$$

where M_g denotes the total mass of pyrolysis gas, M_{gn} represents the gas mass of a period time during pyrolysis, V_{gn} is the gas volume of that period, φ_{ni} is the volume fraction of pyrolysis gas component, and m_i denotes the corresponding molar mass for gas components.

After separating the liquid pyrolysis products, the mass of purified tar can be measured by weighing, and the water yield can then be evaluated by difference. To this end, we follow the following specific calculations:

$$Y_t = M_t / M \quad (5)$$

$$Y_w = 1 - Y_s - Y_g - Y_t, \quad (6)$$

where M_t denotes the mass of tar, Y_t represents the yield of tar, and Y_w is the yield of water.

2.4. Pyrolysis Gas Analysis

The pyrolysis gas is analyzed via an Agilent 490 Micro gas chromatography (Agilent, Santa Clara, CA, USA). Multiple sets of sample gases were employed to calibrate the linear standard curve, which ensured the accuracy of gas analysis. There are two channels inside the device to measure different components, and the gas components are mainly H_2 , CH_4 , CO , and CO_2 , which are of great interest in this research. The exact composition of pyrolysis gas can also be obtained through the external standard method with the previously calibrated standard curve.

2.5. Tar Analysis

Shimadzu GCMS-TQ8040 (Shimadzu Corporation, Kyoto, Japan) was utilized for tar analysis. An HP-5 ms ultra-inert chromatography column (30 m $\times$ 250 μ m $\times$ 0.25 μ m) was adopted to evaluate the tar products. Before injecting the tar sample, the original tar

product was diluted with acetone at a dilution ratio of 1:10. In addition, impurities were filtered through a filter membrane whose pore size was 0.45 μm . The heating procedure for the chromatographic column can be taken as follows: the initial column temperature was set at 55 $^{\circ}\text{C}$, maintained for 2 min, increased to 90 $^{\circ}\text{C}$ at a heating rate of 4 $^{\circ}\text{C}/\text{min}$ and maintained for 1 min, then increased to 170 $^{\circ}\text{C}$ at a heating rate of 3 $^{\circ}\text{C}/\text{min}$ and maintained for 1 min, and finally increased to 280 $^{\circ}\text{C}$ at the heating rate of 3 $^{\circ}\text{C}/\text{min}$ and maintained for 5 min, and the split ratio was set equal to 1:50. The NIST2008 standard [34] spectral library was utilized for computer retrieval qualitatively, and the peak area normalization approach was employed to evaluate the relative content of each component quantitatively.

2.6. Char Analysis

Thermo Scientific FlashSmart (Thermo Fisher Scientific, Waltham, MA, USA) was implemented for the ultimate analysis of char. By utilizing this device, the relationship between particle size and char element content can be accurately mastered.

The Micromeritics ASAP2460 aperture analyzer (Micromeritics, Norcross, GA, USA) was also implemented to analyze the surface area and pore size distribution of char. It should also be highlighted that N_2 is the adsorbate, and samples should be degassed at 300 $^{\circ}\text{C}$ for 6 h before the test.

Microcrystalline structural parameters of char were appropriately measured by an X-ray diffractometer (Bruker D8 advance, Bruker Corporation, Billerica, MA, USA). The $\text{Cu-K}\alpha$ radiation ($\lambda = 0.15406 \text{ nm}$, 40 kV, 40 mA) was utilized as the X-ray source, the scanning range was taken as 10° to 80° , and the scanning rate was set as $8^{\circ}/\text{min}$. In addition, Bragg's equation and Scherrer's formula [35] were employed to obtain microcrystalline structural parameters including interlayer spacing (d_{002}), stacking height of layers (L_c), and size of aromatic layers (L_a). The calculation formula is as follows:

$$d_{002} = \lambda / 2\sin(\theta_{002}) \quad (7)$$

$$L_a = k_1 \lambda / \beta_{100} \cos(\theta_{100}) \quad (8)$$

$$L_c = k_2 \lambda / \beta_{002} \cos(\theta_{002}) \quad (9)$$

where λ represents the wavelength of the X-ray, θ denotes the diffraction angle, k is the Scherrer parameter ($k_1 = 1.84$, $k_2 = 0.90$), and β signifies the angular width of the diffraction peak at half maximum intensity.

The Raman spectra of the char were then recorded by means of the Raman spectrometer (HORIBA Ltd., Kyoto, Japan). The excitation wavelength was set as 532 nm, and the Raman shift range was 800–2000 cm^{-1} . The spectra acquisition time was set as 30 s, whereas the measurement of every sample was conducted at least three times to achieve higher accuracy.

The morphology of chars was characterized by the scanning electron microscope (Zeiss EVO, Carl Zeiss AG, Oberkochen, Germany). The extra high tension and the magnification were set at 5 kV and 500 $\times$, respectively.

3. Results and Discussion

3.1. Heat Transfer Performance of Coal Bed

The heating rate in the center of the coal bed generally reflects the effect of particle size on the heat transfer performance. Figure 3 presents the bed heating rate of varying coal particle sizes at 600 $^{\circ}\text{C}$. It can be observed that the larger coal particle size exhibits a faster heat transfer rate. In the fixed-bed reactor, the increase in the coal particle size is commonly accompanied by more coal seam porosity. The rise in the porosity in the coal bed facilitates the circulation of gaseous products, thereby enhancing convective heat

transfer [36]. Due to the low thermal conductivity of coal itself, the increase in convective heat transfer substantially accelerates the heating rate of the coal bed.

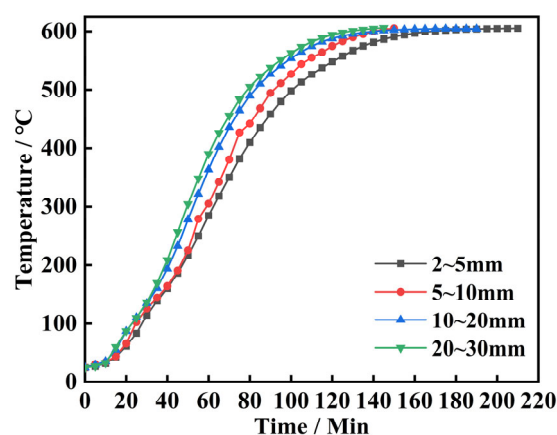


Figure 3. Effect of particle sizes on the heat transfer property in the 30 kW coal pyrolysis device.

3.2. Distribution of Pyrolysis Products

Table 2 summarizes the material balance of pyrolysis products under different particle size conditions. Figure 4 demonstrates the variation in the distribution of pyrolysis products under different coal particle sizes. It is evident that the char yield gradually rises with an increase in particle size. This fact is essentially attributed to the slower heat transfer rate from the surface of coal particles to their interior, which leads to a lower pyrolysis completion of the larger-sized coal block. During large-scale coal pyrolysis, the release of volatiles is less efficient, thereby leading to a higher mass fraction of solid products. The coal pyrolysis experiment with a coal particle size of 20~30 mm exhibits a maximum gas yield of 6.99 wt.%. The maximum tar yield is obtained as 14.66 wt.%, which mainly corresponds to the coal particle size range of 5–10 mm. With the growth of the particle size, the tar yield first demonstrates a tendency to rise and then decline; therefore, the particle size is also capable of affecting the tar yield in a complex way. Analysis of the experimental results concludes that coal beds with smaller coal blocks have poor permeability. The increase in coal particle size could effectively enhance the fluid mobility in the coal bed and facilitate the timely transfer of gaseous products. The residence time of the tar in the reactor is reduced, and the degree of tar cracking is also mitigated, yet the enhancement effect is limited [37]. Further, an increase in particle size complicates the tar release from the coal blocks, and the larger temperature difference between the inside and outside of the coal aggravates the tar cracking during the exit, leading to a further decrease in tar production [27].

Table 2. Quantitative material balance of pyrolysis products under varied particle size conditions.

Particle Size/mm	Pyrolysis Products/wt. %			
	Coke	Gas	Tar	Water
2~5	76.05	6.08	13.12	4.75
5~10	76.32	5.34	14.66	3.68
10~20	76.91	6.57	13.21	3.31
20~30	77.43	6.99	12.43	3.15

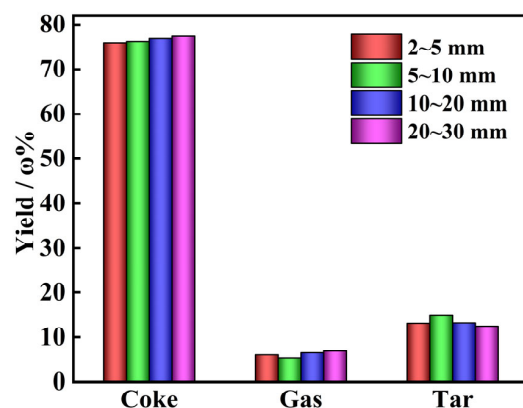


Figure 4. Yield distribution of three-phase products under different particle sizes.

Figure 5 illustrates the variations in pyrolysis gas yield at different times and coal particle sizes, which shows that the effect of particle size on pyrolysis gas production is not remarkable. As the size of the coal blocks increases, the final pyrolysis gas production exhibits a descending trend first and then an ascending trend, where two factors have contributed to this result. On the one hand, from the content of Section 3.1, in the case of larger particle sizes, heat can quickly be conveyed from the high-temperature walls to the coal blocks located in the reactor's center, as the convective heat transfer increases. Therefore, the coal blocks in the radial inner and outer layers of the reactor are rapidly heated, reaching the reaction stage where the pyrolysis gas production is faster. On the other hand, for a coal block, the increase in the coal particle size incorporates the reduction in the heat transfer rate from the coal surface to the interior. This leads to a rapid rise in the temperature of the coal surface, whereas the temperature of the coal center is still very low under the condition of large particle size. In general, due to the influence of these two factors, an increase in the particle size does not contribute to a rapid increase in the global temperature of the coal bed, and the corresponding pyrolysis production does not increase uniformly.

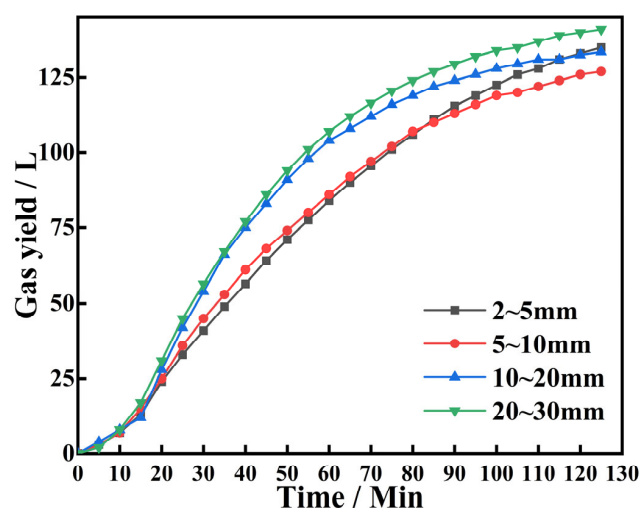


Figure 5. Effect of particle sizes on the pyrolysis gas yield.

The effects of coal particle size on the composition of pyrolysis gas are presented in Figure 6. The components of coal pyrolysis gas mainly include H_2 , CH_4 , CO , and CO_2 . It is obvious that the yields of H_2 and CO_2 initially follow an ascending trend and then a descending trend, whereas the yields of CO and CH_4 demonstrate exactly the opposite trend. In general, the particle size primarily affects the overall heating rate

of the coal bed and subsequently affects the performance of some components of the pyrolysis gas. Appropriate particle size could not only accelerate the heat transfer of the high-temperature wall facing the center of the coal bed but also prevent excessive temperature differences between the inside and the surface of a coal block caused by the low thermal conductivity of coal. When the pyrolysis temperature is higher than 600 °C, low-rank coal mainly undergoes condensation polymerization reactions, during which the gas products are dominated by H₂ [38]. The coal bed, which is stacked with 10~20 mm particles, enables the coal bed to reach the condensation polymerization reaction stage most quickly, which leads to a remarkable increase in the overall H₂ yield. The production of CH₄ chiefly originates from the cleavage of aliphatic side chains and methoxy groups [39], which mainly occurs in the coal depolymerization reaction stage, corresponding to the temperature range of 280~550 °C. A rapid temperature rising rate makes the coal bed pass through this reaction stage faster, resulting in a decreasing trend in CH₄ yield. By observing the relationship between the CH₄ and H₂ yields and particle size, it is evident that with the increase in particle size, the overall heating rate of the coal bed first grows before subsequently declining. The optimal heat transfer effect is achieved as the coal particle size reaches 10~20 mm. The production of CO₂ mainly comes from the decarboxylation of aliphatic hydrocarbons in the low-temperature range of 200~460 °C [40], and could also be produced through the cleavage of aliphatic hydrocarbon side chains with oxygen-containing functional groups mainly in the higher range of 460~610 °C [41]; therefore, the effect of particle size on CO₂ yield is unclear. Last but not least, CO mainly originates from the secondary cracking of tar [42]. The experimental group with coal particle sizes of 5~10 mm demonstrates the lowest CO yield, which is consistent with previous results indicating it has the highest tar yield.

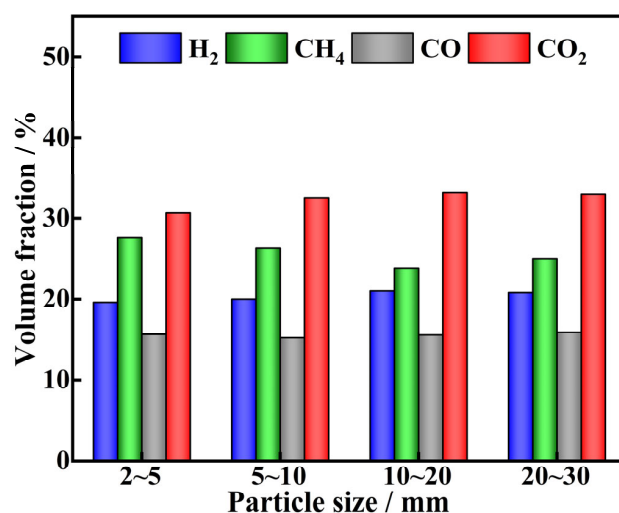


Figure 6. Effect of particle size on the yield of main pyrolysis gas components.

The coal tar component was analyzed using gas chromatography–mass spectrometry, and the results are listed in Table 3. It can be evidently observed that coal tar is mainly composed of aromatic compounds, with representative substances categorized based on their characteristic structures or functional groups. Table 3 presents these representative components and their corresponding categories, while the ion chromatogram of the total tar has been illustrated in Figure 7. The peak area normalization approach was utilized to analyze the GC-MS results. According to Figure 8, the primary components of the tar can be reasonably divided into four groups: benzenes, phenols, naphthalene, and polycyclic aromatic hydrocarbons (PAHs), which are aromatic compounds containing three or more rings. From the data presented in Figure 8, it is evident that with increasing particle size,

the proportions of benzenes, phenols, and naphthalene initially demonstrate a decreasing trend and then an increasing trend. On the contrary, PAHs follow the opposite pattern. Notably, all the reversals for these trends are observed in the experimental results for the 10~20 mm group. Benzene compounds primarily originate from the primary pyrolysis reaction of coal and the secondary cracking reactions of bicyclic aromatic hydrocarbons or PAHs [43]. As mentioned earlier, a coal bed consisting of small-sized particles prolongs the residence time of volatiles and makes them more susceptible to secondary reactions. As a result, the tar contains a higher proportion of monocyclic aromatic compounds; however, an excessive increase in particle size complicates the removal of volatile components from coal and leads to the cracking of aromatic compounds with higher ring numbers into benzenes. The dehydroxylation of phenols reduces their proportion in tar, and the acceleration of the heating rate facilitates this process [44]. This once again demonstrates that the group of particle sizes 10~20 mm, which has the lowest proportion of phenols, exhibits the best overall heat transfer result. The impact of particle size on the content of naphthalene compounds is mainly attributed to the secondary cracking reactions, which include the formation of naphthalene through the cracking of PAHs and the depletion resulting from the cracking of naphthalene itself. It is clear that increasing the heat transfer rate is more effective in consuming naphthalene. The PAHs are consumed through secondary cracking and other reactions. In addition to being formed during the initial reaction of coal pyrolysis, polycondensation also produces numerous heavy tar components. The accelerated overall heating rate forces the coal bed to proceed to the polycondensation stage more rapidly, leading to a higher proportion of PAHs. In addition, the plotted results in Figure 8 are essentially attributed to this fact.

Table 3. Structure and classification of representative substances in tar components.

Classification of Tar Components	Representative Substance
Benzenes	Toluene, p-xylene, o-xylene, m-xylene, mesitylene, styrene, and 4-methylstyrene
Phenols	Phenol, o-cresol, m-cresol, 2,4-dimethylphenol, 3,5-dimethylphenol
Naphthalene	naphthalene, α -methylnaphthalene, β -methylnaphthalene, 1,2-dimethylnaphthalene, 2,6-dimethylnaphthalene, and 1,5-dimethylnaphthalene
Aliphatic hydrocarbon	Undecane and tetradecane
PAHs	Acenaphthene, fluorene, phenanthrene, anthracene, fluoranthene, pyrene, 9,10-phenanthrene, and dibenzofuran

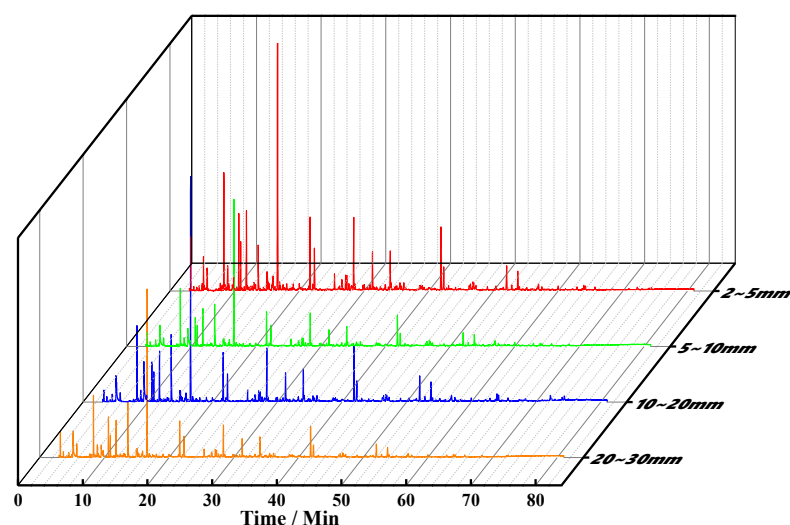


Figure 7. Total ion chromatogram of tar at different particle sizes.

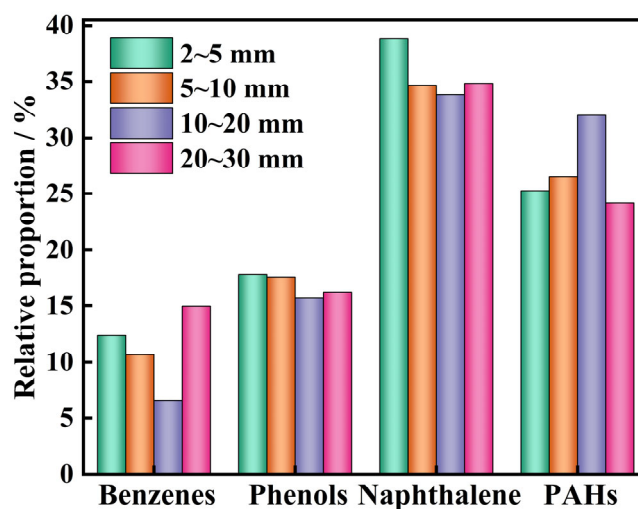


Figure 8. Proportion of tar components under different particle sizes.

3.3. Properties of Pyrolysis Char

The combined characterization techniques, including ultimate analysis, X-ray diffraction (XRD), Raman spectroscopy, Brunauer–Emmett–Teller (BET), and scanning electron microscopy (SEM), are utilized to systematically analyze the obtained char, aiming to realize the influence of particle size on char properties during lump coal pyrolysis.

The ultimate analysis results of char obtained from various particle sizes of coal pyrolysis are listed in Table 4. Former investigations have revealed that raw coal with larger particle sizes retains higher ash content [45]. However, ultimate analysis for coal blocks requires grinding solid materials to smaller sizes, and the ash content has already changed during this process. Therefore, it cannot reflect the true relationship between ash content and particle size in the char obtained from the pyrolysis of large-sized lump coal. The dry basis content of carbon appears to have no clear relationship with particle size. However, it can be seen that the 10~20 mm group exhibits the highest C/H ratio. This is essentially attributed to the fact that the coal blocks in this experiment proceeded to the condensation reaction stage more rapidly, which resulted in a higher degree of coal graphitization.

Table 4. Ultimate analysis of char from different particle size pyrolysis experiments.

Particle Size/mm	C _d	H _d	O _d	N _d	S _d	A _d	C/H
2~5	81.01	1.99	4.16	0.63	0.32	11.89	40.709
5~10	83.38	2.08	5.10	0.65	0.18	8.60	40.087
10~20	82.08	1.97	4.50	0.69	0.27	10.49	41.665
20~30	83.14	2.03	4.99	0.63	0.23	8.98	40.956

The microcrystalline structural features of char were characterized by d_{002} , L_c , and L_a , which were calculated using Scherrer's formula and Bragg's equation. The XRD data for the sample were deconvoluted into individual peaks, and the fitting results for each sample are presented in Figure 9a–d. There are two broad diffraction peaks at 2θ of 20–30° and 40–46°, which in order correspond to the peak 002 and peak 100 of graphite crystallites. The asymmetry of the peak 002 is mainly ascribed to the superposition of the γ -band, which is attributed to the aliphatic hydrocarbon side chains, various functional groups, and alicyclic hydrocarbons bonding with condensed aromatic rings [46]. Therefore, the peak fitting approach is utilized to separate the γ -band and the 002 band. By identifying the relative positions of Peaks 002, 100, and γ , their corresponding half-height widths were determined,

enabling the calculation of specific structural parameters (d_{002} , L_c , L_a) of the aromatic layer based on Bragg's equation and Scherrer's formula. Generally, it is believed that a larger microcrystalline size (L_a), a higher stacking height (L_c), and a smaller interlayer spacing (d_{002}) indicate closer proximity to the ideal interlayer spacing of graphite crystals, thereby reflecting a higher degree of order in the microcrystalline structure of the char sample and consequently lower reactivity [47]. As illustrated in Table 5, the variation of d_{002} exhibits a decreasing trend before an increasing trend, whereas L_c and L_a demonstrate opposite trends. All these data indicate that with the rise in coal particle size, the graphitization degree of the produced coal firstly follows an ascending trend and then arrives at a descending trend. The highest graphitization degree is observed in char with a particle size of 10~20 mm.

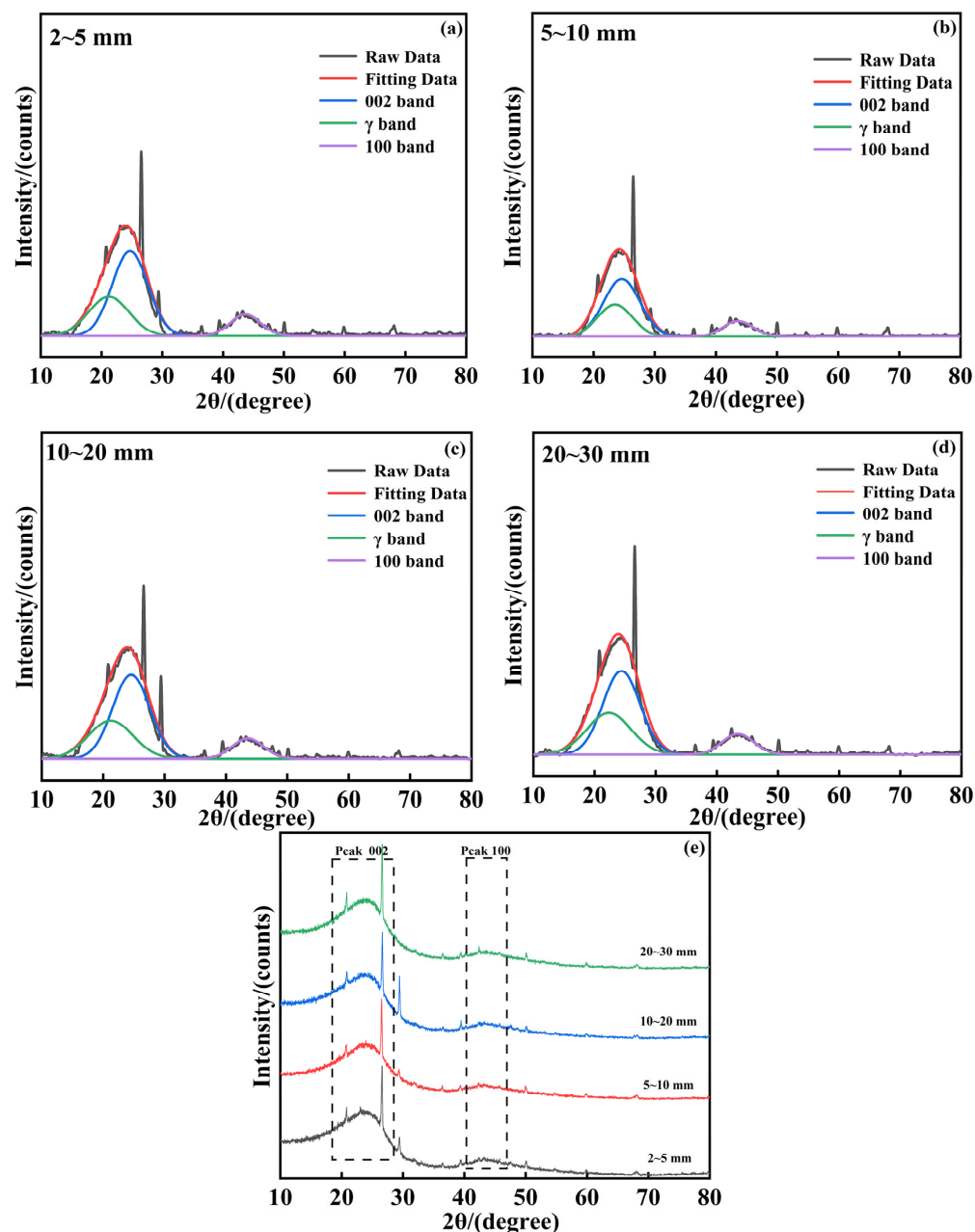


Figure 9. (a–e) Multi-Gaussian fitting XRD spectra of char produced at different particle size conditions and integrated XRD spectra.

Table 5. Microcrystalline structural parameters of char with different particle sizes at 600 °C.

Particle Size/mm	d_{002}/nm	L_c/nm	L_a/nm
2~5	0.3679	1.1748	2.8730
5~10	0.3645	1.1953	2.9125
10~20	0.3606	1.2260	2.9579
20~30	0.3626	1.2121	2.9426

Raman spectroscopy was employed to investigate the microscopic carbon chemical structure of the semi-coke. As shown in Table 6, four D bands and one G band were utilized in the fitting process [48]. Herein, I_{D1} and I_G represent the integrated areas under their respective peaks, while I_{all} denotes the sum of all fitted peak areas. The intensity (peak area) of each Raman peak is strongly correlated with the degree of ordering and reactivity of the graphite structure. In this study, Raman spectra were collected for each sample, followed by peak deconvolution, data fitting, analysis, and calculation to determine the ratios of I_{D1}/I_G , I_G/I_{all} , and generate Raman parameter maps using Origin 2018 software. Specifically, the ratio I_G/I_{all} reflects the ordering degree of the char structure, whereas the ratios I_{D1}/I_G indicate the extent of disorder in the carbonized texture of the char [49]. Additionally, peak-fitting analysis was conducted on the Raman spectra to elucidate the contributions of different carbon forms. The Raman spectra peak fitting of coke obtained from various particle size experiments is shown in Figure 10a–d. As shown in Table 7, the graphitization degree grows with particle size until the size range reaches 10–20 mm. Consistent with the previously attained results, char with a particle size of 10–20 mm possesses the most severe graphitization.

Table 6. Summary of Raman peak/band assignment.

Band Name	Approximate Band Position/ cm^{-1}	Description
D1	1380	C-C between aromatic rings and aromatics with no less than six rings
D2	1620	Aromatics with three–five rings; amorphous carbon structures
D3	1500	Methylene or methyl; semi-circle breathing of aromatic rings; amorphous carbon structures
D4	1200	$C_{\text{aromatic}}-C_{\text{alkyl}}$; aromatic (aliphatic) ethers; C-C on hydroaromatic rings; C-H on aromatic rings
G	1580	Graphite E_{2g2} ; aromatic ring quadrant breathing;

The BET test is used to characterize the specific surface area, total pore volume, and average pore size of the coal [50]. As presented in Table 8, the char formed by the larger particle size obtains a larger specific surface area and greater pore volume. It is obvious that the release of volatiles in larger coal particles faces greater resistance. More pores are easily formed in the coal, which appears as an increase in the specific surface area and pore volume. Abnormal phenomena occurred in 10~20 mm, with the specific surface area lessening to 2.0002 m^2/g and the pore volume decreasing significantly. As mentioned earlier, when discussing the composition distribution of pyrolysis gas, the 10~20 mm group exhibited the highest hydrogen content in the pyrolysis gas, indicating that the polycondensation reaction of coal blocks was more severe in this group of experiments. Some of the pores generated during the volatile removal process were closed due to polycondensation, making the coal structure more compact and leading to a noticeable reduction in the measured specific surface area and pore volume.

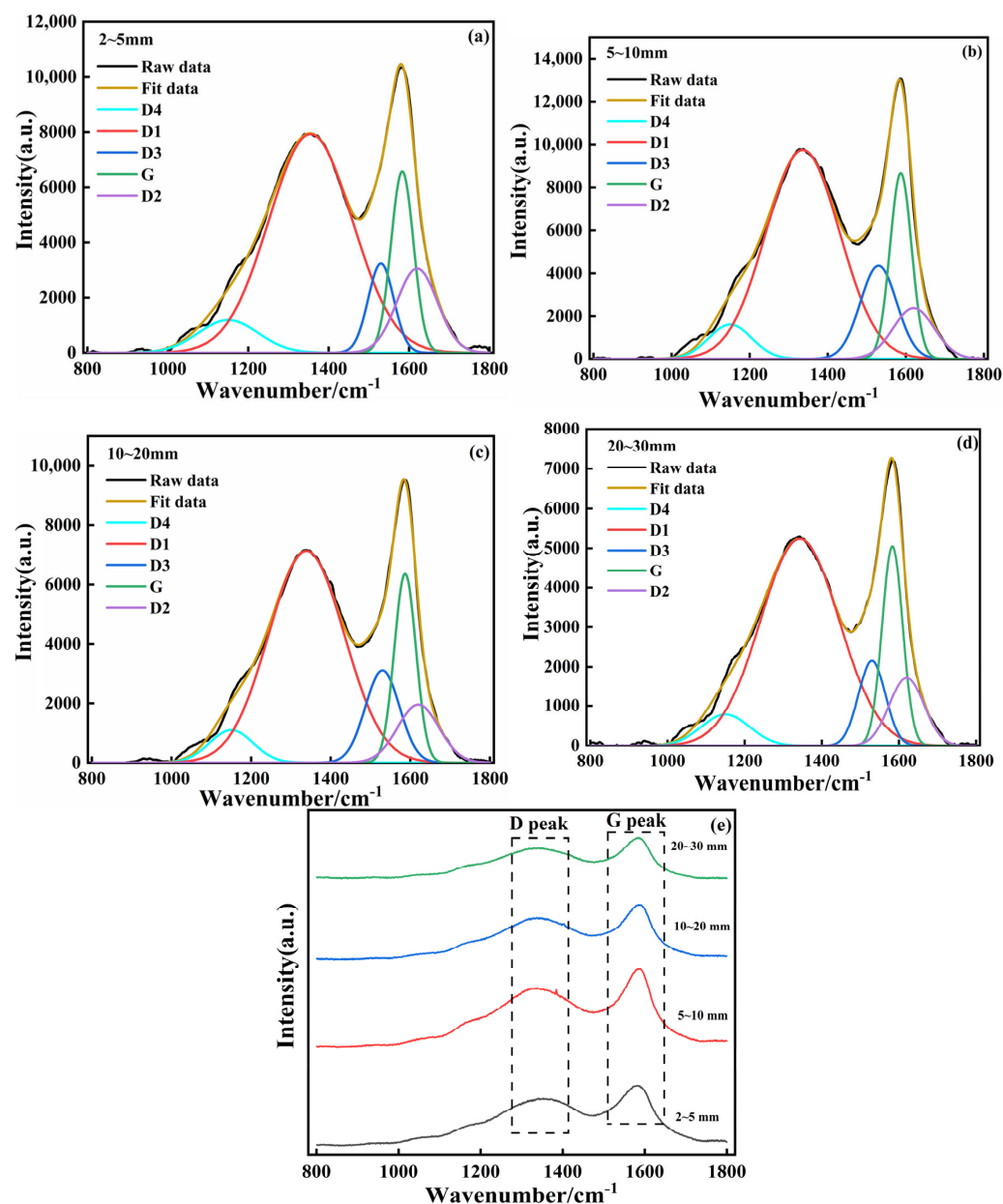


Figure 10. (a–e) Multi-Gaussian fitting of Raman spectra of chars produced at different particle size conditions and Integrated Raman spectra.

Table 7. Raman spectra analysis of char.

Particle Size/mm	I_{D1}/I_G	I_G/I_{all}
2~5	4.478	0.1359
5~10	3.925	0.1514
10~20	3.858	0.1547
20~30	3.975	0.1505

Figure 11 represents the SEM images of coal and char. According to Figure 11a, it can be seen that the surface of raw coal is relatively smooth and without obvious pores and cracks. The pores of 2~5 mm char are small and flat, and the pore size of the 5~10 mm group is slightly expanded. As the particle size reaches 10~20 mm, the surface of the char becomes fragmented and rugged. The pore structure of the 10~20 mm group is not intuitive, and instead, many gullies appear, which may be related to the rapid release of volatiles.

The surface of 20~30 mm char possesses obvious and larger pores, which also confirms the experimental results that it has a high gas production and a fast gas production rate.

Table 8. Specific surface area and pore structure characteristics of char.

Particle Size mm	Specific Surface Area m ² /g	Total Pore Volume cm ³ /g	Average Pore Size nm
2~5	1.3717	0.0028	8.1595
5~10	3.1622	0.0086	10.8799
10~20	2.0002	0.0003	0.6249
20~30	12.3560	0.0106	3.4415

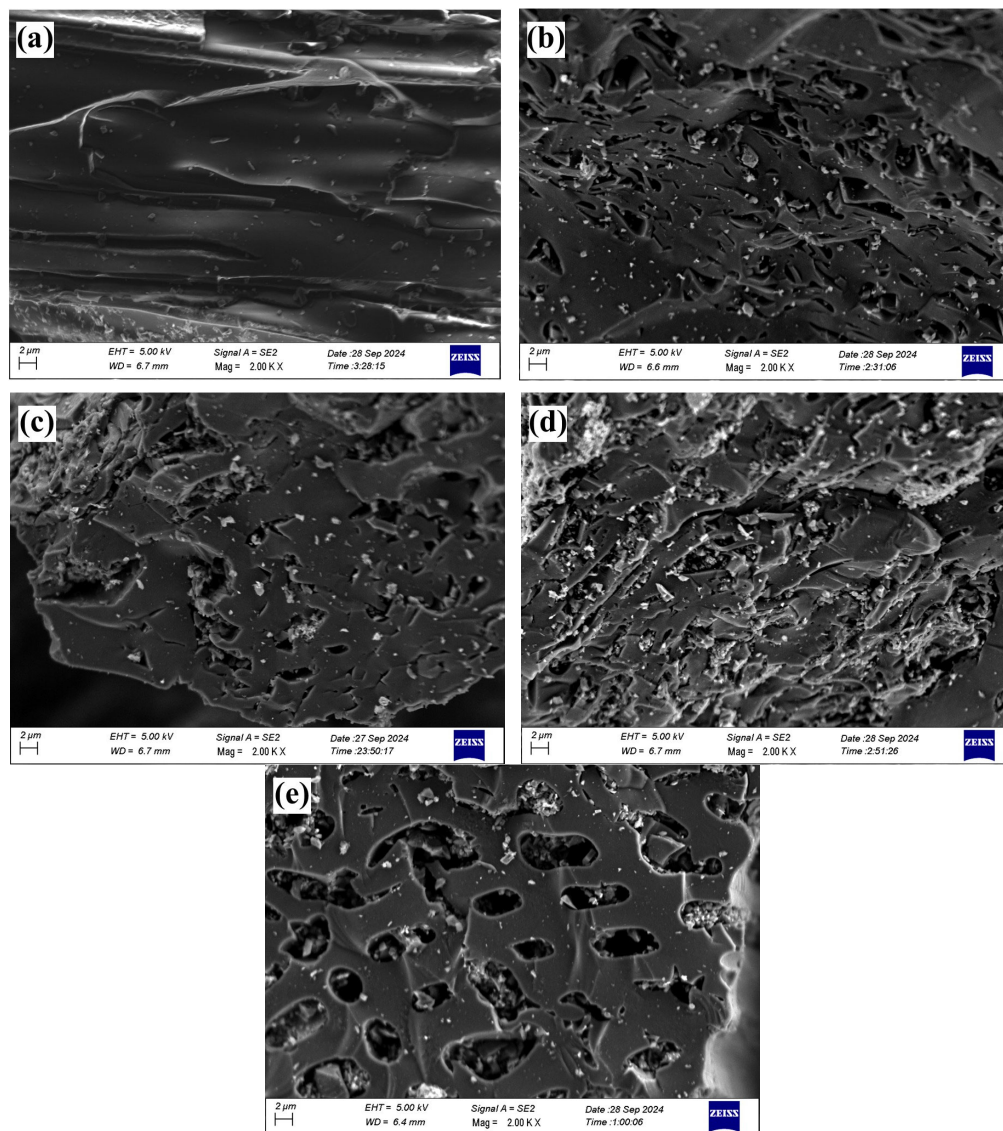


Figure 11. SEM analysis (a) Raw coal, (b) 2~5 mm, (c) 5~10 mm, (d) 10~20 mm, and (e) 20~30 mm.

4. Conclusions

The influence of particle size on the distribution of low-rank coal pyrolysis products and char characteristics was methodically examined based on a fixed-bed reactor. Increasing the particle size led to widening the gaps between the coal blocks, allowing heat to be transferred rapidly from the heated wall to the center of the coal bed. However, due to the low thermal conductivity of the coal itself, large coal blocks resulted in slow heat transfer from the coal surface to the coal interior. Therefore, there would exist an appropriate particle

size that allows the coal block in the reactor's center to be heated rapidly without causing a remarkable temperature difference between the surface and the core of the coal block. Among the four particle size ranges selected in this study, the 10–20 mm group exhibited the best overall heat transfer performance. Actually, this group allows the coal in this experiment to reach the final heating temperature of 600 °C faster, which is the temperature range in which polycondensation reactions are more likely to occur. Among the four experimental groups, the 10–20 mm particle size range exhibited the highest total hydrogen content in pyrolysis gas at 20.99 vol%, while simultaneously showing the maximum relative abundance of heavy tar components at 31.99 percent as determined by GC-MS peak area comparison. In addition, the graphitization degree of the resulting char was demonstrated to be the highest level. This work provides the first systematic investigation into particle size effects across the 2–30 mm range for lump coal pyrolysis—a critically underexplored domain where prior research has predominantly confined itself to pulverized coal particles below 2 mm. However, lump coal pyrolysis faces several constraints: (1) relatively limited lump coal resource availability versus globally dominant powdered coal, and (2) significant char byproduct accumulation requiring efficient conversion solutions. Future research should optimize lump coal pyrolysis protocols to enhance the industrial applicability of derived coke products.

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