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Change of the Petrographic Characteristics of Semi-Coke in the Iron Ore Sintering Process

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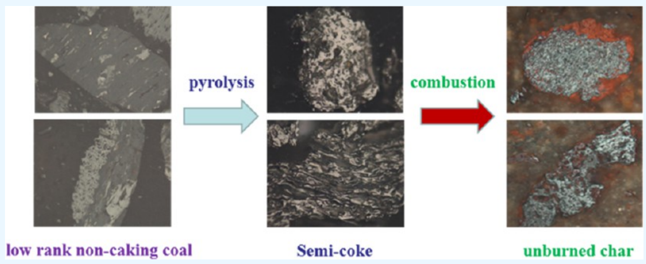
ABSTRACT: This study focuses on the petrographic analysis method to evaluate semi-coke and its combustion behavior in the sintering process, which has been seldom conducted before. Semi-cokes are different in morphological features, porosity, pore structure, and wall thickness, because of differences in the vitrinite and inertinite of the raw coal. Semi-coke displayed isotropy, and even after the drop tube furnace (DTF) and sintering process, it still retained its optical properties. Eight kinds of sintered ash were observed using reflected light microscopy. Petrographic analyzes for the combustion properties of semi-coke were based on its optical structure, morphological development, and unburned char. The results indicated that microscopic morphology was an important characteristic when trying to understand the behavior and burnout of semi-coke. These characteristics can be used to trace the origin of the unburned char in fly ash. The unburned semi-coke mostly existed in the form of inertoid, mixed dense and mixed porous. Meanwhile, it was found that most of the unburned chars were melted into sinter, resulting in inefficient fuel combustion.



1. INTRODUCTION

Iron ore sintering is an important operation unit in the iron and steel industry; traditionally, expensive coke breeze and coal are used as fuels for the iron ore sintering. Meanwhile, a large number of harmful gases are produced by fuel combustion. Therefore, it is necessary to select a clean and high-quality fuel to reduce the operating costs and control the gaseous pollutants released during the iron ore sintering. Specifically, the fuel is required to have a higher heating value, lower ash yield, and better flammability, and release less-harmful gas.[1]

China has abundant reserves of low-rank coal, and pyrolysis is one of the most effective ways to efficiently utilize low-rank coal and transform it into clean products.[2] The solid product obtained from low-temperature pyrolysis of low-rank non-caking coal is semi-coke, which has the characteristics of high chemical activity, low ash, low aluminum, low sulfur, and low phosphorus, and has been identified as clean energy.[3] In the steel industry, with the increasing demand for high-quality anthracite and coke, semi-coke has attracted great attention due to its excellent characteristics,[4] and researchers[5,6] have begun to explore the use of low-cost semi-coke as an alternative fuel for blast furnace injection and sintering processes. Nevertheless, there are still some problems in the steel production of semi-coke as an alternative fuel, such as the increase in fuel ratio and decrease in the economy. In order to solve these problems, the combustion behaviors of the fuel were studied.[7−9]

Coal is a kind of organic biological rock, and its petrographic composition has an important influence on the pyrolysis and gasification process of coal.[10,11] The petrographic analysis is widely used in the coal industry and is one of the main methods to evaluate the performance of coal and coke.[12−18] Meanwhile, a number of research works[19−21] have been carried out on the petrographic analysis of coal and coke, which is helpful to better understand their combustion behaviors. Daniel[22] investigated the petrographic composition of blast furnace waste dusts and it could be applied to identify the pollution source or recycle the dusts. According to microscopic analysis, Wu[23] found that the structure and proportion of unburned coal and coke particles in blast furnace dust were different, and the structure of the unburned particle in blast furnace dust was divided into four parts. The petrographic analysis of fly ash can provide details that cannot be obtained by chemical analysis, such as the shape of carbon, its relationship with inorganic ash, and the combustion efficiency.[24] In order to ensure the good performance of the blast furnace, Xing[25] explored the degradation mechanism of coke by petrographic analysis of coke reacting under simulated blast furnace conditions. Accordingly, the petrographic analysis

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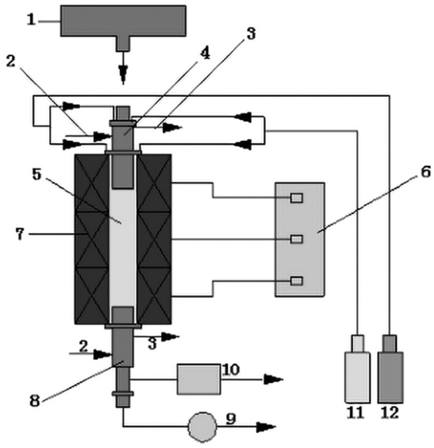

Figure 1. Schematic diagram of the DTF.

Table 1. Chemical Properties of the Samples

sample	M_{ad}/%	A_{ad}/%	V_{ad}/%	FC_{ad}/%	C/%	H/%	O/%	N/%	S/%	Q_{DW}/(kJ/kg)
XJ1	8.35	10.80	14.24	66.61	72.83	3.17	3.25	1.12	0.48	23.61
XJ3	10.58	8.78	12.76	67.89	73.05	2.63	3.67	0.96	0.33	24.16
SX1	8.06	11.17	12.42	68.36	77.76	3.35	3.53	1.22	0.43	24.15
SX2	6.83	9.26	11.24	72.67	78.71	2.47	1.25	1.02	0.46	26.44
XS	7.28	9.64	11.33	71.75	77.34	2.40	1.35	1.02	0.97	26.05
JB	1.57	14.35	3.27	80.80	83.60	1.12	1.07	0.97	0.93	22.98
YS	2.50	12.88	3.33	81.29	84.62	1.26	0.94	0.85	0.91	22.59
YK	0.73	12.34	2.34	84.59	86.25	1.10	1.24	1.12	0.98	23.48
Si	0.50	13.03	1.62	84.84	86.06	0.95	1.12	1.04	1.12	22.18

is a reliable method to analyze the fuel and its combustion behavior.

The content of unburnt char in fly ash has always been regarded as one of the indicators of the combustion performance of pulverized fuel. However, most of studies were focused on the combustion of coal and coke. Semi-coke is a new alternative fuel with excellent performance, and few studies have been conducted to investigate its microscopic analysis and combustion behaviors. In this paper, five kinds of semi-cokes and eight kinds of sintered ash were studied to determine the feasibility of a microscopic method to characterize the unburnt char in fly ash. Based on the microscopic characteristics of semi-coke and unburned char, the changes of its microscopic characteristics in the iron ore sintering process were explored. The microscopic analysis method to evaluate the combustion behavior of semi-coke in the sintering process can be used as a feasible scientific method for evaluating and selecting semi-coke as a fuel.

2. MATERIALS AND METHODS

2.1. Materials. Five semi-cokes and four coke breezes as the iron ore sintering fuels were collected from Nangang Industry Development Company in China. They were Xinjiang semi-coke 1 (XJ1), Xinjiang semi-coke 3 (XJ3), Shanxi semi-coke 1 (SX1), Shanxi semi-coke 2 (SX2), Xinsheng semi-coke (XS), Jiubao coke breeze (JB), Youse coke breeze (YS), Yankuang coke breeze (YK), and Sieve coke breeze (Si). Eight kinds of sintered ash from different sintering processes in the same steel plant were collected. The raw fuels were pulverized and sieved to <1.0 mm particle size; the mass of the sample less than 0.1 mm did not exceed 10%. The sintered ashes taken from the electric dust collector were not crushed due to their small particle size. All of the samples were dried in a drying oven at 110 °C for 5 h. Then, 100−200 g of dry samples less than 1.0 mm was weighed, and they were divided into 10−20 g by the cone-quarter method for petrographic analysis.

2.2. Char Preparation. Chars from semi-coke and coke breeze were prepared in a drop tube furnace (DTF) to identify the microscopic optical characteristics of different single fuels after combustion. The details of the reactor were described in some studies,[26] and the DTF experimental system is shown in Figure 1. The reactor was operated at 1000 °C under air atmosphere (O_2/N_2 = 1:4) and the powder flow rate was 0.6 g ± 0.05 g/min. The residence time of the fuel in the DTF was about 2−3 s. The chars were collected by the filter cartridge for petrographic analysis.

2.3. Petrographic Analysis. The optical thin slices for microscopic optical analysis were prepared according to GB/T 16773−2008. The samples were mixed with an epoxy resin in ring-shaped molds. Once cured, the sample was demoulded, ground, and polished to obtain an optical thin slice with a surface size of 25 mm × 25 mm. The sample particle area on the working area should be more than 2/3.

The petrographic analysis of the optical thin slices was performed using a Zeiss microscope (Axio Scope1&MSO) with reflected light, 50× magnification, and oil immersion. The petrographic composition of coal and coke based on the

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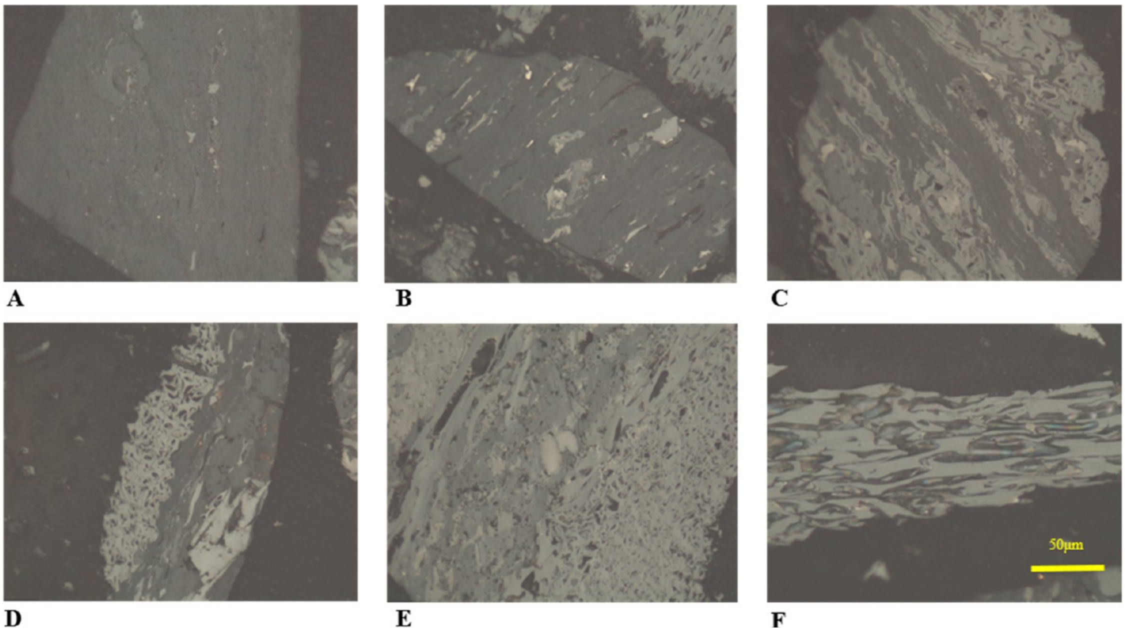

Figure 2. Images of low-rank non-caking coal: (A, B) vitrinite; (C, D) vitrinite and fusinite; (E) fusinite and semifusinite; and (F) fusinite. Optical microscope photos were obtained under reflected light and oil immersion.

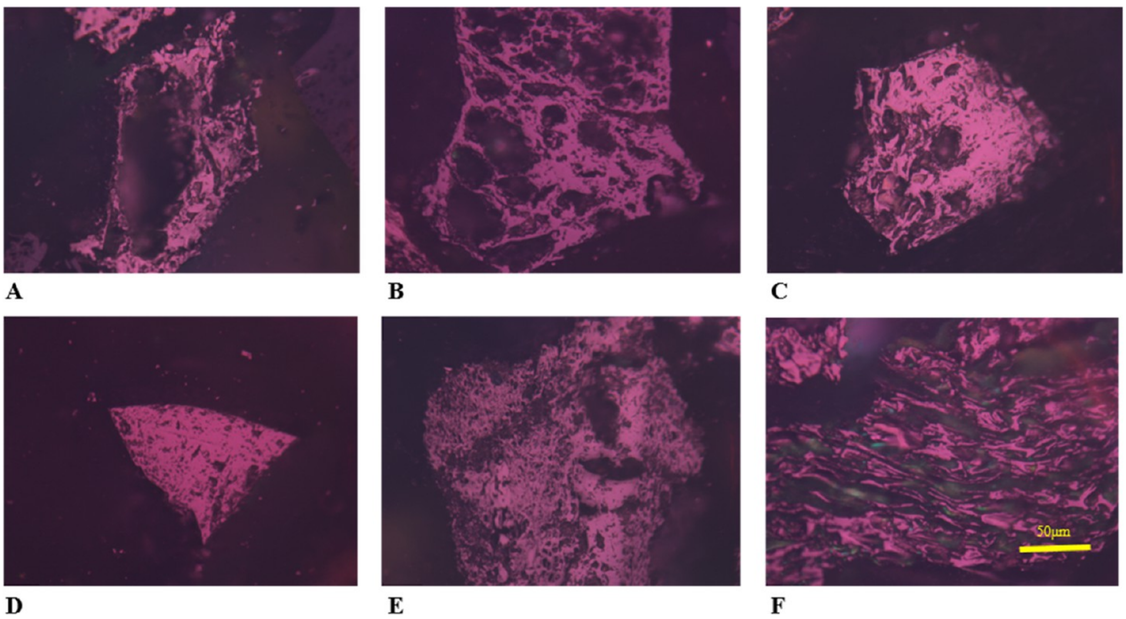

Figure 3. Images of semi-coke. (A) Larger cavity, vitrinite-derived; (B) network, vitrinite-derived; (C) fused and unfused particles; (D) unfused solid particle; (E) massive particle with more porosity; and (F) unfused particle with partially original structure. Optical microscope photos under crossed polars, oil immersion, and a 1λ retarder plate.

microscopic composition was determined according to the ICCP guidelines.[27−29] According to the classification scheme for the combustion coal, the morphology of the unburned char was characterized.[30] The quantitative studies were carried out using point counters; the maceral content analysis was carried out on the surface of the optical thin slices with equal spacing points, and the effective points of each optical slice were not less than 500.

3. RESULTS AND DISCUSSION

3.1. Characteristics of Semi-Coke. Semi-coke is a solid carbonaceous product obtained from low-rank non-caking coal by low-temperature pyrolysis. The proximate and ultimate analyzes of the samples are presented in Table 1. Compared with coke breeze, semi-coke has the characteristics of low carbon, high hydrogen, low ash, low sulfur, and high gross calorific value, and it can provide more heat and reduce the number of SO_x released in the sintering process. This indicates that semi-coke is a more environment-friendly alternative fuel for the sintering process.

The formation of coke structures during pyrolysis depends on the petrographic composition of the original coal.[31,32] Semi-coke is the low-temperature pyrolysis product obtained from low-rank non-caking coal (Figure 2). Figure 2A,B shows

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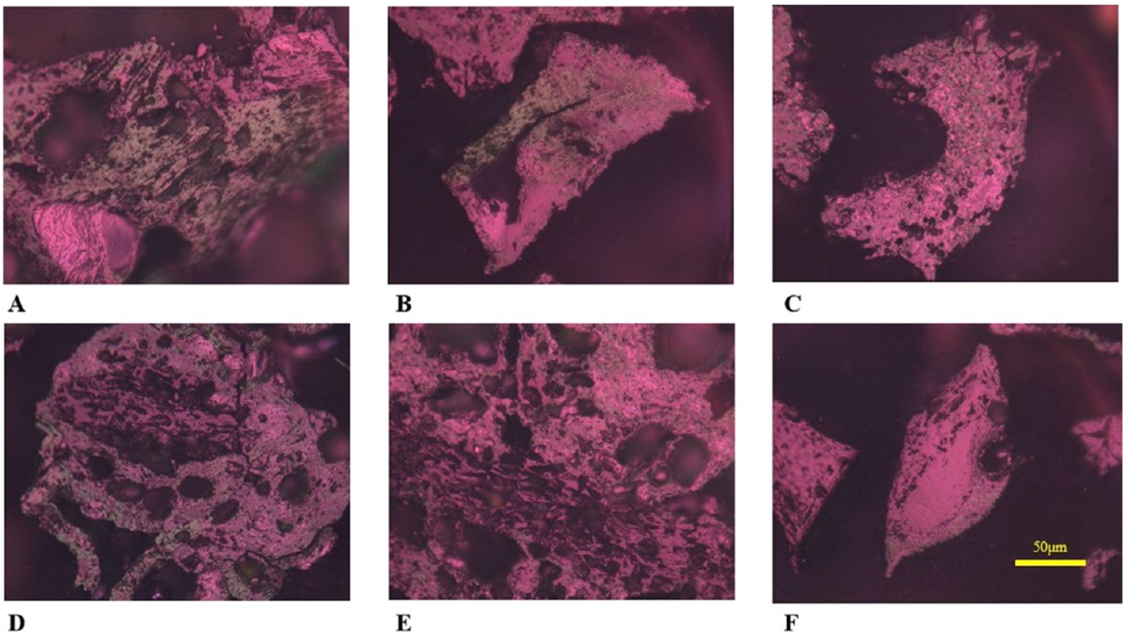

Figure 4. Images of coke breeze. (A−C) Anisotropic and (D−F) anisotropic, with included isotropic images. Optical microscope photos under crossed polars, oil immersion, and a 1λ retarder plate.

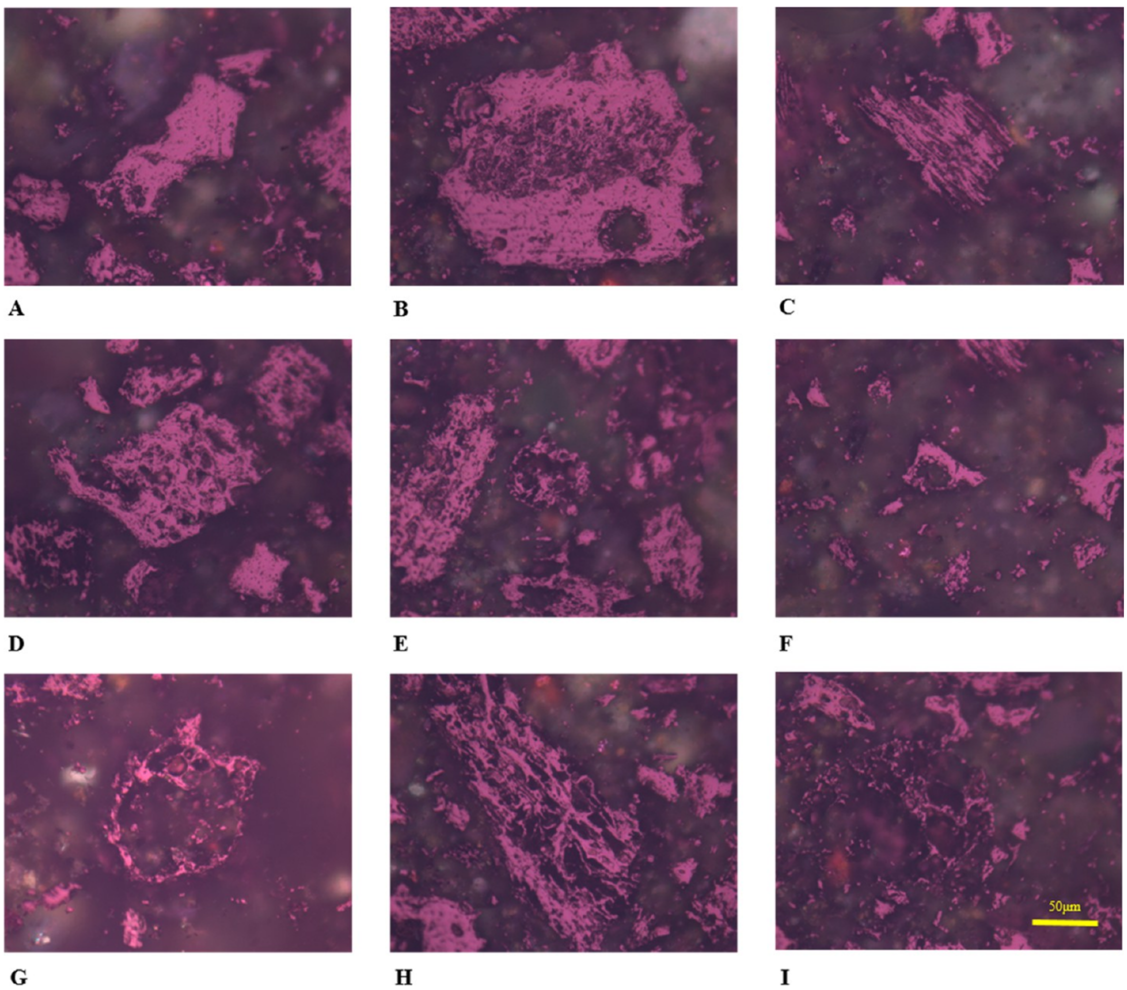

Figure 5. Images of unburned chars from semi-coke. (A) Solid; (B) inertoid; (C) fusinoid; (D) mixed dense; (E) mixed porous; (F) crassispheres; (G) tenuispheres; (H) crassinetwork; and (I) tenuinetwork. Optical microscope photos under crossed polars, oil immersion, and a 1λ retarder plate.

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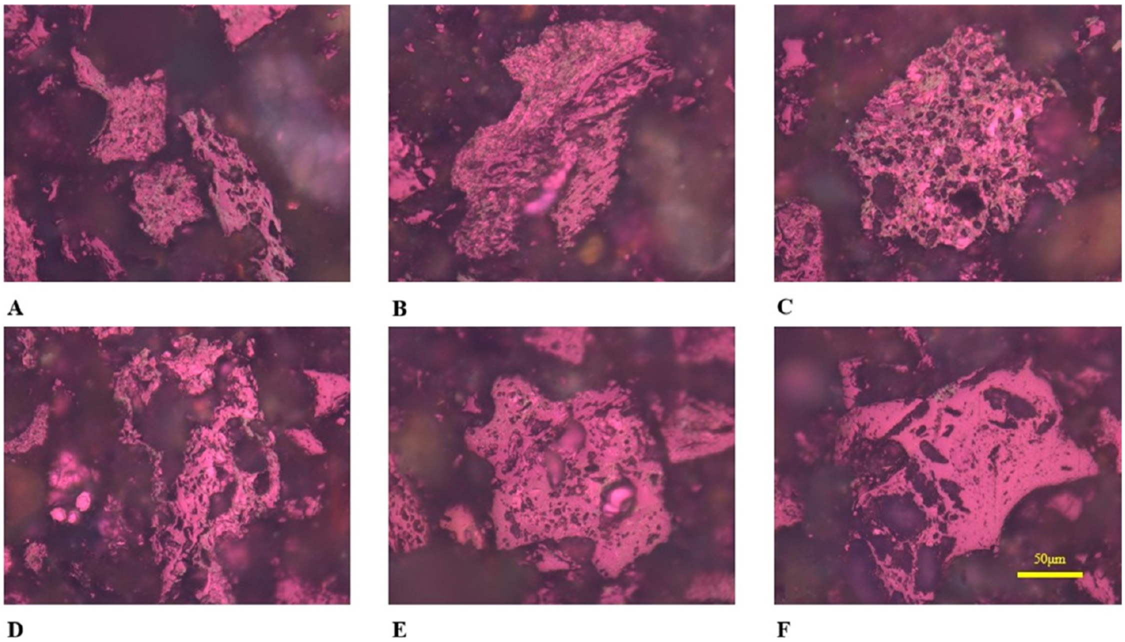

Figure 6. Images of unburned chars from coke breeze. (A, B) Anisotropic, mixed dense; (C, D) anisotropic, vitrinite-derived; (E) anisotropic, mixed porous; and (F) isotropic, with little anisotropic, inertinite-derived. Optical microscope photos under crossed polars, oil immersion, and a 1λ retarder plate.

vitrinite, which is the reactive component of coal. Figure 2E shows fusinite and semifusinite, and Figure 2F shows fusinite, which are the nonreactive components of coal, while the particles are both vitrinite and fusinite in Figure 2C,D.

Based on the observation of samples, the typical morphology and optical texture of semi-coke are presented in Figure 3.

Although many different types of carbons have been described in the literature, there are essentially three fundamental characteristics that are used to differentiate between them: porosity, pore structure, and wall thickness distribution.[33] The larger cavity size is observed in Figure 3A, which indicates that porous particles may be derived from the vitrinite of the raw coal (Figure 2A,B), which lead to fused particles with porous developing structures that are mixed porous. Figure 3B shows the semi-coke in the form of a carbon network, which also originated from vitrinite. During the pyrolysis process, the reactive component of coal softens, melts, and devolatilizes to form porous particles. The mixed dense particle with some cavities is observed in the sample in Figure 3C; this particle has fused and unfused parts, and its wall thickness varies from a few microns to tens of microns; this particle may be derived from the mixed particle (Figure 2C). The same semi-coke shows a dense structure without any porosity or devolatilization pores (Figure 3D); this particle with a relatively flat surface and <5% porosity was an unfused solid particle. Compared to Figure 3D, the massive particle with more porosity (Figure 3E) may be derived from the raw coal in Figure 2D; it is a totally isotropic particle, unfused (left from fusinite with partially original structure) or partially fused with variable porosity (right from vitrinite). The inertinite retains its original structure, and the unfused particle with a partially original structure (Figure 2F) can be clearly seen in Figure 3F. According to Alonso[32] and Isabel,[34] the fusinite structures tend to remain unchanged during heating and exhibit relatively isotropic solid materials with little sign of plasticization or swelling.

In Figure 3, it was found that semi-coke was a porous carbonaceous solid and displayed isotropy. This was consistent with Guo,[35] who found that the char of low-rank coal showed isotropy, and that the high temperature and the increased residence time did not change the optical isotropy of the char.

The semi-cokes display an isotropic nature, obtained during low-rank non-caking coal pyrolysis and poly-condensation, whereas coke is the carbon material derived from caking coal and it displays anisotropy (Figure 4). The coke observed in this study has similar porosity to that observed by previous authors' studies.[13,36] Meanwhile, Malumbazo[37] reported that the reflectance increases with increasing pyrolysis temperature, and that the semi-coke from low-temperature pyrolysis had a lower reflectance than coke from high-temperature pyrolysis. Comparing Figures 3 and4, it is easy to distinguish semi-coke from coke by the optical structure.

The optical structure and morphology of the semi-coke are clearly displayed by the optical microscope, and it provides details that cannot be obtained by the chemical analyses. This indicates that the petrographic analysis is a reliable means to analyze semi-coke.

3.2. Petrographic Characteristics of Chars. Since the mixture fuels are used in iron ore sintering, in order to trace the origin of unburned char in sintered ash, the fuel combustion process in an industrial furnace simulated by DTF was studied, and the petrographic characteristics of the char produced by a single fuel in DTF were observed. The residence time of the fuel stay in DTF is only a few seconds; therefore, part of the char retains the original structure, which can be found in the similarity of the petrographic features between Figures 3 and 4.

The changes of the petrographic characteristics of char from semi-coke and coke breeze are shown in Figures 5 and6, respectively.

From the images, it can be seen that nothing changed in the optical texture of the chars and that the chars from semi-coke displayed isotropy, while the chars from coke breeze displayed

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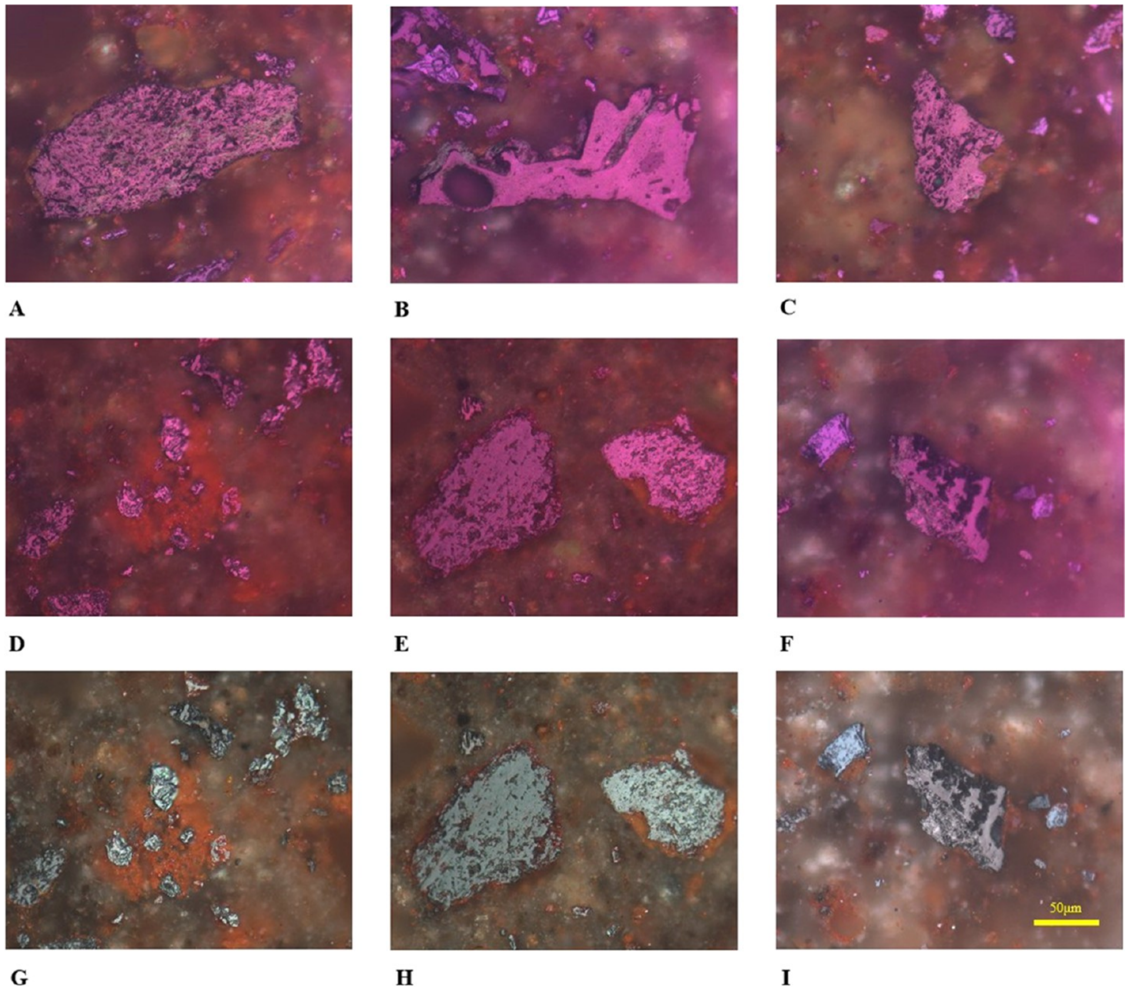

Figure 7. Images of fly ash from iron ore sintering. (A−C) Anisotropy; (D, E) isotropy; and (F) the intermediate particle is anisotropy, whereas the left is isotropy. (A−F) Optical microscope photos under crossed polars, oil immersion, and a 1λ retarder plate. (G−I) Optical microscope photos under reflected light and oil immersion.

anisotropy. Based on the classification system for combustion chars,[30] we can observe that the typical petrographic characteristics of chars from semi-coke are solid (Figure 5A), inertoid (Figure 5B), fusinoid (Figure 5C), mixed dense (Figure 5D), mixed porous (Figure 5E, center of the particle), crassispheres (Figure 5F, center of the particle), tenuispheres (Figure 5G), crassinetwork (Figure 5H), and tenuinetwork (Figure 5I). The morphologic information of char can be used to predict the burnout of char.[38]

Although there were many types of chars as described above, it was observed that there were fewer crassisphere, tenuisphere, and tenuinetwork. Meanwhile, most of the types with many unburned parts were inertoids, fusinoids, and mixed dense. These chars were mostly derived from the inertinite of low-rank coal, and had low flammability, large particle size, and dense and low porosity. This petrographic characteristic of char is similar to that presented by Shibaoka,[39] who reports that some inertinite does not vesiculate and its combustion rate is usually slower.

The unburned chars derived from coke breeze are shown in Figure 6. The unburned chars still retained the anisotropy. In Figure 6F, the fusinoid derived from the inertinite of caking coal still contains the isotropy part, but a small amount of anisotropy can be seen in the upper part of the particle. Compared to the semi-coke and coke breeze (Figures 3 and4), it was observed that the size of unburned char decreased, the pore became larger, the porosity increased, and the pore wall became thinner.

3.3. Sintered Ash Characterization.

The fuel used for sintering in the steel plant is a mixed fuel, and the ratio of semi-coke to coke breeze is 39:61. However, in the sintered ash, the unburned chars showed mostly optical isotropy, and a very small amount of optical anisotropy particles were found.

The pictures of unburned char with different optical structures are shown in Figure 7.

Anisotropy was observed in Figure 7A−C, while isotropy was observed in Figure 7D,E. From Figure 7F, it is seen that the intermediate particle showed anisotropy, whereas the left showed isotropy. The same picture is observed under reflected light and without the 1λ plate (Figure 7I), and the unburned char from isotropy char or anisotropy char can be distinguished by its reflected color. According to the previous research on the optical properties of unburned char in DTF, the optical properties of the burned char are unchanged. This indicates that they may be come from semi-coke and coke, respectively. Figure 7G−I shows the unburned char with isotropy under reflected light, compared to Figure 7D−F.

The unburned char in fly ash depends not only on the combustion conditions but also on certain properties of the fuel, such as particle size and distribution.[40] The particle size

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distribution and proportion of sintering fuel is shown in Table 2.

Table 2. Particle Size Distribution and Proportion of Sintering Fuel

sample	>3 mm	0.5−3 mm	<0.5 mm
XJ1	55.25	32.32	12.43
XJ3	71.80	21.27	6.93
SX1	40.04	41.75	18.21
SX2	73.53	14.57	11.90
XS	62.44	26.76	10.80
JB	38.99	38.60	22.41
YS	11.98	55.53	32.49
YK	42.62	42.29	15.09
Si	24.39	49.00	26.61

The semi-coke particles with size more than 3 mm are 40.04−71.80% and those with size less than 0.5 mm are 6.93−18.21%, while the coke particles with size more than 3.0 mm are 11.98−42.62% and those with size less than 0.5 mm are 15.09−32.49%. The particle size of semi-coke is generally larger than that of coke breeze. The smaller the particle size of the fuel, the easier it is to burn out. The specific surface area and pore volume clearly increased with decrease of the pulverized fuel particle size. This leads to higher heat and mass transfer, which provides a reaction surface during combustion.[41] Therefore coke breeze burns out more easily than semi-coke. Meanwhile, the proportion of coke breeze in the mixed fuel is 60%, the unburned char in fly ash has less anisotropy, and the optical properties of the burned char are unchanged; these indicate that the unburned chars are basically derived from semi-coke.

It is indicated in Figure 7 that the unburned semi-cokes showed isotropy under crossed polars and a 1λ retarder plate. In order to clearly observe the forms of unburned semi-cokes and their relationship with the inorganic ash, the unburned semi-cokes were viewed without a 1λ retarder plate. The typical petrographic characteristics of the unburned semi-cokes are shown in Figure 8. According to the new system for the microscopic classification of fly-ash components developed by the Fly-Ash Working Group, Commission III of the ICCP,[42] a dense particle with little porosity is shown in Figure 8A. Compared to Figure 8A, more porosity is observed in Figure 8B. The typical petrographic characteristic of these two particles is inertoid, and the unfused parts are greater than 75%; this indicates that few of these particles have changed during the combustion. The mixed dense particle has fused

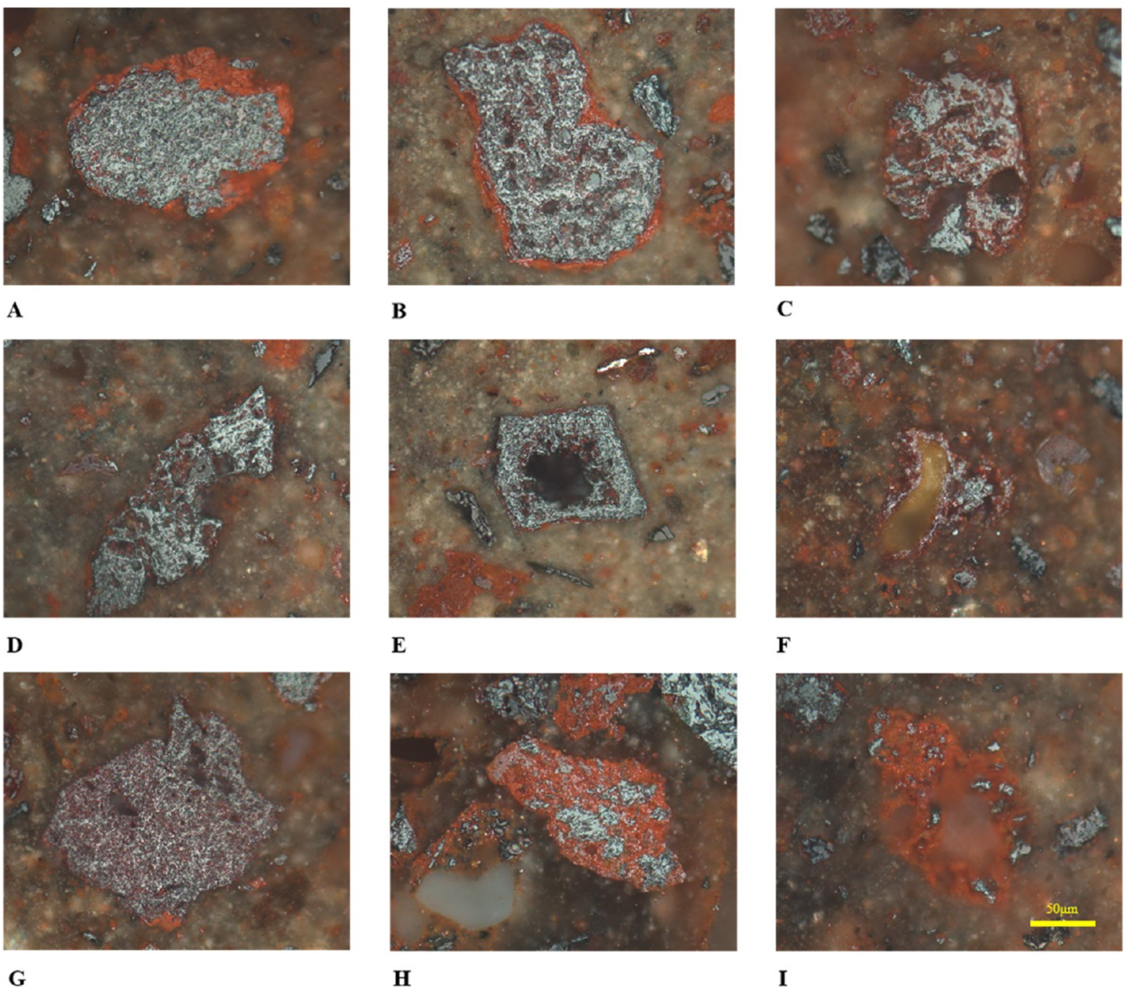

Figure 8. Images of unburned char derived from semi-coke of the iron ore sintering. (A) Dense particle; (B) dense particle with more porosity; (C) mixed dense; (D) crassisphere; (E) crassisphere; (F) tenuisphere; and (G−I) the unburned char and sinter. Optical microscope photos under reflected light and oil immersion, and ×500.

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Table 3. Petrographic Analysis of the Unburned Char in the Sintered Ash (Vol %)

		isotropy							
samples	anisotropy	tenuisphere	crassisphere	tenuinetwork	crassinetwork	mixed porous	mixed dense	inertoid	fusinoid/solid
1	2.83	5.66	7.55	0	0.94	21.70	28.30	32.08	0.94
2	0	0	5.88	0	0	30.88	19.12	44.12	0
3	0	0	2.22	0	0	13.33	20.01	64.44	0
4	0	0	0	0	0	11.11	13.68	75.21	0
5	1.30	0	1.30	0	0.65	7.79	24.03	64.28	0.65
6	4.76	0	0.95	0	0.95	5.71	29.53	58.10	0
7	1.75	0	0	0	1.75	1.75	36.85	54.39	3.51
8	0	0	2.44	0	0	13.41	26.83	57.32	0

and unfused parts with porosity 40−60% (Figure 8C). Figure 8D shows the crassisphere unburned char with the internal network structure, which is partially porous. The unburned char shows a spherical form (Figure 8E,F), and the difference is that Figure 8E is a crassisphere, while Figure 8F is a tenuisphere. As seen in Figure 8G−I, the unburned char and sinter are integrated, even if the unburned char with a small particle size is melted into the sinter (Figure 8I), which makes it difficult to burn out.

According to the classification system for combustion chars,[30] the petrographic composition of the unburned char in the sintered ash is shown in Table 3.

The unburned char had little optical anisotropy; this indicates that the coke breezes in the fuel are almost burned out, and most of the unburned chars come from semi-coke. This is due to the small particle size of coke breeze, which makes the breeze-particle fuel burn faster. Specifically, it can be observed from Table 3 that most of the unburned chars are inertoid, mixed dense, and mixed porous. These unburned chars are dense, highly granular, thick-walled, and have low porosity, resulting in a low fuel burn rate. These unburned chars may be derived from the semi-coke (Figure 3D,E) originating from the fusinite of the raw coal, whereas oxygen diffusion to, and within, the vesicular particle causes it to be more quickly burnt than the dense particle. The network or spherical unburned chars with high porosity, high internal surface area, and thin pore walls are easy to burn out, so only a small amount is observed. This is in agreement with the results of Bailey,[43] who found that inertoid chars contributed most mass to the unburned carbon in the combustion residue. This proves that the characteristics of semi-coke will significantly influence the burnout rate. Since the form of the semi-coke affects the combustion process, in order to improve the burnout rate of the semi-coke, the particle size and petrographic characteristics of the semi-coke must be adjusted accordingly. In addition, most of the unburned chars are encased in the sinter to prevent them from burning, which is one of the reasons for the low fuel burn rate.

4. CONCLUSIONS

In this paper, the use of optical microscopy to observe semi-coke and its changes of petrographic characteristics during sintering was studied. Semi-coke displayed isotropy, and even after the DTF and sintering process, the semi-coke still retained its optical properties. The optical structure and morphology of the semi-coke were clearly displayed by the optical microscope, and the petrographic characteristics of semi-coke with a different porosity, wall thickness distribution, pore structure, and optical properties were sufficient to distinguish from coal and coke.

The unburned char in fly ash indicates inefficiency in combustion. Most of the unburned semi-cokes with isotropy were inertoid, mixed dense, and mixed porous, and the form of semi-coke significantly affected the burnout rate. Meanwhile, most of the unburned chars were melted into the sinter, which make them difficult to burn out and lead to inefficient combustion. This article showed the method of optical microscopy to research semi-coke and the unburned char of the sintered ash. The petrographic analysis of fly ash can provide the same details, which can help to better understand their combustion behavior. The knowledge of the origin and types of unburned char indicates that the burnout performance can be improved. Finally, it provides a quick and easy method to study the combustion of fuel, and can be used to choose semi-coke as an alternative fuel and optimize the ratio of the fuel structure in the iron and steel industry.

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Notes

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