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# Simultaneous investigation of coal ignition and soot formation in two-stage $O_2/N_2$ and $O_2/CO_2$ atmospheres



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# ABSTRACT

Due to the intermittent nature of most renewable energy sources, the flexible oxy-coal combustion technology equipped with carbon capture, utilization, and storage (CCUS) will play an indispensable role in achieving carbon neutrality. In this work, we fundamentally investigated coal stream ignition and soot evolution in a series of staged  $O_2/N_2$  and  $O_2/CO_2$  atmospheres for two kinds of coal samples. A novel two-stage flat-flame burner can provide a controlled temperature (1500 K) and atmosphere that are comparable to those in practical boilers. We incorporated in-situ Mie scattering of coal particles, visible light of coal flame, laser-induced incandescence (LII) of soot, as well as online soot particle sampling (in combustion) and gas analysis (in pyrolysis). Compared with conventional  $O_2/N_2$  atmosphere, the  $O_2/CO_2$  case under the same oxygen fraction ( $X_{O2} = 0.3$ ) substantially delays the ignition of coal streams (i.e., 10.1 ms for lignite and 8.9 ms for bituminous coal) on the more representative two-stage burner, and reduces soot volume fraction in the coal flame (i.e., by 26% for lignite and 48% for bituminous coal). The growth and oxidation of primary soot are retarded by the  $O_2/CO_2$  ambience, which might be attributed to the lowered coal/char surface temperature and the smaller O<sub>2</sub> diffusion coefficient in oxy-mode. In particular, we find that elevating oxygen fraction reduces soot volume fraction in the O2/N2 case, but exceptionally promotes soot formation in the  $O_2/CO_2$  case. When the effect of coal rank is concerned, the bituminous coal ignites much later than the lignite due to less amount of combustible light gases released during pyrolysis. The lowered local temperatures in bituminous coal combustion lead to reduced soot forming ability in the  $O_2/CO_2$  ambience, but in  $O_2/N_2$  conditions, burning bituminous coal is sootier.

# 1. Introduction

In the 2050s, coal generation is projected to still account for a nonnegligible fraction in some developing countries where coal is now dominant [1]. But there will be a complete role reversal with renewables dominating electricity production and coal generation stabilizing the grid. With surging economic pressures, coal-fired power plants are burning more often low-rank coals which are abundant and low cost [2]. To achieve carbon neutrality, oxy-coal combustion is regarded as one of the promising technologies [3–7]. Substitution of ambient N<sub>2</sub> in the air with CO<sub>2</sub>, either by flue gas recirculation or introducing pure oxygen as the oxidizer, facilitates efficient centralized CO<sub>2</sub> processing in the power plant. However, the change of atmosphere greatly affects the physicochemical processes in the boiler, posing huge challenges to flexible operation of the unit and calling for lasting fundamental research efforts for flexible oxy-coal combustion.

The key to unit flexibility is the flame stability at reduced outputs, which is strongly associated with entrained coal stream ignition in the hot ambience. Compared to air combustion, this is even more crucial in oxy-coal combustion because of weakened ignition and coal/char reaction caused by the physicochemical effects of  $CO_2$ , including thermal conductivity, specific heat capacity, oxygen diffusion rate, and chemical reaction activity [8–13]. It was found that, when replacing N<sub>2</sub> with CO<sub>2</sub>, the presence of CO<sub>2</sub> delays coal particle ignition and lowers the coal/char surface temperature [8–10]. As for the coal rank effect, a single-particle-based study revealed that heterogeneous ignition is enhanced with increasing coal rank for both air- and oxy-combustion conditions [11].

Another closely related early-stage process in coal combustion is the formation, evolution, and oxidation of soot (or termed black carbon).

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Received 16 October 2021; Received in revised form 29 November 2021; Accepted 1 December 2021 Available online 9 December 2021 0016-2361/© 2021 Elsevier Ltd. All rights reserved. Soot has a remarkable effect on coal flame temperatures in the furnace due to radiation and involves in the  $NO_x$  conversion pathway [14–16]. Once emitted, soot itself is a risky air pollutant causing global warming and human cancer [17,18]. Different from gaseous and liquid fuel combustion where soot is formed through the HACA (hydrogenabstraction-carbon-addition) mechanism by small molecules [19,20], coal-derived soot is mainly generated in the high-temperature fuel-rich zone by large-molecular-weight tar released during devolatilization [14]. Numerous studies have been carried out to investigate the effects of coal rank [21,22], temperature [14,23], atmosphere [24-27], and minerals [16,23,28,29] on soot formation. It should be pointed out that soot evolution and coal stream ignition are both consequences of volatile releasing outwards and oxygen diffusion inwards. But unlike ignition, the effect of CO<sub>2</sub> ambience on soot formation in oxy-coal combustion is not even qualitatively clear. It was found that, in a 100 kW combustor burning bituminous coal, the soot yield is reduced at low stoichiometric ratios under oxy conditions, which may be attributed to lower particle temperatures in oxy-combustion [30]. By contrast, in a 100 kW propanefired oxy-fuel furnace, the soot volume fraction becomes greater with increasing oxygen concentration in the oxidizer when the overall oxygen-to-fuel equivalence ratio keeps constant [31]. Again, this could result from the higher temperature. On the other hand, temperaturecontrolled coal pyrolysis experiments revealed increased soot yield in the CO<sub>2</sub> atmosphere than in the N<sub>2</sub> atmosphere, and the resulting soot reactivity is distinct under varied atmospheres [25,32]. Hence, temperature-controlled combustion experiments are needed for coals of different ranks to clarify the ambience effect on soot formation but are rarely reported so far.

For this purpose, the flat flame burner turns out to be an appropriate option due to the ability to control temperature and atmosphere independently [33]. Previous works on the coal-fired flat-flame burner identified soot as a major component of early-stage ultrafine particles in air conditions [34]. In the fuel stream injected into the boiler, particles are subject to a local reducing atmosphere before entering the bulk oxidized ambience [35]. This process affects ignition and soot formation prominently, but cannot be properly manifested in the existing single-

stage flat-flame burner. To address this issue, we designed a novel two-stage Hencken burner with two concentric surrounding flames that can be configured in any reducing-to- $O_2/N_2$  or reducing-to- $O_2/CO_2$  ambience. This novel device has preliminarily demonstrated its ability to study early-stage particulate matter (PM) formation in air combustion [36] and is promising to elucidate the unclear soot formation characteristics in oxy-coal combustion for coals of different ranks.

Based on this powerful setup, we performed thorough investigations of ignition and soot evolution for lignite and bituminous coal in a series of carefully designed, staged O<sub>2</sub>/N<sub>2</sub>/CO<sub>2</sub> atmospheres. Visual light signals of the intensified-CCD (ICCD) camera were processed to evaluate the ignition delay time, which gives clues to early-stage devolatilization processes. In-situ laser-induced incandescence (LII) and Mie scattering were applied to investigate the evolution and spatial distributions of primary soot and injected coal particles, respectively. Thermophoretic sampling, together with TEM (Transmission Electron Microscope) characterization, revealed detailed morphologies of soot particles. Finally, the influence of coal rank was studied for coal samples having similar volatile contents, and the devolatilization process and products were carefully examined to interpret our findings.

# 2. Experimental section

#### 2.1. Two-stage flat-flame burner and experimental conditions

Fig. 1 depicts the novel two-stage flat-flame burner and the coal feeder. The two-stage Hencken-type flat-flame burner consists of four chambers as fuel and oxidizer passages, and two coaxial cylindrical honeycombs fixed with hundreds of tiny tubes (inner diameter 1.2 mm). Two supporting flame sheets, both being spatially uniform in temperature and composition, are formed in the outer ( $\Phi$  80 mm) and inner ( $\Phi$ 13 mm) regions [16,33]. Pulverized coal stream is fed in from the central tube with an inner diameter of 2 mm by a stepping feeder. The coal feed rate is precisely controlled by the motor stepping rate of the feeder, and the coal carrier gas (nitrogen) was fixed at 188 mL/min to ensure a nearly consistent post-flame speed of the injection with the flue gas.



Fig. 1. Schematics of the two-stage flat flame burner and the coal feeder.

The advantage of the burner is that the flowrates of fuel gas (CO in this work), O<sub>2</sub>, and balance gas (either CO<sub>2</sub> or N<sub>2</sub>) entering the four passages are controlled separately, making it possible to reproduce the reducing-to-O2/CO2 or reducing-to-O2/N2 ambience the coal stream experiences in the boiler. Fig. 1 shows a typical snapshot of coal flame in staged atmospheres. Four different atmospheres denoted N1, N2, C1, and C2 were designed for this work. Cases N1 and N2 represented air combustion, whereas Cases C1 and C2 replaced nitrogen with CO<sub>2</sub> as the balance gas. Table 1 lists the flow rates of all gases (except the coal-laden gas) into the inner and outer zones. The flowrates were such chosen that the constant-pressure adiabatic flame temperature was kept as 1500 K and the post-flame speed as 1 m/s in both regions of all cases. The molar fraction of oxygen downstream of the flat flame sheet is denoted  $X_i$ ,  $o_2$ . and  $X_0$ ,  $o_2$  for the inner and outer region, respectively. With different values of  $X_{i}$ ,  $o_{2}$ , and  $X_{0}$ ,  $o_{2}$ , the investigated cases featured reducing-to-O<sub>2</sub>/N<sub>2</sub> and reducing-to-O<sub>2</sub>/CO<sub>2</sub> environments for entrained coal particles.

Fig. 2a plots the post-flame gaseous temperature as a function of the height above the burner (HAB). A 0.5-mm bare B-type thermocouple was used for the measurement, and the radiation loss has been amended [37]. For all cases, the temperature is close to the designed value of 1500 K and keeps nearly constant for as long as 30 mm beneath the burner rim.

To convert HAB into the residence time of coal particles, we measured the coal particle up-flow velocity through diving the trajectory length of coal particles in the images taken by a Nikon D300s camera by an exposure time of 1/200 s for all cases [38]. As shown in Fig. 2b, the pulverized coal particles have accelerated from 0 to 70 mm and then decelerated further downstream (70–90 mm). Expansion of the carrier gas and coal devolatilization are likely the causes for the initial acceleration. Also given in Fig. 2b is the obtained profile of particle residence time for *Case* **N1**.

A thermophoretic sampler was used to collect combustion-generated ultrafine PMs. Details of the sampling system are referred to [39]. In most cases soot particles on the centerline (with reducing or low oxygen atmosphere) of a certain HAB were sampled, while in some cases the probe collected particles in the outer oxidizing region of the same HAB by adjusting the travel distance of the probe. The microstructure of sampled soot particles was visualized through a TEM (model JEM-2010) for further analyzing the impact of atmosphere on particle morphology.

#### 2.2. Coal properties and devolatilization tests

Low-rank Zhundong lignite (denoted ZD) and bituminous coal (denoted SH) were used in this study. The coal properties are listed in Table 2. The two coals contain almost the same content of volatile matter. The raw coal was ground and sieved, and the size cut of 65–74  $\mu$ m was used for combustion experiments. The coal feed rate was set at 0.06 g/min ( $\pm$ 2%). We further characterized the size distribution of sieved samples by Mastersizer 2000, indicating  $d_{0.5}$  as 24.59  $\mu$ m and the volume-weighted mean diameter as 44.13  $\mu$ m.

To obtain deeper insights on the coal rank effect, Thermogravimetry (TG), Mass spectrum (MS) and Fourier transform infrared spectrometer

Table 1			
Gas flowrates in	n the	investigated	cases

Case	Oxygen molar fraction	CO (SLM)	O <sub>2</sub> (SLM)	N <sub>2</sub> (SLM)	CO <sub>2</sub> (SLM)
N1	$X_{i}o_2 = 0.0$	0.34	0.17	0.99	0.00
	$X_o, o_2 = 0.2$	6.64	11.11	24.52	0.00
N2	$X_i, o_2 = 0.0$	0.34	0.17	0.99	0.00
	$X_o, o_2 = 0.3$	6.64	15.00	20.63	0.00
C1	$X_i, o_2 = 0.0$	0.28	0.14	0.00	1.04
	$X_{o}, o_2 = 0.3$	7.79	15.58	0.00	19.47
C2	$X_i$ , $o_2 = 0.1$	0.35	0.24	0.00	0.19
	$X_{\alpha}o_2 = 0.3$	7.79	15.58	0.00	19.47



**Fig. 2.** (a) Ambient temperature as a function of the height above burner (HAB) for all cases; (b) Typical profiles of coal particle velocity and residence time as a function of HAB for *Case* **N1**.

Table 2 Coal properties.

	ZD lignite	SH bituminous coal
Proximate analysis (wt.%, dry b	asis)	
Fixed carbon	63.54	62.89
Volatile matter	30.58	29.96
Ash	5.88	7.15
HHV (MJ/kg)	28.83	29.88
Ultimate analysis (wt.%, dry an	d ash-free basis)	
С	71.60	80.93
Н	3.16	4.63
N	0.78	2.07
S <sub>total</sub>	0.52	0.71
O (by difference)	23.94	12.37
Ash composition (wt.%, ash bas	is)	
SiO <sub>2</sub>	28.53	54.23
Al <sub>2</sub> O <sub>3</sub>	3.27	19.97
CaO	32.78	1.51
SO <sub>3</sub>	21.47	20.81
Na <sub>2</sub> O	6.19	0.14

(FTIR) were applied in combination to study the thermal conversion (especially devolatilization) of the coal samples [40,41]. The TG-MS-FTIR analyses of ZD and SH coals were employed in  $N_2$  atmosphere with a heating rate of 50 K/min.

#### 2.3. Two-dimensional optical diagnostics system

Fig. 3 shows the in-situ diagnostics system used in this work, including Mie scattering, LII, and flame intensity. The 5 Hz Nd: YAG laser generator emits radiation at 532 nm. The laser energy was set at 120 mJ/pulse so that the maximum intensity of the LII signal can be achieved in the coal flame, following the method used in [42]. A group of cylindrical lenses with a focal length of 50 mm converted the light source into a laser sheet with energy uniformly distributed in a vertical area of 60 mm  $\times$  1.9 mm. An ICCD camera (Princeton Instruments, PIX-



Fig. 3. Schematic illustration of optical diagnostics of Mie scattering, LII, and flame intensity.

MAX4 emlICCD, 1024  $\times$  1024 pixels) perpendicular to the laser sheet was triggered by the laser generator simultaneously and recorded the two-dimensional optical signals of the flame. The Mie scattering signals of coal particles were detected through a 532-nm filter (Thorlabs FL532-10, CWL = 532  $\pm$  2 nm, FWHM = 10  $\pm$  2 nm) in front of the ICCD, whereas the LII signals were collected through a 405-nm filter (Thorlabs FB405-10, CWL = 405  $\pm$  2 nm, FWHM = 10  $\pm$  2 nm). To eliminate the noise signals from polycyclic aromatic hydrocarbons (PAHs) interference and coal particle reflection, we carefully adjusted an ICCD camera detection delay time to be 200 ns after the laser emitted. The gate width was set as 70 ns, and the gain of ICCD was set to 5000. For flame intensity signals (to determine the ignition delay time), we used no laser or any filter before the ICCD camera.

For each measurement, the exposure time of the ICCD camera was set as 20  $\mu$ s to acquire instantaneous 2-D pictures. We then averaged 100 images continuously taken to improve the signal-to-noise ratio (see Fig. 4a). The margin of error is less than 5% for each signal. The axial time evolution of measured signals was derived by summing the radial signals at a fixed HAB (see Fig. 4b).

# 3. Results and discussion

# 3.1. The effect of ambience on soot formation and coal stream ignition

In this section, we performed systematic investigations on earlystage soot formation and coal ignition in the combustion of ZD coal at



Fig. 4. (a) 2-D spatial distributions and (b) normalized 1-D time evolution of Mie, LII, and flame intensity signals in the combustion of ZD coal for *Cases* N1, N2, C1, and C2 at a post-flame temperature of 1500 K produced by the burner. The gray line in (b) is the signal obtained in the absence of coal steam, while other experimental conditions remain unchanged. The shaded areas highlight the stages of initial heating up, devolatilization/soot formation, and soot oxidation/ignition, respectively. We remark that in the experiments, the laser sheet only covered the area with HAB greater than 5 mm to avoid reflection/scattering interference by the burner. Consequently, the Mie scattering signal at the very initial stage with HAB less than 5 mm cannot be recorded, but the initiation of both LII and flame intensity signals is not affected in any case. Also note that in (b) the LII signal intensities are normalized by the maximum of *Case* N1, while other signals in (b) the Mie and Flame signal intensities are subject to min–max normalization by the extreme values of each curve.

1500 K. The effect of ambience is revealed with the aid of several in-situ optical diagnostics including Mie scattering [42], LII [42,43], and flame intensity [10,38] signals. Fig. 4a and 4b show, respectively, the 2-D spatial distribution and normalized 1-D time evolution of Mie scattering, LII, and flame intensity signals during stable combustion (ambience 1500 K) for the investigated atmospheres (see Table 1). Note that in Fig. 4b, the LII signal intensities are normalized by the maximum of Case N1, while other signals (Mie and flame intensity) are subject to min-max normalization using the extreme values of each curve. The background signals for each measurement are also presented in Fig. 4b as grey lines, implying that the LII and flame signals at the very early stage (between 0 and 10 ms) are not statistically distinguishable from the noise. Therefore, we define the characteristic ignition time when the flame intensity reaches 10% of the peak, as marked in Fig. 4b-Flame [38]. A similar definition is given for the LII initiation time, as marked in Fig. 4b-LII.

First, it is seen that the Mie scattering signal of coal streams has the maximum value at the burner surface, decreases along the time for all cases, and almost vanishes at a position of  $\sim$  55 ms. This continuous decrement is due to the coal particle consumption by devolatilization, ignition, and combustion [24,42,44]. By contrast, the LII signal, positively correlated to soot volume fractions, and the flame intensity both emerge after some tens of milliseconds. In terms of coal stream ignition, it is found that increasing oxygen concentration from Case N1 (inner reducing, outer 0.2O2-0.8N2) to Case N2 (inner reducing, outer 0.3O2-0.7N<sub>2</sub>) drops the ignition delay time by  $\sim 1 \text{ ms}$  (see Fig. 4a). The same trend exists in the O2/CO2 ambience where the ignition delay time of *Case* C2 (inner 0.1O<sub>2</sub>, outer 0.3O<sub>2</sub>-0.7CO<sub>2</sub>) drastically reduces by  $\sim 11$ ms as compared with that of Case C1 (inner reducing, outer 0.302-0.7CO<sub>2</sub>). When switching from Case N2 to C1, however, it is found that the flame brightness is reduced, and the ignition delay time largely increases by  $\sim 10$  ms for O<sub>2</sub>/CO<sub>2</sub> ambience. Previous studies on singlestage atmospheres reported the difference of ignition delay times between  $O_2/N_2$  and  $O_2/CO_2$  ambience no greater than 5 ms at the oxygen molar fraction of 0.3 [45]. Thus, our two-stage flat-flame burner tends to make the difference more prominent due to the presence of inner reducing zones. This is closer to the time-atmosphere history of coal particles in the practical boiler.

The LII intensity in Fig. 4b reveals the dynamic process of soot evolution which roughly coincides in time with coal stream ignition for all cases: The rise of LII signal occurs ahead of coal stream ignition, implying an accumulation of soot volume fraction in a local reducing atmosphere; After ignition the LII signal rapidly falls below the detection limit, reflecting massive oxidation by the oxygen diffusing in. Note that the soot formed between  $\sim 15$  to  $\sim 40$  ms even acts to slow down the declining rate of Mie scattering curves. As for the ambience effect, it is seen that in Case N2, the peak of the LII curve decreases by  $\sim$ 5% and occurs  $\sim 3$  ms ahead as compared to Case N1. However, in the O<sub>2</sub>/CO<sub>2</sub> ambience, the LII peak of Case C2 emerges ~6 ms earlier than that of Case C1 and has increased by  $\sim 17\%$ . While previous works tried to clarify the role of oxygen, only on the two-stage burner can the oxygen fraction be manipulated more precisely, and its effect in O2/CO2 ambience be clearly revealed. The inner stage oxygen in Case C2 promotes volatile release by increasing the coal particle heating rate and surface temperature and thus accelerates both soot formation and oxidation [8,10,12]. It then causes the earlier emergence of a higher LII peak, as compared with Case C1.

Furthermore, a comparison between *Cases* N2 and C1 gives clues to the effect of  $O_2/CO_2$  ambience. It is found that in *Case* C1, the LII signal intensity becomes weakened with the peak reduced by 36% and delayed in time by ~4 ms, implying an inhibition effect of  $CO_2$  ambience on total soot yield in coal combustion. The  $O_2/CO_2$  ambience has led to a lower coal/char surface temperature, a lower particle envelope flame temperature, and a remarkably delayed ignition [10,26]. On the other hand, it was found in temperature-controlled coal pyrolysis works that more tar and soot can be produced in  $CO_2$  ambience than in  $N_2$  ambience due to chemical effects [25,32]. If this still holds in coal combustion, one then expects some soot particles to be oxidized more slowly in  $O_2/CO_2$  ambience, even though the total accumulated soot volume fraction is less than the  $O_2/N_2$  condition. We need to resort to particle sampling and characterization to verify this assumption.

#### 3.2. Soot characterization in reducing-to-oxidizing atmospheres

Here we sample and characterize soot particles morphology for all cases based on the procedure described in Section 2.1. Fig. 5 shows typical ZD-derived soot sampled in Case C1 (inner reducing, outer  $0.3O_2$ - $0.7CO_2$ ) and HAB = 20 mm (corresponding to a residence time of 26 ms). The mature soot agglomerate collected in the inner reducing zone (see Fig. 5a) is fractal and consists of spherical primary particles ranging from 20 to 60 nm in size. The magnified high-resolution TEM image of the primary soot (Fig. 5b) reveals concentrically-stacked graphitic layers and a disorganized core. By contrast, the soot sampled in the outer oxidizing region (see Fig. 5c) becomes much tinier in size, and the high-resolution image in Fig. 5d shows no typical graphitic layers, implying that soot is oxidized, or that the precursor fails to support nascent particles to grow. Previous research has shown that compared with an oxygen-free atmosphere, the soot yield decreased in a downward flat-flame burner at a post-flame atmosphere of 1500 K and 20% O<sub>2</sub> [28]. The released tar made up by PAHs is the building block of coal-derived primary soot via high-temperature secondary reactions at the initial stage of combustion [14]. Two competitive pathways for primary tar, polymerization, and thermal cracking, are crucial for soot formation [14]. In the reducing zone, the mature soot chain larger than 100 nm in size can be readily produced through successive processes of tar polymerization, soot nucleation (see the core in Fig. 5b), surface growth (ordered shell in Fig. 5b), and agglomeration. Whereas, in the oxidizing O2/N2 and O2/CO2 region, tar and soot are oxidized, prohibiting the growth of primary soot, as exhibited in Fig. 5c&d.

We further analyze the size of primary soot collected on the centerline to reveal the competition between soot formation/growth and oxidation. As shown in Fig. 6a, we approximated the primary soot by the ellipse fitting (yellow line), and the circle-equivalent particle diameter  $(d_{p})$  is defined as the mean value of its major axis *a* and minor axis *b*:  $d_p = (a+b)/2$ . More than 400 primary soot particles from different soot aggregates were counted to derive the average diameter (in Fig. 6b) and size distributions (in Fig. 6c) for each sampling position (HAB = 20 mm, 50 mm, 80 mm) and all cases N1, N2, C1, and C2 (see Table 1). Notice that the statistics in Fig. 6b&c are biased in that we excluded any tiny oxidized residuals (usually smaller than 10 nm) whose boundary is difficult to distinguish even in high-resolution TEM images; see a typical example in Fig. 5c&d. In addition, trace quantities of soot can be detected through thermophoretic sampling at HAB = 80 mm even when the concentration drops below the detection limit of LII (see Fig. 4a). In this way, the results presented here describe the evolution of larger, spherical primary soot particles.

It is first seen that in the  $O_2/N_2$  ambience (*Cases* N1 and N2), the average diameter of larger primary soot particle decreases monotonically with the increment of sampling HAB (see Fig. 6b), and the particle size distribution curves move to smaller size fractions (see Fig. 6c). In particular, for HAB greater than 50 mm, soot particles larger than 80 nm vanish; For HAB = 80 mm, the most likely size drops from ~55 nm (at HAB = 20&50 mm) to ~ 40 nm. This is a consequence of soot oxidation due to a higher fraction of oxygen diffusing inwards. Compared with *Case* N1, *Case* N2 with a higher surrounding oxygen fraction features a larger primary soot size at all sampling HABs, but the relative difference between two cases rapidly shrinks with increasing HAB (8.7% for HAB = 20 mm, 5.6% for HAB = 50 mm, and 1.3% for HAB = 80 mm). This again verifies that oxygen promotes both soot particle growth and oxidation.

When it comes to  $O_2/CO_2$  ambience (*Cases* C1 and C2), a different pattern emerges. For both cases, the average diameter of larger primary



Fig. 5. Morphology of soot agglomerates sampled in *Case* C1 for ZD coal at a post-flame temperature of 1500 K produced by the burner and HAB = 20 mm: (a & b) soot sampled in the inner reducing region; (c & d) Soot collected in the outer oxidizing region.



**Fig. 6.** ZD-derived primary soot under varied atmospheres **N1**, **N2**, **C1**, and **C2**: (a) Morphology of nearly-circular primary soot, whose boundary is fitted by an ellipse (the red curve) and the circle-equivalent particle diameter ( $d_p$ ) is defined as the mean value of its major axis *a* and minor axis *b*; (b) Average diameter of primary soot particle in all cases; (c) Size distribution of primary soot particle in all cases.

soot increases from HAB = 20 mm to 50 mm, and then decreases from HAB = 50 mm to 80 mm. The particle size distribution Fig. 6c shows that in *Case* C1, the 60-nm fraction increases from HAB = 20 mm to 50 mm, while greatly reduces at HAB = 50 mm. The same trend can be observed for the fraction ranging from 60 nm to 90 nm in *Case* C2. In addition, the average diameter of larger primary soot in *Case* C1 is smaller than *Case* N2 at all sampling HABs. This distinct pattern under  $O_2/CO_2$  condition can be interpreted as a combined effect of temperature and oxygen concentration. Because of the lower heating rate and coal surface temperature in *Case* C1, devolatilization is slower at the most initial stage, resulting in a smaller average diameter of primary soot at HAB = 20 mm. Then, from HAB = 20 mm to 50 mm during which the LII signal is dropping significantly (see Fig. 4b), the lower diffusion rate of oxygen in  $O_2/CO_2$  mixtures, as well as the delayed volatile release, still permits the

existence of micro reducing environments for some primary soot to grow. The trace amount of soot particles is finally being oxidized from HAB = 50 mm to 80 mm.

In a nutshell, for the first time, we reveal that the  $O_2/CO_2$  ambience prohibits the accumulation of soot volume fractions in ZD-coal flame under a post-flame temperature of 1500 K, and retards the growth and oxidation of some primary soot particles. The lowered coal surface temperature and the smaller diffusion coefficient of  $O_2$  in  $O_2/CO_2$  (than in the  $O_2/N_2$  atmosphere) may be the main causes. More detailed characterization of the coal flame temperature is thus needed to clarify the evolution and oxidation of coal-derived soot. This will be addressed in the future work.

# 3.3. Effect of coal rank on coal stream ignition and soot formation

We are now in a position to elucidate the effect of coal rank by studying two samples, ZD lignite, and SH bituminous coal, with close volatile contents. Fig. 7 exhibits the normalized 1-D time evolution and 2-D spatial distributions of LII and flame intensity signals at 1500 K and varied atmospheres. Note that the LII curves in Fig. 7a&c are normalized by the peak value SH in Case N1, while each flame intensity curve in Fig. 7b&d is subject to min-max normalization by its extreme values. Table 3 lists the characteristic times for coal stream ignition and soot formation. We first remark that the ambience effects on ignition and soot evolution of SH coal combustion are the same as on ZD coal. Elevating oxygen concentration accelerates ignition and the emergence of LII peak for both O<sub>2</sub>/N<sub>2</sub> and O<sub>2</sub>/CO<sub>2</sub> atmospheres. A higher oxygen fraction reduces the soot volume fraction under the O2/N2 condition but contributes to soot accumulation for the  $O_2/CO_2$  atmosphere. When switching from O<sub>2</sub>/N<sub>2</sub> (Case N2) to O<sub>2</sub>/CO<sub>2</sub> atmosphere (Case C1), ignition delay time surges, and the LII peak is delayed with a value reduced by 48%.

The comparison between ZD and SH coals helps reveal the coal rank effect. Most remarkably, the ignition delay time of SH is increased by 2 ms, 3 ms, 2 ms, and 11 ms in Cases N1, N2, C1, and C2 as compared with ZD coal, whereas the LII peak of SH coal is delayed by 8 ms, 9 ms, 3 ms, and 11 ms in Cases N1, N2, C1, and C2. In hot ambience, coal ignition depends on surrounding gas temperature, oxygen concentration, and volatile concentration [8]. In our experiments, the surrounding gas temperature and oxygen concentration are similar for ZD and SH combustion in the same case, while the devolatilization process is different. As will be revealed later in TG-MS-FTIR analyses, ZD produces more light gases such as CH<sub>4</sub> and CO than SH during the pyrolysis stage. Thus, ZD is easier to lignite. Besides, SH coal is less productive in soot than ZD in oxy Cases C1 and C2 (see the LII peak), which can be attributed to the lowered local temperatures. However, in O2/N2 Cases N1 and N2, SH generates more soot than the lignite, indicating that other factors are playing a prominent role. And extra efforts are required to explore the early-stage devolatilization process.

For this purpose, we performed integrated TG-MS-FTIR analyses of the coal samples in  $N_2$  ambience. Such characterization gives clues to the

species and chemical bonds of coal devolatilization products, which are difficult to measure directly on the flat-flame burner. We shall remark that the joint use of bench-scale furnaces and TGA measurements is still a common practice in state-of-the-art coal combustion/pyrolysis studies [46,47]. Fig. 8 presents the TG-DTG curves with a heating rate of 50 K/ min. Three weight-loss stages in the DTG curve can be identified for both coal samples, with the parameters listed in Table 4. Stage #1 at 323  $\sim$ 521 K for ZD and 323-521 K for SH corresponds to the moisture release. Stage #2 at 514–795 K for ZD and 521–945 K for SH generate tar as the dominant building block of coal-derived soot [14]. Therefore, the much larger DTG value for Stage #2 during SH coal devolatilization implies a greater capacity of producing soot precursors at the early stage of combustion, as compared with ZD coal. Stage #3 at 795-1500 K for ZD and 945-1500 K for SH is associated with secondary devolatilization during which tar cracking and char transformation occurs [48,49], and the mass weight loss of ZD is about 4 times that of SH at this stage. Because SH coal has a higher temperature and a smaller DTG value corresponding to Stage #3, it is reasonable to infer that the tar generated by SH is more stable than that of ZD. With a less propensity to decompose in the early stage of combustion, the SH-generated tar, more abundant than ZD, can fuel a more intensive accumulation of soot in the flame, as manifested in Fig. 6.

The main gaseous products during coal pyrolysis are analyzed by MS, as shown in Fig. 9. The first stage of weight loss between 323 and 521 K (corresponding to Stage #1 in Fig. 8 and Table 4) mainly produces H<sub>2</sub>O for both coals. Notice that the peak value of ion current of H<sub>2</sub>O in ZD pyrolysis is more than twice that of SH, which is consistent with the weight loss from TG results. Also formed in the first stage are light gases of CH<sub>4</sub>, H<sub>2</sub>, CO, and CO<sub>2</sub> during ZD coal pyrolysis, while little light gases can be detected for SH coal at this stage. The ease of ZD to generate light gases can be a cause for the shorter ignition delay time than SH coal in any atmosphere (see Fig. 6). The amount of CO<sub>2</sub> from ZD reaches its peak at around 700 K, while for SH, the amount of CO<sub>2</sub> is much smaller. In devolatilization, CO<sub>2</sub> is generated by the thermal decomposition of oxygen-containing functional groups such as carboxylic acid, ester, and acid anhydride groups [51]. Hence, the distinct CO<sub>2</sub> yields reflect the difference in the type and content of oxygen-containing functional



**Fig. 7.** Time evolution of LII and flame intensity signals under varied atmospheres **N1**, **N2**, **C1**, and **C2**: (a) ZD, LII; (b) ZD, flame intensity; (c) SH, LII; (d) SH, flame intensity. Insets are the two-dimensional spatial distributions of LII and flame intensity signals. Note that all LII signals in (a) and (c) are normalized by the peak value of SH in *Case* **N1**, while each flame intensity curve in (b) and (d) is subject to min–max normalization by its extreme values.

## Table 3

Characteristic times and peak value for ignition and soot formation.

	Ignition	delay time	e (ms)		LII initiation time (ms)			LII peak time (ms)			LII peak value (a.u.) <sup>a</sup>					
	N1	N2	C1	C2	N1	N2	C1	C2	N1	N2	C1	C2	N1	N2	C1	C2
ZD SH	31.5 33.6	30.5 32.8	40.6 41.7	30.3 42.2	15.7 25.8	13.3 22.3	18.2 28.3	16.8 28.1	37.3 45.6	34.2 43.0	37.6 45.5	31.9 43.3	0.77 1.00	0.72 0.76	0.46 0.28	0.57 0.41

<sup>a</sup> This panel is normalized by the peak value of SH coal in Case N1.



Fig. 8. Thermogravimetric results of (a) ZD and (b) SH coal in  $N_2$  atmosphere under a heating rate of 50 K/min. The DTG curve is the derivative of the mass with time.

#### Table 4 TG-DTG results of coal samples

Weight- loss	Temperatur	e (K)	Maximu value (1	um DTG mg/s)	Description
stage	ZD	SH	ZD	SH	
#1	323–514	323–521	0.030	0.011	Release of free water, bound water, and small molecule volatiles [49].
#2	514–795	521–945	0.065	0.077	Breakage of aromatic alkyl side chains, bridge bonds, and aliphatic chains. Forming tar and light hydrocarbon [49,50].
#3	795–1500	945–1500	0.057	0.027	Secondary devolatilization is associated with thermal cracking of large- molecular-weight species including oil and tar, forming light gases [48,49].



**Fig. 9.** Ion current of fragments versus temperature from TG-MS results of (a) ZD and (b) SH pyrolysis. The mass-to-charge ratio m/e is used to deduce species of the fragment.

groups between the coal of varied ranks, which is consistent with the significant difference in oxygen content of the raw coal samples (see Table 1). The end of the second stage of TG weight loss sees ion current peaks for CH<sub>4</sub>, CO, and H<sub>2</sub>O around 850 K. The peak value of H<sub>2</sub>O from SH pyrolysis is about 3 times that of ZD. The formation of H<sub>2</sub>O at the second stage is due to the thermal decomposition of phenolic OH groups, an important pathway for the evolution of tar/soot precursors [52]. Then, at the third stage of TG weight loss, H<sub>2</sub> reaches its peak at around 1000 K due to secondary devolatilization [53,54].

Fig. 10 shows the FTIR spectrum of pyrolyzing gas products at different temperatures (400–1400 K) for both coal samples. The most likely species have been marked in the figure based on Refs. [40,55,56]. The absorption peak occurs between 400 and 1200 K, especially from 600 to 1000 K, which is consistent with the MS spectrum. Compared with SH coal, the absorption peak of H<sub>2</sub>O (4000–3500 cm<sup>-1</sup>) and CO<sub>2</sub> (2400–2250 cm<sup>-1</sup>, 700–600 cm<sup>-1</sup>) is higher in ZD-generated pyrolyzing gas. By contrast, the absorption peaks of C = O (1900–1650 cm<sup>-1</sup>), benzene skeleton (1590–1450 cm<sup>-1</sup>), and C–H (3100–2600 cm<sup>-1</sup>) are higher for SH coal devolatilization than those from ZD coal, implying more PAHs generation. This again supports our observations that SH coal is more prone to produce soot precursors at the early stage of



Fig. 10. TG-FTIR analysis of the pyrolysis gaseous products of (a) ZD and (b) SH coal.

combustion and thus has higher soot yields than ZD coal in air conditions.

#### 4. Conclusions

We experimentally investigated soot formation and coal stream ignition of different rank coals under carefully designed oxy-fuel  $O_2/N_2$  and  $O_2/CO_2$  atmospheres in a novel two-stage flat-flame burner. An online thermophoretic sampling of soot particles and in-situ optical diagnostics including Mie scattering, LII, and flame intensity are incorporated to obtain insights on the ambience effects. The integrated TG-MS-FTIR analyses are performed to reveal the coal rank effect. We draw the following conclusions.

- (1) Under the same ambient oxygen concentration and temperature (1500 K in this work), the  $O_2/CO_2$  atmosphere delays the coal stream ignition and prohibits the accumulation of soot volume fraction in the flame for both lignite and bituminous coals. The growth and oxidation of primary soot particles are retarded by the  $O_2/CO_2$  ambience. The two-stage flame ambience makes more prominent the difference in ignition delay time between  $O_2/CO_2$  and  $O_2/N_2$ , and this is closer to the practical situation in the boiler.
- (2) With other ambience parameters unchanged, elevating oxygen concentration promotes coal stream ignition for both  $O_2/N_2$  and  $O_2/CO_2$  atmospheres. A higher oxygen fraction reduces soot volume fraction under the  $O_2/N_2$  condition by accelerating soot oxidation but contributes to soot accumulation in the  $O_2/CO_2$  atmosphere, most likely due to the higher coal flame temperature.
- (3) Having similar content of volatile matter, the bituminous coal (SH) ignites later than the low-rank lignite (ZD) because ZD coal is more likely to produce light gases during pyrolysis, such as CH<sub>4</sub> and CO. Under O<sub>2</sub>/CO<sub>2</sub> conditions, burning SH is less sooty than

ZD due to the lowered local temperatures. On the contrary, SH combustion produces more soot in air conditions, which can be attributed to a greater tar yield in bituminous coal devolatilization. Besides, the bituminous-coal-produced tar is likely to be more stable than that from lignite and is thus more efficient in forming soot particles.

#### CRediT authorship contribution statement

**Peng Ma:** Methodology, Data curation, Investigation, Writing – original draft. **Qian Huang:** Conceptualization, Investigation, Writing – review & editing. **Yuanping Yang:** Methodology, Investigation. **Renshan Ji:** Methodology, Resources. **Shuiqing Li:** Conceptualization, Methodology, Funding acquisition, Writing – review & editing.

## **Declaration of Competing Interest**

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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