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Investigating the effect of flue gas temperature and excess air coefficient on the size distribution of condensable particulate matters

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ABSTRACT

Primary particles emitted from fuel combustion mainly involve filterable particulate matter (FPM) and condensable particulate matter (CPM). Particularly, CPM has emerged as a subject for further emission control. This study investigated the effects of the sampling temperature and excess air coefficient (EAC) on the total mass, chemical speciation, and particle size distribution of CPM by integrating Electrical Low-Pressure Impactor⁺ (ELPI⁺) sampling devices with the EPA Method 202 (dry impinger method). The total mass of CPM increased with the sampling temperature and EAC. Specifically, the total concentration of CPM was $10.51-39.93 \text{ mg/m}^3$, in which the mass fraction of organic species varied between 8.74 and 49.80%, and the organic components in CPM followed the ranking order of alkanes/alkenes (62.6–78.6%), oxygen-containing volatile organic compounds (OVOCs) (19.7–35.4%), and aromatics (5.6%). Compared with other inorganic species such as HCl and NO_X, SO₃ had a higher migration tendency from the flue gas to CPM. The particle size distribution suggested that heterogeneous condensation was responsible for the whole size range of particles in CPM, whereas the homogeneous condensation led to the increase of finer particles (smaller than 0.2 µm). Accordingly, adjusting the emission temperature and EAC could help to control the emission of CPM.

1. Introduction

Particulate matter emitted from coal combustion is a continuing environmental issue in China. Total particulate matter (TPM) from fuel combustion involves two contributions: filterable particulate matter (FPM) and condensable particulate matter (CPM) [1]. In the flue gas, while FPM is already in the particle state, CPM initially exists in the gaseous state and is then condensed into liquids or solids at a lower temperature. Emission standards and characterization of TPM have been focused largely on FPM [2,3], of which its emission has gradually dropped thanks to the development of ultra-low emission control technology. In particular, China proposed new regulations in 2014 to enforce "ultra-low emission (ULE)" by setting the emission limit of particulate matter to 10 mg/Nm³ [4].

On the other hand, regulations on the emission of CPM are limited despite its potential to cause atmospheric pollution due to the small particle size (usually less than $2.5 \ \mu m$) [5]. Studies have suggested that

the emission of CPM could be equal to or even higher than that of FPM in some power plants. For example, Pei [6] found comparable emissions of CPM (21.2 \pm 3.5 mg/Nm³) and FPM (20.6 \pm 10.0 mg/Nm³) in three power plants (300, 325, and 1000 MW). Likewise, Corio and Sherwell [7] reported three times higher emissions of CPM than FPM in coal-fired boilers. Notably, Li [8] reported a much higher emission ratio between CPM and FPM (7.9 mg/Nm³ vs. 1.6 mg/Nm³) in an ultra-low emission (ULE) power plant (1030 MW). Noticeably, Zheng [9] showed that CPM accounted for 76.73% of the total particulate concentration from an ultra-low emission power plant. In particular, Cano [10] found that the CPM/FPM concentration ratio was around 1.5 (7.8 mg/Nm³ vs. 3.2 mg/ Nm³). Song [11] investigated CPM emission from an ultra-low emission power plant and showed that the CPM for 76.43%, 77.89%, and 82.96% of TPM at the outlet of LLT-ESP, WFGD, WESP, respectively. Wu [12] conducted measurements at an ultra-low power plant and stated that the CPM court for 59% of TPM. These studies highlight the significant contribution of CPM to the particulate matter, which, in turn, emphasize

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Fig. 1. Experimental setup of the entrained flow reactor and sampling device.

the importance of characterizing CPM in the flue gas.

Characterization of CPM usually follows the U.S. EPA method 202 [13] (dry impinger method), which is a manual wet chemistry method that measures the total mass of CPM and the organic–inorganic mass fraction. Importantly, the EPA method 202 (dry impinger method) and several other variations do not tell the size distribution of CPM, which could be important for evaluating its hazardous effects. For example, the condensation of flue gas could generate CPM particles of different sizes, to which the adsorption of toxic species (e.g., heavy metal and polycyclic aromatic hydrocarbons) could vary substantially due to the difference in specific surface area. Also, compared with larger CPM particles, finer ones could persist longer and travel further in the atmosphere [5,14,15], thus presenting more significant risks to human health.

Here, a modified version of the EPA method 202 was reported for robust characterization of CPM including the size distribution. Compared with the EPA method 202 (dry impinger), the only change in this setup was the replacement of the CPM filter with ELPI⁺, which can divide particulate matter with sizes between 0.006 and 10 μ m into 14 groups and measure the mass and number concentration of particles in each group [16–19]. In addition to the size-specific particle concentration, ELPI⁺ also yields the total mass and number of CPM particles in the flue gas simply by combining results from all 14 groups. Particularly, the total mass of CPM determined by this modified method agreed well with that from the EPA method 202 (dry impinger).

The concentration of CPM from the different coal-fired boilers is different. Small values of excess air coefficient (EAC) could lead to incomplete combustion and thus more organic compounds in CPM, which, in turn, affect the condensation process that forms CPM particles. Using the EAC as a single variable could analyze its contribution to CPM. Outlets of the electrostatic precipitator (ESP), wet flue gas desulfurization (WFGD), and wet electrostatic precipitator (WESP) in power plants usually have different temperatures, ranging from 45 – 60 °C at the WESP, 50–80 °C at the WFGD, and 110–150 °C at the ESP. The temperature variation could cause condensation of the flue gas and reduce the emission of CPM and affect the size distribution of CPM. Most current studies usually only took the effect of different air pollution control devices (APCDs) on the reduction of CPM, which did not consider the temperature effect separately.

This study uses both the EPA method 202 and a modified CPM sample system to analyze the size distribution characteristic of CPM. Two parameters of the combustion process were further investigated to clarify their effects on the CPM properties. Three values of EAC (1.2, 1.4, and 1.8) were chosen to represent conditions in a power plant, heating plant, and industrial enterprise, respectively. Also, the flue gas was sampled at 150, 120, 100, 80, and 50 °C to identify the correlation of CPM size distribution and the flue gas temperature.

2. Experimental section

2.1. The entrained flow reactor

Fig. 1 shows the experimental setup of the entrained flow reactor and the sampling scheme of the flue gas. The feed rate of coal at the coal feeder (top of the furnace) was fixed at 4 g/min. Coal flowed through a 130 mm diameter, 2000 mm long heated tube at 1300 °C to generate the flue gas, while the experimental parameter of the EAC (1.2, 1.4, and 1.8) was controlled by the flow meter to simulate combustion conditions in the coal-fired power plant. After the cyclone separator, the flue gas was sampled at different temperatures (50, 80, 100, 120, and 150 °C) in the entrained flow reactor. The particle size distribution and the



Fig. 2. Comparison of ELPI⁺ results from entrained flow reactor (Result1, Result2, and Result3) and the inlet of SCR at 1000 MW power plant (SCRinlet1 and SCRinlet2).

The concentration of several selected organic compounds in the entrained flow reactor.

Compounds	Concentration(µg/m ³)	Compounds	Concentration(µg/m ³)
acetone	102.22	2-heptanone	7.33
benzene	134.25	Styrene	65.97
N-heptane	2.73	O-xylene	0.29
Toluene	57.56	Anisole	1.72
Ethylbenzene	0.19	Benzaldehyde	14.04
M/p-xylene	0.07	Dodecene	0.73

concentration of organic compounds were collected under the different experimental parameters.

It is challenging to adjust experimental parameters during daily power generation in the coal-fired power plant. Instead, in this study, flue gas was provided by the entrained flow reactor with continuous combustion of coal. This choice was justified based on the following analysis that implied that the physicochemical properties of flue gas from the entrained flow reactor and coal-fired power plant were similar. First, the ELPI⁺ sampling results from the 1000 MW power plant at the selective catalytic reduction (SCR) inlet were similar to those from the entrained flow reactor (Fig. 2). Second, based on the characterization results of organic emission (Table 1), the entrained flow reactor yielded similar organic compounds to those reported previously from the power plant [20,21], but only at a slightly higher concentration that was possibly due to incomplete combustion.

2.2. Sampling methods

Two different methods were used to analyze the CPM in this study. The first one was the EPA method 202 (dry impinger method) alone (Fig. 1a) and the second one was the dry impinger EPA method 202 integrated with the ELPI⁺ sampling instrument (Fig. 1b). Operation details of these two methods are presented below:

2.2.1. EPA method 202 (dry impinger method) [13]

The whole sampling process of 1 m³ of flue gas took 1–1.5 h. Before entering the CPM sampling train, the flue gas first passed through the FPM filtration system involving a nozzle and FPM filter (Advantec thimble filter 86R, which has a greater than 99.9% collection efficiency for particles with a size larger than 0.3 μ m). The heated probe used by the EPA method 202 sampling system was maintained at 120 °C to make sure the gaseous CPM stayed in the flue gas. Before reaching the CPM filter (Whatman glass microfiber filters 934-AH), the flue gas went through a vertical condenser and then passed through a set of two initially empty impingers (dropout and backup) that were immersed in a water bath.

Following the sampling process, compressed N_2 of ultrahigh purity continuously purged the sampling train (more than 14 L/min) for 1 h to reduce the bias caused by dissolved SO₂. After the purge, the glassware (including the probe extension, condenser, impingers, the connecting glassware, and the front half of the CPM filter housing) before the CPM filter was washed by deionized water (twice), acetone (once), and hexane (twice) in series. The water and organic solutions were collected in separate containers. A sonication bath was used to extract particles on the CPM filter with sufficient deionized water and hexane as the extracting medium. Each extraction step took two minutes and was repeated three times.

After all the washing and extraction steps, aqueous (all the deionized water) and organic (all the acetone and hexane) solutions were collected in separate containers. 30 mL hexane was used to extract from the aqueous solution three times. A separating funnel was used to separate these two solutions after each test. The aqueous solution was first heated to 105 $^{\circ}$ C and evaporated to around 10 mL. Then, the remaining water solution and the organic solution were dried in separate containers in the hood at room temperature (not to exceed 30 $^{\circ}$ C). The weight was measured every 6 h until a constant value was obtained. After recording this mass information, the aqueous and organic powders at the bottom of the container were dissolved in water and methanol, respectively, for further inorganic and organic compounds analysis.

2.2.2. EPA method 202 (dry impinger method) integrated with the ELPI+

The information on the size distribution of CPM was obtained with ELPI⁺. In the EPA method 202 (dry impinger method), partial CPM was condensed in the empty impinger before reaching the CPM filter (Fig. 1). Part of the CPM was collected in the CPM filter. In the modified system, this part of the CPM was analyzed by the EPA method 202 (dry impinger method). Meanwhile, CPM leaving the empty impinger into the ELPI⁺ system usually has higher flow-ability than that condensed in the impinger and could be more harmful to the environment. This part of the CPM was analyzed by ELPI⁺ to provide its particle size distribution. These two parts of the characterization were combined to yield more comprehensive information on the formation of CPM. The only difference from the EPA method 202 (dry impinger method) was the replacement of the CPM filter with the ELPI⁺. In this modified method, the condensed flue gas flowed into the inlet of the ELPI⁺ instead of being collected by the CPM filter, while the sampling operation before the CPM filter was the same as the EPA method 202 (dry impinger method). A drying tube was also added before the ELPI^+ to minimize the effect of water vapor.

After being charged to a known level by the corona charger at the inlet of the ELPI⁺, CPM particles in the flue gas were separately collected at 14 impactor stages according to their aerodynamic diameter (Da).

Table 2

Industrial and elemental analysis of the coal sample (w/w %, air dry).

Industrial analysis	ndustrial analysis			Elemental an	alysis			
Moisture	Volatile	Ash	Fixed carbon	С	н	Ν	S	0
3.79	38.70	13.48	44.03	58.57	4.16	0.73	0.64	35.90

During this collection process, charged particles were discharged at each stage and the electrical current was recorded by an electrometer channel, yielding the number and mass concentration of particles at each stage. Adding the ELPI⁺ to the EPA method 202 (dry impinger method) provided a 14-stage characterization of CPM particles between 6 nm and 10 μ m with the number- and concentration-based size distribution.

2.3. Sample analysis

The industrial and elemental analysis of coal is shown in Table 2. Organic and inorganic compounds collected by the EPA method 202 (dry impinger method) and the modified EPA method 202 were extracted by different processes. The gas chromatography/mass spectrometer (GC/MS, model: PE-SQ8) was used to analyze organic compounds and to provide the organic composition of species in the CPM. Inorganic compounds were analyzed by ion chromatography (IC, model: ICS-1100) and inductively coupled plasma atomic emission spectroscopy (ICP-AES, model: Leeman-Prodigy). The determined anions include the concentrations of F, Cl⁻, NO₃, and SO₄²⁻. The elements tested by ICP-AES are Ca, Na, Mg, Al, Fe, K, Cr, Mn, Ti, Sr, Ba, As, Se, Pb, Cd, and Si.

2.3.1. Gc/Ms

A fused silica capillary column with dimethyl polysiloxane (Extile-1, 60 m × 0.25 mm × 0.25 µm film thickness) was used in the GC/MS system. The temperature of the column was first kept at 50 °C for 5 min, then increased to 300 °C at a rate of 10 °C/min, and held at 300 °C for another 5 min. Helium was used as a carrier gas with a flow rate of 1.0 mL/min. The information of the mass spectra was collected with the *m*/z range from 35 to 500. Both the information of the mass spectra and the retention time were used to identify the organic compound in CPM.

When separated by the chromatographic column and detected by the detector, each component in the sample yielded a peak. Analytes were first identified based on their retention time by comparing the obtained mass spectra with the standard from the NIST Mass Spectral Library. The relative amount of each analyte (RA_i) was evaluated with the semiquantitative area normalization method [20],

$$\mathbf{RA}_{i} = \mathbf{A}_{i} / \sum_{i=1}^{n} \mathbf{A}_{i} \times 100\%$$
(1)

where A_i is the area of the peak corresponding to species i in the spectrum of GC/MS and *n* is the total number of detected peaks or species.

2.3.2. ICP-AEs

The ICP-AES equipped with a salt atomizer was used with the following working parameters: the carrier gas pressure at 50 psi (1 psi = 6.895 kPa) and flow rate of the auxiliary gas, cooling gas, and sample lift volume at 0.5 L /min, 19 L/min, and 1.4 mL/min, respectively.

2.3.3. Ic

An ion chromatograph from DIONIX Company was used for analyzing cations in the sample. The eluent involves the mixture of Na₂CO₃/NaHCO₃ solutions. The flow rate of the sample injection was 1.2 mL/min. The type of cation column was IonPic AS23. The type of suppressor was ATRS 300 (4 mm), and the current of the suppressor was 36 mA. The background conductivity was 17 μ s.

2.4. Calculation of species concentrations

The mass concentration of species in the standard state (C_i , mg/m³) is calculated in the CPM following Eq. (2):

$$C_i = M_i / V_i \tag{2}$$

where M_i is the mass (mg) of species i, and V_i is the dry gas volume

 (m^3) under the standard state. Also, the baseline mass concentration of particulate matter (C_i ', mg/m³) in the flue gas is computed using Eq. (3) [22]:

$$C_i' = C_i \times (21 - C(O_2))/(21 - C'(O_2))$$
 (3)

where $C(O_2)$ and $C'(O_2)$ are the measured oxygen content and the baseline oxygen content (6%) [22], respectively.

2.5. Quality assurance and control

Before and after each test, all filters were equilibrated for 48 h under constant conditions (temperature: 20 ± 5 °C, relative humidity: 40 ± 2 %). Then, they were weighed by a balance with a precision of 0.01 mg (Model: Sartorius BT 25 S). Before the analysis, all the samples were stored at 4 °C. Before the experiment, the glassware (including the probe extension, condenser, impingers, the connecting glassware, and the front half of the CPM filter housing) and containers were cleaned with soap and water and rinsed sequentially with tap water, deionized water, acetone, and hexane. All cleaned glassware and containers as mentioned above were dried at 300 °C for 6 h before use.

The airtightness of the system was also checked at each test run. For each condition at the sampling location, three samples were collected. Before each test, a blank sample (without the flue gas) was taken by rinsing the probe extension, condenser, each impinger, the connecting glassware, and the front half of the CPM filter housing twice with deionized water, once with acetone, and twice with hexane (the same process as sampling without flue gas). The average value of the blank sample was taken into consideration to obtain the final analysis results of CPM to exclude contamination from the chemical solutions and operations during the experiments.

The concentration of each element in the CPM sample was identified by ICP-AES, in which each element has its correlation coefficient and detection limit. The calibration details are shown in Table S1. 11 duplicated blank samples (5% HNO3) for each element were tested by ICP-AES. As shown in Table S1, the detection limit was calculated by three multiples of the standard deviation of the test result, and the quantification limit of the method was calculated by 10 multiples of the standard deviation. The correlation coefficients of the linear calibration curve for each element were above 0.995. The accuracy and precision of the ICP-AES method were investigated by the standard material GBW07407 (GSS-7). Then, 5 mL $\rm HNO_3 + 3$ mL $\rm HF + 30$ mL $\rm H_3BO_3$ were added to digest a 0.2 g sample, with the total volume adjusted to 50 mL by deionized water. Six duplicated samples were prepared in parallel and determined by ICP-AES. The accuracy of the ICP method was expressed in terms of the relative error (RE, %), which is calculated by comparing the average of the 6 results and the concentration of the standard materials, and the precision of the method was expressed by the relative standard deviation (RSD, %) of the 6 analysis results. The results are shown in Table S2. It can be seen from the table that the RE (%) determined by ICP-AES is 0.45% - 5.88% and the RSD (%) determined by ICP-AES is 1.29% - 5.45%.

Similarly, each anion has its correlation coefficient and detection limit in IC. The detail of the detection limit is shown in Table S3. 11 blank samples (deionized water) were tested by IC. As shown in Table S3, the detection limit was calculated by 3 times the standard deviation of the test result, and the quantification limit of the method was calculated by 10 times the standard deviation. In general, the correlation coefficients of the linear calibration curves for each anion were above 0.999. The precision of the IC method was investigated, and three different concentrations of the standard solution were selected for verification. For each concentration, six duplicate samples were prepared in parallel and determined by IC. The precision of the 6 analysis results. The results are shown in Table S4. It can be seen from the table that the RSD (%) determined by IC is 0.88% - 2.93%.

Concentrations of condensable particulate matter under different experimental conditions.

Conditions Excess air coefficient/ Temperature	Inorganic fraction (mg/m ³)	Organic fraction (mg/m ³)	Total CPM* (mg/m ³)	CPM _p (mg/ m ³)	ELPI ⁺ Total PM (mg/m ³)	Compare ELPI ⁺ with CPM filter (%)
1.2–50 °C	6.89 ± 0.94	5.96 ± 0.88	12.85 ± 1.82	$\textbf{4.22} \pm \textbf{0.72}$	3.78 ± 1.02	89.62
1.4–50 °C	7.31 ± 1.02	3.20 ± 0.45	10.51 ± 1.47	$\textbf{8.08} \pm \textbf{1.27}$	6.55 ± 1.34	81.10
1.8–50 °C	12.34 ± 2.44	1.54 ± 0.36	13.88 ± 2.80	$\textbf{9.27} \pm \textbf{1.30}$	$\textbf{7.58} \pm \textbf{1.88}$	81.84
1.2–80 °C	6.39 ± 1.06	6.34 ± 1.05	12.73 ± 2.11	5.66 ± 0.69	4.93 ± 0.91	87.08
1.4–80 °C	$\textbf{7.68} \pm \textbf{1.26}$	3.47 ± 0.62	11.15 ± 1.87	9.77 ± 1.65	8.63 ± 1.00	88.31
1.8–80 °C	15.56 ± 2.30	2.52 ± 0.58	$\textbf{18.08} \pm \textbf{2.98}$	12.15 \pm	10.88 ± 2.42	89.56
				2.61		
1.2–100 °C	$\textbf{8.42} \pm \textbf{1.23}$	$\textbf{7.12} \pm \textbf{0.85}$	15.54 ± 2.08	$\textbf{8.49} \pm \textbf{1.25}$	6.89 ± 0.99	81.19
1.4–100 °C	9.13 ± 1.65	3.56 ± 0.58	12.69 ± 2.23	$11.23~\pm$	10.24 ± 2.52	91.20
				1.85		
1.8–100 °C	18.75 ± 3.59	2.57 ± 0.37	21.32 ± 3.94	16.74 \pm	13.55 ± 2.02	80.97
				2.08		
1.2–120 °C	12.19 ± 2.62	8.02 ± 1.21	20.21 ± 3.83	10.45 \pm	9.14 ± 1.13	87.50
				2.39		
1.4–120 °C	15.02 ± 2.75	3.64 ± 0.60	18.66 ± 3.35	$12.93~\pm$	11.321.53	87.54
				1.87		
1.8–120 °C	$\textbf{28.62} \pm \textbf{5.18}$	$\textbf{2.84} \pm \textbf{0.40}$	31.46 ± 5.56	16.50 \pm	14.932.02	90.47
				2.63		
1.2–150 °C	21.32 ± 4.86	8.34 ± 1.57	$\textbf{29.66} \pm \textbf{6.41}$	$13.23~\pm$	11.28 ± 1.93	85.29
				1.71		
1.4–150 °C	23.11 ± 3.64	$\textbf{4.12} \pm \textbf{0.48}$	$\textbf{27.23} \pm \textbf{4.11}$	16.43 \pm	14.15 ± 2.40	86.12
				2.85		
1.8–150 °C	$\textbf{36.84} \pm \textbf{6.88}$	3.09 ± 0.44	$\textbf{39.93} \pm \textbf{7.31}$	$18.27~\pm$	16.19 ± 4.25	88.61
				2.47		

* Total CPM (4th column) involves all the CPM including particles collected by the CPM filter (CPMp).

In the case of GC/MS, the calibration standards were prepared by four different concentrations of standard solution (CDGG-121172–02-1 mL, a mixture containing 22 different VOCs, diluted by chromatographic grade methanol), 20, 50, 100, and 200 μ g/mL. The correlation coefficient, retention time, mass spectrum (*m*/*z* ratio), and detection limit of the standard solution are shown in Table S5. The spectra of four different concentrations of standard solution are shown in Fig. S1. This standard calibration procedure confirmed that the GC/MS was in good working condition. However, some compounds identified (with the NIST 14 Mass Spectral Library) in the CPM samples do not exist in the standard solution. Therefore, the area normalization method was used for semiquantitative analysis in this study [20].

3. Results and discussion

3.1. Results of dry EPA method 202 (dry impinger method)

3.1.1. Total mass of CPM

The total mass concentration of CPM from the modified (from ELPI⁺, Table 3 Column 6) and the EPA method 202 (dry impinger method) (on the CPM filter, Table 3, Column 5) showed a good agreement (over 80%). The total mass of CPM from the ELPI⁺ was slightly less than that of the EPA method 202 (dry impinger method). The reason could be the condensation of a small number of particles in the CPM before entering the sampling probe of the ELPI⁺. It was also noticed that the sampling results showed a standard deviation from 10% to 20% between the two sampling methods under the tested combustion conditions, which could be due to the difference between the two methods.

The total mass concentration of CPM from both methods increased with the EAC and sampling temperature. More importantly, the inorganic fraction of CPM increased with the EAC, whereas the organic fraction underwent the opposite trend. Meanwhile, increasing the EAC from 1.2 to 1.8 yielded a more dramatic change in the inorganic fraction (increase) than that of the organic components (decrease). In the next section, a more detailed analysis is presented on the change of inorganic and organic compound speciation with the sampling temperature and EAC.

3.1.2. Inorganic components of CPM

The top ten detected inorganic components in the CPM are shown in Fig. 3a. Of the anions, SO_4^2 was the most concentrated in all the tested conditions, followed by Cl⁻ and NO₃. F⁻ had the lowest concentration, which was an order of magnitude less than that of SO_4^{2-} . Similarly, Ca^{2+} was identified as the main cation in the CPM, followed by Na⁺, Mg²⁺, Al³⁺, and Fe³⁺. While the reason for such a high concentration of cations $(300-2000 \ \mu g/m^3)$ at low temperature still needs further investigation, several other studies have also reported a relatively high concentration of cations in CPM [8,12,23] and proposed plausible explanations. First, Wu [12] suggested that during combustion of the coal, the salts (such as sodium salt and calcium salt) evaporated into the flue gas and then condensed into CPM as ionic species. Second, Wang [3] attributed the calcium ion to uncertainties in the measurement because calcium was not a volatile element. Third, Yang [24] reported that calcium is the dominating inorganic species in CPM. Furthermore, these authors argued that the enrichment of cations (such as Ca²⁺, Na⁺, and K⁺) was induced by the vapourization of volatile components in the fly ash, after which the vapour could condense or be adsorbed by fly ash. Therefore, the mechanism of the existence of metal cations at low temperatures needs further investigation.

Given that only a small portion (about 0.5% - 2%) [25] of the flammable sulphur element in the coal is converted to SO₃, the dominant concentration of SO₄² in the CPM from the experiments may seem surprising at first glance. There are several possible reasons for this counter-intuitive result. First of all, almost all the flammable sulphur elements in the coal turn into SO₂ during the combustion process at temperatures above 1500 °C [26]. Second, SO₂ is converted into SO₃ in the combustion through different mechanisms [26] that occur at temperatures higher than 1200 °C, as shown in Eq. (4), and that occur between 670 °C and 1250 °C, as shown in Eq. (5) & (6).

$$2SO_2 + O_2 \rightarrow 2SO_3 \tag{4}$$

$$SO_2 + OH \rightarrow HOSO_2$$
 (5)

$$HOSO_2 + O_2 \rightarrow SO_3 + HO_2 \tag{6}$$

Furthermore, the conversion from sulphur dioxide to sulphur trioxide can be enhanced under many conditions. For example, when the



Fig. 3. (a) Concentration of top ten ions in different experimental conditions, (b) Composition and the total amount of the organic fraction. (The highest data was used as a baseline (100%). The other data was the relative value to the baseline).



Fig. 4. The concentration of chemical species in different conditions.

coal has a low sulphur content as in the experiments, studies have shown that the conversion rate increases [27]. Additionally, the conversion of SO_2 to SO_3 may be promoted by the catalyst (V_2O_5 or TiO_2) in the Select Catalytic Reduction (SCR) system, which was originally designed to reduce the formation of NO_X and installed in power plants [28]. Finally, most of the SO_3 in the flue gas condensed during the sampling process.

In the meantime, chloride and nitride are released during coal combustion as gaseous HCl and NO_X because of their high volatility [29,30]. Similar to SO₄², these gaseous compounds are easily condensed during the sampling process, leading to the detection of Cl⁻ and NO₃ anions. For chloride, NaCl and CaCl₂ are the two most likely inorganic formations in coal, accounting for 95% of the generated HCl. The rest of the chloride component could be in the form of chlorine gas or free radicals generated by combustion [29].

Furthermore, NO_3^- detected in the CPM was mostly converted from NO_X . There are three main routes for NO_X production during the combustion process: fuel NO_X , thermal NO_X , and rapid NO_X [31]. Fuel NO_X , accounting for 60% – 80% of the total NO_X , starts with the pyrolysis of organic nitrogen in the fuel to HCN, NH₃, and CN, as well as other intermediate products, followed by oxidization of these pyrolysis products to NO_X . Thermal NO_X is generated by the oxidation of nitrogen from the air at high temperatures (above 1300 °C) and represents 20% of the total

Detected organic compounds of CPM.

Alkanes/Alkenes	Oxygen-containing volatile organic compounds (OVOCs)	Aromatics
1-Nonadecene	Fumaric acid, pent-4-en-2- vl tridecvl ester	1-Methylnaphthalene
1-Docosene	Methyl stearate	1,2,3,4,5- Pentamethylbenzene
(E)-9-Octadecene	2-Octyldecanol	1,3-Dimethyl-5-(1- methylethyl)-benzene
2,6,11,15- Tetramethylhexadecane	Methyl 3-(3,5-di- <i>tert</i> -butyl- 4-hydroxyphenyl) propionate	Naphthalene
C16H32	Methyl hexadecanoate	1-Methyl-4-(1- methylpropyl)- benzene
Nonadecane	1,2-Benzenedicarboxylic acid, di(2-methylpropyl) ester	(1,1,3,3,5- Pentamethylhexyl) benzene
Tetradecane	2-Ethylhexyl benzoate	1,3-Dimethyl-5- ethylbenzene
1,2,4,5- Tetraethylcyclohexane	Carbonic acid, eicosyl vinyl ester	2,3-dihydro-4-methyl- 1H-indene
5,8-Diethyldodecane	1-Hexadecanol	1,2,4,5- Tetramethylbenzene
2,6,11- Trimethyldodecane	1-Hendecanol	1-Methyl-3-(1- methylethyl)-benzene
3,6-Dimethyldecane	2-Methyl-1-hexadecanol	1,2,3- Trimethylbenzene
Hexylcyclohexane 2,8-Dimethylundecane	2,5-Di- <i>tert</i> -butylphenol 2,6-Bis(1,1- Dimethylethyl)-2,5- cyclohexadiene-1.4-dione	Styrene
(2-Cyclohexyl-1- methylpropyl) cyclohexane	2-Butyloctanol	
Hendecane	3,7,11-Trimethyl-1- dodecanol	
3,5-Dimethyloctane	2,4-Dimethylbenzaldehyde	
4-Methyloctane	Isophorone	
2,4-Dimethylhept-1-ene	3,7-Dimethyl-1-octanol	
3-methylhexane	hydroperoxide	
2-Methylheptane	2,4-Dimethyl-3-hexanol	
1,1,3- Trimethylcyclopentane	3-Methylcyclopentanone	

*All compounds are in the liquid or solid state after being cooled and condensed.

 NO_X . Rapid NO_X contributes to less than 5% of the total NO_X [32] and is formed by a two-step reaction. N_2 in the air and CH radicals in the fuel react first to form HCN and N radicals, which then react with oxygen to

generate NO_X.

After analyzing the origin of the ions detected in CPM, now the effects of the sampling temperature and EAC on their concentration are discussed. The inorganic species in CPM are shown in Fig. 4. First of all, because the production of most ions (e.g., SO_4^2 , SO_3 , NO_3) involved oxidation, a smaller EAC yielded fewer anions and metal cations in the CPM, which was inconsistent with previous studies [9,33]. The EAC could still affect the concentrations of ions that were not generated by oxidation. For example, one study [34] reported that a higher oxygen concentration increased chloride in the flue gas by reducing its deposition on fly ash.

Also, the total concentration of both metal cations and anions decreased at lower sampling temperatures, where the most significant reduction (over 50%) occurred in the temperature range of 150–100 °C. In particular, the change of the metal cation and anion concentration with temperature was mostly contributed by their representative ions, i. e., SO_4^2 and Ca^{2+} , and the change of the Cl⁻ concentration with the temperature only becomes clear when EAC greater than 1.2. When sampled at a lower temperature, part of the SO_4^2 , Cl⁻, and NO_3^- was condensed and collected at the FPM filter, leading to fewer anions being detected in the CPM. This was more dramatic for $SO_4^2^-$ due to the higher concentration. 8.2–81.4% of SO₃ dissolved in the CPM. The concentration of chloride species in the coal was 110–130 ppm, 0.7–4.3% of which entered the CPM. SO₃ had the greatest tendency to be converted to CPM compared with Cl and NO_X.

3.1.3. Organic components of CPM

Organic compounds in the coal entered into the flue gas mainly due to incomplete combustion, where decomposition, cyclization, and condensation of these species could occur [35,36]. Most of these compounds in the flue gas would be either oxidized or captured when passing through the APCD. However, a very small amount could still escape into the atmosphere and form CPM, as demonstrated by this paper. The organic components were detected by GC/MS (software: Tubemass). Due to the difficulty of getting standard samples for all the compounds, area normalization was used for qualitative analysis. The results indicated three major groups of chemicals, alkanes/alkenes (70% signal intensity), oxygen-containing volatile organic compounds (OVOCs, 29% signal intensity), and aromatics (less than1% signal intensity), as shown in Fig. 3b.

The strong signal intensity of alkane/alkene and OVOCs, coinciding with previous studies [8,11], suggested that these chemicals in the flue gas had a great tendency to be emitted into the CPM. Also, compound analysis (Table 4) showed over 50 species of VOC in the CPM. The major species were C_{15} and C_{18} - C_{22} for alkanes, 2,4-Dimethyl-3-hexanol, and



Fig. 5. Effects of EAC and sampling temperature on the particle total distribution of CPM: (a) mass concentration; (b) number concentration.

Number concentration at different particle sizes at 50 $^\circ C$ with different EAC values (1/cm³).

	PM _{10~2.5}	PM _{2.5~1}	$PM_{1\sim0.2}$	PM _{0.2}
50 °C	53	144	1496	6.62E + 06
-1.8EAC 50 °C	(0.00080%) 45	(0.0021%) 121	(0.023%)	(99.97%) 4.66E + 06
−1.4EAC 50 °C	(0.00097%) 26	(0.0026%) 64	(0.025%) 773	(99.97%) 3.34E + 06
-1.2EAC	(0.00078%)	(0.0019%)	(0.023%)	(99.97%)

Methyl-hexadecanoate for OVOCs, and Naphthalene and 1-Methylnaphthalene for aromatics. Among these organic compounds, alkanes/alkenes tend to condense at low temperatures due to their high boiling points. Therefore, the total mass concentration of the alkanes/alkenes dropped by more than 13% as the sampling temperature reduced from 150 °C to 50 °C. However, the vapour pressure of the OVOCs did not change significantly [37], so their concentration reduction was less than that of alkanes/alkenes. Increasing the EAC promoted the oxidation of VOCs and the combustion of the coal. Therefore, it reduced the organic compounds in CPM more dramatically than lowering the sampling temperature. The total mass concentration of alkanes/alkenes dropped by more than 35% as the EAC increased from 1.2 to 1.8, and the reduction of OVOCs was enhanced by more than 10%. The main reason was that the large EAC promotes the oxidation of VOCs and the combustion of coal. 3.2. Integrated EPA method 202 (dry impinger method) and ELPI + sampling system

3.2.1. Total CPM formation

Fig. 5 shows the total mass and number concentration of CPM at different sampling temperatures and EAC values. Both the total mass and number concentration of the CPM increased at higher sampling temperatures, where more species remained in a gaseous state and passed through the FPM filter to be detected as CPM. Additionally, the mass concentration was dominated by PM_{10} (all particles whose size is less than $10 \,\mu$ m). However, the number concentration was dominated by $PM_{0.2}$ (all particles whose size is less than $0.2 \,\mu$ m), as shown in Table 5. The percentage of PM_{10} , $PM_{2.5}$, and PM_1 in the total collected PM was 0.0008, 0.002, and 0.023%, respectively. 99.97% of the collected PM was less than $PM_{0.2}$.

3.2.2. CPM size distribution

As shown in Fig. 5, the mass- and number-based size distribution of CPM particles showed two groups of curves separated by the sampling temperature at 100 °C, below which the distribution curves were almost identical and above which the CPM had more particles smaller than 0.2 μ m. The effect of the EAC was further examined by plotting the particle size distribution in the CPM at two different sampling temperatures (Fig. 6). The change of size distribution with the EAC was not significant when the sampling temperature was 50 °C, whereas the increase in smaller particles was clear when the sampling temperature was 120 °C.

3.2.3. Discussion

The results from the modified sampling system highlight three key



Fig. 6. Mass particle size distribution at different EAC at (a) 50 °C, (c) 120 °C and number particle size distribution at different EAC at (b) 50 °C, (d) 120 °C.



Fig. 7. Effect of different sampling temperatures at (a) 1.2 EAC, (c) 1.4 EAC, and (e) 1.8 EAC on mass particle size concentration, and the effect of different sampling temperatures at (b) 1.2 EAC, (d) 1.4 EAC, and (f) 1.8 EAC on number particle size concentration.

observations. First of all, the mass-based distribution was mainly dominated by large particles, while the number-based distribution in the CPM was contributed mostly by particles smaller than $0.2 \,\mu$ m. Also, both the number and mass of the CPM particles increased with the sampling temperature, and this effect was more significant for smaller particles at sampling temperatures higher than 100 °C. Finally, the EAC had a similar effect to that of the sampling temperature and was highly sensitive to the actual sampling temperature. Starting from a high sampling

temperature, there would be very minimal condensation before passing through the FPM filter because the temperature was above the boiling point of most of the flue gas components. Lowering the sampling temperature caused more condensation. In particular, heterogeneous condensation could be prevalent at this stage due to its lower energy barrier [38]. This means that most of the condensed species were associated with large particles and collected by the FPM filter, yielding fewer particles (number and mass) detected by the ELPI⁺, as shown in Fig. 7. Lowering the sampling temperature further promoted more homogeneous condensation and produced liquid droplets in the flue gas before reaching the FPM filter. Importantly, according to Kelvin's equation [39],

$$r^* \sim (T \ln p/p_{sat})^{-1}$$
 (7)

the critical size of a condensed droplet r^{\star} becomes smaller with a larger reduction in the saturated vapour pressure p_{sat} caused by a temperature T drop (p is the vapour pressure). Therefore, more condensed droplets could pass through the FPM filter and be detected by the ELPI⁺ as CPM. This explained why varying the sampling temperature below 100 $^\circ$ C did not induce significant changes in the particle size distribution of the CPM.

Because particles entering the ELPI⁺ were formed by vapour condensation in the condenser, the measured particle size distribution of the CPM also reflected the temperature difference between the flue gas sampling position and the condenser (maintained at 30 °C). A higher sampling temperature generated a larger temperature drop in the condenser, which, in turn, promoted smaller droplets in the homogeneous condensation. Therefore, small changes in the sampling temperature favored the heterogeneous condensation that was responsible for CPM particles of all sizes. This mechanism closely affected the total mass concentration of the CPM. Moreover, homogeneous condensation was enhanced with larger temperature drops, leading to finer particles smaller than 0.2 µm. Therefore, the total number concentration was more sensitive to the process of homogeneous condensation. Combining the results of both chemical species and particle size distribution provided guidelines to further reduce or control the CPM emission. For example, researchers have reported that SO₄²⁻, NO₃⁻, and NH₄⁺ are the three main ions from atmospheric aerosols [3,40-42]. This result not only coincided with the measured ionic composition in the CPM, but also suggested that substantial finer particles in the CPM tended to form aerosols. While lowering the outlet temperature and increasing the oxygen content help to reduce the concentration of these ions, an understanding of the condensation dynamics of flue gas is necessary to better control the formation and migration of fine particles in CPM. Also, the ultra-low emission (ULE) technologies usually involve SCR, which could enhance the formation of SO_3 and thus, the concentration of SO_4^{2-} ions. Therefore, further studies on the effect of ULE technologies on the formation of CPM are needed.

4. Conclusions

This study presented a modified sampling system that combines the US EPA method 202 and ELPI⁺ to characterize the chemical species and measure the real-time particle size distribution of CPM. The results of total mass concentration and chemical speciation in CPM from the modified system agreed well with the classic EPA method 202 (dry impinger method). The sampling temperature reduced the total concentration of CPM, the inorganic fraction of CPM increased, while the organic fraction decreased with the EAC. A high sampling temperature or EAC can increase the concentration of fine particles (smaller than 0.2 μm), and this effect was only significant when the sampling temperature was above 100 °C. Heterogeneous condensation was attributed to the whole size particles in CPM, while the homogeneous condensation only led to the increase of finer particles. Considering the challenges in controlling these fine particles and their tendency to migrate and form aerosols, more studies are necessary to further investigate the dynamics of homogeneous condensation during the flue gas sampling and transport processes.

CRediT authorship contribution statement

Yue Peng: Methodology, Formal analysis, Writing - original draft. Nan Shi: Methodology, Writing - review & editing. Tao Wang: Conceptualization, Funding acquisition. Jiawei Wang: Resources. Yongsheng Zhang: Project administration. Wei-Yin Chen: Writing review & editing. Baharak Sajjadi: Writing - review & editing. Wei-Ping Pan: 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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Appendix A. Supplementary data

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