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Real-time analysis of soot emissions from bituminous coal pyrolysis and combustion with a vacuum ultraviolet photoionization aerosol time-of-flight mass spectrometer

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ABSTRACT

This paper reports on-line analyses of the soot emissions from the Inner Mongolia bituminous coal combustion and pyrolysis processes with a vacuum ultraviolet photoionization aerosol time-of-flight mass spectrometer (VUV-ATOFMS). The soot particles are generated by heating a small amount of screened coal powder in synthetic air and nitrogen atmosphere in a tubular oven. The vacuum ultraviolet photoionization time-of-flight (VUV-TOF) mass spectra of the soot particles emitted from combustion and pyrolysis at different oven temperatures and different stages are obtained. The VUV-TOF mass spectra are assigned with the references of the results of the off-line GC/MS analysis.

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1. Introduction

Coal smoke makes a large contribution to global air pollution due to the fact that coal is one of the major fossil fuels. Coal accounts for approximately 27% of the total primary energy consumption of the world (Cooke et al., 1999). Both coal combustion and pyrolysis processes emit large amounts of carbonaceous aerosols. Aromatic compounds in these aerosols get special attention due to their great potential carcinogenic, mutagenic and genotoxic effects on human health (Lighty et al., 2000).

Many researchers have focused on the emission characteristics of both the particulate phase and the gas phase from coal combustion and pyrolysis using a variety of conventional scientific instruments such as GC/MS (Greenwood et al., 1993; Olivella et al., 2002; Chen et al., 2005), HPLC (Ledesma et al., 2000) and FTIR (Ibarra and Moliner, 1991; Baxter et al., 1993; Arenillas

et al., 2004) etc. All of these methods involve the isolation of particles on filters with subsequent analysis performed in the laboratory. These isolation processes often disturb the original constituents and thus render the data questionable because of potential evaporation or reaction of the particles before analysis. To overcome these difficulties, fast and in situ mass spectrometric techniques, especially aerosol mass spectrometers combining with soft ionization methods, e.g. vacuum ultraviolet (VUV) photoionization techniques (Woods et al., 2001; Sykes et al., 2002; Oktem et al., 2004; Mysak et al., 2005; Gloaguen et al., 2006; Canagaratna et al., 2007; Northway et al., 2007; Streibel et al., 2007) have been developed, enabling on-line analyses of the chemical compositions of aerosols. VUV photoionization is a soft ionization technique; the organic molecules undergo much less fragmentation during the VUV photoionization process compared with the electron impact (EI) ionization (Northway et al., 2007). Therefore, VUV photoionization mass spectra are

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more easily assigned than EI mass spectra. However, large variations in photoionization potentials and photoionization cross-sections between molecules result in large variations of the sensitivity of this method for detecting different molecules (Gao et al., 2008).

Since the first on-line mass spectrometric analysis of single particles in 1973 (Davis, 1973), many researchers have focused on the emission factors of different sources including gasoline and diesel engine (Oudejans et al., 2004; Toner et al., 2006), wood burning (Schneider et al., 2006) and cigarette smoke (Dall'Osto et al., 2007) etc., whereas relatively little has been carried out on the characterization of coal soot with an aerosol mass spectrometer. This paper presents on-line analyses of the coal soot produced at different combustion/pyrolysis times and temperatures with a newly home-built vacuum ultraviolet photoionization aerosol time of flight mass spectrometer (VUV-ATOFMS). This real-time analysis mainly focuses on the PAH compounds contained in the soot particles as the ATOFMS is more sensitive to PAH compounds than others (Gao et al., 2008).

2. Experiment

The schematic diagram of the experimental setup is shown in Fig. 1. The VUV-ATOFMS was newly home-built to analyze organic particles on a real-time, quantitative basis. A detailed description has been given elsewhere (Shu et al., 2008). Briefly, samples are introduced into the instrument through a nozzle with ~ 0.12 mm orifice, and then the aerosol particles are focused into a particle beam with a diameter of ~ 1 mm by an aerodynamic lens assembly. A three stage differential pumping system, which is composed of a source chamber, a differential chamber, and a detection chamber, is used to directly sample the particles at a flow rate of ~ 1.3 cm³ min⁻¹ at atmospheric pressure. An 8 mm diameter copper rod coupled with a cartridge heater is used to vaporize the particles. A VUV lamp that has a

main energy output at 10 eV (123.6 nm, Kr resonance line) is used as the ionization source with a total photon flux output of about 2×10^{12} photon/s. A 0.41 V/cm electric field is used to drive the ions into the second stage of ion optics where a positive electric pulse repels the ions into the reflectron mass spectrometer with a field free flight distance of 1.4 m, an ion mirror and a chevron multichannel plate detector. The pulse is provided with a HTS31 fast high voltage transistor switch (Behlke Electronic GMBH, Germany). The repetition rate of the pulses is optimized to 10 kHz in this experiment.

The Inner Mongolia bituminous (IMB) coal is selected because of its abundant content in China. The IMB coal is ground into a 75-mesh. A tubular oven with a quartz tube insert (i.d. 30 mm) is used for the combustion and pyrolysis experiments. In these experiments, the heating of coal in air is defined as combustion, while heating in nitrogen is defined as pyrolysis. The oven temperature is set to 673, 873, 1073 and 1273 K respectively. A ~ 1.5 l/min flow of synthetic air (80% nitrogen, 20% oxygen) is provided for the combustion experiment while a 1.5 l/min flow of high purity nitrogen is maintained for the pyrolysis experiment. After passing through a 2.5 m long conductive rubber tube, the soot particles entrained in the flow are continuously sampled and analyzed with the VUV-ATOFMS. The temperature of the thermal heater is optimized to be 393 K in order to minimize the fragmentation and maximize the signal intensities of the organic compounds. Generally, the complete heating process for ~ 10 g coal powder in air or nitrogen atmospheres ranges in time from tens of seconds to minutes with different oven temperatures. For off-line analysis, a glass fiber filter is placed at the outlet of the furnace (873 K) to separate particles from the gas stream. Then the filter is ultrasonically extracted 3 times in dichloride methane (DCM), a process which lasts for 20 min. The extracts are combined and concentrated to about 1 ml on a rotary evaporator. Finally, the extracts are dried under a gentle stream of high purity nitrogen and re-dissolved in hexane to about 1 ml. Interfering species such as the sticky oily matter contained in the extract are removed by liquid–solid chromatography using a silica column (the silica was preheated at 423 K for 12 h). We speculate that these are from coal tar which contains lots of organic compounds with high molecular weights and small solubility; identifying such compounds is beyond the scope of this research. Finally, the aliphatic and aromatic fractions are respectively eluted out by 100 ml hexane and 150 ml mixture of DCM and hexane (1:1 V/V). The two fractions are respectively concentrated to approximately 0.5 ml using the same procedure described above. All samples are analyzed using a Hewlett-Packard 6890 gas chromatograph equipped with a 30 m $\times$ 0.25 mm $\times$ 0.25 μ m HP-5 capillary, and coupled to a HP-5973 quadrupole mass filter with a 70 eV electron impact ionizer. The chromatographic conditions are as follows: injector temperature, 553 K; ion source temperature, 453 K; the temperature program begins at 338 K, after a hold for 5 min, then increases to 563 K at 5 K/min, with another isothermal hold at 563 K for 20 min. The carrier gas is helium at a constant flow rate of 1.5 ml/min. The sample of 1 μ l is injected with a splitless model. A mass range from 50 to 510 Da is used for qualitative determination. The chromatographic peaks of the samples are identified by comparison of mass spectra with the literature and library data (NIST mass search, version 2005) (matching degree > 90%).

Fig. 1 – Schematic diagram of the experimental setup.

The original organic contents contained in coal are analyzed with the VUV-ATOFMS and the GC/MS. ~20 g 75-meshed coal powder is extracted ultrasonically 3 times each with 100 ml DCM. Each extraction lasts for 20 min. The organic extracts are filtered with filter paper. The three filtered extracts are mixed together. One portion of the extract is introduced into the VUV-ATOFMS after atomization. The other is fractionated, concentrated and stored at 277 K for GC/MS analysis.

All reagents are of chromatographic grade, from J. T. Baker Co. The anhydrous sodium sulfate (analytical grade) and the silica gel (chromatographic grade 100–200 mesh) are purchased from QingDao Chemical Plant in China. The IMB coal is purchased from a coal storage and transportation station in Beijing.

3. Results and discussion

The GC/MS total ion chromatogram and the VUV-TOF mass spectrum of the IMB coal extracts are shown in Fig. 2(A and B), respectively. Each GC/MS TIC and the VUV-TOF mass spectrum is normalized with the value of the most intense peak. The dominant peak in Fig. 2(A) is 3,4,5,6-tetramethyl-phenanthrene assigned by matching its daughter ion pattern with the standard database (NIST mass search, version 2005). Other major peaks shown in Fig. 2(A) which can be identified

with the standard database are naphthalene, 2-methyl-naphthalene, 2,3,6-naphthalene, 4-isopropyl-1,6-dimethyl-naphthalene, 1,4,5,8-naphthalene, pyrene, and, 4-isopropyl-1,3-dimethyl-phenanthrene. The VUV-TOF mass spectrum shown in Fig. 2(B) shows a very intense peak at 234 Da, which is assigned to 3,4,5,6-tetramethyl-phenanthrene according to the GC/MS TIC results. The mass peaks of pyrene and 4-isopropyl-1,3-dimethyl-phenanthrene also appear in Fig. 2(B). However, naphthalene and its analogues are absent in the VUV-TOF mass spectrum shown in Fig. 2(B). We think that the chemicals with lighter molecular weights might be lost to gas phase during atomization, and thus are not detected by the ATOFMS. Contrary to this phenomenon, the chemicals with heavier molecular weights (>250) are not detected by GC/MS sensitively. We think that the capillary temperature of the GC/MS may be not high enough to vaporize the chemicals with heavier molecular weights efficiently. The difference between Fig. 2(A) and (B) may reflect the difference of the sensitivities between the GC/MS and the VUV-ATOFMS. Fig. 2 shows that 3,4,5,6-tetramethyl-phenanthrene is abundant in the IMB coal. The VUV-TOF mass spectrum of the coal extract offers a good reference for the VUV-TOF mass spectra of soot particles below.

Fig. 3 shows the VUV-TOF mass spectra of coal soot particles generated from pyrolysis (a–d) and combustion (A–D) with oven temperatures of 673 K (a, A), 873 K (b, B), 1073 K (c, C), and 1273 K (d, D), respectively. The mass spectra are all collected for 30 s when the density of soot generated roughly reaches its maximum. They are each normalized with the value of the most intense peak of the individual spectrum. Fig. 3(a, b, A) shows a very intense peak at 234 Da, which is also observed in Fig. 2(B). These phenomena indicate that 3,4,5,6-tetramethyl phenanthrene, the original organic content contained in coal, is vaporized from coal powder and condenses on soot particles at low or medium temperatures during combustion or pyrolysis process (673–873 K). Fig. 3(b) shows the signals of aryl-PAHs prevailing while Fig. 3(B) shows obvious signals of oxygen containing PAHs (O-PAHs). This may be an indication of the participation of oxygen in the reaction during combustion process. Fig. 3(D) shows that the main peaks are at 178 Da, 202 Da, 226 Da, 228 Da, 252 Da and 276 Da, which are assigned to phenanthrene, pyrene, cyclopenta[cd]pyrene, benzo[a]anthrene, benzo[a]pyrene and indeno[1,2,3-cd]pyrene respectively according to the GC/MS TIC results. In Fig. 3(d), besides the mass peaks shown in Fig. 3(D), it shows much stronger mass peaks at 152 Da, 166 Da, 190 Da, 216 Da, 242 Da, which respectively represent acenaphthylene, fluorene, 4H-cyclopenta[def]phenanthrene, mono-methylated pyrene and mono-methylated chrysene (Gao et al., 2008).

The spectral differences between Fig. 3(a) and (A) as well as Fig. 3(b) and (B) indicate obviously different reaction mechanisms for pyrolysis and combustion at relatively lower oven temperatures (i.e. <873 K). Compared with Fig. 3(b and B), the mass spectra of the soot particles generated from the pyrolysis (Fig. 3(c)) and combustion (Fig. 3(C)) processes of the coal at 1073 K become sparser. This means that the unstable compounds in soot particles are consumed by pyrolysis or combustion at much higher oven temperatures. Fig. 3(d) and (D) shows that most of the substituted PAHs such as aryl-PAHs, and oxygen containing PAHs (O-PAHs) are reduced to their skeletal structures, and only the most thermally stable compounds

Fig. 2 – GC/MS total ion chromatogram (A) and VUV-TOF mass spectrum (B) of the coal extract. The GC/MS total ion chromatogram and the VUV-TOF mass spectrum are normalized with the values of their most intense peaks, respectively.

Fig. 3 – VUV-TOF mass spectra of coal soot particles generated from pyrolysis (a–d) and combustion (A–D) at different oven temperatures. The mass spectra are all collected for 30 s when the density of soot generated reaches around its maximum. They are all normalized with the value of the most intense peak of each. The temperatures of the tube oven are 673 K (a, A), 873 K (b, B), 1073 K (c, C), and 1273 K (d, D) for pyrolysis and combustion.

without any functional groups prevail on the VUV-TOF mass spectra of soot particles. This indicates a more thorough combustion or pyrolysis process at 1273 K (Cao et al., 2003). These results reveal that the oven temperature is critical to the measurement of the composition of soot emissions.

Fig. 4 shows the VUV-TOF mass spectra of the coal soot particles generated at different stages of pyrolysis (Fig. 4(a–c)) and combustion processes (Fig. 4(A–C)) at 873 K. The oven temperature of 873 K is selected because the reaction times of pyrolysis and combustion at that temperature are in the response range of the ATOFMS. Fig. 4(a), (b) and (c) are respectively collected at 140 s, 350 s and 525 s while Fig. 4(A), (B) and (C) are respectively collected at 65 s, 135 s and 240 s. The time is recorded after the coal sample is delivered into the center of the tube oven. It takes about 1 min for the carrier gas to transport the nascent soot particles from the tubular oven to the VUV-ATOFMS. The times are selected to represent three different stages of the pyrolysis or combustion experiment, i.e. initial stage, intermediate stage and terminal stage. Fig. 4(a) is similar to Fig. 2(B), which means that the constituents of soot particles formed in early stage of pyrolysis are mainly contributed from non-decomposed organics contained in coal. Fig. 4(b) shows that there are many new species formed in the mid stage of the pyrolysis. These new species may come from

the large pyrolysis fragments or the repolymerization of some small pyrolysis fragments. A detailed description of the PAHs formation mechanism has been given by Richter and Howard (2000). Fig. 4(A–C) shows significant differences from Fig. 4(a–c). The intense mass peaks at the m/z 234 shown in Fig. 4(a–c) are absent in Fig. 4(A–C). This phenomenon indicates the vapor that the organics emitted from coal decomposes completely at the early stage of combustion. Fig. 4(A–C) shows that the shapes of the mass spectra of the soot particles produced at different stages of the combustion at 873 K are similar, which means that chemical compositions of those particles are similar.

Fig. 5 shows the particle-phase emissions of phenanthrene, 3,4,5,6-tetramethylated phenanthrene, benzo[b]naphtho[2,3-d]furan/benzo[k,l]-xanthene at different combustion and pyrolysis times at 873 K. Every data point shows the peak intensity of the corresponding compound on the VUV-TOF mass spectra. Here we assume that the ionization efficiencies of the three compounds are equal based on their benzenoid structures. These three chemicals show the general emission characteristics of PAHs, aryl-PAHs, and O-PAHs, respectively. Different emission characteristics are observed during the combustion and pyrolysis processes at lower oven temperatures. The minor emissions of O-PAHs observed in the pyrolysis process may be caused by the oxygen or oxygen

Fig. 4—VUV-TOF mass spectra of the coal soot particles generated in different stages of pyrolysis (a–c) and combustion (A–C) at 873 K. Fig. 4(a–c) are collected at 140 s, 350 s, and 525 s, respectively, while Fig. 4(A–C) are collected at 65 s, 135 s, and 240 s. The time is recorded after the coal sample is delivered into the center of the tube oven. All the mass spectra are collected for 30 s.

containing chemicals contained in coal. Fig. 5 shows that the combustion process at 873 K emits more PAHs and O-PAHs than the pyrolysis process, whereas the pyrolysis process produces more aryl-PAHs than the combustion process. The emissions of the three kinds of the chemicals from combustion at 873 K decrease faster than those from pyrolysis. These phenomena can be explained by the formation of oxygen-containing radicals in the combustion process (Cao et al., 2003). The oxygen-containing radicals often react with the organic species in the form of O, OH and O₂H (Hucknell, 1985), and this results in the production of O-PAHs and more stable products (PAHs). The formation of oxygen-containing radicals leads to a faster decomposition of the organic compounds due to the increase of radical concentration or the increase of the reaction pathways (oxidation).

There are two limitations in this experiment. First, we did not measure the temperature of coal directly when burning. The combustion process is exothermic. We think that the temperature of the coal when burning should be higher than the temperature of the tube oven presented. Secondly, the VUV-ATOFMS is not uniform in the sensitivity to aromatic compounds and aliphatic compounds. GC/MS results show that there are mainly two classes of organic compounds in the particle phase of the IMB coal emissions, one is the aromatic compounds (1–7 rings) which is mainly divided into four sub-series, e.g. PAHs, aryl-PAHs, cyclopenta-fused PAHs (CP-PAHs) and oxygen containing PAHs (O-PAHs) such as phenols, furans,

aromatic alcohols, aromatic ketones and aromatic acids, another class is the aliphatic compounds including long chain alkanes and alkenes with molecular weights ranging from 170 Da (Dodecane) to 507 Da (Hexatriacontane). However, only mass peaks of the aromatic compounds are dominant in the mass spectra obtained, which means that the VUV-ATOFMS is more sensitive to aromatic compounds. Apparently, these two problems may bring about some distortion to understanding of the soot emissions of coal combustion and pyrolysis. Nevertheless, the capability of the on-line analysis of the VUV-ATOFMS is a good supplement to the instruments of off-line analyses such as GC/MS.

4. Conclusions

This paper reports the on-line analysis of the soot emission characteristics of the coal combustion and pyrolysis with a vacuum ultraviolet photoionization aerosol time-of-flight mass spectrometer. Based on the combination of the GC/MS TIC results and VUV photoionization mass spectra, we conclude that at lower oven temperatures (<873 K), the soot particles generated in combustion processes contained more O-PAHs, while those from pyrolysis contain more aryl-PAHs. At high temperatures, however, the soot particles from both combustion and pyrolysis contain mainly PAHs without any functional groups. The organics contained in coal undergo

Fig. 5 – The particle-phase emissions of phenanthrene, 3,4,5,6-tetramethylated phenanthrene, benzo[b]naphtha[2,3-d]furan/benzo[k,l]xanthene at different combustion (A) and pyrolysis (B) times at 873 K. Every data point shows the peak intensity of the corresponding compound on the VUV-TOF mass spectra, which is collected for 30 s.

more rapid dissociation during the combustion process than the pyrolysis process at lower oven temperatures. The contents of soot particles generated in pyrolysis vary with the pyrolysis time. However, the chemical composition of the soot particles generated at different stages of combustion show no apparent differences under this experimental condition.

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