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Spatial Characteristics and Major Sources of Polycyclic Aromatic Hydrocarbons from Soil and Respirable Particulate Matter in a Mega-City, China

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Abstract As one of China's great metropolises, Taiyuan is generally recognized to be one of the most polluted cities from polycyclic aromatic hydrocarbons (PAHs) in the world. Therefore, this study was conducted to determine a total of 16 PAH concentrations in various environmental media in Taiyuan. The total PAHs concentration ranged from 1.0 to 26 μg g^{-1} in soil, 1.2 × 10^2 to 1.4 × 10^3 ng m^{-3} in PM 2.5 and 76 to 1.1 × 10^3 ng m^{-3} in PM 10, respectively. Furthermore, the primary source of PAHs was coal combustion, but the samples were also affected to varying degrees by traffic emissions.

Keywords Polycyclic aromatic hydrocarbons · Respirable particulate matter · Soil · Taiyuan

Polycyclic aromatic hydrocarbons (PAHs) are an important class of organic pollutants that have received considerable attention due to their carcinogenic and mutagenic properties (Martin et al. 2007). PAHs are produced by the incomplete combustion of organic materials or during pyrolisis (Bernal-Martinez et al. 2007). Though they may have natural origins, the main pollution sources of PAHs are anthropic actions, such as combustion of fossil materials, coal combustion, industrial combustion, smoke of cigarettes and so on (Bernal-Martinez et al. 2007). Therefore, PAHs are discharged into the atmosphere as both gases and aerosols, after which they are transported through the atmosphere until they are ultimately deposited in the terrestrial environment (Franz et al. 1991). Many regulations on PAH emissions have been proposed in the world in order to improve environmental quality. The United States Environmental Protection Agency (USEPA) has proposed a reference concentration of naphthalene in ambient air of 3 μg m^{-3} to prevent harmful respiratory tract, ocular, and blood effects. Most studies on environmental contamination by PAHs in China have focused on their levels in environmental media, such as soils, sediments, and water. To the best of our knowledge, however, the contamination status of various environmental media within an area have yet to be assessed for metropolis in China.

Taiyuan city, the capital of China's Shanxi Province, is generally recognized to be one of the most polluted cities in the world (Mestl et al. 2005). Taiyuan is located in the middle reaches of the Yellow River in North China and is surrounded by hills to its west, north and east. It occupies an area of 6,956 km^2, with 180 km^2 classified as urban, and has a population of about 3.6 million inhabitants. Taiyuan produces a quarter of China's raw coal. China clay is the main representative soil type in Taiyuan. The climate is dominated by temperate semi-wet monsoon, with a mean annual temperature of –5.2°C during the sampling period. In 1998, the average annual concentration of total suspended particulates was 498 μg m^{-3} (Mestl et al. 2005). This is approximately five times higher than the threshold for health risk set by the World Health organization (WHO) and the European Union (EU) (EQRT 2000). Moreover, the rapid economic growth in recent years has sharply increased coal consumption in Taiyuan: the annual

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coal consumption is around 25 million tons, of which 9.6 million tons are for energy use, accounting for 95% of the total energy production (Environmental Monitoring Center of Taiyuan 2004). The amount of coal consumed within such a small area is likely to result in high levels of PAHs. Furthermore, Taiyuan is located in a basin surrounded by mountains on three sides, and frequent inversions and low wind velocities concentrate pollutant levels. Because of the paucity of data on PAH levels from various environmental media within an area in Chinese cities (Wang et al. 2008), we undertook this study to determine both the concentration and profile of PAHs in soil and respirable PM (PM 2.5 and PM 10) samples from Taiyuan urban areas. The main objectives were to establish the urban area's contamination status, explore the interaction of PAHs among these environmental media in the terrestrial ecosystem and identify the possible sources of pollution. It may help in environmental policy making regarding PAHs pollution in the mega-cities in China.

Materials and Methods

Sampling sites were set to represent the area of Taiyuan. The sampling locations were chosen by a symmetrical grid (25 km^2 per grid), in an attempt to evenly distribute sampling over the region. Surface soil (0–10 cm) was obtained from 15 sites (site 1–15) in Taiyuan city, Shanxi province (Fig. 1), in January 2006. Surface soil (0–10 cm) was sampled in triplicate using a hand-held coring device from 15 sites (sites 1–15) in Taiyuan (Fig. 1), in January 2006. Each composite soil sample was 16 sub-samples from corresponding grids of sampling sites (Fig. 1). Overlying vegetation was removed prior to collection of the sample. The samples were freeze-dried, thoroughly mixed, sieved to 60-mesh, transferred to pre-cleaned amber glass and maintained at –18°C before analysis. The soil water content was determined gravimetrically after oven-drying individual composite soil samples at 105°C for 12 h.

Sampling was done in combination with the PAHs soil study, using the same sampling sites. From the corresponding grid for soil samples (sites 1–17, Fig. 1) in Taiyuan, 34 respirable PM ($PM_{2.5}$ and PM_{10}) samples were taken in December 2006. To combine with the PAHs soil study, the PM sampling was also done in winter. The PM samples were taken with a modified medium-volume TH-150 sampler with dual sampling module, which is in two main parts, filter holder and glass fiber filters, located on the top of the sampler (Wuhan Tianhong Instrument Factory, Wuhan, China). The overall average volume was approximately 24 m^3 (average flow rate: 100 L min^{-1}) and each sample was a 4-h composite. There were 17 of $PM_{2.5}$ and 17 of PM_{10} samples taken. Before the experiment, the fiber filters were annealed for 4 h at 550°C to remove organic material and equilibrated in desiccators, weighed and put into acetone pre-washed aluminous envelopes. After sampling, the filters were removed from the inlet and folded in half and returned to the pre-clean aluminous envelopes and stored them at –18°C before analysis.

A mixed PAH standard solution (1,000 μg mL^{-1}) that included naphthalene (Na), acenaphthylene (Acy), acenaphthene (Ace), fluorene (Fl), phenanthrene (Phe), anthracene

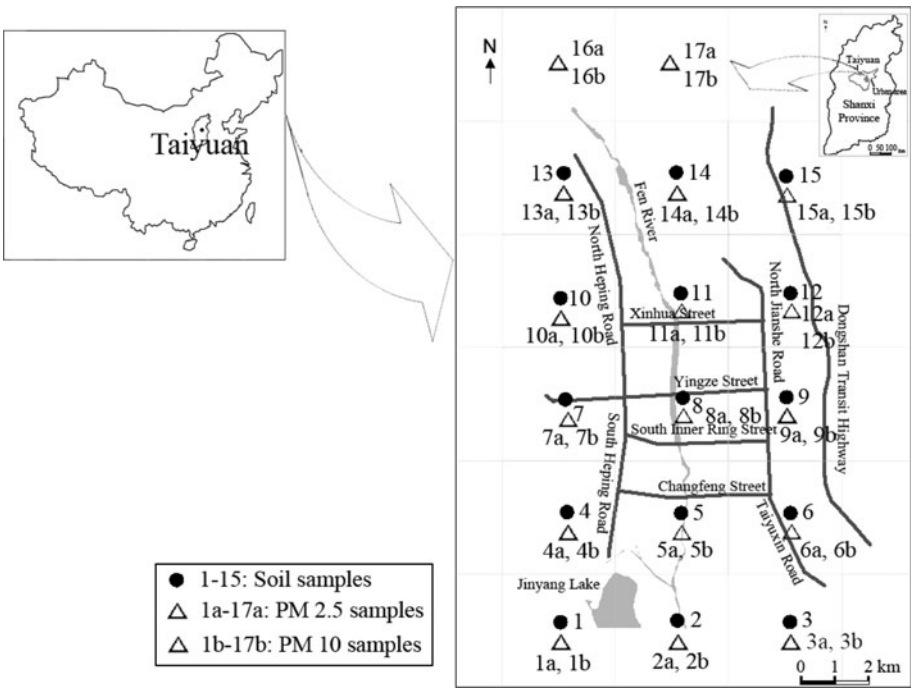

Fig. 1 Sampling sites of various environmental media in urban areas of Taiyuan

(An), fluoranthene (Flu), pyrene (Pyr), benz[a]anthracene (BaA), chrysene (Chr), benzo[b]fluoranthene (BbF), benzo[k]fluoranthene (BkF), benzo[a]pyrene (BaP), indeno-[1,2,3-cd]pyrene (InP), dibenz[a,h]anthracene (DBA), and benzo[g,h,i]perylene (BghiP) was purchased from Sigma-Aldrich, St. Louis, MO (Milwaukee, WI, USA). In addition, 2-flurobiphenyl (2-FBP), which was used as a surrogate standard, was purchased from Supelco (Bellefonte, PA, USA). The standards were then diluted to the desired concentration using isooctane and then used as working standards. Silica gel (100–200 mesh) was purchased from Qingdao Haiyang Chemical Co. (Qindao, China) and then activated in a drying oven at 550°C for 6 h. Anhydrous sodium sulfate (Beijing Chemical Factory, China) was heated at 600°C for 12 h prior to being used to eliminate organic contamination. All solvents used were of pesticide grade and were purchased from J.T. Baker (Phillipsburg, NJ, USA).

Five grams of each soil sample or each PM sample was ground with anhydrous sodium sulfate into a free-flowing powder. Each sample was extracted with 30 mL of hexane/acetone (1:1, vol/vol) by ultrasonication for 4 min and then centrifuged at 3,000*g*. During the extraction, actived copper was added for desulfurization. This process was repeated three times and the extracts were combined. Before extraction, 100 μL of 2-FBP (1 μg mL^{-1}) was added in each sample as a surrogate standard and balanced at desiccator for 2 h. The concentrated extracts were evaporated to 1 mL in a Kuderna-Danish concentrator under a gentle N_2 stream for cleanup.

The concentrated extracts were cleaned by using a chromatography column (30 cm × 10-mm internal diameter) containing 6 g of activated silica gel and 2 g of anhydrous sodium sulfate. The column was pre-eluted with 15 mL of hexane before loading the sample. The fraction containing PAHs was eluted using 25 mL of hexane/dichloromethane (3:2, v/v). The solvent was evaporated to 100 μL in the Kuderna-Danish concentrator under a gentle N_2 stream for analysis.

PAHs were measured with an Agilent 6890 gas chromatograph (GC) equipped with flame ionization detector (FID). Separation was performed on a 30 m DB-5 (30 m × 0.25 mm i.d., 0.25 μm film thickness) capillary column. The injector and detector temperatures were 280 and 300°C, respectively. The GC column was maintained at 70°C for 2 min, then increased by 4°C min^{-1} to 280°C and held at this temperature for 15 min. The total run time was 54.5 min. Quantification of the samples was performed using an external standard method. To confirm the PAH results, selected typical samples were checked using an Agilent 6890 series gas chromatograph coupled to an Agilent 5973 mass spectrometer (MS) using an electron impact ionization source (EI) in the selected ion monitoring (SIM) mode. In EI mode, the MS source temperature was 230°C, the transfer line was 300°C and the electron energy was 70 eV. Gas chromatographic separation was performed as mentioned above.

All analytical procedures were monitored using strict quality assurance and control measures. Field blanks consisted of pre-clean soil and pre-clean glass fiber filters which were taken to the sampling site. A total of six field blanks were extracted and analyzed in the same way as samples. The target compounds were not detected when the field blank samples were analyzed. A solvent blank, a procedural blank and a standard mixture were run per 10 samples to check for contamination, peak identification and quantification. The target compounds were not detected in the solvent blanks and the procedural blanks. Duplicate samples in the laboratory were analyzed along with regular samples to determine repeatability and reproducibility, for additional quality-control assessment to ensure valid results. Instrument stability and relative response factor variance were determined by analyzing the calibration standard solutions in each sample batch.

Identification of PAHs was confirmed, and concentration was measured using an external quantification standard consisting of known amounts of all the target compounds. The limits of detection for PAHs were defined by a signal-to-noise ratio greater than three times the average baseline variation and were within 0.58–1.03 ng g^{-1} (dry weight). At the same time, prior to extraction, each sample was spiked with a known concentration of 2-FBP as a surrogate to compensate for the loss of components. The recoveries of the 2-FBP surrogate were in the range 70%–95%. The recoveries of the surrogate were satisfactory and no correlation of analytical data was applied to the samples.

The standard reference material sample including 5 PAHs (GBW 08403, National Research Center for Certified Reference Materials, China) was analyzed to validate the analytical method used. The results were satisfactory, with a z score ≤ 1 for all congeners (range of 0.01–0.56 for the PAHs; $p < 0.05$). Recovery of 2-FBP surrogate was 85% with the GBW 08403 sample.

Results and Discussion

Sixteen PAHs were identified in 15 urban soil samples (Table 1). The detection rates of some PAHs in the urban soils reached 100%, indicating their wide distribution in Taiyuan. The total PAH concentration (defined as the sum of the 16 PAHs) ranged from 1.0 to 26 μg g^{-1} dry weight (median: 5.1 μg g^{-1}) (Table 1). The highest concentration was found at site 12 (26 μg g^{-1}) and the lowest value was found at site 1 (1.0 μg g^{-1}). When compared with other areas in China, the concentration of the ΣPAHs in the soil

Table 1 Total PAHs, low molecular weight PAHs (2–3-ring PAHs, LMW PAHs), high molecular weight PAHs (4–6-ring PAHs, HMW PAHs), selected PAHs ratios in soil, PM 2.5 and PM 10 in Taiyuan

Sampling sites	Total PAHs ($\mu g\ g^{-1}$)	LMW PAHs ($\mu g\ g^{-1}$)	HMW PAHs ($\mu g\ g^{-1}$)	ΣCOMB/ ΣEPA-PAHs	An/(An + Phe)	Flu/(Flu + Pyr)	BaP/BghiP
Surface soil							
1	1.0	0.1	0.8	0.87	0	0.59	0.80
2	1.0	0.2	0.8	0.82	0	0.55	0.63
3	2.2	0.3	1.9	0.84	0	0.56	0.75
4	11	1.3	10	0.87	0.09	0.59	0.95
5	2.2	0.3	1.8	0.82	0	0.58	1.15
6	1.8	0.3	1.6	0.81	0	0.51	1.06
7	5.6	0.5	5.1	0.88	0.12	0.57	0.97
8	15	2.0	13	0.85	0.12	0.59	0.94
9	5.1	0.9	4.2	0.80	0.10	0.57	0.86
10	3.7	0.3	3.4	0.88	0	0.53	0.56
11	14	1.5	13	0.87	0.14	0.58	0.86
12	26	1.9	24	0.89	0.12	0.58	0.80
13	10	1.1	9.5	0.87	0.14	0.58	0.87
14	26	3.0	23	0.86	0.11	0.58	0.82
15	4.5	0.7	3.8	0.76	0.92	0.60	0.30
Sampling sites	Total PAHs ($ng\ m^{-3}$)	LMW PAHs ($ng\ m^{-3}$)	HMW PAHs ($ng\ m^{-3}$)	ΣCOMB/ ΣEPA-PAHs	An/(An + Phe)	Flu/(Flu + Pyr)	BaP/BghiP
PM 2.5							
1	1.2×10^2	1.8	1.2×10^2	0.98	0	0.92	0.58
2	4.2×10^2	22	4.0×10^2	0.90	0.21	0.48	0.59
3	6.2×10^2	8.4	6.1×10^2	0.96	0	0.53	0.82
4	7.0×10^2	17	6.8×10^2	0.96	0.14	0.91	0.51
5	1.5×10^2	1.6	1.5×10^2	0.96	0	0.87	0.63
6	6.7×10^2	12	6.6×10^2	0.96	0.15	0.92	0.63
7	6.3×10^2	18	6.1×10^2	0.94	0.13	0.90	0.59
8	4.1×10^2	96	3.2×10^2	0.62	0.96	0.71	0
9	1.4×10^3	29	1.4×10^3	0.96	0.19	0.92	0.43
10	3.8×10^2	7.5	3.8×10^2	0.92	0.21	0.73	0.33
11	6.1×10^2	26	5.9×10^2	0.92	0.13	0.57	0.83
12	3.5×10^2	16	3.4×10^2	0.93	0.16	0.56	0.83
13	4.1×10^2	19	3.9×10^2	0.92	0.10	0.52	0.59
14	3.4×10^2	9.0	3.3×10^2	0.94	0	0.53	0.74
15	4.1×10^2	1.8×10^2	2.2×10^2	0.43	1.00	0.63	0
16	1.0×10^3	21	9.8×10^2	0.94	0.17	0.54	0.56
17	2.8×10^2	11	2.7×10^2	0.94	0	0.49	2.04
PM 10							
1	3.4×10^2	9.3	3.2×10^2	0.93	0.17	0.51	0.68
2	2.0×10^2	12	1.9×10^2	0.90	0.15	0.50	0.44
3	8.1×10^2	45	7.7×10^2	0.91	0.08	0.59	0.58
4	6.7×10^2	22	6.4×10^2	0.95	0.13	0.93	0.47
5	7.6×10^2	3.2	73	0.91	0	0.52	0.28
6	2.8×10^2	19	2.6×10^2	0.91	0	0.60	0.65
7	5.9×10^2	31	5.6×10^2	0.91	0.13	0.57	0.50
8	1.1×10^3	50	1.0×10^3	0.91	0.10	0.84	0.47
9	3.3×10^2	22	3.1×10^2	0.89	0.10	0.57	0.54

Table 1 continued

Sampling sites	Total PAHs (ng m^{-3})	LMW PAHs (ng m^{-3})	HMW PAHs (ng m^{-3})	ΣCOMB/ ΣEPA-PAHs	An/(An + Phe)	Flu/(Flu + Pyr)	BaP/BghiP
10	2.8×10^2	6.2	2.8×10^2	0.94	0	0.58	1.16
11	5.2×10^2	37	4.8×10^2	0.91	0.10	0.83	0.89
12	4.8×10^2	23	4.6×10^2	0.93	0.11	0.80	0.81
13	6.9×10^2	23	6.7×10^2	0.96	0.17	0.92	1.06
14	5.1×10^2	13	5.0×10^2	0.95	0.17	0.90	0.42
15	1.8×10^2	6.6	1.7×10^2	0.92	0	0.56	0.51
16	2.9×10^2	11	2.8×10^2	0.93	0.13	0.57	0.51
17	3.6×10^2	19	3.4×10^2	0.91	0.10	0.58	0.59

from Taiyuan was higher than those in surface soil in outskirt areas and urban area of Beijing (0.467–5.470 μg g^{-1}) (Li et al. 2006). Furthermore, the median concentration of PAHs in Taiyuan (0.4 μg g^{-1}) was much higher than that in vegetable soil in Guangzhou Province (160–370 ng g^{-1}) (Cai et al. 2007). Compared to urban soils from other countries, the levels were similar to those from urban soil in New Orleans (3.731 μg g^{-1} as median) (Mielke et al. 2001), but it was higher than those from soil in Tokushima in Japan (0.611 μg g^{-1} as mean) (Yang et al. 2002).

There were 16 PAHs identified in 34 respirable PM samples (17 of PM 2.5 and 17 of PM 10, respectively; Table 1). The total PAH concentration ranged from 1.2×10^2 to 1.4×10^3 ng m^{-3} (median: 4.1×10^2 ng m^{-3}) in PM 2.5 and 76 to 1.1×10^3 ng m^{-3} (median: 3.6×10^2 ng m^{-3}) in PM 10, respectively (Table 1). The medians concentration in PM 2.5 was higher than those obtained in PM 10 in Taiyuan. When compared with other areas in China, the median concentration of the ΣPAHs in the respirable particulate matter from Taiyuan was higher than those in urban area of Nanjing (62.6 pg m^{-3} for PM 2.5 and 86 ng m^{-3} for PM 10) (Wang et al. 2006). Few studies have been conducted on PM 2.5 and PM 10 to date; however, it is known that atmospheric pollution directly influences respirable particulate matter. Therefore, we compared the concentrations of PAHs in the samples collected for this study to atmospheric concentrations that have been previously reported. Compared to air from other countries, the levels were higher than those obtained from a traffic site in Turkey (412 ± 537 ng m^{-3} and 44 ± 39 ng m^{-3} for gas and particle phases, respectively) (Tasdemir and Esen 2007) and urban and industrial areas in Germany (1.87–7.12 ng m^{-3}) (Rehwagen et al. 2005). The investigation showed that PAHs levels in the present study were obviously high in composition with previous studies reported elsewhere in the literature.

As shown in Fig. 2, the concentrations of the ΣPAHs in the soil samples are higher in northeast Taiyuan. On the other hand, the spatial distribution of ΣPAHs in the PM 2.5 and PM 10 samples shows in Fig. 2 revealing an increasing trend in the south Taiyuan. This may be due to the distribution of industrial plants, which are primarily located in the south portion of the city. The industrial activities in Taiyuan result in the consumption of a large amount of energy. This energy consumption results in waste gas or particles that contain PAHs produced as a result of incomplete combustion being emitted into the atmosphere. Furthermore, the wind direction during the sampling period was northwest in Taiyuan. In the transmission process,

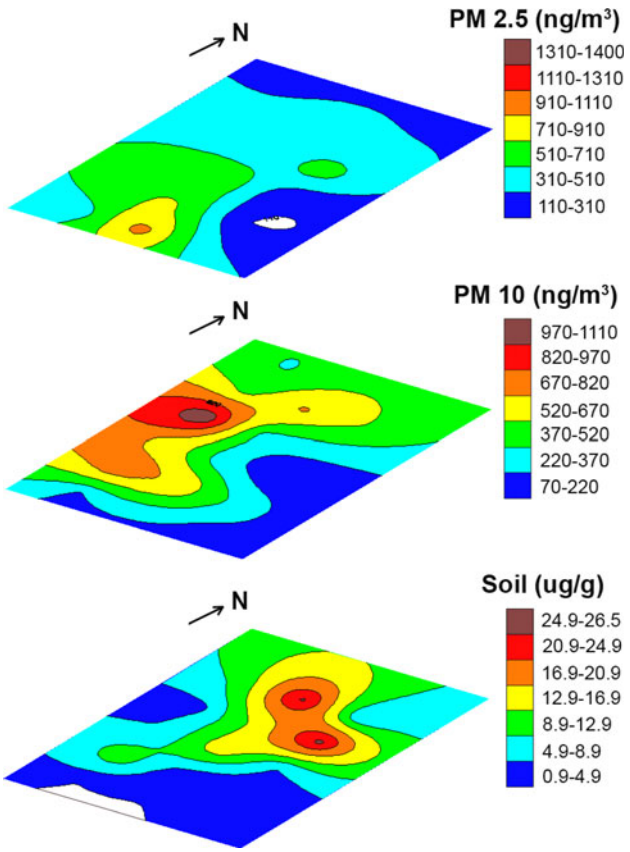

Fig. 2 The distributions of PAHs in PM 2.5, PM 10 and soil in urban areas of Taiyuan

PAHs could be absorbed by respirable particulate matter passing though the urban area, transported to the northeast area of Taiyuan, partially resulting in higher concentrations in the soil samples in this area.

There were some correlations between soil and respirable PM ($PM_{2.5}$ and PM_{10}). Concentration of ΣPAHs showed that soil was relatively the most polluted media (Table 1). The fluctuations in the distribution of PAHs were similar in soil, PM 2.5 and PM 10. The observed PAHs show that low molecular weight PAHs (LMW PAHs) such as Na, Acy, Ace and Fl in this study presented a lower level of concentrations, whereas high molecular weight PAHs (HMW PAHs) presented a higher level of concentrations, which was probably attributed to their different volatility (Bernal-Martinez et al. 2007). When compared with other report, Omar had reported higher proportions of lower molecular weight PAHs in soil particles, whereas high molecular weight PAHs were more abundant in atmospheric particles (Omar et al. 2002). However, the dominant PAHs detected from soil, PM 2.5 and PM 10, in this study were all Flu accounting for 12.3%, 12.4% and 13.4% as a median, respectively, followed by BbF and BghiP.

The composition of PAHs produced by different sources varies significantly. Therefore, the characteristic spectrum of PAHs can be used to identify their sources. The ratios of the sum of the major combustion specific compounds (ΣCOMB, including Flu, Pyr, BaA, Chr, BbF, BkF, BaP, InP and Bghip) to the sum of the 16 EPA-PAHS (ΣCOMB/ΣEPA-PAHs), as well the An/(An + Phe) and Flu/(Flu + Pyr) ratios are generally used to identify the sources of PAHs (Table 1) (Zhang et al. 2004). In general, combustion induces the production of relatively higher concentrations of ΣCOMB; therefore a large proportion of ΣCOMB is characteristic of PAHs that originated from combustion processes (Zhang et al. 2004). Conversely, an An/(An + Phe) ratio of <0.10 generally indicates that the PAHs were formed by petroleum combustion while an An/(An + Phe) ratio >0.10 indicates that the source of the PAHs was coal combustion (Budzinski et al. 1997). Furthermore, cruel oil samples generally produce a Flu/(Flu + Pyr) ratio of <0.40 while coal combustion generates a Flu/(Flu + Pyr) ratio of >0.40 (Zhang et al. 2004). In the present study, the ratios of the ΣCOMB/ΣEPA-PAHs ranged from 0.43 to 0.98 in soil, PM 2.5 and PM 10, which indicates that extensive combustion activities affected the PAHs in the samples. In addition, 70% of the PM 2.5 and PM 10 samples had the An/(An + Phe) ratio of 0.1–1.0. Moreover, the Flu/(Flu + Pyr) ratio ranged from 0.48 to 0.93 in the PM 2.5 and PM 10 samples. These findings further demonstrate that the PAHs originated from coal combustion sources.

The BaP/BghiP ratio can be used to distinguish traffic exhausts from coal combustion sources. For example, a BaP/BghiP of 0.3–0.44 indicates that the PAHs originated from auto exhaust, whereas a ratio of 0.9–6.6 indicates that the source was coal combustion (Sawicki 1962). Furthermore, a Flu/(Flu + Pyr) ratio of <0.40 is characteristic of crude liquid fossil fuel (traffic emission) combustion, whereas a ratio of >0.4 is characteristic of grass, wood or coal combustion (Zhang et al. 2004). In the present study, the ratio of BaP/BghiP ranged from 0 to 1.16 in all samples. In addition, 100% of Flu/(Flu + Pyr) were >0.4. As shown in Fig. 3, a plot of the BaP/BghiP ratio versus the Flu/(Flu + Pyr) ratio revealed that 80% of the samples fell within the combustion zone (III), which suggest that the source of the PAHs was a mixture of coal combustion and traffic emission. However, about 20% samples had a Flu/(Flu + Pyr) ratio of > 0.4 and a BaP/BghiP ratio of >0.9, which indicated that coal combustion is the primary source of the PAHs. Taken together, these results indicate that the primary combustion source was coal combustion, but that traffic emissions also influenced the presence of PAHs in soil, PM 2.5 and PM 10 samples collected from urban areas in Taiyuan.

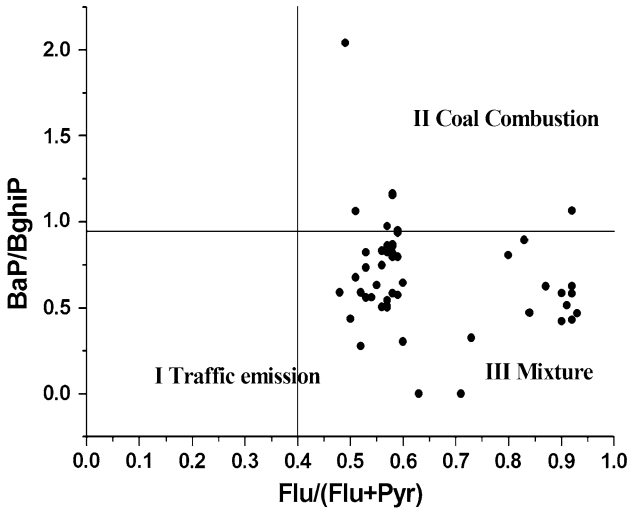

Fig. 3 PAH cross plot for the ratios of Flu/(Flu + Pyr) versus BaP/BghiP in soil, PM 2.5 and PM 10 in Taiyuan

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