



## Soil pollution by polycyclic aromatic hydrocarbons: A comparison of two Chinese cities

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### Abstract

Soil samples from Huizhou and Zhanjiang, China were analyzed for 16 priority polycyclic aromatic hydrocarbons (PAHs) with harmonized sampling, sample extraction and analysis quantification methods. The concentrations and sources of PAHs in soil samples of the two cities were compared. Almost all of the PAH components were detectable in 103 soil samples. The concentrations of  $\Sigma$ PAHs ranged from 35.40 to 534.5  $\mu\text{g}/\text{kg}$  in soil samples from Huizhou, and ranged from 9.50 to 6618.00  $\mu\text{g}/\text{kg}$  in samples from Zhanjiang. Evident differences of concentrations, compositions and sources of PAHs in soils were observed between the two cities. The average concentrations of individual component and the sum of a group of PAHs in soil samples from Zhanjiang were significantly higher than those in Huizhou ( $P < 0.05$ ). Phe, Flu, Pyr, Bbf and Baa were the dominant PAH components both in soil samples from Huizhou and Zhanjiang. Except for these five components, Bap, I1p, Daa and Bgp were also the dominant PAH components in soil samples from Zhanjiang. Coal combustion and liquid fossil fuel combustion were the same sources of PAHs in the two cities with different contributions, and petroleum played a key role in PAHs release in Zhanjiang.

**Key words:** polycyclic aromatic hydrocarbons (PAHs); urban soil; sources; principal component analysis (PCA)

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### Introduction

Polycyclic aromatic hydrocarbons (PAHs) are a group of persistent organic compounds composed of two or more fused aromatic rings. US Environmental Protection Agency (EPA) has identified 16 PAHs as priority pollutants, some of these PAHs are considered to be possible or probable human carcinogens, and their distribution in the environment have been the focus of much attention (Menzie et al., 1992).

Soil, comprising of mainly mineral particles and organic matters, is a major reservoir and sink for environmental pollutants because of their quantity and holding capacity (Ockenden et al., 2003). Wild and Jones (1995) estimated that at least 90% of the environmental PAH burden in Great Britain is stored in soil. Soil is also a good indicator of pollution and environmental risks. Accumulation of PAHs in soils may lead to contamination of vegetables and food chains (Kipopoulou et al., 1999), leading to direct or indirect human exposure. It was accepted that the amount of human exposure to PAHs through soil was higher than through air or water (Menzie et al., 1992). Hence, study on

PAHs in soil is important, and numerous studies have been conducted since PAHs were first detected at the surface of agricultural soils (Blumer, 1961). But in previous studies, sample collection, extraction and analysis of PAHs, and other methodology varied greatly, it is difficult to compare levels of PAHs contamination in different places reported by different authors (Morillo et al., 2007).

PAHs have been found to be the most abundant pollutants in soils (up to 28 mg/kg dry weight) of China (Cai et al., 2008). In the present study, two representative cities (Huizhou and Zhanjiang), located in Guangdong Province, south China were selected as study areas. Using the same methodology for sampling, extraction and chemical analysis to (1) investigate the degree of PAH pollution in the soils of the two cities; (2) identify the dominant PAH components and the major sources of PAHs in the two cities; (3) analyze the differences and causation of soil pollution by PAHs in the two cities.

### 1 Materials and methods

#### 1.1 Description of study areas

Huizhou located in the fast-growing Pearl River Delta

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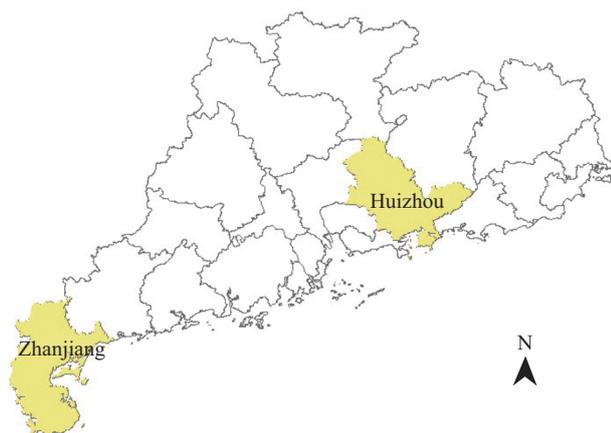


Fig. 1 General map showing the locations of the two studied cities.

of Guangdong Province, South China. Zhanjiang located in the northeastern part of the Leizhou Peninsula, South China (Fig. 1). The major soils from Huizhou and Zhanjiang are latosol, red earth, and paddy soil (GSSO, 1993). Characteristics of the two cities were listed in Table 1.

## 1.2 Sampling and preparation

Sampling of Zhanjiang was carried out from April to September 2004, and sampling of Huizhou was carried out from December 2004 to May 2005. Totally 103 composite samples were collected from surface soils (0–20 cm), in which 42 samples from Huizhou and 61 samples from Zhanjiang, respectively. Upon return to the laboratory, soil samples were placed in glass jars and stored in refrigerators at 4°C. Soil samples were air dried in room at 25°C before analysis, and then sieved to pass a brass mesh of aperture 2 mm and stored in cleaned glass jars with aluminium caps.

## 1.3 Analysis of PAHs

Totally 16 PAHs were analyzed and for brevity the following abbreviations for PAHs are employed: naphthalene (Nap), acenaphthylene (Acy), acenaphthene (Ace), dibenzo[a,h]anthracene (Daa), fluorene (Flu), phenanthrene (Phe), anthracene (Ant), fluoranthene (Fla), pyrene (Pyr), benzo[a]anthracene (Baa), chrysene (Chr), benzo[b]fluoranthene (Bbf), benzo[k]fluoranthene (Bkf), benzo[a]pyrene (Bap), indeno[1,2,3-cd]pyrene (I1p) and benzo[g,h,i]perylene (Bgp).

All solvents used for the extraction and cleanup procedures were residue-analysis grade and were obtained from Fisher Scientific (Fair Lawn, NJ, USA). Extraction of PAHs from soil samples were performed according to the standard method of US EPA (3540C method). Spiked with the surrogate standards and Soxhlet extracted with 200 mL of methylene chloride for 48 hr. A piece of activated Cu were added for desulfurization. The extraction was concentrated and solvent-exchanged to hexane, the rest

was blown down to 1 mL with nitrogen for cleanup. A alumina: silica (1:2) gel glass column was used for cleanup, with methylene chloride: hexane (3:7) as elution solvent. The 0–70 mL eluent was collected and concentrated to 1 mL, then internal standards (IS) was spiked to aid in quantification of the PAHs before gas chromatographic mass spectrometry (GC/MS) analysis.

Analysis of PAHs were performed according to the standard method of US EPA (8270C method). The analysis was carried out on GC (5890II, HP, USA) system equipped with a mass selective detector (MSD) (5973, HP, USA). A DB-5 capillary column (30 m × 250 μm × 0.25 μm) was used to separate the analyte. The GC oven was temperature-programmed from 70 to 200°C (at 3°C/min) to 285°C (at 5°C/min) and held isothermally at 285°C for 12 min. Helium was used as carrier gas. The temperature of the injection port and the ion source was maintained at 300 and 230°C respectively. GC injection was made in the splitless mode, and 1 μL of prepared sample was injected by an auto-injector. Identification of PAHs was conducted by comparing the relative retention time (RT) to IS. Only those peaks located within the proper range (2%) of RT were integrated for qualification and quantification, and further qualification was performed in selective ion monitoring (SIM) mode using three different ion masses. Quantifications were performed using external standards of a mixture of PAHs (Chem. Service Inc., USA). Final amount of corresponding PAHs were calculated from the relative signal ratio of PAHs in samples to those from IS.

Limit of detection was defined as three times the standard deviation from the mean blank, and concentration smaller than limit of detection was reported as not detected (n.d.).

## 1.4 Quality control

All data were subject to strict quality control procedures. Laboratory analytical blank and Certified Reference Material (CRM) were added in every two batches (18 samples) of Soxhlet extraction to assess the recoveries and procedural performance.

## 1.5 Data analysis

SPSS 13.0 was employed for data analysis. A two-sided *P* value < 0.05 was considered statistically significant.

# 2 Results and discussions

## 2.1 Concentrations of PAHs in soil samples

A total of 103 soil samples from Huizhou and Zhanjiang were analyzed for 16 individual PAHs. Descriptive statistics of concentrations of PAHs are listed in Table 2. Means, standard deviations (SD), minimums, maximums

Table 1 Characteristics of Huizhou and Zhanjiang

City	Population	Area (km <sup>2</sup> )	Annual average temperature (°C)	Annual precipitation (mm)	Reference
Huizhou	2,699,972	11,158	21–22	2069.5	Ma et al., 2008
Zhanjiang	7,100,000	13,000	22.9	1711	Zhang et al., 2007

of samples were reported for individual component and the sum of PAHs.

As shown in Table 2, all 16 PAH components were detectable in most soil samples with different detectable ratios. Nap, Flu, Pyr and Baa were 100% detected in soil samples from Huizhou, while in Zhanjiang, only Fle was 100% detected in soil samples. Concentrations of individual components varied greatly, depending probably on the location of the sampling point and its distance to contamination sources.

As analyzed above, in soil samples from Huizhou, the low molecular weight PAHs were found in all samples at lower concentrations than the higher molecular weight ones, presumably due to their greater volatility. Concentrations of PAHs in soil samples from Zhanjiang ranged from 9.50 to 6618.00  $\mu\text{g}/\text{kg}$  dw with the mean of 552.82  $\mu\text{g}/\text{kg}$  dw. In Huizhou, concentrations of PAHs ranged from 35.4 to 534.5  $\mu\text{g}/\text{kg}$  dw with the mean of 123.09  $\mu\text{g}/\text{kg}$  dw (Table 2). Both the concentrations of individual component and the sum of PAHs in Zhanjiang were much higher than those in Huizhou, which may be correlated to the fact that the population of Zhanjiang (7,100,000) is more than two times to that of Huizhou (2,699,972) (Table 1). Significant relationship between concentrations of PAHs in urban soils and the size of the population has been observed (Saltiene et al., 2002). Wilcke (2000) combined data of concentrations of PAHs in urban areas from 11 different publications and calculated a mean of 4420  $\mu\text{g}/\text{kg}$ , which are much higher than those of Huizhou (123.09  $\mu\text{g}/\text{kg}$ ) and Zhanjiang (552.82  $\mu\text{g}/\text{kg}$ ).

According to the classification of Maliszewska-Kordybach (1996) of soil pollution by PAHs (< 200, 200–600, 600–1000 and > 1000  $\mu\text{g}/\text{kg}$ , for “not contaminated”, “weakly contaminated”, “contaminated” and “heavily contaminated” samples, respectively), in Huizhou 33 soil samples (78.57%) were not contaminated, 9 samples (21.43%) were weakly contaminated; and in Zhanjiang 42 soil samples (68.85%) were not contaminated, 7 samples (11.48%) were weakly contaminated, 4 samples (6.56%) were contaminated and 8 samples (13.11%) were heavily contaminated.

In soil samples from Huizhou, Phe, Flu, Pyr, Bbf and Baa were the predominant components with high concentrations, the sum of these five components account for 52.49% of the total concentration of PAHs. In soil samples from Zhanjiang, except for Phe, Flu, Pyr, Bbf and Baa (the sum of these five components account for 51.08% of the total concentration of PAHs), Bap, I1p, Daa and Bgp were also the predominant contributors of PAHs (the sum of these components account for 34.82% of the total concentration of PAHs). The results were in line with previous study conducted in temperate region, where Phe, Flu and Pyr were predominant PAH components (Motelay-Massei et al., 2004).

The detectable ratios of low molecular weight PAHs (such as Nap) in Huizhou were higher than that in Zhanjiang. On the contrary, while the detectable ratios of high molecular weight PAHs in Zhanjiang were higher than that in Huizhou. This could be attributed to the influence

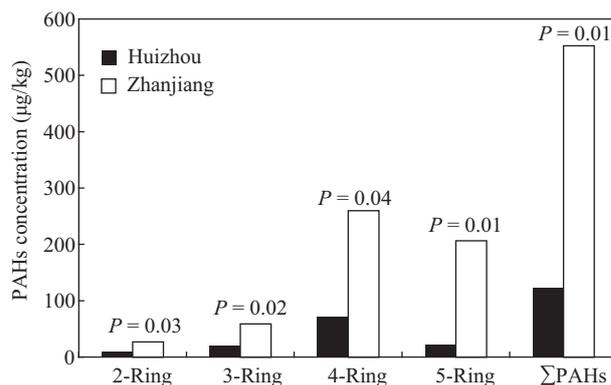


Fig. 2 Comparison of concentrations of PAHs in Huizhou and Zhanjiang ( $P$  value,  $T$  test).

of climate on the environmental behaviors of the low molecular weight PAHs. Compared to high molecular weight PAHs, low molecular weight PAHs are more easily to volatilize (Morillo et al., 2007). Zhanjiang located in tropical and subtropical zone, and Huizhou located in subtropical zone, the sunshine intensity and temperature of Zhanjiang are both higher than those of Huizhou (Table 1), which is propitious to volatilize for low molecular weight PAHs, resulting in low molecular weight PAHs could not be detected in some soil samples from Zhanjiang.

Comparisons of concentrations of PAHs in soil samples between Huizhou and Zhanjiang are shown in Fig. 2. The concentrations of 2-, 3-, 4-, 5-ring PAHs in soil samples from Zhanjiang were all significantly ( $P < 0.05$ ) higher than those of Huizhou, with  $P$  value Sig (2-tailed) of 0.03, 0.02, 0.04, 0.01, respectively. The concentrations of low rings (2-, 3-ring) PAHs were relatively low, while the concentrations of high rings (4-, 5-ring) PAHs were relatively high in both of Huizhou and Zhanjiang. Especially in Zhanjiang, the concentrations of 4- and 5-ring PAHs were much higher than that of 2- and 3-ring PAHs. Hence, higher rings PAHs were the dominant PAH components in the two cities, especially in Zhanjiang. The results were contrary to previous study (Gillian et al., 2007), which found that compared to temperate areas, tropical regions appear to have a higher relative abundance of 2- and 3-ring PAHs in soils.

Some previous studies on soil pollution by PAHs were selected and listed in Table 3. Concentration of PAHs in soil samples from Huizhou was very close to the determined values of Switzerland, and much lower than those of Guangzhou, Beijing, Yangtze River Delta, China and other countries such as UK, Japan and Norway. While concentration of PAHs in soil samples from Zhanjiang was much higher than those of other countries and regions except for Beijing. Generally, concentrations of PAHs in tropical soils are generally found to be much lower than those in soils of temperate zone (Wilcke, 2000). Zhanjiang located in subtropical and tropical regions with high temperature, plentiful rainfall and strong ultraviolet radiation (Table 1), which are helpful for PAHs to volatilize and degrade. Even though, concentrations of PAHs in soil samples from Zhanjiang were higher than those of other regions, which indicated that soil pollution by PAHs in

**Table 2** Concentrations of individual component and the sum of PAHs in soil samples from Huizhou and Zhanjiang (unit:  $\mu\text{g}/\text{kg dw}$ )

PAHs	Huizhou ( $n^a = 42$ )			Zhanjiang ( $n^a = 61$ )		
	$n^b$	Mean $\pm$ SD	Range	$n^b$	Mean $\pm$ SD	Range
Nap	42	5.10 $\pm$ 4.13	1.00–27.63	41	6.51 $\pm$ 13.32	n.d.–67.50
Ace	28	1.76 $\pm$ 1.17	n.d.–4.74	18	2.55 $\pm$ 5.40	n.d.–30.00
Acy	40	0.54 $\pm$ 0.76	n.d.–1.90	52	4.58 $\pm$ 9.25	n.d.–39.50
Fle	41	2.28 $\pm$ 0.88	n.d.–4.90	61	19.25 $\pm$ 41.49	1.00–287.50
Phe	41	18.63 $\pm$ 16.07	n.d.–57.50	47	50.93 $\pm$ 94.40	n.d.–445.00
Ant	22	1.10 $\pm$ 1.34	n.d.–4.31	46	26.24 $\pm$ 52.39	n.d.–321.50
Flu	42	18.53 $\pm$ 15.48	3.05–56.50	40	64.81 $\pm$ 129.68	n.d.–686.50
Pyr	42	11.11 $\pm$ 10.43	2.31–51.50	58	44.22 $\pm$ 103.69	n.d.–613.50
Chr	38	6.03 $\pm$ 4.99	n.d.–24.50	51	29.50 $\pm$ 63.77	n.d.–383.50
Baa	42	11.54 $\pm$ 12.40	1.93–62.50	40	56.70 $\pm$ 127.55	n.d.–615.50
Bbf	41	21.78 $\pm$ 27.76	n.d.–140.50	59	115.26 $\pm$ 282.28	n.d.–1838.00
Bkf	22	2.44 $\pm$ 3.81	n.d.–22.00	45	3.03 $\pm$ 1.91	n.d.–9.00
Bap	36	5.67 $\pm$ 7.23	n.d.–40.00	51	55.59 $\pm$ 123.14	n.d.–686.00
I1p	33	7.98 $\pm$ 9.71	n.d.–50.50	59	50.30 $\pm$ 132.22	n.d.–878.00
Daa	16	1.32 $\pm$ 2.87	n.d.–14.00	53	50.91 $\pm$ 75.44	n.d.–380.50
Bgp	30	7.27 $\pm$ 10.36	n.d.–51.00	59	69.45 $\pm$ 153.81	n.d.–873.00
2-Ring		9.68 $\pm$ 5.57	1.00–34.82		27.16 $\pm$ 51.26	1.00–359.50
3-Ring		19.73 $\pm$ 15.60	n.d.–57.50		59.02 $\pm$ 107.73	n.d.–527.00
4-Ring		71.43 $\pm$ 63.90	15.48–316.00		260.11 $\pm$ 592.80	3.00–3649.00
5-Ring		22.24 $\pm$ 28.96	n.d.–155.50		206.53 $\pm$ 432.37	3.00–2748.50
$\Sigma$ PAHs		123.09 $\pm$ 196.28	35.4–534.5		552.82 $\pm$ 1114.32	9.50–6618.00

<sup>a</sup> Total sample number; <sup>b</sup> detectable sample number of individual component.  
n.d.: not detected.

Zhanjiang is serious.

## 2.2 Identification of sources

PAHs are ubiquitous pollutants in the environment with various sources. Fraction of PAHs arise from endogenesis combustion such as forest fires, volcanic eruptions and diagenetic processes (Wilcke, 2000). Anthropogenic combustion of fossil fuels is the by far most important source of PAHs input in the environment (Baek et al., 1991).

It was estimated that endogenesis concentration of PAHs ranged from 1 to 10  $\mu\text{g}/\text{kg}$  in soil (Edwards, 1983). In the present study, concentrations of PAHs in soil samples were much higher than 10  $\mu\text{g}/\text{kg}$  with the exception of one samples (9.50  $\mu\text{g}/\text{kg}$ ), which indicated that anthropogenic source was the most important contributor of PAHs in Huizhou and Zhanjiang.

Compared to endogenesis source, anthropogenic source is more complex, which can be divided into petroleum and combustion sources (Yunker et al., 2002). Within combustion source, coal and liquid fossil fuel combustion were the major contributors. Generally, the petroleum source contain relatively higher concentrations of 2–3 ring PAHs compounds (Tolosa et al., 1996), while a large proportion of high molecular weight parent PAHs is typical characteristic of a combustion origin (Budzinski

et al., 1997). The ratios of low to high molecular weight PAHs (LPAHs/HPAHs) was generally used as a tool for discriminating the petroleum/combustion sources of PAHs. For example, 0.4, the ratio of Flu/(Flu+Pyr), was defined as the boundary of petroleum and combustion source. Indeed, the petroleum boundary ratio appears closer to 0.40 than 0.50 for Flu/(Flu+Pyr), and ratios between 0.40 and 0.50 are more characteristic of liquid fossil fuel (gasoline, diesel oil and crude oil) combustion, whereas ratios > 0.50 are characteristic of grass, wood or coal combustion (Yunker et al., 2002). The ratios of Flu/(Flu+Pyr) in the present study are listed in Table 4.

As shown in Table 4, in Huizhou samples, there were 33 out of 42 samples with the ratio of Flu/(Flu + Pyr) > 0.5, only 9 samples with the ratio ranged from 0.4 to 0.5, suggesting that PAHs in soil samples from Huizhou mainly come from grass, wood or coal combustion, with little contribution of liquid fossil fuels. Compared to Huizhou,

**Table 4** Number of soil samples from Huizhou and Zhanjiang in different Flu/(Flu + Pyr) ratios

City (total soil sample number)	Ratio < 0.4	Ratio = 0.4–0.5	Ratio > 0.5
Huizhou ( $n = 42$ )	0	9	33
Zhanjiang ( $n = 61$ )	28	12	21

**Table 3** Comparison of concentrations of PAHs in soils from different regions

Study area	Year	PAHs ( $\mu\text{g}/\text{kg dw}$ )	Mean ( $\mu\text{g}/\text{kg dw}$ )	Reference
Beijing, China	2006	467–5470	1637	Li et al., 2006
Guangzhou, China	1999, 2002	42–3077		Chen et al., 2005
Yangtze River Delta, China		8.6–3881	397	Ping et al., 2007
Hong Kong	2000	7.0–410		Zhang et al., 2006
United Kingdom	2008	42–11,200	640	Nam et al., 2008
Norway	2008	8.6–1050	150	Nam et al., 2008
Japan	1959–2002	52.9–2180	496	Honda et al., 2007
Switzerland	2002	50–619		Bucheli et al., 2004

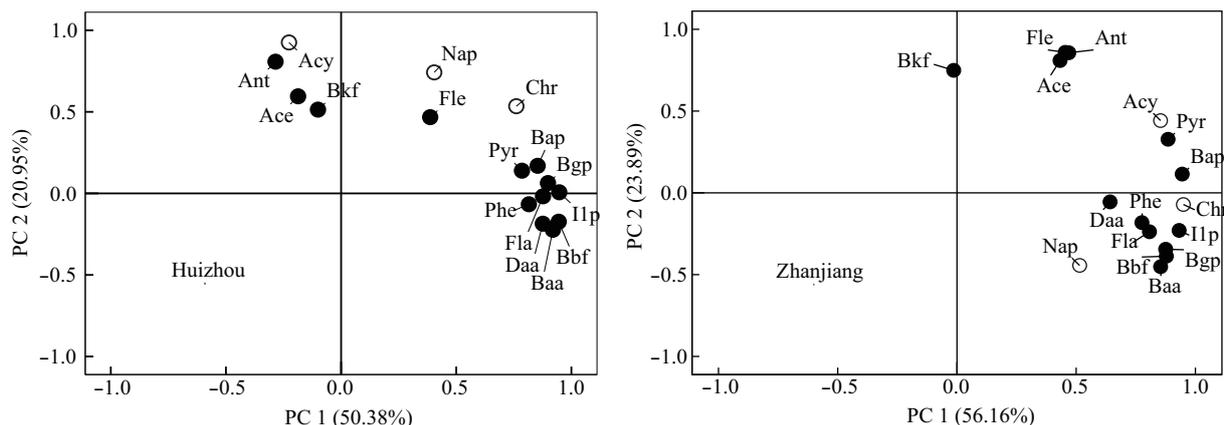


Fig. 3 Loading plots of PAHs in soil samples from Huizhou and Zhanjiang.

sources of PAHs in Zhanjiang are more complex: 28 samples with the ratio  $\text{Flu}/(\text{Flu} + \text{Pyr}) < 0.4$ , 21 samples with the ratio  $> 0.5$  and 12 samples with the ratio ranged from 0.4 to 0.5. Hence, the sources of PAHs in soil samples from Zhanjiang were in the following order: petroleum  $>$  grass, wood or coal combustion  $>$  liquid fossil fuels combustion.

Based on the analysis above, we deem that grass, wood or coal combustion and liquid fossil fuel combustion were the same main contributors of PAHs in Huizhou and Zhanjiang. According to our investigation, coal combustion maybe the most important contributor of PAHs in our study areas. Up to the present, coal is still the major energy origin of Guangdong Province, it was reported that 51.9% energy of Guangdong Province originate from coal combustion (Zhang et al., 2007). Except for coal, oil is another important energy origin of Guangdong Province, it was estimated that 5,448,500 tons oil were used for power generation in 2002 in Guangdong Province (Zhang et al., 2007). Hence, power generation plants (coal-fired and oil-fired) become important sources of PAHs in the two cities. As mentioned above, Phe, Flu and Pyr were the predominant PAH components both in soil samples from Huizhou and Zhanjiang, which further confirms the pyrogenic origin, cause Phe, Flu and Pyr are typical pyrogenic products derived from high temperature condensation of lower molecular weight aromatic components (Azimi et al., 2005).

Huizhou locates in the Pearl River Delta, the rapidest developing region in China in recent decades. The amount of vehicles increased rapidly in the area, and vehicles exhausts have been a major source of PAHs. It was estimated that there were 591,150 vehicles in Huizhou at the end of 2005 (Qiu et al., 2009). This is helpful to explain the fact that liquid fossil fuel combustion become a major source of PAHs in Huizhou. It is noteworthy that PAHs pollution in traffic and urban influenced areas originated not only from automobile exhausts, but also from tyre abrasion and tailpipe soot (Pyr is the most prominent PAH component from tyre abrasion) (Glaser et al., 2005), and this could be another explain for the high contribution of Pyr in soil samples from Huizhou.

Petroleum was also found to be another important source of PAHs in Zhanjiang. This can be explained by

the fact that Zhanjiang has a strong petroleum industrial heritage with a large amount of oil refineries, which have been proved to be important sources of PAHs (Bakker et al., 2000).

Principal component analysis (PCA) is a multivariate analytical tool used to reduce a set of original variables, and to extract a small number of latent factors (principal components, PCs) for analyzing relationships among the observed variables. PCA is an effective technique used for source identification and has the ability to focus on the most important or principal components (Harrison et al., 1996). In the present study, PCA was performed with the software SPSS 13.0 (Fig. 3).

As shown in Fig. 3, two PCs were extracted from the raw data of Huizhou and Zhanjiang, respectively. The two PCs explained 71.33% and 80.05% of the total variance of the raw data of Huizhou and Zhanjiang, respectively. PC1 was the most important contributor both in Huizhou (50.38%) and Zhanjiang (56.16%). Phe, Pyr, Bbf, Baa, Bgp, I1p, Fla and Daa were the same PAH components comprised in PC1 of the two cities. While, it is noteworthy that there were evident differences in the compositions of PC1 of the two cities. Compared to PC1 of Huizhou, there were more low rings (2- or 3-ring) PAH components (Nap, Acy and Chr) in PC1 of Zhanjiang. It is accepted that PAHs derived from petroleum spills characterized by the predominance of 2-, 3-ring PAHs (Tolosa et al., 1996). The result further demonstrated that petroleum was one of the main sources of PAHs in Zhanjiang.

### 3 Conclusions

In summary, 16 priority PAHs were detectable in most soil samples. Concentrations of PAHs in soils of the two cities were in high levels, which can be attributed to the large amount of PAHs come from anthropogenic sources. The average concentrations of individual component and the sum of a group of PAHs in soil samples from Zhanjiang were significantly higher than those of Huizhou. Phe, Flu, Pyr, Bbf and Baa were the dominant PAH components in soil samples from Huizhou and Zhanjiang. In addition, Bap, I1p, Daa and Bgp were also the dominant PAH components in soil samples from Zhanjiang. Coal combustion and liquid fossil fuels were the same sources of PAHs in

the two cities, and petroleum was another important source of PAHs in Zhanjiang. Further research is warranted to evaluate human exposure to PAHs and health risks in the two cities.

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