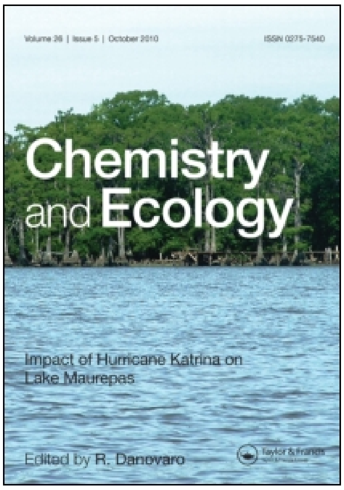

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RESEARCH ARTICLE

Polycyclic aromatic hydrocarbons in soils around Guanting Reservoir, Beijing, China

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The concentrations of 16 polycyclic aromatic hydrocarbons ($\sum$16PAHs) were measured by gas chromatography equipped with a mass spectrometry detector (GC-MS) in 56 topsoil samples around Guanting Reservior (GTR), which is an important water source for Beijing. Low to medium levels of PAH contamination (mean $= 394.2 \pm 580.7\,\mathrm{ng\,g^{-1}}$ dry weight (d.w.)) was evident throughout the region. In addition, localised areas of high PAH contamination near steel and cement factories were identified, with $\sum$16PAHs concentrations as high as 4110 ng/g, dry weight (d.w.). There was a significant positive correlation ($r^2 = 0.570$, $p < 0.01$) between total organic carbon content and $\sum$16PAHs concentrations. Phenanthrene was the predominant compound, accounting for 27.2% of the $\sum$PAH concentration, followed by chrysene > pyrene > benzo[a]anthracene≈benzo[b]fluoranthene≈benzo[a]pyrene. Four-ring PAH homologues (39%) were dominant. The higher proportion of 4–6 ring homologues, molecular indices, and the spatial distribution of PAH indicated that industrial discharges, incineration of wastes and traffic discharges were the major sources of soil PAHs around the water reservoir.

Keywords: PAHs; soil contamination; drinking water, Guanting Reservoir

1. Introduction

Polycyclic aromatic hydrocarbons (PAHs) are a group of organic compounds with two or more fused aromatic rings, and are some of the widespread contaminants throughout various environmental media [1–4]. Because of their toxic, mutagenic, and carcinogenic characteristics, the United States Environmental Protection Agency (US EPA), has listed 16 PAHs as priority pollutants. PAHs have received considerable attention from environmental contamination and human exposure points of view for over three decades [1,5,6,7]. PAHs arise from both natural and anthropogenic emissions including the incomplete combustion of organic matter, emissions of non-combustion-derived petrogenic sources, natural petroleum seeps, and products of biogenic

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transformations [1,8]. The diversity of PAH sources result in its ubiquity and complexity of origin in various environmental media. Once PAHs are released into the atmosphere, they attach to particles and, via dry and wet deposition, settle on soil. PAH concentrations in soil correlated significantly with atmospheric [9] and water concentrations [10]. Therefore, PAH concentrations in soil may provide important information on pollution in various environmental media. There are increasing efforts in the scientific reporting of PAHs in recent years in China [10–12]. However, few reports investigated PAH contamination in soils from conservation zones.

Guanting Reservoir (GTR) is Beijing's second largest water source for agricultural and industrial purposes. This reservoir was a source of drinking water for Beijing prior to 1997. However, industrial pollution and contamination from farming have substantially degraded the quality of water in this reservoir [13,14]. This is of great concern because Beijing currently has a severe shortage of drinking water. As the host of the summer Olympic Games in 2008, both Beijing and the national government have established a Beijing Olympic Action Plan, which includes water environmental protection. One of the action plans was the resumption of GTR as a drinking water source by 2010. Although regional surveys performed the analysis of PAHs in water and sediment [14], residue concentrations of PAHs in watershed area soils are less well known. Since watershed soils are the major sources of contamination, this study focused on the analysis of PAHs in soils from the watershed areas of the reservoir. Since 2002, we have conducted a comprehensive and systematic survey on measuring the levels of various inorganic and organic pollutants in the study area [15,16], assessing the health risks for the residents in the neighborhood of this important reservoir. This study is a pioneering survey of soils contaminated by PAHs around the reservoir. We have also analysed waterborne and sedimentary PAHs, which will be reported in another paper. The main objective of this study was to determine the concentrations and distribution characteristics and sources of 16 priority PAHs in soils around the reservoir. The baseline data will help to provide information for regulatory action concerning PAH pollution in water and soils.

2. Materials and methods

2.1. *Description of study area*

This study focused on a 920 km^2 watershed area around the Guanting Reservoir located northwest of Beijing, China (E115.43° $\sim$ 115.97°, N40.19° $\sim$ 40.50°), and includes 98 km^2 of reservoir and 820 km^2 of the surrounding land. The area has a cool continental monsoon climate, with an average annual temperature between 3 and 9 °C, and average annual precipitation between 370 and 480 mm. A northwest wind prevails in the reservoir area. Agricultural production is the major land use in the watershed of the reservoir, where corn and cash crops are the main crops. A small area of the land is used for industrial activities. The burning of crop residues including straw/stalk in the open air and/or the use of straw for domestic cooking are potential sources of PAHs in the watershed.

2.2. *Soil sampling and preparation*

Fifty-six composite samples were obtained in the Spring of 2007 (Figure 1). Each sample was a composite of subsamples taken from the top 20 cm of five sites and was mixed well to obtain a representative sample. The sample was then dried at room temperature [17], sieved through a 100-mesh sieve, and stored in glass bottles prior to analysis. At the time of sampling, site description including land use pattern and major environmental features were recorded. To eliminate the

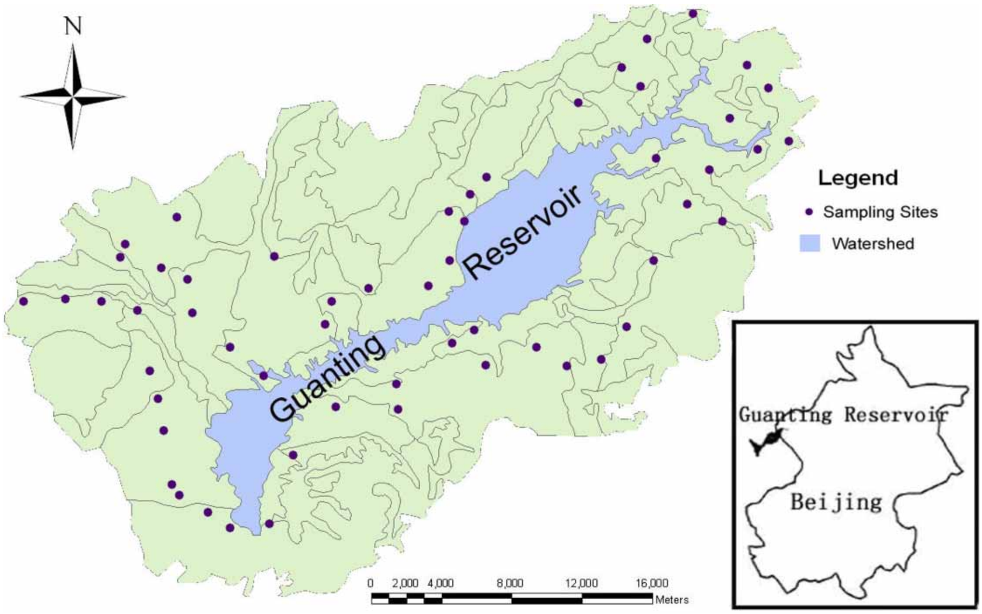

Figure 1. Location of the sampling sites around the Guanting reservoir.

effects of vehicle exhaust emissions, each sample was collected at least 50 m away from the nearby highway or road. Some sampling sites were specifically selected to cover point sources of contamination such as industrial areas.

2.3. *Sample extraction and fractionation*

Soil samples (about 5.0 g) were mixed with anhydrous sodium sulfate (2 g) and activated copper (1 g), and extracted using a Soxhlet apparatus. Each sample was spiked with a surrogate standard, phenanthrene-d_{10} (Supelco, Bellefonte, PA, USA), and then extracted with 210 ml of methylene chloride for 48 h. The extract was concentrated to 2 ml by a rotary evaporator and solvent-exchanged to hexane. The hexane extract was fractionated and cleaned by a silica gel column (Supelco). Sodium sulfate (1 cm) was added to the top of silica gel, and the column was eluted with n-hexane and 5 ml of methylene chloride/hexane ($v/v = 3 : 7$). The PAH fraction was concentrated to 1 ml under a gentle stream of nitrogen.

2.4. *PAHs analysis*

Concentrations of PAHs in soil extracts were determined by an Agilent 6890 gas chromatograph (GC) equipped with a 5973 mass selective detector (MSD) under selected ion monitoring (SIM) mode. A HP-5 fused silica capillary column (60 m × 0.25 mm inner diameter ×0.25 μm film thickness) was used with helium as the carrier gas at a constant flow rate of 1 ml/min. Splitless injection of 1 μl of the extract was made using an autosampler. The GC oven temperature was programmed from 50°C (2 min) to 200 °C (2 min) at 20 °C/min, then to 240 °C (2 min) at 5 °C/min before reaching 290 °C at 3 °C/min and then held for 15 min. The injector and detector temperatures were 280 °C and 300 °C, respectively. Mass spectra were acquired in the electron ionisation (EI) mode with an electron multiplier voltage of 906 eV. Before sample analyses, the instrument was tuned daily with decafluorotriphenylphosphine (DFTPP).

PAHs in the samples were identified by both the retention time and the abundance of quantification and confirmation ions with respect to authentic PAH standards. Standards of 16 US EPA priority PAHs [naphthalene (Nap), acenaphthylene (Acy), acenaphthene (Ace), fluorine (Fl), phenanthrene (Phe), anthracene (An), fluoranthene (Flu), 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 (Inp), dibenz[a,h]anthracene (DBA), and benzo[ghi]perylene (BgP)] in a mixture solution of 200 mg/ml and surrogate standard phenanthrene-d_{10} were purchased from Supelco (Bellefonte, PA, USA). Automated library searching was performed using the National Institute of Standards and Technology (NIST) Mass Spectral Database. Quantification was made using a five-point calibration curve for 16 individual PAHs. Detection limits were 1.7–4.9 ng/g dry weight for individual PAHs (Nap, 1.8 ng/g; Acy, 2.2 ng/g; Ace, 1.8 ng/g; Fl, 1.7 ng/g; Phe, 2.2 ng/g; An, 2.9 ng/g; Flu, 3.7 ng/g; Pyr, 3.1 ng/g; BaA,3.6 ng/g; Chr, 3.4 ng/g; BbF, 2.6 ng/g; BkF, 4.9 ng/g; BaP, 3.9 ng/g; Inp, 4.7 ng/g; DBA 3.5 ng/g and BgP, 2.7 ng/g). All the results are expressed on a dry weight basis.

2.5. *Quality assurance and quality control*

Laboratory quality control procedures included analyses of method blanks (solvent), spiked blanks (standards spiked into solvent), and matrix spikes. The analysis of matrix spikes and samples was performed in duplicate. Instrument stability and response were checked by injecting NIST standard solutions (DFTPP) periodically. The instrument was calibrated daily, and the relative percentage differences between the five-point calibration and the daily calibrations were $< 20\%$ for all of the target analyses. The recoveries of surrogate standards fell within a narrow range, and for individual PAHs, it was between 58.7 ± 7.4% (Nap) and 99.3 ± 5.8% (Flu).

2.6. *Soil chemical analysis*

Soil pH was measured (10 g of air-dried soil suspended in 25 ml deionised water) using a pH meter. Total organic carbon (TOC) was analysed using an Universal CHNOS Elemental Analyzer (Elementar Vario EL III, Germany).

2.7. *Statistical analyses*

The software used for mapping the sampling sites was ArcGis (ESRI, US). An interpolation method called ordinary Kriging was adopted for the mapping of geographical PAH contamination. SPSS 11.5 for Windows was employed for statistical analysis. Correlation analysis was used to explore the relationship between PAHs and TOC.

3. Results and discussion

3.1. *Soil PAH concentration in GTR*

Table 1 shows the concentrations of total PAH (sum of 16 priority PAHs, $\sum$ 16PAHs) and individual PAHs in 56 soil samples from the GTR. The concentrations below the limits of detection were assigned a value of half the detection limit for the calculation of mean. Concentrations of $\sum$ 16PAHs varied considerably among the soil samples, from 62.8 to 4110 ng/g dry weight (d.w.), with an average of 394 ng/g. The detectable ratios (DR, detectable ratio, is the ratio above the detection limit of each PAH) of the carcinogenic PAH compounds (BaA, Chr, BbF, BkF, BaP,

Table 1. Measured PAH concentration (ng/g dry weight (d.w.)) in the surface soils from the Guanting Reservoir.

PAHs	Rings	Mean	Min	Max	SD	Median	DR[c](%)	OR[d]
Nap	2	2.06	ND[b]	23.1	3.68	0.248	53.60	0.00
Acy	3	1.32	ND	13.5	1.81	ND	1.78	0.00
Ace	3	1.62	ND	42.5	5.68	ND	16.10	0.00
Fl	3	15.50	ND	251.0	33.80	8.03	91.10	0.00
Phe	3	90.40	ND	407.0	82.80	71.4	98.20	16.07
An	3	13.50	ND	337.0	49.90	1.97	87.50	3.57
Flu	4	22.20	ND	168.0	26.60	16.3	98.20	3.57
Pyr	4	39.80	5.46	336.0	51.30	27.7	100.00	5.36
BaA	4	36.30	3.52	404.0	58.10	19.6	100.00	5.36
Chr	4	56.20	6.32	493.0	84.70	31.1	100.00	10.71
BbF	5	29.90	4.06	168.0	32.50	18.8	100.00	3.57
BkF	5	17.70	1.34	337.0	44.60	8.67	100.00	1.79
BaP	5	32.40	4.61	405.0	55.90	17.60	100.00	3.57
Inp	6	11.40	3.58	409.0	54.10	4.07	100.00	1.79
DBA	5	7.99	0.34	226.0	29.80	3.16	100.00	1.79
BgP	6	18.40	2.06	184.0	26.80	9.91	100.00	1.79
Σ16PAHs[a]		394.00	62.80	4110.0	581.00	261.00		

Notes: [a] Sum concentration of 16 PAH compounds.
[b] Not detectable.
[c] Detectable ratio (detectable ratio is the ratio above the detection limit of each PAH).
[d] Over-limit ratio (over-limit ratio is the ratio above 100 ng/g for each PAH).

Inp, and DBA) were all 100%. The DR of Fl, Phe, An, Flu and Pyr were between 87.5 ∼ 98.2%. The DR of Ace was only 16.1%, and Acy was detectable in only one sampling site.

Concentrations of $\sum$ 16PAHs were higher than that (about 100 ng/g d.w.) reported for remote or rural soils from Estonia by Trapido [18]. The greatest concentrations of $\sum$ 16PAH in between the given numbers (4110 ng/g and 1610 ng/g) were found at the northeastern area of the GTR. The locations with the highest $\sum$ 16PAH concentrations in the GTR watershed are close to steel and cement factories, which use coal for energy. The emissions from the nearby factories are probably the main sources of PAHs in the GTR watershed. The difference between the two highly contaminated sites is the wind direction, as the most highly contaminated site is located downwind, while the second most highly contaminated site is located upwind, of the industrial point source.

In comparison with the results of previous studies in China, $\sum$ 16PAH concentrations of the GTR soils were lower than those reported for urban soil (mean 3917 ng/g) [11] and suburban soil (1308 ng/g) [19] in Beijing, China. PAH concentrations in the urban area were higher than in rural and agricultural areas due to intensive traffic and centralised house-heating [17]. $\sum$ 16PAH concentrations of the GTR soils were also lower than that in a southern rural area (464 ng/g) [19] of Beijing. The distance between downtown Beijing and the GTR or the rural area is one possible reason for this difference in the contamination, as the GTR is about three times farther from Beijing than southern rural area. The prevailing wind direction (northwest) is another possible reason for the difference. However, $\sum$ 16PAH concentrations in the GTR soils are higher than those found in soils around the Miyun Reservoir, China (58.3 ng/g) [20]. This is mainly because the Beijing government enforces more stringent pollution control measures around the Miyun Reservoir than the GTR, as the former is the current drinking-water source for Beijing. This suggests the need for controlling industrial and agricultural-related emissions of PAHs in the GTR watershed. In 1999, three different protection areas around the Miyun Reservoir were established. In contrast, industrial pollution and contamination from farming have substantially degraded the quality of water in the GTR [13,14], and the GTR could no longer be a drinking water source for Beijing after 1997.

Organic matter content is known to be one of the most important factors related to PAH accumulation in soils [11,21], by influencing chemical and biological processes of PAHs through sorption/desorption and/or biodegradation, etc. However, there is no agreement regarding this; some studies showed a positive relationship [5,11], while some showed a poor or no relationship between total PAH concentration and TOC content [22]. We further determined occurrence and concentrations of $\sum$ 16PAHs and PAHs with different rings by looking closely at the total organic content (TOC) in the samples and their relationships. TOC measured in the GTR samples varied considerably from 0.66% to 3.74% and there was significant positive correlation ($r^2 = 0.570$, $p < 0.01$) between TOC and $\sum$ 16PAH concentrations (Table 2). This positive correlation also existed between TOC and PAHs with different rings. TOC content in the industrial area was higher than the agricultural area; however, there is no other relationship between TOC and land use pattern or agricultural or industrial activities. Soil pH was measured between 7.17 and 8.16, and no correlation was observed between soil pH and $\sum$ 16PAH concentration.

3.2. *Spatial distribution of PAHs*

A soil pollution map of $\sum$ 16PAHs was generated by using a Geographic Information System (GIS). A clear spatial distribution pattern of PAHs around the GTR was observed. The sites with high PAH concentrations are mainly distributed northwest of the reservoir, which is the upstream of the reservoir. The PAH concentrations in the soils declined significantly from the northwest to the southeast (Figure 2). These results are in agreement with the PAH distribution in the water

Table 2. Correlations between PAH concentration and TOC content in soil from the Guanting Reservoir.

	t-PAHs	2-ring	3-ring	4-ring	5-ring	6-ring
TOC	0.570**	0.603**	0.570**	0.568**	0.565**	0.470**

Note: **Correlation is significant at the 0.01 level (2-tailed).

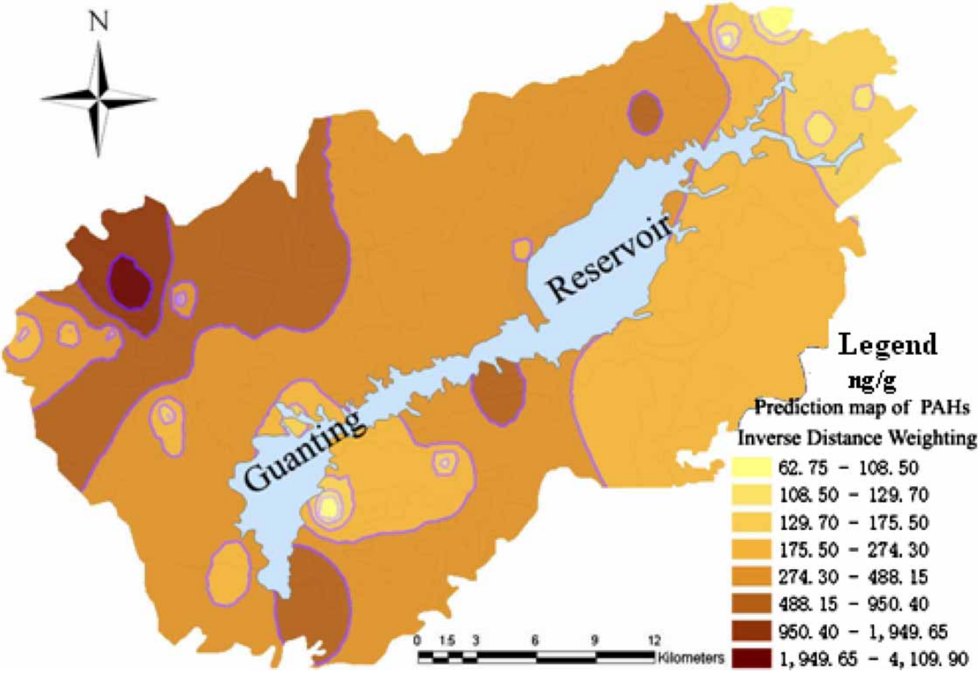

Figure 2. Distribution map of $\sum$16PAH concentrations in soils around the Guanting Reservoir.

from the GTR [14]. The locations with high PAH concentrations are close to steel and cement factories, which rely on coal for energy, and the discharge from the factories are the main sources of PAHs. Except for this industrial area, agricultural production is the major land use pattern in the GTR watershed. The prevailing northwest wind contributes to the apparent gradient of PAH found in the GTR watershed. There are more trees to the southwest of the reservoir, which can prevent the transport of PAHs through air movement. Since GTR will resume the function of a drinking water source by 2010, control over the discharge from nearby factories in the northwest of the reservoir is important to reduce the pollution.

Because environmental standards for PAHs in soils are not established in China and the Guanting Reservoir is a conservation area, we looked over the standards from some developed countries, such as the Netherlands [22] and Canada [23–26], and chose the strictest environmental standards for each PAH (100 ng/g). When comparing the PAHs values, only a few values were above 100 ng/g (Table 1). In this regard, 100 ng/g was chosen as a criterion for assessing the potential ecological effects of PAH levels found in the Guanting Reservoir. In this study (Table 1), Phe in 16% of the soil samples and Chr in 10 % of the samples were found to exceed the recommended limits (100 ng/g). The ratios of the other PAHs, whose concentrations are higher than 100 ng/g, were no more than 5%, which are mainly distributed around the industrial area. It is therefore believed that, based on the above criteria, the general levels of native PAHs in the GTR would not impose adverse health effects on humans. However, locations close to the steel and cement factories should be remediated to protect the reservoir as discussed.

3.3. *Soil PAH profiles and potential sources*

The contribution of individual PAHs to the $\sum$16PAHs concentrations varied greatly (Figure 3). Phe was the predominant compound, accounting for 27.2% of the $\sum$16PAHs concentration in our study, whereas the dominant compound in water was Nap reported by Wang [14]. Phe was followed by Chr>Pyr>BaA≈BbF≈BaP in our study, which was similar to the relative proportion of PAHs reported by Zuo [27] for the soils from the western watershed of the Bohai Sea, China

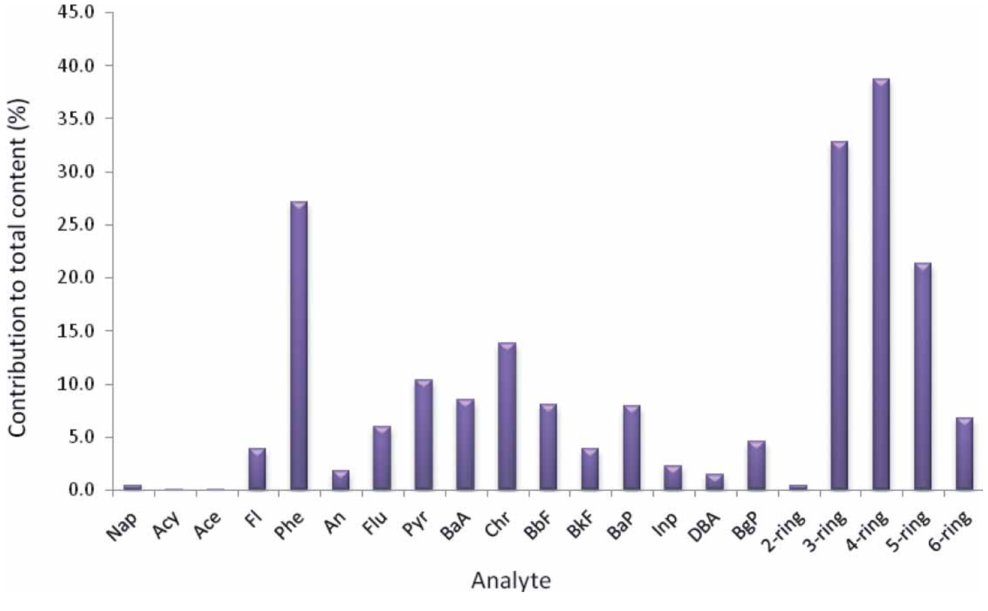

Figure 3. Profiles of PAH in the soils from the Guanting Reservoir.

(GTR included). The contribution of Nap in our study was lower than in the western watershed of the Bohai Sea reported by Zuo [27], which was probably due to the high volatility and different sources of Nap.

Among the PAHs with different benzene rings, 4-ring PAH homologues (39%) were the main contributors to $\sum$ 16PAH concentration (Figure 4), which is in agreement with that reported previously [19]. The 4–6 ring PAHs, which are considered as combustion-origin products, represented about 67% of the $\sum$ 16PAHs found in the GTR. This indicates that combustion may be the major source of soil PAHs around the GTR reservoir.

Molecular indices based on the ratios of individual PAH levels in soil can be used to distinguish PAHs from pyrogenic (e.g. fossil fuel combustion and vegetation fires) and petroleum (e.g. oil spill and petroleum products) inputs. Two specific PAH ratios were calculated for the studied samples: BaA/(BaA+Chr) and Inp/(Inp+BgP), which have been frequently employed in published studies [28–30].

Figure 4 presents these two isomer ratios in our samples. Yunker et al. [30] suggested that values of BaA/(BaA+Chr) ratio < 0.2 were indicative of origin PAHs, those > 0.35 were indicative of combustion origin PAHs, and results between these two critical values implied mixed origins. Most of the values of the BaA/(BaA+Chr) ratios are above 0.35, which indicates that combustion is the main source of PAHs in the reservoir. On the basis of Inp/(Inp+BgP) ratios, most of which were > 0.2, which further indicated that combustion is the main source of PAHs in this area. About half of the values are above 0.35, biomass (for example wood, grass or coal) may represent the major origin of PAHs in the region. The locations with high PAH concentrations are close to the steel and cement factories which rely on coal for energy, and agricultural production is the major land use pattern in the watershed of the reservoir where corn, wheat, soy bean, apricots and grapes are the main crops. Therefore, coal combustion from factory and straw stalk burning in open areas after harvest and/or use of crop residues/firewood for cooking are the main sources of PAHs around the reservoir. About one quarter of the values are between 0.2 and 0.35, so

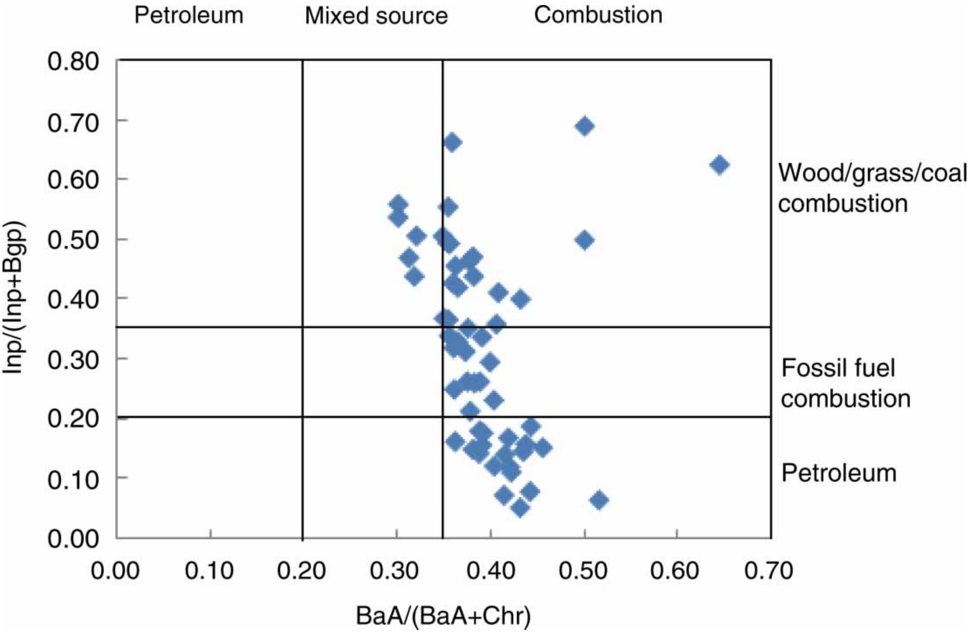

Figure 4. Plots of PAH index pair ratios for source identification.

liquid fossil fuel combustion may be another source of PAHs around reservoir. There are several highways going through the reservoir, and automobile emissions may be sources of PAHs.

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