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SEDIMENTS, SEC 1 · SEDIMENT QUALITY AND IMPACT ASSESSMENT · RESEARCH ARTICLE

Seasonal variations of concentrations, profiles and possible sources of polycyclic aromatic hydrocarbons in sediments from Taihu Lake, China

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Abstract

Purpose Obtaining a better understanding of the concentrations and origins of polycyclic aromatic hydrocarbons (PAHs) in the sediments Taihu Lake, China, is of paramount importance for the environmental protection and remediation of this lake. To investigate temporal and spatial variations in PAH concentrations, composition and possible source categories, 29 sediment samples were collected from Taihu Lake during both the flooding and dry seasons of the lake.

Materials and methods Fifteen US Environmental Protection Agency priority PAHs were detected in 58 surface sediments (29 for each season) by gas chromatograph/mass selective detection, following extraction by accelerated solvent extraction.

Results and discussion The concentrations of the total and individual PAHs in the flooding season were higher than those in the dry season, suggesting that high levels of fishing activity may be an important contributor to PAH pollution in the flooding season. The fractions of high molecular weight PAHs in the flooding season ranged from 63 % to 71 % and were higher than those in the dry season (which ranged from 52 % to 65 %). These results indicate that vehicle exhaust may be a more important pollutant source in the flooding season than in the dry season. Diagnostic ratios, principal component analysis and hierarchical cluster analysis were used to study possible source categories in the different seasons. Consistent results were obtained for all techniques. Seasonal and spatial variations were also investigated by the coefficient of divergence method. The results of previous studies support the conclusion of source identification.

Conclusions Vehicle emissions were the dominant contributor to PAHs in the flooding season, while PAHs in the dry season sediments may have come from multiple sources. The findings of this study may provide a theoretical basis for seasonal PAH control strategies for Taihu Lake.

Keywords PAHs · Profile · Seasonal variation · Sediments · Source identification

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1 Introduction

Polycyclic aromatic hydrocarbons (PAHs) have been listed as priority pollutants by international environmental protection agencies, drawing considerable attention to their effects on human health. These compounds have elicited serious concern due to their persistence in the environment, long-range transport properties, toxicity and health risk as potential carcinogens and mutagens (Christensen and Bzdusek 2005; Meng et al. 2011). PAHs have consequently become the subject of studies by a number of scientific communities and governments (Fernández et al. 2000; Yu et al. 2009; Oliva et al. 2010; Wang and Yang 2010; Mikac et al. 2011). PAHs are known for their prevalence and persistence in the environment due to their chemical stability and hydrophobic nature (Beg et al. 2003; Sun et al. 2011), especially in sediments in petroleum-impacted environments (Lei et al. 2005; Pies et al. 2008). Studying PAHs in aquatic systems is of considerable significance due to the sensitivity of aquatic systems to pollutants; investigating the behaviour of PAHs in sediments is particularly important because sediments can easily adsorb PAHs (Cornelissen et al. 2006; Witt et al. 2010).

This work investigates PAHs in Taihu Lake, which has an area of 2,250 km^2 and is the third largest freshwater lake in China. With the rapid industrialisation and urbanisation of the surrounding region, Taihu Lake has been plagued in recent years by pollution, such as vehicle exhaust emissions from fishing boats, industrial wastewater, municipal sewage, roadway runoff and agricultural non-point sources. Developing an understanding of the concentrations and origins of PAHs in sediments is essential to the management of the aquatic environmental of Taihu Lake. High levels of PAHs in Taihu Lake sediments have attracted considerable attention from the Chinese government and several researchers (Wang et al. 2003; Qiao et al. 2006; Liu et al. 2009). There has been very limited work performed, however, on the temporal and spatial variations in PAH profiles and possible sources of PAHs in Taihu Lake sediments.

To investigate the seasonal and spatial variations of the levels, profiles and potential sources of PAHs, 15 US Environmental Protection Agency (US EPA) priority PAHs were studied in 29 sediments from Taihu Lake for each of two seasons. The PAH levels and profiles for different regions of the lake in the flooding season (September) and the dry season (May) were analysed. Possible sources for PAHs were then identified using PAH diagnostic ratios, principal component analysis (PCA) and hierarchical cluster analysis (HCA). Finally, the coefficient of divergence (CD) method was used to analyse the dissimilarity in PAH profiles in sediments to study seasonal variations. The findings of this study may provide a theoretical basis for PAH control strategies for Taihu Lake in different seasons.

2 Materials and methods

2.1 Sediment sampling

A total of 58 surface sediments were collected from Taihu Lake in 2010, corresponding to 29 sediments collected for the dry season (May) and 29 sediments collected for the flooding season (September). Locations of the sampling sites are shown in Fig. 1. A global positioning system (GPS) was used to locate the sampling sites during the survey. All surface sediment samples (0–20 cm) were collected with a stainless steel grab sampler and homogenised on site. The samples were then immediately stored in glass bottles with Teflon-lined caps at 4 °C. The samples were transported on ice to the laboratory. The upper aqueous sediment layers were removed by high-speed centrifugation, and the remainder of the sediment samples was stored at −20 °C until further treatment.

2.2 Extraction and instrumental analyses

The samples were extracted by accelerated solvent extraction (ASE; Fisher et al. 1997). The sediment samples were freeze-dried and homogenised. Five grams of each sediment sample, combined with 5 g of copper powder and 2 g of diatomite, was extracted by an automated Dionex ASE 200 (Sunnyvale, CA, USA), using a hexane-dichloromethane mixture (50/50, *v*/*v*) as the extraction solvent. The extraction temperature was 100 °C and the extraction pressure was 1,500 psi. Both the preheating time and static time were set to 5 min. A total flush volume of 100 % of the cell volume was used by purging with nitrogen for 60 s. The final extraction volume was approximately 20 ml after two extraction cycles. Complete condensation of the extracts to approximately 2 ml was followed by solid phase extraction (SPE; HyperSep C18 cartridges, 500 mg/6 ml, Thermo Electron Corporation) for clean-up. The extracts were then concentrated to 1 ml for analysis. Further details on the SPE cleaning process can be found in Zhang et al. (2011).

PAH detection was performed on an Agilent 6890 N gas chromatograph, equipped with a 5975C mass selective detector (GC/MSD), an Agilent 7683B automatic liquid sampler, and a HP-5 MS capillary column (30 m, 0.25 mm i.d., 0.25 μm film thickness). Helium was used as the carrier gas, with a column flow rate of 1.2 ml min^{-1} in constant-flow mode. The injector, ion source and transfer line temperatures were set at 250 °C, 230 °C and 280 °C, respectively. The GC oven temperature was programmed from 70 °C (2 min) to 260 °C (8 min) at 10 °C min^{-1}; the temperature was then increased to 300 °C at 5 °C min^{-1} and held for 5 min. The electron impact energy was set at 70 eV. A 1-μl sample was injected in splitless mode. PAHs were analysed in the selected ion monitoring (SIM) mode. The following 15 compounds were detected in this study: naphthalene (Nap), acenaphthene (Ace), fluorine (Flu), phenanthrene (Phe), anthracene (Ant), fluoranthene (Flua), pyrene (Pyr), benzo[a]anthracene (BaA), chrysene (Chr), benzo[b]fluoranthene (BbF), ben-zo[k]fluoranthene (BkF), benzo[a]pyrene (BaP), indeno[1,2,3-c,d]pyrene (IcdP), dibenzo[a,h]anthracene (DahA) and benzo[g,h,i]perylene (BghiP).

2.3 Quality assurance and quality control

An internal calibration procedure was used to quantify PAH concentrations. The correlation coefficients for the five concentration gradients ranged from 0.995 to 0.999. A calibration standard was analysed daily to determine the accuracy of the calibration curves. To estimate the repeatability and accuracy

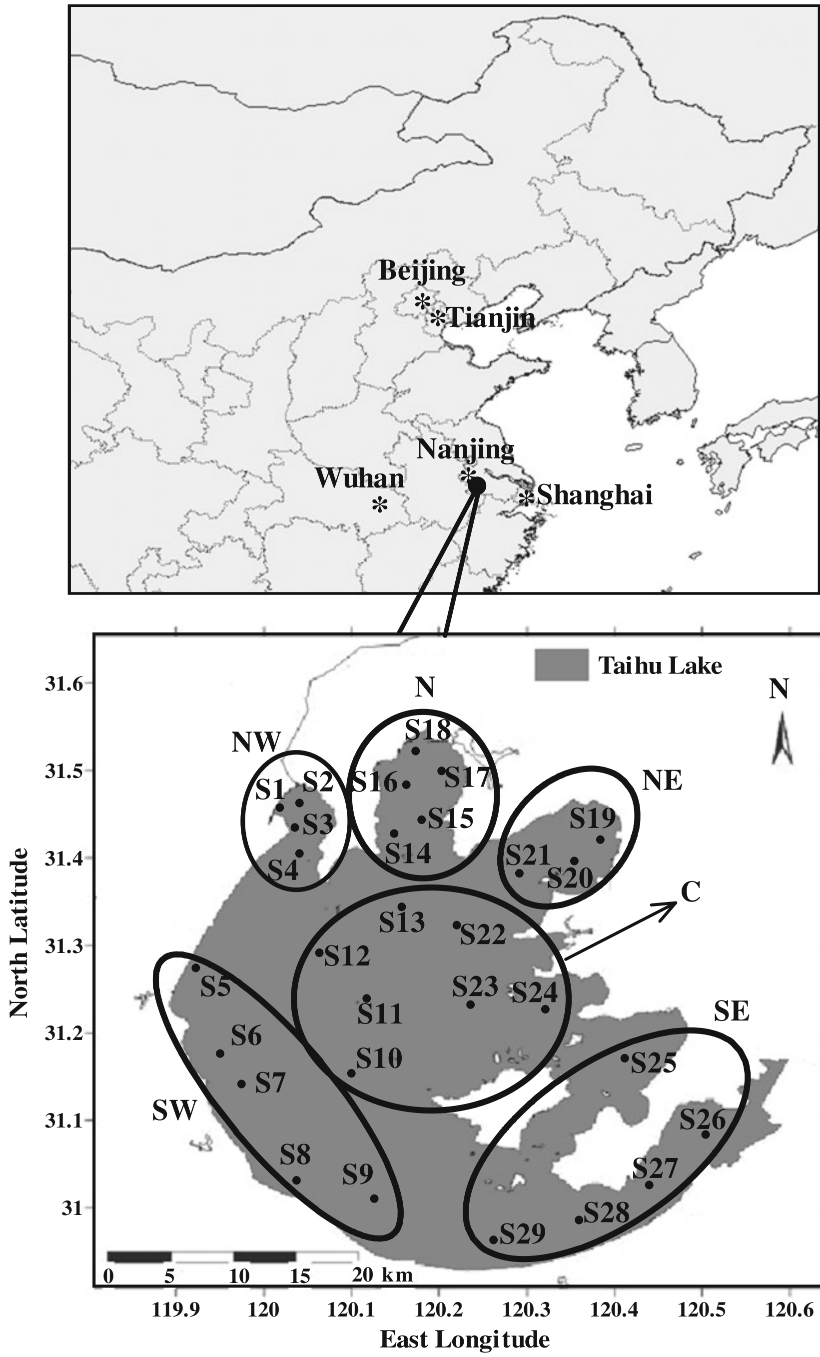

Fig. 1 Sampling sites in Taihu Lake, China: *NW* (northwest, *S1–4*), *N* (north, *S14–18*), *NE* (northeast, *S19–21*), *SW* (southwest, *S5–9*), *SE* (southeast, *S25–29*) and *C* (center, *S10–13* and *S22–24*)

of the analytical method, every sample was spiked with known amounts of surrogate standard mixtures prior to extraction. The surrogate recoveries in the sediment were 70.2±7.5 % for naphthalene-d8, 84.5±5.6 % for acenaphthene-d10, 92.5±5.6 % for phenanthrene-d10, 92.4±10.2 % for chrysene-d12 and 89.0±9.4 % for perylene-d12. Quality control for the method was maintained by analysing the following controls for each batch of samples: method blanks (solvent), spiked blanks (standard spiked into solvent), matrix spikes/matrix spike duplicates, and sample duplicates. The method detection limit was 1.0-6.0 ng g^{-1} dry weight (dw) for the sediment. For data reduction purposes, masses calculated from areas below the limit of detection were equated to zero. Further details can be found in Zhang et al. (2011).

3 Results and discussion

3.1 PAH concentrations in sediments

The levels of 15 priority PAHs were measured in surface sediment samples taken from Taihu Lake for each season, at

29 sites spanning the lake. Figure 2 shows that the sum of 15 PAH concentrations (∑PAHs) for the whole lake in the flooding season ranged from 255.07 ng g^{-1} dw (S10) to 1,059.39 ng g^{-1} dw (S2), averaging 580.54±236.99 ng g^{-1} dw. Figure 2 also shows that the ∑PAHs for the dry season ranged from 208.94 ng g^{-1} dw (S10) to 1,002.78 ng g^{-1} dw (S2), with an average value of 472.14±95.55 ng g^{-1} dw. The average concentrations for the individual PAHs are shown in Fig. S1 (Electronic Supplementary Material). The histogram shows that the concentrations of the individual PAHs were all higher in the flooding season than in the dry season.

The spatial distribution of the ∑PAHs was studied by grouping the 29 sites spanning Taihu Lake into six regions (see Fig. 1): NW (northwest: S1–4), N (north: S14–18), NE (northeast: S19–21), SW (southwest: S5–9), SE (southeast: S25–29) and C (centre: S10–13 and S22–24; see Fig. 2). Figure 2 shows that for all six regions, as well as for the whole lake, the average concentrations of ∑PAHs in the flooding season were higher than those in the dry season. The average concentrations of ∑PAHs for the NW, N, NE and SE regions were also higher than for the whole lake in the flooding season. In the dry season, however, only the ∑PAHs of the NW and N regions were higher than that of the whole lake, possibly because September is usually fishing season in Taihu Lake. There is increased motor boat activity on the lake in September, especially in the NW, N, NE and SE regions, which are important fishing areas. Fishing activities may therefore exert a stronger influence in the flooding season than in the dry season.

To explain the PAH levels in the Taihu Lake sediments, the ∑PAHs concentrations found in this study were compared with those documented in other studies. Table S1 (Electronic Supplementary Material) shows that the ∑PAHs concentrations for both seasons in this study were higher

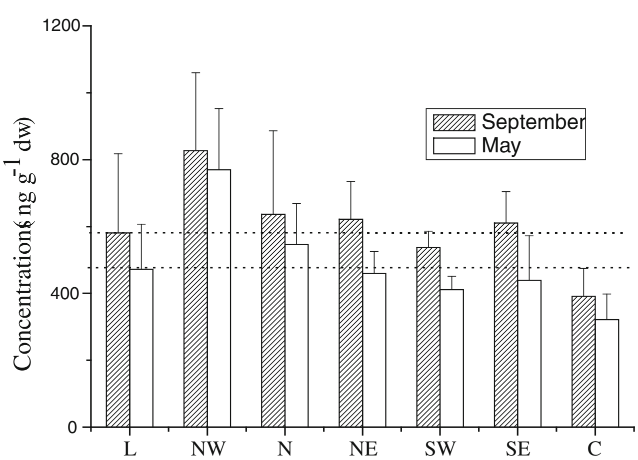

Fig. 2 The average concentrations (ng g^{-1} dw) of ΣPAHs in sediments from different parts of Taihu Lake for two different seasons. *L* whole lake, *NW* northwestern part, *N* northern part, *NE* northeastern part, *SW* southwestern part, *SE* southeastern part, *C* center part

than those in the Henan Reach of the Yellow River (16.4–1,358 ng g^{-1} dw), China (Sun et al. 2009), but lower than the ∑PAHs for most other watersheds in China and other countries. Some examples of ∑PAHs are: Yangtze River (72.4–3,995.2 ng g^{-1} dw) and Yellow River (464–2,621 ng g^{-1} dw) in China, Shinano River (102–10,450 ng g^{-1} dw) in Japan, Gomti River (5.24–3,722.87 ng g^{-1} dw) in India and Saginaw River watershed (50–7,590 ng g^{-1} dw) in the USA (Koh et al. 2004; Feng et al. 2007; Xu et al. 2007; Hori et al. 2009; Horii et al. 2009; Shen et al. 2009; Vinas et al. 2009; Fu et al. 2011; Malik et al. 2011; Wang et al. 2011; Li et al. 2012). In summary, the ∑PAHs in the Taihu Lake sediments were lower than those documented for other watersheds in China, as well as for watersheds in other countries.

3.2 PAH profiles in sediments

PAH profiles can reveal possible pollutant sources (Manoli et al. 2004). Figure 3 shows average PAH sediment profiles for six sampling sites in Taihu Lake for both seasons. For all the PAH profiles, the high molecular weight PAHs (4–6 rings) show the highest fractions, ranging from 52 % to 71 %, while the low molecular weight PAHs (2–3 rings) fractions ranged from 29 % to 48 %. Petroleum-derived residues generally contain relatively high concentrations of low molecular weight PAH compounds, whereas high molecular weight PAHs are formed in high temperature combustion processes (Mai et al. 2002; Xu et al. 2007). Our findings therefore indicate that high temperature combustion processes are the dominant PAH source category in Taihu Lake.

Seasonal variability of the individual PAH fractions was also investigated (see Fig. 3). In the flooding season, fractions of high molecular weight PAHs ranged from 63 % to 71 %, which were higher than those in the dry season which ranged from 52 % to 65 %. Some high molecular weight PAHs, such as IcdP, DahP and BghiP, were more abundant in the flooding season (23–26 %) than in the dry season (14–15 %). These high molecular weight PAH species can be used as source markers for vehicle emissions (Harrison et al. 1996; Larsen and Baker 2003; Valavanidis et al. 2006), indicating that vehicle exhaust emission sources may be more important in the flooding season than in the dry season.

3.3 Source identification

3.3.1 Diagnostic ratios of PAHs

The concentrations of some PAH species and their ratios can provide information about the possible source categories of PAH pollutants (Guo et al. 2003; Grigoriadou et al. 2008; Shi et al. 2009). In this study, the ratios of Flua/(Flua + Pyr) and BaA/(BaA + Chr) were examined to identify possible

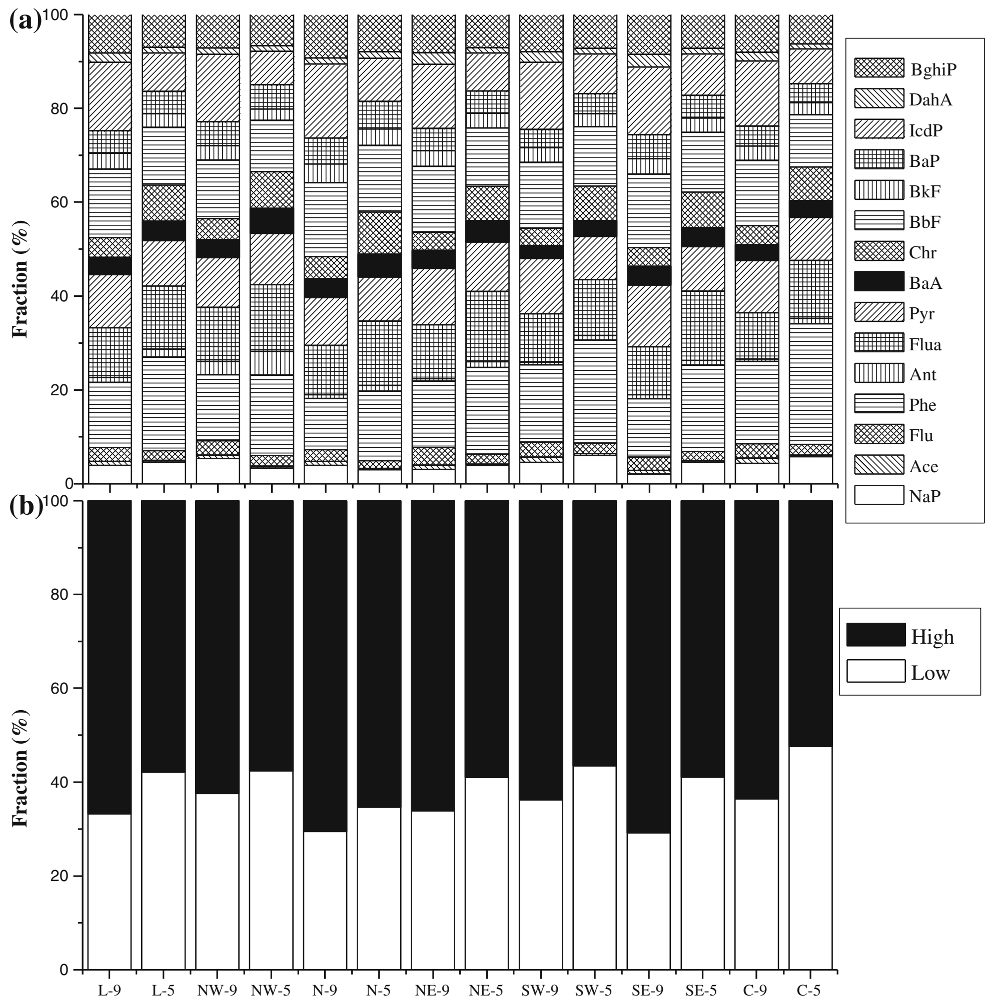

Fig. 3 Average PAH profiles in sediments for different parts of Taihu Lake for two different seasons. (**a**): fractions of 15 individual PAHs; (**b**) fractions of high and low molecular weight PAHs. L-9 (−5): all 29 sediments in Taihu Lake for September (for May). NW-9 (−5): sediments in northwestern part for September (for May). N-9 (−5): sediments in northern part for September. (for May). NE-9 (−5): sediments in north-eastern part for September (for May). SW-9 (−5): sediments in southwestern part for September (for May). SE-9 (−5): sediments in southeastern part for September (for May). C-9 (−5): sediments in center part for September (for May)

sources of PAHs. Figure 4 presents a PAH cross-plot for these two ratios, for all the sediments in Taihu Lake in both seasons. Generally, a ratio of BaA/(BaA + Chr) <0.2 suggests petroleum-based pollutants, a ratio >0.35 indicates that combustion sources are dominant, while a ratio between 0.2 and 0.35 indicates mixed sources (Hu et al. 2011). For Flua/(Flua + Pyr), a ratio <0.4 indicates a petroleum-based origin, a ratio from 0.4 to 0.5 suggests petroleum combustion sources and a ratio >0.5 implies coal/grass/wood combustion sources (Yunker et al. 2002). Figure 4 shows that combustion sources may significantly impact sediments in both seasons. Vehicle exhaust emissions (petroleum combustion) may be the dominant PAHs source in the flooding season, while PAHs may originate from multiple sources in the dry season.

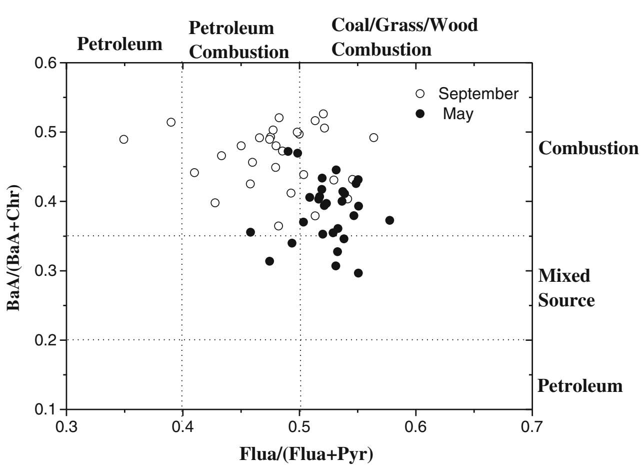

Fig. 4 PAHs cross plot for Flua/(Flua + Pyr) and BaA/(BaA + Chr) in sediments of Taihu Lake for two seasons

The results of the diagnostic ratios are reasonable because the fishing season occurs every September in Taihu Lake. The heavy fishing activities of motor boats on the lake are the most significant source of PAHs in September. In the dry season, the residential, industrial and agricultural activities around the lake are important sources for PAH pollutants, in addition to the vehicle emissions on the lake (Qiao et al. 2006).

3.3.2 Principal component analysis and hierarchical cluster analysis

PCA and HCA were used to further characterise potential source categories for PAHs in Taihu Lake sediments. PCA is a sophisticated statistical technique for source identification (Schaanning et al. 2011). In this study, PCA was performed on a PAH profile matrix to obtain factor loadings and scores. The matrix was composed of 15 columns (the number of individual PAH species) and 58 rows (the number of PAH profiles in 29 sediments for two seasons). Loading and score plots have been used previously to study the similarity/dissimilarity of sediment profiles (Lam et al. 1997; Dahle et al. 2003; Manoli et al. 2004; Cao et al. 2005). Dahle et al. (2003) used a combination of loading and score plots to identify possible PAH source categories; this combination of plots also produced good results in our previous work (Cao et al. 2005).

PCA revealed three principal components with eigenvalues >1.0, accounting for 78 % of the total variance. Factor loading and score plots are displayed in Fig. 5a, which shows that almost all of the September data were positively correlated with the first principal component (PC 1) or the second principal component (PC 2). Figure 5b shows that some PAH species, such as BbF, BkF, IcdP and BghiP, exhibited high loadings for PC 1; thus, the September data, which positively correlated with PC 1, are associated with BbF, BkF, IcdP and BghiP. Some species, such as Pry and DahA, showed high levels for PC 2, so the September data, which positively correlated with PC 2, are associated with Pry and DahA. These species (BbF, BkF, IcdP, BghiP, Pry and DahA) have been used previously as source markers for diesel-powered or gasoline-powered vehicle emissions (Harrison et al. 1996; Larsen and Baker 2003). Thus, we propose that vehicle emissions may be an important source category for PAHs in September (the flooding season). On the other hand, some of the data points for the dry season (May), contained within the ellipse, also positively correlated with PC 1 and PC 2, indicating that vehicle emissions might also have contributed to these sites.

The plots of PC 1 vs. PC 3 (see Figs. 5c, d) show that some of the data for May were positively correlated with PC 3, while the loading plot (see Fig. 5d) shows that the source markers, including Ant, Flua and BaA, exhibited high loadings. These results suggest that coal combustion/coke production/wood combustion may be important source categories for sediments in the dry season (Harrison et al. 1996).

In summary, factor loading and score plots indicate that vehicle emission is an important source for PAHs in sediments

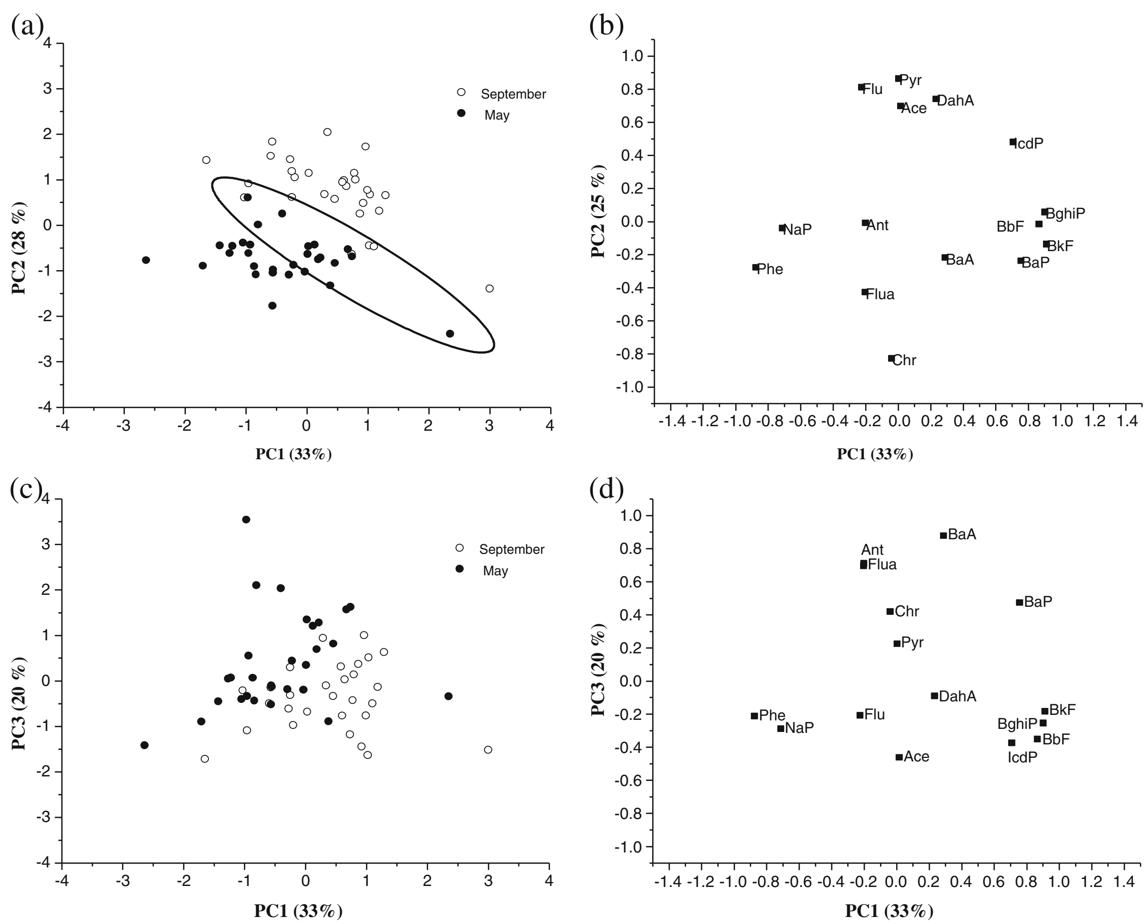

Fig. 5 Scatter plots for factor loading and score. **a** Factor score plot of PC1 and 2. **b** Factor loading plot of PC 1 and 2. **c** Factor score plot of PC1 and 3. **d** Factor loading plot of PC 1 and 3

during the flooding season, while PAHs in sediments from the dry season may come from multiple sources. The results of the loading and score plots are in agreement with the diagnostic ratio results.

HCA was conducted, in addition to PCA, using complete linkage and Euclidean distances (Fig. 6). HCA showed that similar PAH profiles clustered together; the similarity of the profiles suggests that these sediments had similar source categories (Lee et al. 2004; Shi et al. 2009). The dendrogram shows that almost all of the flooding season profiles clustered into one group, suggesting that the PAHs in the flooding season sediments could be attributed to a similar source category. Portions of the dry season profiles resembled this group, indicating that the PAHs in sediment from these sites might derive from sources similar to those in the flooding season. These sites correspond to the data points within the ellipse in Fig. 5a. Figure 1 shows that these sites are located at the lakeside. The other profiles were clustered in another group, implying that different source categories might have contributed to the sediments from these sites.

3.4 Seasonal and spatial variation analyses

The CD method was used to study the similarity/dissimilarity of the PAH profiles in both seasons, in order to investigate seasonal variations in the PAH sediment composition in the different regions. The CD is self-normalising and can be calculated from short-term measurements or long-term averages. The CD is defined as follows (Zhang and Friedlander 2000):

$$CD = \sqrt{\frac{1}{15}\sum_{i=1}^{15}\left(\frac{x_{if}-x_{ij}}{x_{if}+x_{ij}}\right)^2} \quad (1)$$

where x_{if} is the average fraction (%) of the *i*th individual PAH species in the flooding season, x_{ij} is the average fraction (%) of the *i*th individual PAH species in the dry season, and 15 is the number of the individual PAH species. Equation (1) shows that if the average fractions of the individual PAH species in the two seasons are similar, the CD approaches 0; if the average fractions of the individual PAH species in the two seasons are quite different, the CD approaches 1. Similar PAH profiles may indicate similar sources (Manoli et al. 2004; Shi et al. 2009).

Table 1 Coefficient of divergence (CD) values for different parts of Taihu Lake

Spatial parts	Sites	CD values
NW (Northwest)	S1–4	0.17
N (North)	S14–18	0.19
NE (Northeast)	S19–21	0.24
SW (Southwest)	S5–9	0.22
SE (Southeast)	S25–29	0.22
C (Center)	S10–13 and S22–24	0.25

Table 1 presents the mass fraction diagrams and CD values for the six regions. The CD values ranged from 0.17 to 0.25 across the six regions. Portions of the NW and N regions showed relatively lower values (0.17 and 0.19), suggesting less dissimilarity in seasonal variation among the six regions. There are some areas in the NW and N regions where the vehicle emissions were also high in the dry season. The NE and C regions exhibited the highest CD values, corresponding to high seasonal dissimilarity. The NE region is one of the most important fishing areas in the lake, so motor boat activity ought to be much greater in the flooding season compared to the dry season, leading to the relatively high seasonal variation in PAH profiles. There is limited motor boat activity in the C region in the dry season, while the contribution of vehicle emissions becomes more significant in the flooding season due to

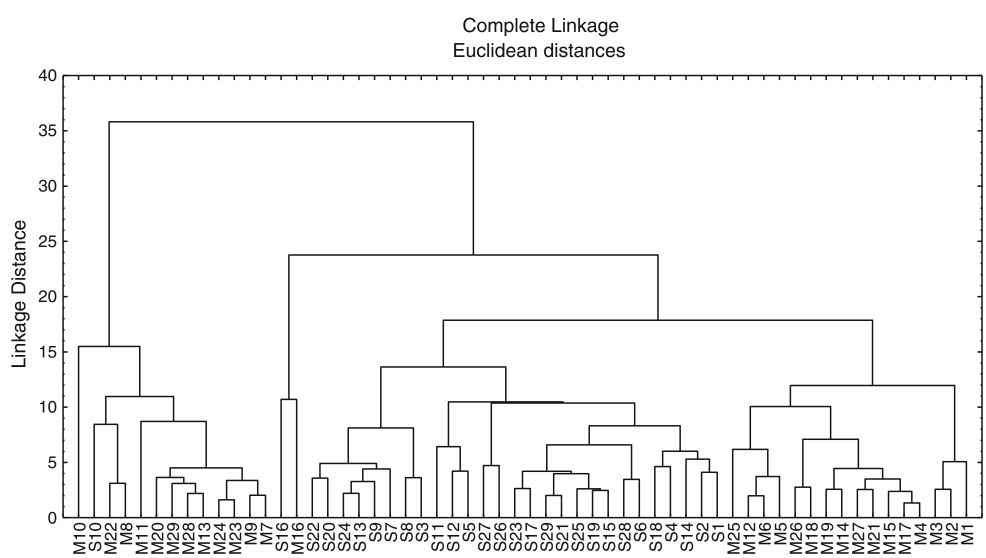

Fig. 6 Hierarchical clustering of PAH profiles of sediments in two seasons. *M1–M29* PAH profiles of sediment from 29 sites in the dry season (May); *S1–S29* PAH profiles of sediment from 29 sites in the flooding season (September)

fishing boat activity, resulting in relatively large seasonal variations.

4 Conclusions

In this study, the levels of 15 PAHs species were measured in 29 sediments from Taihu Lake, in both the flooding and dry seasons. The concentrations of both total and individual PAHs were higher in the flooding season than in the dry season, due to high fishing activity in the flooding season. Analysis of the PAH profiles demonstrated that the fractions of high molecular weight PAH species were more abundant in the flooding season for six regions in the lake, showing that the dominant source contribution was vehicle emissions from motor boats in the flooding season. Source identification was studied by diagnostic ratios, PCA and HCA, suggesting that vehicle emissions was the dominant source category for PAHs in sediments in the flooding season, while PAHs in the dry season came from multiple sources. The CD values for seasonal and spatial variations were in agreement with the source identification results.

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