Polycyclic aromatic hydrocarbons (PAHs) in surface sediments from the largest deep plateau lake in China: Occurrence, sources and biological risk

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Abstract

The unprecedented rate at which economy, urbanization and industrialization have occurred in China in the last few decades has resulted in environmental pollution in China. Polycyclic aromatic hydrocarbons (PAHs) are of great concern because of their persistence, long-range transport properties, and health risk as potential carcinogens and mutagens. Plateau lakes are special ecosystem and located in remote western China. To our knowledge, studies on PAHs in sediments of plateau deep lakes in China are scarce. Therefore, PAHs in surface sediments from Fuxian Lake, the largest deep plateau lake in China, were investigated. The total PAH concentrations ranged from 83.2 to 261.7, with a mean concentration of 143.3 ng/g. Four-ring PAHs (39.1–73.9%) were most abundant in all samples. The composition of PAHs was characterized by high molecular weight PAHs, and FA and PHE were the dominant

constituents. A correlation analysis revealed that TOC was positively significantly correlated with total PAHs, while percentages of grain size fraction were poorly correlated with total PAHs. PAH diagnostic ratios demonstrated that PAHs mainly originated from biomass, coal, and petroleum combustion. Taking as a whole, surface sediments of Fuxian Lake had a 9% incidence of adverse biological effects based on the mean effects range-median quotient.

Keywords: PAHs; Sediment; Sources; Biological risk; Fuxian Lake

Introduction

Sediments are effective sinks for pollutants such as polycyclic aromatic hydrocarbons (PAHs) in aquatic ecosystems, but also act as a source of pollutants (White and others 2005; Guo and others 2010; Lu and others 2012; Gregg and others 2015). PAHs are a large group of organic compounds with two or more fused aromatic rings, and they primarily originate from the incomplete combustion of carbonaceous materials (Boström and others 2002; Martins and others 2010; Shen and others 2013; Gu and others 2016). PAHs are of great concern because of their persistence, long-range transport properties, and health risk as potential carcinogens and mutagens (Perera 1997; Zhang and others 2012; Gu and others 2013).

Freshwater deep plateau lakes are special ecosystems. Inputs of PAHs into lakes are generally predominated by atmospheric deposition rather than direct intake (Yang and others 2016). Few studies have been conducted to investigate concentrations and

sedimentary record of PAHs in plateau lakes in China (Guo and others 2010; Zhao and others 2014; Yang and others 2016), but studies on PAHs in sediments of deep plateau lakes in China and the resulting biological risks are scarce.

Fuxian Lake, situated in the central part of Yunnan Province on the Yunnan-Guizhou Plateau in Southwest China (Fig.1), covers an area of 212 km², with a maximum depth of 155 m, and water residence time of 167 years (Liu and others 2008; Zhao and others 2012; Zhang and others 2015). As the largest deep freshwater lake in China, it has been seriously influenced by anthropogenic activities such as industrial and agricultural development, deforestation, and tourism since the 1980s (Liu and others 2008; Zeng and Wu 2009; Zhang and others 2015). Previous studies show that anthropogenic activities in the areas, related to increasing nutrient inputs, have resulted in the deterioration of the aquatic ecosystem and environment over the past years (Liu and others 2008; Zeng and Wu 2009). It is therefore necessary to evaluate pollutants, such as PAH levels, in aquatic sediments. Therefore, the present study was designed to (1) determine PAH concentrations in surface sediments of Fuxian Lake, and (2) identify potential sources and evaluate biological risks of PAHs in this region.

2. Materials and methods

2.1 Sample collection

Sediment sampling was undertaken along a transect down the long axis of the lake, as proposed by Mudroch and Azcue (1995). The water depth contour of Fuxian

Lake has been described in detail in previous studies (Liu and others 2008; Zhang and others 2015). Along the transect, eight sites were selected and located for our study. Surface sediment was obtained using a stainless steel Peterson grab sampler in August 2015 (Fig. 1). At each site, two sets of samples were carefully taken from a sediment depth of 0–3 cm with a polyethylene spatula and placed in self-sealing polyethylene bags. Prior to analysis, one set of samples was stored at -4°C and the other at -20°C . Samples at -4°C were used for grain size analysis; those kept at -20°C were freeze-dried to constant weight, homogenized using an agate mortar and pestle, sieved through a 200 mesh stainless steel mesh ($< 74\ \mu\text{m}$), and stored in a glass bottle at -4°C until analysis of total organic carbon (TOC) and PAHs.

Fig. 1. Map showing the sampling sites and distribution of total organic carbon (TOC) in surface sediments from Fuxian Lake, Southwest China.

2.2 Analytical methods

The pre-treatment procedures for grain size analysis were conducted in accordance with the Chinese National Standards (GB/T12763.8–2007). Sample granulometry was analyzed using a Malvern Mastersizer 2000 laser, diffractometer capable of analyzing particle sizes between 0.02 and 2,000 μm . The percentages of grain size were divided into three groups: $< 4 \mu\text{m}$ (clay), $4\text{--}63 \mu\text{m}$ (silt), and $> 63 \mu\text{m}$ (sand). Organic matter content was determined by loss on ignition (LOI) at 550°C for 4 h (Heiri and others 2001). Subsequently, total organic carbon (TOC) content was

calculated by using a correction factor of 1.72 (De Jonge and others 2012).

Pre-treatments and measurements of 16 PAHs in surface sediments were performed as described in our previous study (Gu and others 2013).

For quality assurance and control, surrogate standards were added to all sedimentary samples prior to extraction to quantify the procedural recoveries. PAHs were quantified using the internal calibration method based on five-point calibration curves for individual compounds. The surrogate recoveries (mean $\pm$ standard deviation) were $76. \pm 7.8$, 90.5 ± 8.7 , $101.4 \pm 9.5\%$ for acenaphthene- d_{10} , chrysene- d_{12} , perylene- d_{12} and phenanthrene- d_{10} , respectively. We used Nation Institute of Standard and Technology (NIST) standard material (SRM 1941) and quantified PAHs applying the internal calibration method. Recoveries of 16 PAHs in the NIST 1941 sample varied from 82.3 to 104.6% of the certified values. Blank samples contained no detectable amount of the targeted PAHs, and the relative percent analysis difference of paired duplicate samples was less than 12% (n=12). The PAHs and standard materials were analyzed using a chromatography-mass spectrometer (Agilent 7890 series gas chromatograph/5975C mass spectrometer, Agilent Technologies, USA).

The instrument detection limits varied from 0.2 to 2 ng/g. The 16 US EPA priority PAHs analyzed were: naphthalene (NA), acenaphthylene (ACL), acenaphthene (AC), fluorene (FL), phenanthrene (PHE), anthracene (AN), fluoranthene (FA), pyrene (PY), benzo(a)anthracene (BaA), chrysene (CHR), benzo(b)fluoranthene (BbFA), benzo(k)gluoranthene (BkFA), benzo(a)pyrene (BaP), Indeno(1,2,3-c,d)pyrene (IP), dibenzo(a,h)anthracene (DBahA) and

benzo(g,h,i)perylene (BghiP).

3. Results and discussion

3.1 Sediment characteristics

Total organic carbon (TOC) contents varied between 2.8 and 4.8% of the dry sediment weight, with an average of 3.8% (Fig. 1). The relative higher TOC contents (> 4.3%) were all recorded at sites F3 and F4 in the northeastern part of Fuxian Lake (Fig. 1). The ternary diagram in Fig. S1 categorizes the sediments of Fuxian Lake using the system of Shepard (1954), revealing clear spatial patterns. Apart from sampling sites F2, F6, and F8, which were dominated by clayey silt, the studied surface sediments of Fuxian Lake were predominated by silty clay.

3.2 PAHs levels in sediments

Surface sediments can reflect the current contamination status (Gu and others 2013). Table 1 shows the concentrations of the 16 US EPA priority PAHs in surface sediments from Fuxian Lake. Total PAH concentrations ($\sum$ PAHs) from all sediment samples varied from 83.2 to 261.7, with a mean concentration of 143.3 ng/g. Thirteen individual PAHs were found at all stations, with the exception of BbFA, IP and DBahA. Bap, the most carcinogenic among all PAHs compounds (San José and others 2013), was detected at all stations (0.8–4.4 ng/g, mean 2.3 ng/g). FA was the most abundant PAH with concentrations between 11.4 and 91.9 ng/g (mean 32.9 ng/g),

135 followed by PHE (19.5–32.2 ng/g, mean 25.1), NA (0.5–56.2 ng/g, mean 22.9 ng/g),
136 and PY (8.5–56.9 ng/g, mean 21.7 ng/g). These four groups contributed more than
137 71.6% to the total PAHs concentration. The highest and relatively higher
138 concentrations of some sites in northeast Fuxian Lake may be attributed to the
139 cumulative effects of water discharge from urban runoffs, sewage discharges, oil
140 spills from boats, and petroleum combustion (Fig. 2a). In addition, the TOC might
141 have facilitated PAH concentrations (Fig.1).

142 Fig. 2b shows a comparison of PAH concentrations in surface sediments collected
143 of different plain lakes. Average total PAH concentrations in surface sediments of
144 Fuxian Lake were significantly lower than those in sediments from Chaohu Lake,
145 Dianchi Lake and Taihu Lake, but only slightly lower than concentrations in Poyang
146 Lake and Baiyangdian Lake.

Fig. 2. Spatial distribution of PAHs in surface sediments from Fuxian Lake (a) and mean concentrations of PAHs in surface sediments soils different Chinese lakes (b). CH: Chaohu Lake (Wang and others 2011); PY: Poyang Lake (Lu and others 2012); DC: Dianchi Lake (Zhao and others 2014); BY: Baiyangdian Lake (Hu and others 2010); TH: Taihu Lake (Zhang and others 2012); TS: Fuxian Lake (This study).

161 Table 1 Concentrations of PAHs in surface sediment from Fuxian Lake (ng/g, dry weight).

PAHs	F1	F2	F3	F4	F5	F6	F7	F8	ERL	ERM
NA	56.2	6.5	1.1	50.6	7.0	5.4	55.9	0.5	160	2100
ACL	0.4	0.3	0.9	0.5	0.4	0.5	0.4	0.4	44	640
AC	1.3	1.1	1.4	1.3	1.5	1.6	1.5	1.5	16	500
FL	7.0	7.4	13.0	6.3	8.7	10.3	6.3	10.9	19	540
PHE	24.2	19.5	32.2	20.9	23.8	31.4	22.4	26.3	240	1500
AN	1.3	1.0	2.2	1.1	1.3	1.1	1.1	0.9	853	1100
FA	27.0	25.6	91.9	34.8	17.4	28.6	26.4	11.4	600	5100
PY	19.1	18.3	56.9	22.9	11.3	19.0	17.5	8.5	665	2600
BaA	6.3	5.5	13.0	6.5	4.3	3.6	4.5	2.6	261	1600
CHR	13.8	12.3	31.8	16.0	9.8	8.4	10.5	14.7	384	2800
BbFA	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.a	n.a
BkFA	3.5	2.9	7.1	4.0	3.1	3.6	2.0	2.2	n.a	NA
BaP	1.7	1.3	4.0	2.3	1.9	4.4	0.8	2.0	430	1600
IP	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.a	n.a
DBahA	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	63.4	260
BghiP	1.8	1.6	6.4	1.8	1.5	5.1	1.3	1.1	n.a	n.a
Σ16 PAHs	163.7	103.3	261.7	169.0	92.0	123.0	150.6	83.2	4000	44792

162 n.d: not detected; n.a: not available; ERL: effects range low; ERM: effects range median (Gu et al. 2013).

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164 3.3 Composition profiles of PAHs

165 Fig. 3 shows composition pattern of PAHs by ring size in the surface sediments
166 from Fuxian Lake. Four-ring PAHs (39.1–73.9%) were most abundant in all samples,
167 followed by three-ring PAHs ranging from 17.8 to 48.2%, except in samples from F1,
168 F4 and F7, where the two-ring PAHs ranked second at 34.3, 29.9 and 37.1%,
169 respectively. The composition pattern of PAHs was usually characterized by the
170 dominance of high molecular weight (HMW, 4–6 benzenoid rings) PAHs. In a similar
171 study on surface sediments from lacustrine environment, Zhao et al. (2014) also found
172 higher concentrations of HMW PAHs than of low molecular weight (LMW, 2–3
173 benzenoid rings) PAHs. FA and PHE were the most dominant components in the
174 sediment samples studied, accounting for 13.7–35.1% (mean 21.3%) and 12.3–31.7%

(mean 19.5%), respectively, of total PAHs, followed by NA and PY, which accounted for 0.4–37.1% (mean 15.1%) and 10.2–21.7% (mean 14.3%), respectively, of total PAHs.

Fig. 3. Distribution of 2-, 3-, 4-, 5-, 6-ring in the surface sediments from Fuxian Lake.

3.4 Relationship between sediment characteristics and PAHs

Several studies have shown that TOC and grain size are two important controlling factors for the sorption of PAHs in sediments (Wang and others 2001; Yan and others 2009; Agarwal and Bucheli 2011; Timoney and Lee 2011; Li and others 2012). Table S1 shows the correlations between PAHs concentrations in sediments with TOC and

grain size. In our study, TOC was positively significantly correlated with total PAHs, AN, FA, PY and BaA. In contrast, there were no significant correlation between percentages of sand, silt, and clay and total as well as individual PAH concentrations, suggesting that grain size is only a minor factor influencing PAH accumulation. Our results are in agreement with the findings by Timoney and Lee (2011), who did not observe a correlation between PAH concentration and sediment grain size.

3.5 Source identification of PAHs

PAHs of anthropogenic origin have various sources, including biomass and fossil fuel combustion and oil spills (Guo and others 2010). Diagnostic concentration fractions of PAH isomers are often applied to identify potential sources of PAHs (Yunker and others 2002; Yang and others 2016). In the present work, the molecular diagnostic ratios of $FA/(FA+PY)$ and $BaA/(BaA+CHR)$ were used for this identification, and the results are schematically shown in Fig. 4. According to the source classification by Yunker et al. (2002), of the PAHs in the sediments mainly originate from biomass, coal combustion, and petroleum combustion.

Fig. 4. Bivariate plot of PAH diagnostic ratios in lake sediments. PAH sources identification by Yunker et al. (Yunker and others 2002): $FA/(FA+PY) < 0.4$: petroleum, $0.4 < FA/(FA+PY) < 0.5$: petroleum combustion, $FA/(FA+PY) > 0.5$: biomass and coal combustion; $BaA/(BaA+CHR) < 0.2$: petroleum, $0.2 < BaA/(BaA+CHR) < 0.35$: petroleum combustion, $BaA/(BaA+CHR) > 0.35$: biomass and coal combustion.

3.6 Biological risk assessment

Effects range-low (ERL) and effects range-median (ERM), depending on numerous toxicity tests, field studies, and altered benthic communities are the main parameters to evaluate the adverse biological effects of PAHs in marine and estuary sediments (Long and others 1995). As shown in Table 1, the concentrations of total and individual PAHs at all sites were below the ERL values, indicating that adverse effects on benthic organisms are rare.

As PAHs in sediments usually occur as mixtures, the mean ERM quotient method was proposed as a way to determine the possible biological effect of combined toxicant groups by computing mean quotients for a large range of contaminants

including PAHs (Carr and others 1996; Long 2006); it can be calculated using the formula:

$$\text{mean ERM quotient} = \sum(C_x / \text{ERM}_x) / n,$$

where C_x is the measured concentration of the examined component (x) in sediments, ERM_x is the ERM for PAH x, and n is the number of PAHs. According to the mean ERM quotient method by Long et al. (2000), mean ERM quotients below 0.11 have a 9% probability of being toxic, quotients of 0.11-0.5 have a 21% probability, quotients of 0.51-1.5 have a 49% probability, and quotients greater than 1.50 have a 76% probability. In surface sediments of Fuxian Lake, the mean ERM quotients were below 0.11, indicating that the combination of the studied PAHs has a 9% probability of being toxic (Fig. 5).

Fig.5. Spatial distribution of mean ERM quotient values in surface sediments from Fuxian Lake.

4. Conclusions

Total PAH concentrations in surface sediments of Fuxian Lake, the largest deep plateau lake in China, were in the range of 83.2 to 261.7 ng/g. FA was the most abundant PAH, followed by PHE, NA and PY; together, these four PAHs contributed more than 71.6% to the total PAH concentration. Bap, which has the highest carcinogenic potential, was detected at all sampling sites. The PAH composition pattern was usually characterized by the dominance of high molecular weight PAHs. We found a significant correlation between total PAH concentrations and TOC levels, indicating that TOC is a key property for PAH accumulation in these sediments. PAH diagnostic ratios indicated that PAHs mainly derived from biomass, coal, and petroleum combustion. The concentrations of total and individual PAHs at all sites were below the ERL values, indicating that adverse effects on benthic organisms are unlikely.

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