Historical Pollution and Source Contributions of PAHs in Sediment Cores from the Middle Reach of Huai River, China

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Abstract

To investigate the spatial and historical distributions, and source contributions of polycyclic aromatic hydrocarbon (PAHs) from the middle reach of Huai River, 15 surface sediments and two sediment cores were analyzed. The Σ_{16} PAHs levels in surface sediments varied from 533.15 to 1422.83 ng/g dw, and from 413.27 to 43951.56 ng/g dw in individual sediment layer of sediment cores. The temporal trends of PAHs in sediment cores are the good indicators of the anthropogenic emissions over the last 60 years. The stable carbon isotope ratios of PAHs indicate the primary PAHs sources were the combustion of wood and coal during 1950s–1970s, and automobile exhausts and the coal combustion emissions in recent decades.

Polycyclic aromatic hydrocarbons (PAHs) are a group of ubiquitous environmental contaminant with two or more fused aromatic rings, and derived primarily from anthropogenic origins in modern environments (Neff 1979; Wang et al. 2011, 2013; Ren et al. 2015). Owing to their toxic, carcinogenic and mutagenic properties, United States Environmental Protection Agency (US-EPA) has placed 16 PAHs as the priority contaminants (Menzie et al. 1992; Chen et al. 2016a). In the aquatic system, PAHs tend to absorb to sediment particles, owing to their low solubility and hydrophobicity (Zhang et al. 2013). Thus, sediment serves as a natural reservoir of PAHs (Wade et al. 2008). The PAHs concentrations and distributions in the sediment cores have been used to document the temporal trends of anthropogenic PAHs contamination (Liu et al. 2012a). Many researches indicate that the PAHs deposition rate varied at different times, which was mostly related to the differences in the intensities of anthropogenic activities and economic development (Guo et al. 2010; Xu et al. 2014). Furthermore, compound-specific stable carbon isotope ratios ($\delta^{13}C$) of PAHs have emerged as useful tools to trace the origins of PAHs (O'Malley et al. 1994, 1996; Walker et al. 2005; Nobuyasu and Nobuyasu 2010; Yoon et al. 2017). Compared to the traditional molecular compositions, the $\delta^{13}C$ values of PAHs are not readily altered by chemical and biological processes (Okuda et al. 2002a; Kim et al. 2008; Jautzy et al. 2013).

Huai River (HR) is one of the seven largest rivers in China, and the area adjacent to the middle reach of HR is dominated by coal-processing plants, coal-fired power plants and urbanization (Cui et al. 2004; Chen et al. 2016b; Tang et al. 2013, 2018). Many studies (Fu et al. 2011; Feng et al. 2012; Zhang et al. 2017) indicate municipal effluents and waste water from chemical factories were discharged into the river, and PAHs contamination was closely related to waste water input and industrial activities. Up to now, little information are available regarding the historical PAHs deposition in HR. In present study, our objectives are to (1) construct the PAHs historical trends in sediment cores; (2) trace and quantify the PAHs origins using PAHs $\delta^{13}C$ and the binary model.

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Materials and Methods

Fifteen surface sediments (about 5 cm upper layer) and two sediment cores (depth: ~ 50 cm) (HS1, HS2) (Fig. 1) were collected from the middle reach of HR by a Zn-plated Petersen grab sampler and a gravity corer with a polycarbonate tube of 75 mm internal diameter. Then, two sediment cores were cut into 2 cm slices, and all sediment samples were freeze-dried, homogenized, sieved, and stored at − 20°C until analysis.

Using external standard method, the 16 US EPA priority PAHs (Σ_{16} PAHs) in sediments were extracted by microwave digestion method, and eluted with chromatographic column (Qin et al. 2014; Wang et al. 2015). Then, the PAHs concentrations were analyzed with Gas Chromatography–Mass Spectrometry (GC-MS) (Ammami et al. 2015; Zhang et al. 2015). The method blanks, spiked blanks and sample duplicates were also analyzed. Method blanks operation showed no detectable amounts of PAHs contamination. All experiments were carried out in triplicate. Average recoveries for Σ_{16} PAHs ranged from 71.5% ± 4.7% to 98.3% ± 8.2% in spiked blanks. The average recovery was 90.1% ± 12% for $^2H_8$ naphthalene (NaP–D_8), 86.5% ± 10% for $^2H_{12}$ chrysene (Chry–D_{12}) and 89.3% ± 8.14% for $^2H_{10}$ phenanthrene

(Ph–D_{10}). The PAHs concentrations in reported samples were not corrected for recoveries.

After PAHs separation from the sediments, the PAHs stable carbon isotope ratios were analyzed using Gas Isotope Ratio Mass Spectrometer (GC-IRMS) with helium (1.5 mL/min) (Jautzy et al. 2013). Acenaphtehene–d10 ($\delta^{13}C$: − 23.04‰) was used for the isotopic standard. The carbon isotopic ratio is expressed using the following equation (Peng et al. 2006; Blessing et al. 2015; Nascimento et al. 2017):

$$\delta^{13}C(‰, PDB) = \left(R_{sample}/R_{standard} - 1\right) \times 1000 \qquad (1)$$

where R is $^{13}C/^{12}C$ ratio, and PDB is the Pee Dee Belemnite standard. The measurement uncertainty of the $\delta^{13}C$ values ranged from is better than 0.4‰.

The sediment dating were performed by an Ortec HPGe GWL series well-type coaxial low background intrinsic Ge detector (Guo et al. 2007). Before analysis, all samples were stored in a sealed centrifuge tube more than 3 weeks to keep the radioactive equilibration of ^{226}Ra and its daughter isotope ^{210}Pb (Yuan et al. 2014). The ^{210}Pb, ^{226}Ra and ^{137}Cs radioactivity was determined at 46.5 keV, 352 keV and 662 keV, respectively. The deposition rate were analyzed using a dating model of constant rate of supply (Wang et al.
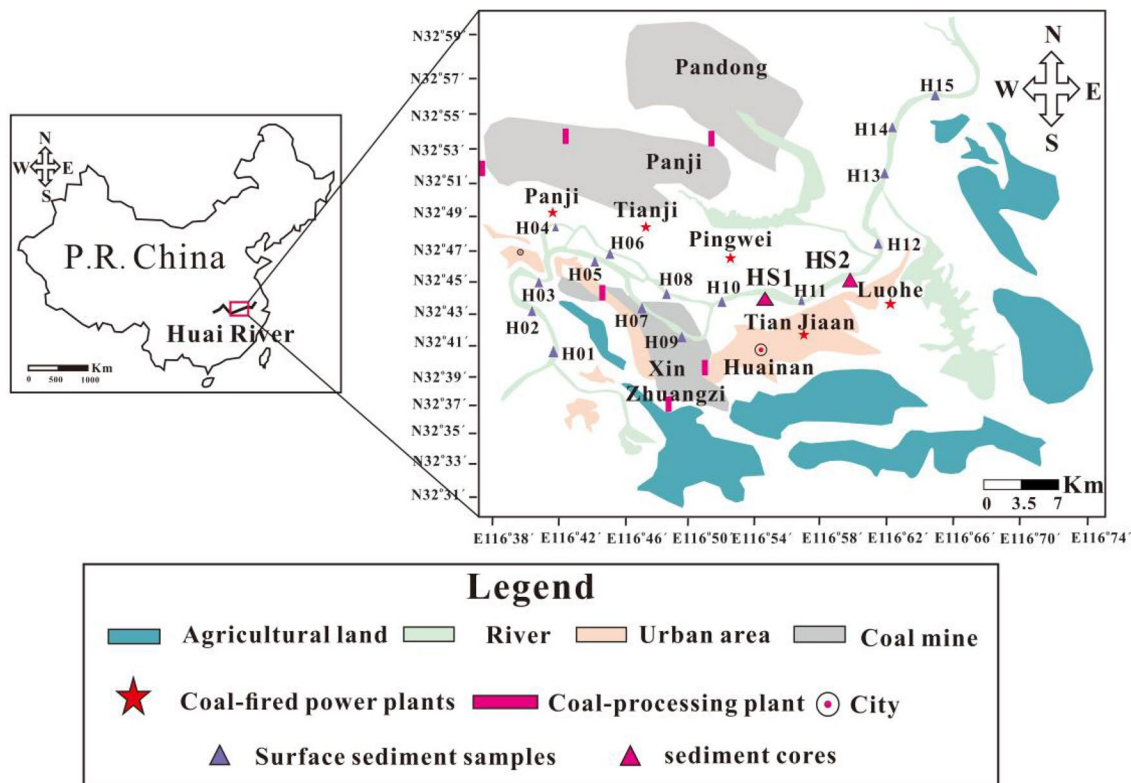

**Fig. 1** Map showing the location of the sediment samples

2017). Across the sediment core, the average rate of sedimentation is 0.5 cm/a.

Results and Discussion

The Σ_{16} PAHs concentrations in 15 surface sediment samples ranged from 533.15 (H03) to 1422.83 ng/g (dry weight, dw) (H10), with a mean value of 970.10 ng/g dw (Fig. 2). The concentrations of seven carcinogenic PAHs (CPAHs: BaA, BaP, BbF, BkF, Ind, DahA, Chry) varied from 201.85 to 1005.30 ng/g dw (mean: 458.11 ng/g dw), which accounted for 38%–58% of Σ_{16} PAHs detected in surface sediments. The high weight PAHs (HPAHs: Flu, Py, BaA, Chry, BbF, BkF, BaP, Ind, DahA, BghiP) accounted for 77% of Σ_{16} PAHs, while the low weight PAHs (LPAHs: NaP, Ace, Ac, Fl, Ph, An) only accounted for 23% of Σ_{16} PAHs. This could be due to the fact that HPAHs have low octanol–water partition coefficients, whereas LPAHs have high water solubility (Yang et al. 2002). Comparing with other regions of the world, the mean content of Σ_{16} PAHs in the present study was higher than that reported for surface sediments from the upper reach of HR in China (371 ng/g; Feng et al. 2012), Yinma River in China (825 ng/g; Sun et al. 2017), Bohai Bay in China (79 ng/g; Wang et al. 2015), Guan River in China (133 ng/g; He et al. 2014), Luan River in China (121 ng/g; Zhang et al. 2016), Pulau Merambong and Muar River in Malaysia (71 ng/g, 76 ng/g; Vaezzadeh et al. 2014). However, the mean value of Σ_{16} PAHs in the present study was lower than the contents of PAHs at Bhavnagar coast of India (345×10^3 ng/g; Dudhagara et al. 2016), urban lake of Bergen from Western Norway (8161 ng/g; Andersson et al. 2014) and Huveaune River of France (1966 ng/g; Kanzari et al. 2014). The results from this study indicate that the measured PAH values in surface sediments from middle reach of the HR were at the midpoint of global ranges.

The total PAHs content profiles across two sediment cores are shown in Fig. 2. The total PAHs contents in the two sediment cores fluctuated vigorously with depth. Σ_{16} PAHs in HS1 spanned a large range from 989.04 up to 43951.56 ng/g dw (mean: 11219.27 ng/g dw), and the CPAHs levels were in the range of 413.27–24902.65 ng/g dw (mean: 5813.45 ng/g dw). At HS2, the Σ_{16} PAHs levels were from 1141.39 to 52,845.03 ng/g dw, with a mean value of 13,016.05 ng/g
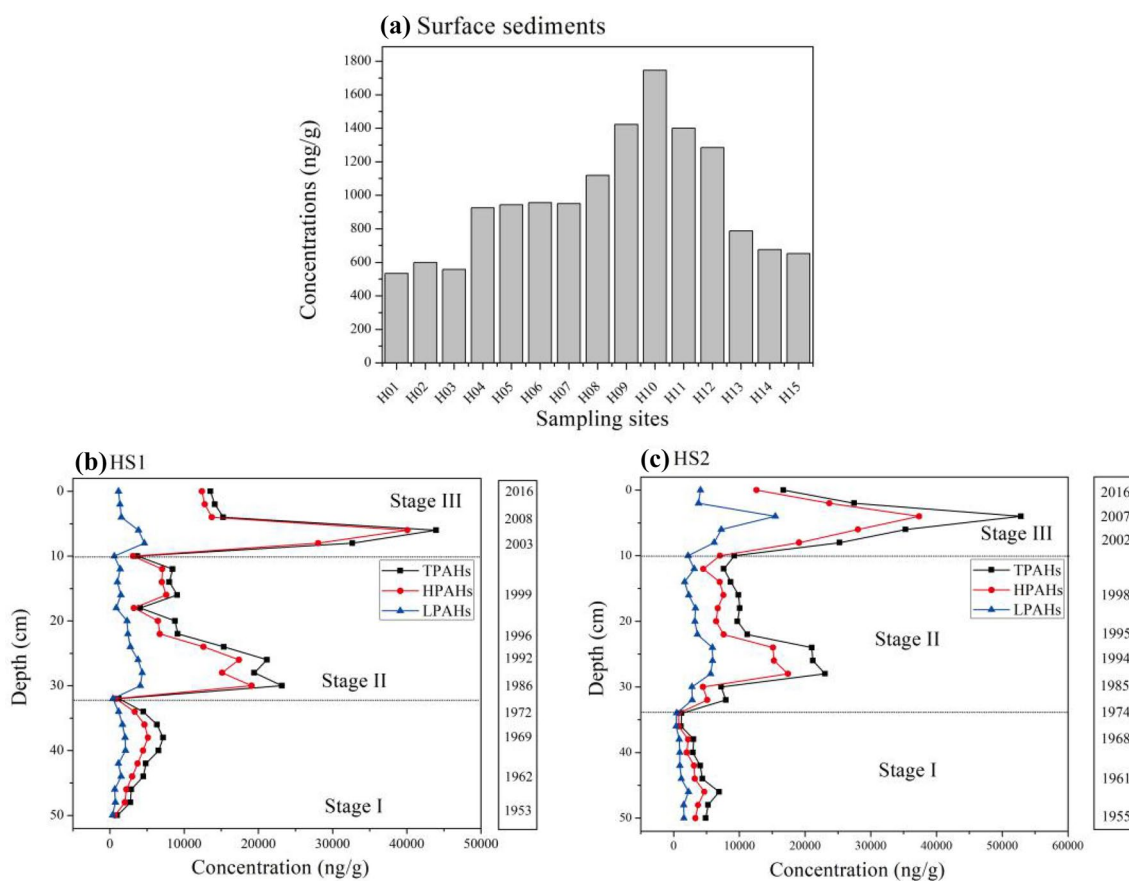
Fig. 2 Temporal trends of PAHs levels in **a** surface sediments, **b** sediment core of HS1, and **c** sediment core of HS2. **a** Surface sediments: 15 surface sediment samples; **b** HS1: 26 sediment samples at the sediment core of HS01; **c** 26 sediment samples at the sediment core of HS2

dw, and the CPAHs concentrations varied from 527.99 to 22,173.80 ng/g dw (mean: 6042.64 ng/g dw). Using isotopic dating, the entire cores (50 cm) cover approximately 60 years (1950s–2010s). According to the historical trends of PAHs in Fig. 2, the temporal trends of the HPAHs and LPAHs levels were similar to that of Σ_{16} PAHs, although the LPAHs levels were constantly low across sediment cores. The historical trends of the PAHs contents between the two sediment cores were similarly. In general, there are three abrupt increase of PAHs across the recent 60 years in HS1 and HS2 (Stage I: 1950s–1970s; Stage II: 1980s–1990s and Stage III: 2000s–2010s) (Fig. 2).

Stage I recorded the largest increase of PAHs to 7177.81 ng/g dw (HS1), and 7953.59 ng/g dw (HS2). After the establishment of the People's Republic of China in 1949, an economic reconstruction period began, especially the Great Leap Forward period (1958–1960) when vast amounts of forest woods were burned as fuel to produce the steel. The largest PAHs peaks in Stage I might be caused by deforestation and wood burning. Similar PAHs peaks were also observed in sediment cores from other studies (Liu et al. 2012b; Guo et al. 2011; Yan et al. 2009). The Cultural Revolution period in China was in the range of 1966–1976 when the industrial production was stagnant. This might result in a constant low PAHs levels between 1968 and 1974.

The resuscitation of China's economy was initiated again with "Reform and Open" policy in 1978 when fast industrialization and urbanization resulted in rising energy consumption (Zhang et al. 2013). The accompanied increase of PAH emissions is likely reflected by the moderate increase of PAHs in the range of 1970s–1990s. Since the mid-1990s, because of the rapid development in China, PAHs levels have continued to increase as evidence from sediment cores collected from various river basins in China (e.g. Taihu Lake, Pearl River, Liangtan River, Bohai Sea) (Liu et al. 2005, 2009, 2012c; Hu et al. 2011). However, the PAHs

contents at HS1 and HS2 in the mid-1990s showed a sharply decreasing trend. The main reasons are the pollution control measures have been implemented, and the cleaner burning fuels have been used.

After entering new millennium, the local government started to transform the economy from coal mining to coal energy. The amount of coal consumption for power generation increased from ~6 million tons in 2000 to 30 million tons in 2010 (Chen et al. 2016b). The increased PAH levels at HS1 and HS2 appeared again in the range of year from 2002 to 2007. The phenomenon indicates that pollution control measures or changes in energy consumption patterns could not be the main factor influencing the PAH trend during this period. In the new decade, the local government has made great efforts to mitigate the pollution of HR (e.g., installation of air pollution control devices in the coal-fired boilers, recirculating the use of wastewater), which may reduce PAH loadings into this system (Stage III; Fig. 2).

Some researchers have argued that the diagnostic PAHs ratios, which would comprise their fingerprinting abilities, have been influenced by some parameters (e.g. sediment chemical conditions, PAHs bioavailability) (Yanik et al. 2003; Kim et al. 2008; Jautzy et al. 2013). Therefore, the PAHs $\delta^{13}C$ values are illustrated in the present study to determine the PAHs origins (Fig. 3) (McRae et al. 2000; Wilcke et al. 2002; Jautzy et al. 2013). From Fig. 3, the $\delta^{13}C$ of Σ_{16} PAHs in the sediment core (HS1) were ranged from $-28.42‰$ to $-24.73‰$, while the Σ_{16} PAHs $\delta^{13}C$ values in another sediment core (HS2) varied from $-28.38‰$ to $-24.27‰$.

Recent studies indicated the C4 biomass combustion have the values of PAHs $\delta^{13}C$ between $-16.6‰$ and $-15.8‰$ (O'Malley et al. 1997; Wang et al. 2016), and the wood combustion emissions have the lower values of PAHs $\delta^{13}C$ between $-26.8‰$ and $-31.6‰$ (Okuda et al. 2002b; Wang et al. 2016). When the values of PAHs $\delta^{13}C$ were between
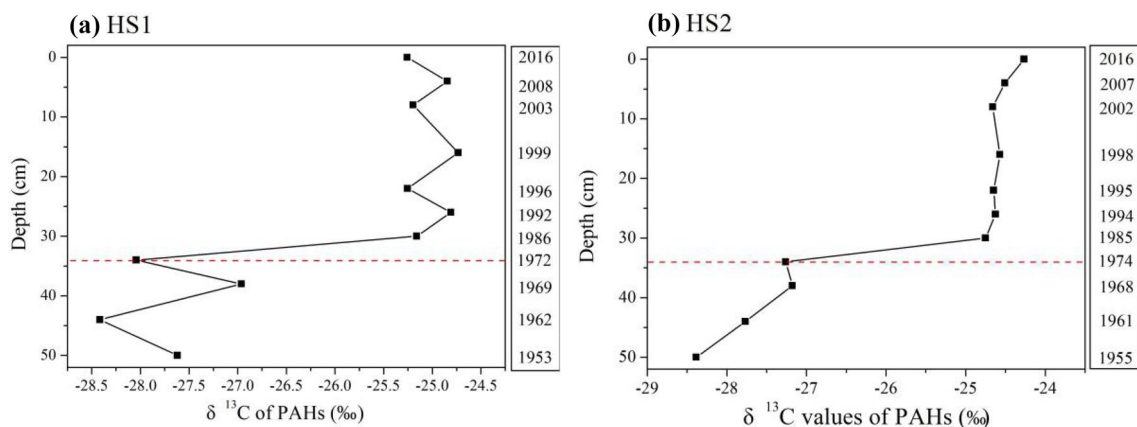
Fig. 3 Stable carbon isotope ratios of PAHs in **a** the sediment core of HS1; and **b** the sediment core of HS2

Table 1 The contributions (%) of PAHs source in the two sediment cores

Depth (cm)	Percentage source contributions					
	Sediment core of HS1 (n = 26)			Sediment core of HS2 (n = 26)		
	Wood combustion (%)	Coal combustion (%)	Automobile exhaust (%)	Wood combustion (%)	Coal combustion (%)	Automobile exhaust (%)
0	–	99.12	0.88	–	78.89	21.11
4	–	90.71	9.29	–	83.83	16.17
8	–	97.87	2.13	–	86.94	13.06
16	–	88.43	11.57	–	85.09	14.91
22	–	99.07	0.93	–	86.68	13.32
26	–	89.96	10.04	–	86.24	13.76
30	–	97.16	2.84	–	88.79	11.21
34	85.68	14.32	–	38.75	61.25	–
38	51.93	48.07	–	41.35	58.65	–
44	97.34	2.66	–	22.93	77.07	–
50	72.5	27.50	–	3.63	96.37	–

n sediment samples

−23‰ and −31.2‰, PAHs were mainly from coal combustion emissions (O'Malley et al. 1997; Peng et al. 2005; Wang et al. 2016). In addition, automobile exhaust was indicated with the values of PAHs $\delta^{13}C$ between −12.9‰ and −26.6‰ (Okuda et al. 2002b, c). Therefore, PAHs in HS1 and HS2 were mainly derived from the combustion of wood and coal before 1970s, and from automobile exhaust and combustion emissions of coal after 1970s.

In order to investigate the contributions of PAHs origins in the dated sediment cores, a binary model were used as follow (Peng et al. 2005):

$$C = AX + B(1 - X) \qquad (2)$$

where C is the $\delta^{13}C$ values of PAHs in the samples; A is the $\delta^{13}C$ values of the coal combustion source; B is the $\delta^{13}C$ values of the automobile exhaust source or wood combustion; X is the fraction of source contribution (%). Based on Peng et al. (2005), A and B were assigned a value of −25.3‰ (coal combustion), −20.4‰ (automobile exhaust) or −28.5‰ (wood combustion), respectively.

The calculated fractions of PAHs sources in sediment cores were shown in Table 1. In the two sediment cores, coal combustion (~78%–99%) and automobile exhaust (~0.8%–21%) were the predominant PAHs sources after 1970s, while the main sources of PAHs were the coal combustion (~14%–96%) and wood combustion (~3%–97%) before 1970s. It is noted that the PAH fractions from automobile exhaust and coal combustion have been increasing after 1970s. The relative PAHs variation from coal combustion and automobile exhaust sources are in consistent with the fast developing of industries, coal-fired power plants and vehicles (Wang et al. 2014; Zhang et al. 2014).

PAH concentrations were studied in sediment cores and surface sediments from the middle reach of HR. The temporal trends of PAHs contents in sediment cores are the good indicators of the historical PAHs inputs into the watersheds. The concentrations of PAHs in two sediment cores are characterized by three temporal trends from 1950s to 2010s. The historical profiles of PAHs indicate that the national and local economic policies, and the development of pollution control measures are all influenced the PAHs trends in the sediment cores from the middle reach of HR. Source identification using PAHs stable carbon isotope with binary mixing model indicated that wood and coal combustion were the dominant sources of PAHs before 1970s, whereas automobile exhausts and coal combustion accounted for the strong contribution afterward.

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