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Sediment record of polycyclic aromatic hydrocarbons in the Liaohe River Delta wetland, Northeast China: Implications for regional population migration and economic development☆

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ABSTRACT

The polycyclic aromatic hydrocarbons (PAHs) of a ^{210}Pb-dated sediment core extracted from the Liaohe River Delta wetland were measured to reconstruct the sediment record of PAHs and its response to human activity for the past 300 years in Northeast China. The concentrations of the 16 U.S. Environmental Protection Agency priority PAHs (∑16PAHs) ranged from 46 to 1167 ng g^{-1} in this sediment core. The concentrations of the 16 PAHs (especially 4- and 5+6-ring PAHs) after the 1980s (surface sediments 0–6 cm) were one or two orders of magnitudes higher than those of the down-core samples. The exponential growth of 4-ring and 5+6-ring PAH concentrations after the 1980s responded well to the increased energy consumption and number of civil vehicles resulting from the rapid economic development in China. Prior to 1950, relatively low levels of the 16 PAHs and a high proportion of 2+3-ring PAHs was indicative of biomass burning as the main source of the PAHs. A significant increase in the 2 + 3 ring PAH concentration from the 1860s–1920s was observed and could be attributed to a constant influx of population migration into Northeast China. It was suggested that the link between historical trend of PAHs and population or energy use involves two different economic stages. Typically, in an agricultural economy, the greater the population size, the greater the emission of PAHs from biomass burning, while in an industrial economy, the increase in sedimentary PAH concentrations is closely related to increasing energy consumption of fossil fuels.

1. Introduction

Polycyclic aromatic hydrocarbons (PAHs) are ubiquitous environmental pollutants that have been the focus of many studies due to their carcinogenic and mutagenic properties. PAHs can be of anthropogenic or natural origin, and atmospheric PAHs are mainly derived from incomplete combustion of fossil fuels and biomass, as well as by coking and metal production in modern society (Yunker et al., 1996; Mai et al., 2003; Pereira et al., 1999; Xu et al., 2006). Energy consumption and emission levels could be reflected in PAH records in the sedimentary deposits, and thus can provide a picture of the economic development in a region (Pereira et al., 1999; Lima et al., 2003; Hartmann et al., 2005; Kannan et al., 2005; Liu et al., 2005; Guo et al., 2006; Itoh et al., 2010; Lin et al., 2012).

Delta environments are one of the ultimate sinks for PAHs from riverine discharge, wastewater and petroleum inputs, urban runoff, and atmospheric deposition (Mai et al., 2003; Lima et al., 2003; Xiao et al., 2014; Yang et al., 2015). The Liaohe River is the largest river in Northeast China and the 25th largest river (coursing 1345 km long) in the world (Milliman and Meade, 1983). Its drainage area covers ~30% of Northeast China, approximately 300,000 km^2, and the annual runoff is approximately 13 billion m^3. From 2001 to 2005, the Liaohe River carried an annual average of approximately 20 million tons of sediments into the Bohai Sea.

Northeast China, consists specifically of the three provinces of Liaoning, Jilin and Heilongjiang, and was the first important heavy-industry base in China (after 1949) because of its bountiful natural resources, in particular there has been considerable exploration and exploitation of petroleum in the region since the 1960s. However, before the 1860s, Northeast China was backward

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economy compared to the rest of China. The industrial development and population migration into Northeast China was forbidden by the Qing government. That prohibition broke down as the Qing began losing power in the mid 1800's (Mi and Jiang, 1996). Since then, a large population influx to Northeast China has been sustaining and the early industrial activities have been growing. Northeast China has its own unique history of energy usage and population movements, which differs completely from that of other areas of China.

In this study, a sediment core was collected from the center of Liaohe River Delta (LRD) wetland situated in the lower reaches of the Liaohe River, (Fig. 1). LRD has a relatively intact wetland environment, where water level is shallow and relatively stable. It is thus the ideal place to extract sediment core for the reconstruction of historical record of anthropogenic contaminants transported mainly from the drainage basin of the Liaohe River. This work aimed to establish the first 300-year sediment record of PAHs in the delta wetland to determine if there is a correlation between the PAH sediment record and socio-economic development (population and energy usage) in Northeast China and to infer the possible factors influencing the temporal PAH variations in the region.

2. Materials and methods

2.1. Sampling

The LRD is a flat topography made up of soft sediment soil, with several meandrous waterways that had a rich history of rerouting. LRD wetland area (121° 25′–122° 31′ E, 40° 40′–41° 25′ N) of nearly 6000 km^2, spanning the Panjin and Yingkou city of Liaoning province, is a deposition plain situated in the lower reaches of the Liao River (Fig. 1). LRD wetland is one of the largest reed coastal wetland area in China, and is also the world's second largest reed origin. Sample core GGLW was located at 41° 09′ 33.75″ N, 121° 47′ 42.71″ E, at the center of LRD wetland reed field area which is nature reserve (Fig. 1). This core was collected using a manual coring equipment in January 2012, and its length was 72 cm. The core was cut into 1-cm samples from 6 to 72 cm using a stainless steel cutter in the laboratory, while the top layer samples (0–6 cm) were sectioned every 2 cm. The samples were wrapped in aluminum foil and stored at −20 °C until analysis of organic compounds.

2.2. Extraction and PAH analysis

Analytical procedures and quality assurance/quality control essentially followed the methods reported by Mai et al. (2003) and Lin et al. (2011). The approximately 8 g of the sample were freeze-dried, ground, and spiked with a mixture of recovery standards of four deuterated PAHs (acenaphthene-d_{10}, phenanthrene-d_{10}, chrysene-d_{12} and perylene-d_{12}). The samples were extracted in a Soxhlet extractor for 72 h with dichloromethane, and then activated copper was added to remove the sulfur in the samples. The extract was fractionated in a silica-alumina (1:1) column using hexane/dichloromethane (1:1) as eluent. The PAH fraction was concentrated to approximately 0.5 ml under a gentle nitrogen stream, and hexamethylbenzene was added to the mixture as an internal standard. The samples were analyzed by the Agilent 5975C

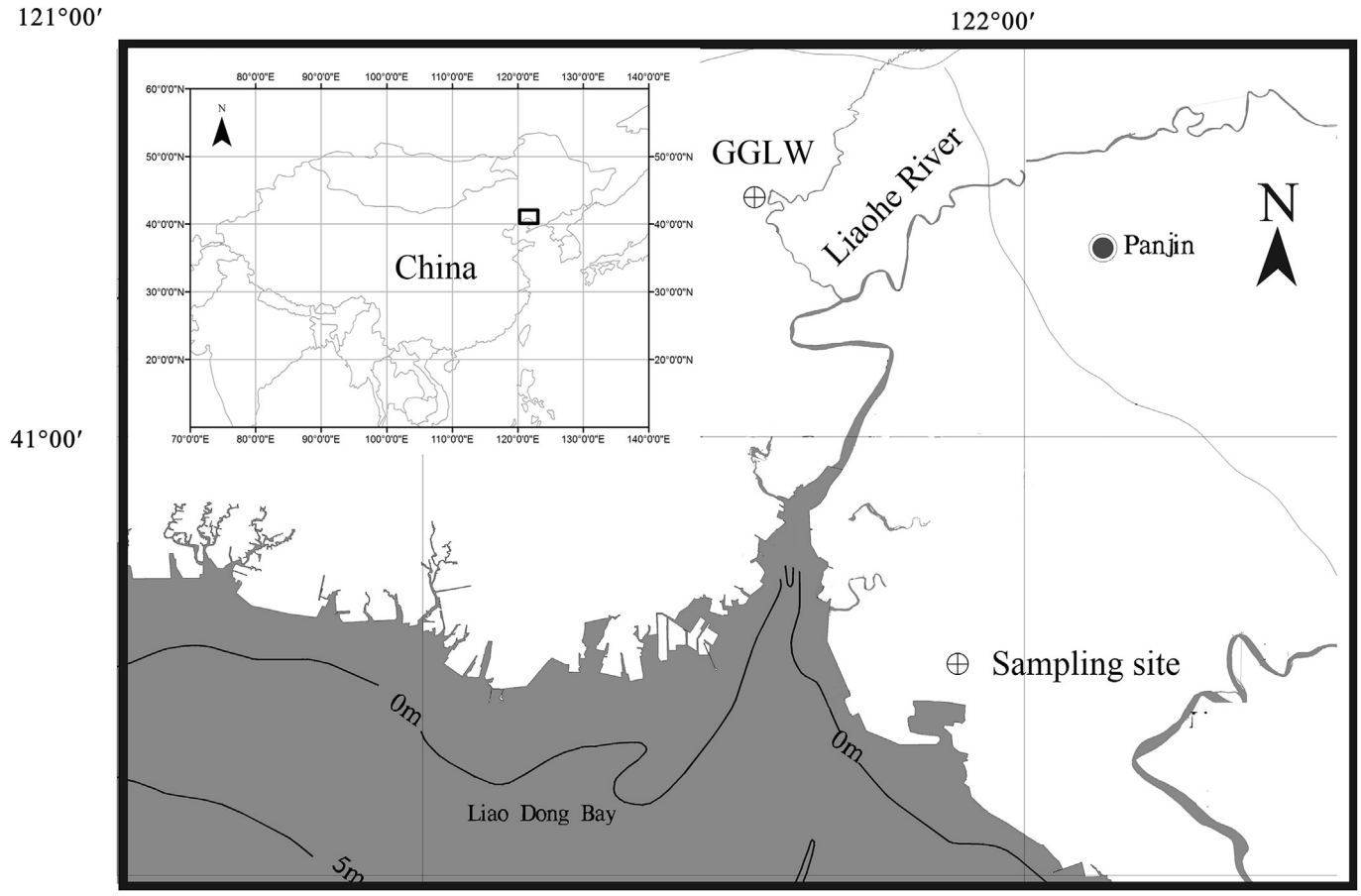

Fig. 1. Study area and location of the sampling site [revised from Ma et al., 2014].

mass spectrometer interfaced with the Agilent 7890A gas chromatograph. Using helium as the carrier gas, the GC was equipped with a DB-5MS capillary column (30 m length × 0.25 mm inner diameter × 0.25 μm film thickness). The chromatographic conditions were: injector temperature, 290 °C; detector temperature, 300 °C; temperature program, held at 60 °C for 3 min, increased from 60 °C to 290 °C at 3 °C/min, and held at 290 °C for 20 min.

PAHs were identified based on the retention times and mass spectra of target compounds against the authentic standards. 16 priority PAHs and four surrogate standards were quantified using the response factors related to the respective internal standards based on five-point calibration curve for individual compounds. The concentrations of 16 priority PAHs (16 PAHs) proposed by the U.S. Environmental Protection Agency and abbreviation are list in Supporting Information Table S1. In the lab, PAHs recoveries of the standard spiked matrix ranged from 85% to 95%. Recoveries of the PAHs for standard reference material (*NIST 1941b*) were between 80% and 120% of the certified values (Mai et al., 2003). Limits of detection and quantification (defined as the mean blank value plus 3 and 10 times the standard deviation, respectively) for individual PAH compounds ranged from 0.2 to 2 ng/g. The average surrogate recoveries were 78 ± 9% for ACE-d_{10}, 83 ± 8% for Phe-d_{10}, 111 ± 16% for Chr-d_{12}, and 81 ± 31% for Pery-d_{12}, respectively. One procedural blank was run for every 10 samples. Procedural blank samples contained no detectable amount of the measured PAHs. The concentrations of PAHs reported in this work were not recovery corrected.

2.3. ^{210}Pb and ^{137}Cs analysis and core dating

The methodology for ^{210}Pb and ^{137}Cs dating of this sediment core was described in detail in Supporting Information S2.3. The average constant sedimentation rate of 0.24 cm/a at site GGLW was calculated using a constant flux-constant sedimentation rate model. The highest ^{137}Cs activity was detected at a depth of 6 cm, which is most likely 1986, due to the nuclear accident at Chernobyl. Fig. 2 shows that the ^{137}Cs dating was nearly consistent with the ^{210}Pb dating, indicating that the dating results are credible. The dating beyond 100 years on the core was used only as a reference.

3. Results and discussion

3.1. Concentrations and historical variations of 16 PAHs

The individual 16 PAH concentrations from sediment core GGLW are listed in Supporting Information Table S1, and the vertical distribution of the total 16 PAH concentrations (∑16PAHs) is shown in Fig. 3. The ∑16PAHs ranged from 46 to 1167 ng g^{-1} in the core samples. The average ∑16PAHs in the core after the 1850s was 2.4-fold higher than that of the background PAH values before the 1850s (except the surface and subsurface sediments) (Fig. 3). The highest PAH concentration observed was 1167 ng g^{-1} in the surface slice, and the second highest was 700 ng g^{-1} in the subsurface sediment (Table S1). These PAH concentrations observed in the surface and subsurface sediments in this study were comparable to those reported in the tidal flat in Haizhou Bay (73–805 ng g^{-1}) (Zhang et al., 2013) and the tropical inner lagoon of Las Matas, Gulf of Mexico (259–1176 ng g^{-1}) (Ruiz-Fernández et al., 2012). Overall, as Liu et al. (2013) reported, the concentrations of PAHs in sediments in China were much lower than those in the "old" industrialized countries (e.g. Germany and UK).

Before the 1850s, the lower ∑16PAHs ranging from 46 to 87 ng g^{-1} (66 ng g^{-1} in average) were observed (Fig. 3). These concentrations were still lower than those during pre-industrial time (53–440 ng g^{-1}, 1680s–1850s) observed in Osaka, Japan (Moriwaki et al., 2005). These low levels of the ∑16PAHs might reflect a closed stable and autarkic economic entity such as an agricultural civilization (Guo et al., 2006). A likely source of PAHs in the sediments was the combustion of wood and other biomass. From the beginning of the 1850s to the early 1870s, the ∑16PAHs increased rapidly from 56 to 121 ng g^{-1}. The Second Opium War (1856–1860) and the Taiping Heavenly Kingdom war (1851–1864) occurred during this period, when China experienced drastic social upheaval (Cao, 2001). During this time, China began to industrialize under the Westernization Movement. In the following 42 years (1876–1918), the levels of the ∑16PAHs remained elevated

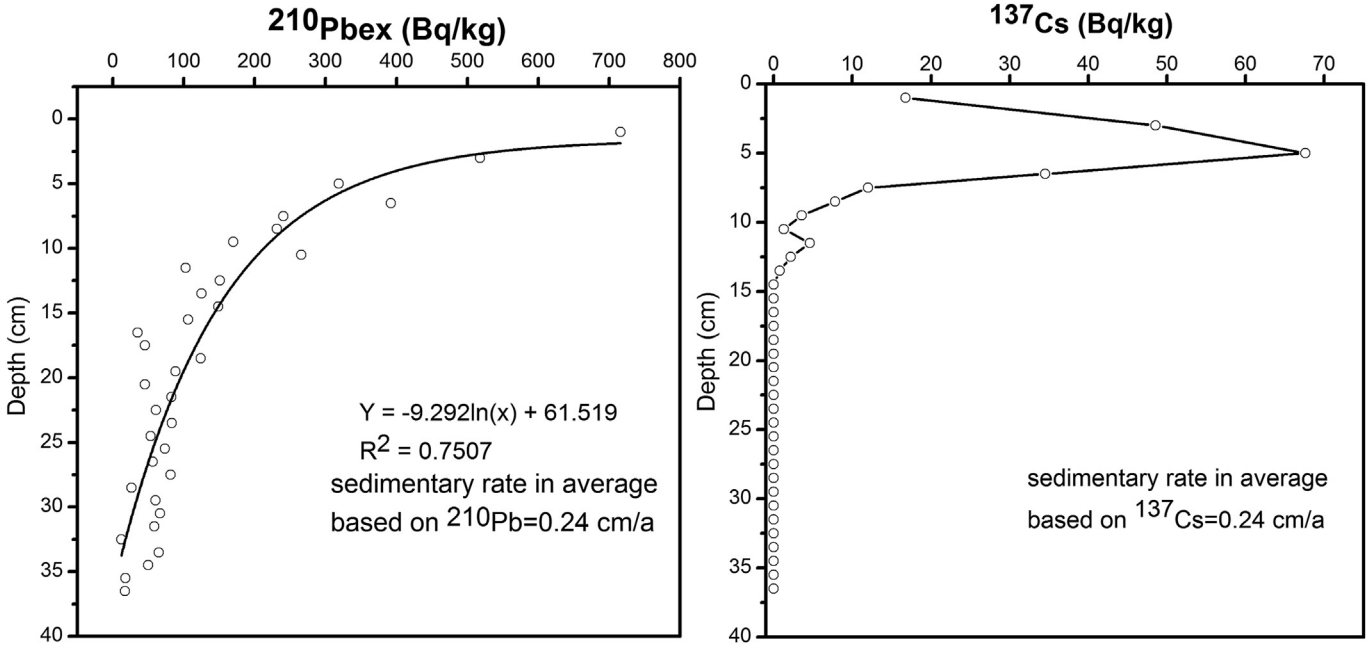

Fig. 2. Sediment records of ^{210}Pb and ^{137}Cs and its recent accumulation rate of sediment core GGLW.

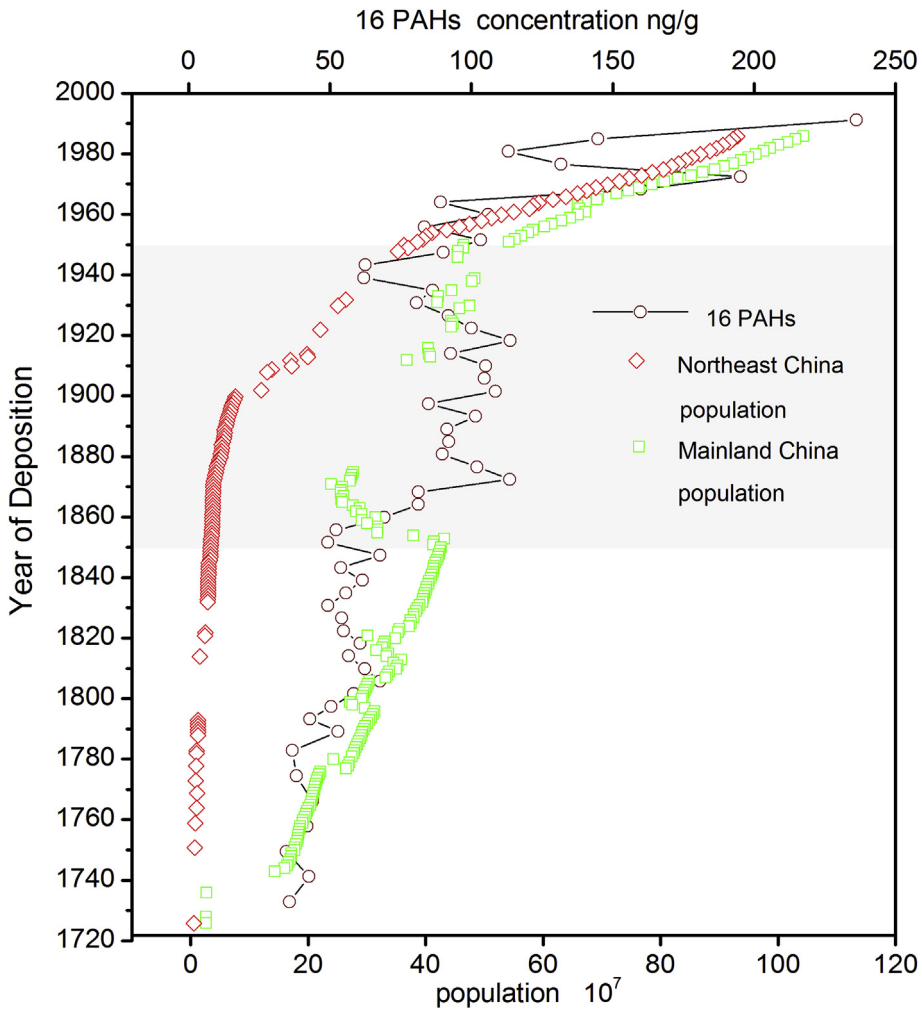

Fig. 3. The profiles of the total 16 PAHs concentration (circles) in the core GGLW and demographic population of Northeast China and China (nationwide) (surface and subsurface sediments 0–4 cm were not included). [The Northeast China data is from Zhao (2004); the 1949–1984 population data for Northeast China is from Liu (2011).].

(ranging from 100 to 131 ng g $^{-1}$, 112 ng g $^{-1}$ in average). The continued rise in the ∑16PAHs was observed during the long period from the 1850s–1920s, corresponding to the most turbulent period in modern China history. From the 1920s to the late 1930s, the ∑16PAHs decreased from 131 to 100 ng g $^{-1}$, reflecting a decreasing trend in PAH emissions. During this period, the warfare among warlords might have influenced economic development which resulted in the decreases in the ∑16PAHs. From 1934 to 1943, the ∑16PAHs further decreased from 100 to 70 ng g $^{-1}$. This might be due to the outbreak of World War II (1937–1945) against Japan, and China economy was completely destroyed (Guo et al., 2006).

The total concentrations of the 16 PAHs increased drastically from 96 to 1167 ng g $^{-1}$ after the 1950s. During this period, there was a short-term reduction in the PAH concentrations after the 1970s, decreasing from 223 to 124 ng g $^{-1}$, then the concentrations increased dramatically to 1167 ng g $^{-1}$. The trend of the ∑16PAHs has mainly followed the reconstruction and rapid economic development in China. The rapid increases of the ∑16PAHs after the 1960s corresponded to the industrialization process, especially the activities of oil field development and production in the delta. The Liaohe Oilfield began exploitation in 1967 and became the third largest oil field in China (Ma et al., 2014). The total concentrations of the 16 PAHs in the top-layer samples (0–6 cm) were one or two orders of magnitude higher than background concentrations observed in the down-core samples (Fig. 3), indicating a strong input in the region since the 1980s. After the 1980s, the rapid increases in the PAH concentrations were widely observed in many sediment record studies in China (Guo et al., 2006, 2007; Liu et al., 2009; Hu et al., 2011; Lin et al., 2012; Liu et al., 2012). This could be due to the rapid development of the economy in China with the initiation of the Reform and Open policy (Guo et al., 2006; Hu et al., 2011; Liu et al., 2012). Northeast China entered the fastest urbanization period ever experienced, and the local emissions have sustained increase and persisted to the present day.

Generally, the trends of the ∑16PAHs during this period in the LRD wetland are clearly different from those in the U.S. (Lima et al., 2003) and Europe (Fernández et al., 2000), chiefly due to the different energy structures of China compared with developed countries. The highest peaks of the PAHs occurred in the 1950s in the U.S. sediment core (Lima et al., 2003) and the European lake sediment core (Fernández et al., 2000), which was attributed to the switch from coal to oil and natural gas as an energy source (Fernández et al., 2000; Lima et al., 2003). Interestingly, we found that the sediment record of the ∑16PAHs in this work is also somewhat different from other PAH sediment records in the East China Sea (Guo et al., 2006; Hu et al., 2011; Liu et al., 2012), Pearl River estuary (Liu et al., 2005, 2012) and Taihu Lake in East China

(Liu et al., 2009). This could be due to the unique population migration history of Northeast China as discussed below.

3.2. Vertical composition and source profiles: implications for energy structure changes

Overall, low molecular weight (LMW) PAHs (2 + 3 ring PAHs) comprised 71–97% of the total 16 PAHs in this study (Figs. 4 and 5). PAHs in the core were dominated by LMW PAHs with no obvious change in the PAH composition throughout history. For the short slices between the 1810s and 1840s, the ratio of LMW to high molecular weight (HMW) PAHs (4–6 ring PAHs) increased gradually over time; then this ratio decreased from the late 1840s (Fig. 5). The LMW/HMW ratio continued to decrease from the late 1840s to recent years, except for a short increase during the 1960s, which was likely associated with the exploitation of the Liaohe Oilfield in the LRD in 1967. The increasing proportion of HMW PAHs suggests that the contribution of high-temperature pyrogenic sources has been increasing gradually, especially in recent decades (Fig. 5, Guo et al., 2006, 2007; Hu et al., 2011; Lin et al., 2012).

PAH isomer ratios, such as Flu/(Flu + Pyr) (m/z = 202), IP/(IP + BghiP) (m/z = 276), and Ant/(Ant + Phe) (m/z = 178), were used as tracers to infer the possible sources of PAHs (Yunker et al., 2002) (Fig. 5). In this study, the ratios of Flu/(Flu + Pyr), IP/(IP + BghiP), and Ant/(Ant + Phe) are shown in Fig. 5. The Flu/(Flu + Pyr) ratio was >0.5, suggesting that the 4-ring PAH sources were mainly grass, wood, and coal combustion (Yunker et al., 2002). Meanwhile, the IP/(IP + BghiP) ratio was also > 0.5. Some previous studies have reported that a value between 0.18 and 0.40 corresponds to vehicle emissions, while values of 0.56 and 0.62 correspond to coal burning and wood combustion, respectively, indicating that in the study area, coal burning was an important source (Saldarriaga-Noreña et al., 2015). Similarly, the Ant/(Ant + Phe) ratio was >0.1 in most samples, also suggesting the dominance of a pyrogenic source in this region (Yunker et al., 2002).

Obvious changes (decreasing trends) in the ratios, for example Ant/(Ant + Phe), Flu/(Flu + Pyr), and IP/(IP + BghiP), occurred after the 1950s. The decreases in the isomer ratios after the 1950s (Fig. 5) reflected a transfer in the energy structure from firewood to coal and oil, and emission source from coal combustion to vehicle exhaust for 5–6 ring PAHs. During this period, the nation's economy transferred from an agricultural to an industrial economy. Variations in PAH compositions and isomer ratios have been regarded as indicators of the shift in PAH sources (Kannan et al., 2005), and this was also confirmed by the rapid increase trend in LMW/HMW ratio relative to that before 1950 (Figs. 4 and 5). This evidence reveals the transformation of sources and an energy structure shift since that time. After the 1950s, the 2+3-ring PAHs increased approximately three-fold, while there were 11.3- and 18.2-fold increases in the concentrations of 4-ring and 5+6-ring PAHs, respectively, during the same period. Thus, the increase rate in 4–6-ring PAHs was much higher than that of 2–3-ring PAHs after the 1950s, suggesting increasing emissions from energy usage at high temperatures, which is a modern industrial pollution

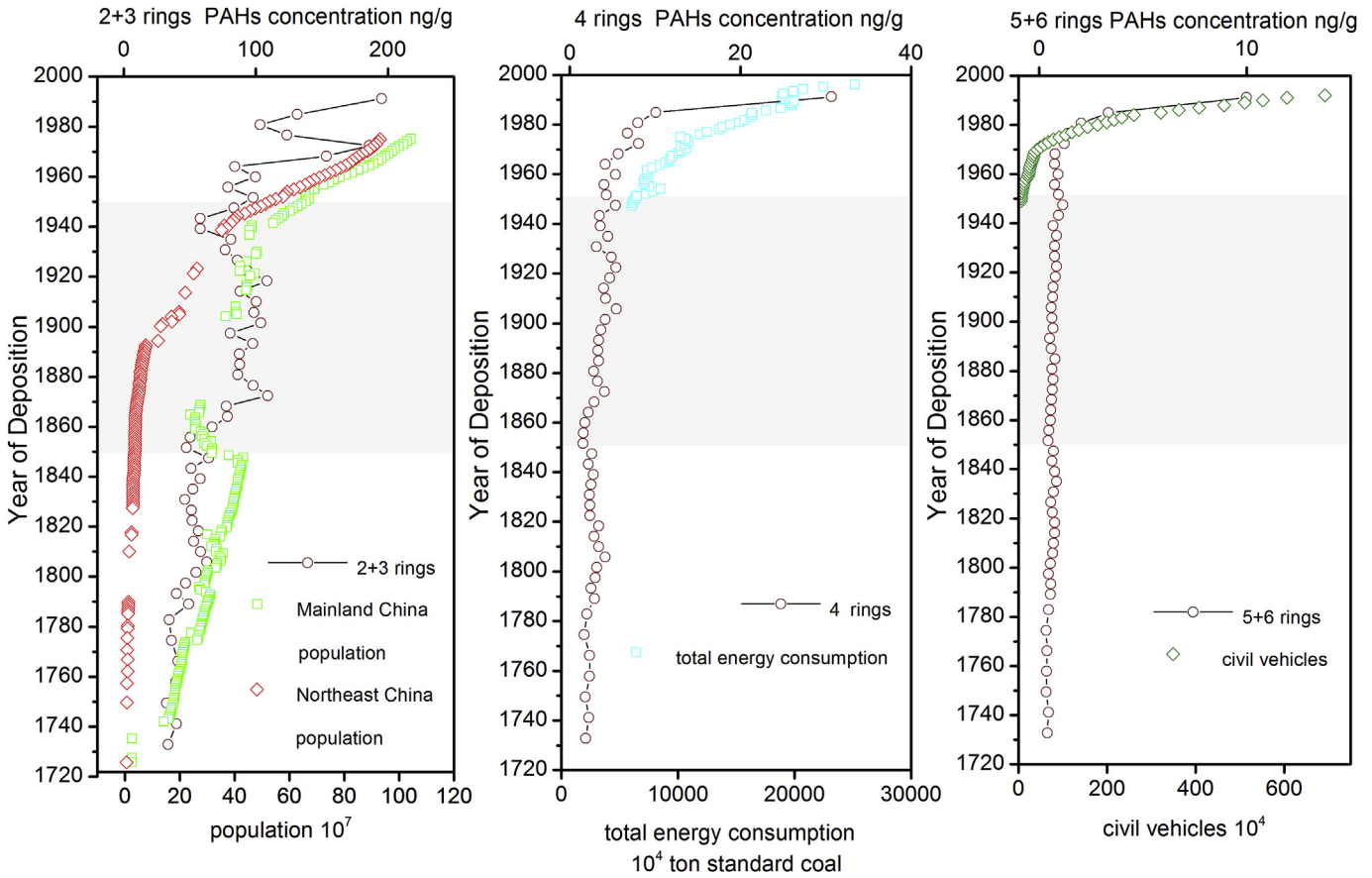

Fig. 4. Vertical distributions of 2+3-ring, 4-ring, and 5+6-ring PAHs concentrations (circles) in core GGLW (surface and subsurface sediments 0–4 cm were not included), compared with population (red), total energy consumption (blue), and the number of civil vehicles (green). [The data for energy consumption and the number of civil vehicles are from the National Bureau of Statistics of China http://www.stats.gov.cn/]. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

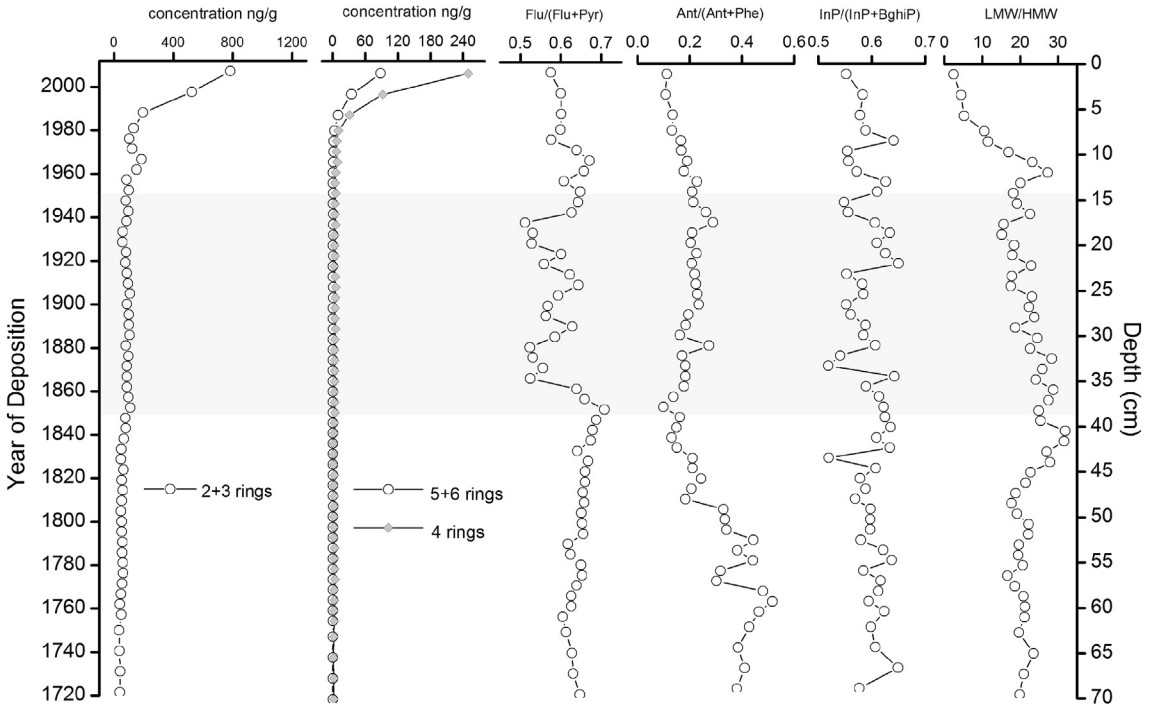

Fig. 5. The PAH concentrations and source diagnostic ratio profiles in core GGLW.

feature (Mai et al., 2003). Specifically, the mode of increase in 4-ring PAHs clearly matched that of energy usage, and the 5+6-ring PAH increase was the same increasing pattern as civil vehicles (Fig. 4). The main energy source in China is still coal (>60%), and total coal consumption increased ca. five-fold from 1980 to 2012 Fig. 4. In general, coal burning emits much higher levels of 4–5-ring PAHs than are released from the combustion of petroleum products or natural gas (Ravindra et al., 2008; Lin et al., 2011, 2012). Besides, 6-ring PAHs, IP, and BghiP are considered as tracers of automobile emissions in traffic tunnel, roadside, and urban environmental studies (Harrison et al., 1996; Nielsen, 1996). Vehicle-emitted PAHs increased three-fold in the past two decades in China, and vehicle emissions were estimated at 15 Gg in 2004, accounting for 13% of the total PAHs emitted in China (Shen et al., 2011). As a result, the large increase in PAHs after the 1950s in Northeast China can be due to the increase in coal consumption and vehicle emissions during the rapid economic development, indicating a significant energy structure change and sustained increase in energy consumption and vehicle numbers in this region.

3.3. *A possible link between population size and LMW PAH variation under an agricultural economic base*

In this study, the temporal trend in the total concentration of 16 PAHs in GGLW core mainly followed the fluctuations of LMW PAHs. The LMW PAHs have been identified as a major source from biomass burning before the 1950s when wide use of fossil fuels was not occurred. During this period, biomass burning only provided the basic heating and cooking requirements for household needs (Zhang et al., 2007). Thus, the general pattern of the LMW PAHs closely follows the population size in Northeast China. The first obvious increasing trend observed for the LMW PAHs was observed during the 1850s and persisted until 1920. This was the period of the Second Opium War (1856–1860) and the Taiping Heavenly Kingdom war (1851–1864) (Cao, 2001). During this time, China suffered several invasions by foreign powers, and the White Lotus Society (1796–1804) and Tianli Society dealt heavy blows to the Qing Dynasty (Cao, 2001). These heavy upheavals caused a severe population decrease during the second half of the nineteenth century in China (Cao, 2001). However, in this work, we observed an increase in the LMW PAH concentrations during this period of ongoing social turbulence (Fig. 3). The development of industry from the Westernization Movement generally promoted the output of the PAHs; however, the Westernization Movement had only a small impact in Northeast China, since this movement was mainly in eastern coastal areas of China such as Shanghai, Tianjin, and Guangzhou. The significant increase in 2–3-ring PAHs from the 1850s to the 1920s could thus possibly be derived from the increase in biomass burning for residential cooking and heating due to the large population migration, mainly from North China (e.g., Shandong, Henan, and Hebei provinces) to Northeast China. In fact, there was a marked increase in the population of Northeast China, significantly differing from the rest of the nation's population during that period (Fig. 3) (Zhao, 2004; Liu, 2011). Prior to 1860, population migration to Northeast China was forbidden by the Qing government (Zhao, 2004). Due to the weakness of the Qing dynasty and intrusions from neighboring countries, Northeast China was opened up for population migration in 1860 (Zhao, 2004). Since then, a large population influx into Northeast China was sustained. From the 1920s–1940s, however, the PAH concentration decreased intensively (Fig. 3), which could be due to the effect of the colonial rule in Northeast China by Japan. A large number of families were compelled to depart Northeast China (Cao, 2001).

The evidence from this study suggests that the increases in the ∑16PAHs before the 1950s changed synchronously with the growing population, suggesting that in an agricultural society, population size is the main factor influencing PAH concentrations, especially for LWM PAHs. However, ∑16PAHs after the 1980s followed the increases in the level of standard coal usage and number of civil vehicles. This indicates that the PAH emissions (mainly HMW PAHs) were directly related to energy consumption rather than population size during the stage of the rapid industrial

economic development in China.

4. Conclusions

This work showed that the ∑16PAHs were low (66 ng g^{-1} in average) and stable before the 1850s. The ∑16PAHs, mainly composed by 2–3 ring PAHs, significantly increased from the 1860s to the 1920s, which was in response to the growing population under an agricultural economic base. The PAH concentrations, especially 4–6-ring PAHs, significantly increased from the 1980s, corresponding to the rapid industrial economic development in China. This suggests a transition in PAH emissions from wood burning to fossil fuel combustion due to transformation from an agricultural to an industrial economy in China. This work revealed that the sediment record of PAHs in the LRD wetland can be used to track the unique population migration history and socio-economic development, especially the energy consumption and energy structure changes, in Northeast China.

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Appendix A. Supplementary data

Supplementary data related to this article can be found at http://dx.doi.org/10.1016/j.envpol.2016.12.065.

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