

Source apportionment of polycyclic aromatic hydrocarbons and *n*-alkanes in the soil-sediment profile of Jiangnan Oil Field, China

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Abstract Surface soil in oil exploration area always contains high contents of polycyclic aromatic hydrocarbons (PAHs) and *n*-alkanes. To investigate the migration possibility of PAHs and *n*-alkanes from surface through aquitard and aquiclude to aquifer, the distribution, together with the source apportionment using several indicators, such as composition pattern, fluoranthene/(fluoranthene+pyrene) (Flt/(Flt+Pyr)), anthracene/(anthracene+phenanthrene) (Ant/(Ant+PA)), and the carbon preference index (CPI) of *n*-alkanes, in a 30-m-deep soil-sediment profile were studied. Results showed that there were considerable PAHs and *n*-alkanes not only in surface soil but also in aquitard, aquiclude, and aquifer sediments. The PAHs and *n*-alkanes in surface soil strongly suggested petroleum pollution. The high molecular weight PAHs and the *n*-alkanes with both long and short chains could not migrate into deep sediments as their sources in surface soil and deep sediment were different. Whereas the aquitard and aquiclude had significant input of low molecular weight PAHs (LMWPAHs) from petroleum sources, the LMWPAHs in confined aquifer suggested pyrogenic sources. Therefore, LMWPAHs migrated from surface to aquitard and aquiclude, but did not cause aquifer pollution in Jiangnan Oil Field. However, the high mobility of LMWPAHs from surface

to aquitard and aquiclude suggested that the long-term risk of groundwater pollution from oil exploration should be concerned.

Keywords Polycyclic aromatic hydrocarbons · *n*-Alkanes · Molecular diagnostic ratios · Petroleum pollution

Introduction

Polycyclic aromatic hydrocarbons (PAHs) and *n*-alkanes, as the major organic pollutants of petroleum, can be accumulated in sediment for many years (Ockenden et al. 2003). Much attention had been paid to PAHs for its high teratogenicity and persistent carcinogenicity (Chiffre et al. 2015; Lu et al. 2012; Perrichon et al. 2014). PAHs and *n*-alkanes can transport into soil, sediment, and groundwater through leaching, evaporation, and migration (Cousins et al. 1999; Zhang et al. 2014), which potentially threatens the environment quality.

It had been reported that the surface soil close to some chemical or oil refinery plants and oil exploration area contained notable PAHs and *n*-alkanes (Oviasogie et al. 2006; Sojinu et al. 2010; Xu et al. 2016). For example, the total PAH content of the surface soil (depth 0–1 cm) from the site adjacent to an oil refinery in Zelzate, Belgium was up to 300 $\mu\text{g g}^{-1}$ (Bakker et al. 2000). The total PAH and *n*-alkane contents in the surface soil from the oil exploration site of Jiangnan Oil Field, China reached 1200 and 380 $\mu\text{g g}^{-1}$, respectively (Xu et al. 2016), which means severely polluted according to Maliszewska-Kordybach's classification about critical contents of soil PAH contamination ($>1000 \text{ ng g}^{-1}$) (Maliszewska-Kordybach 1996).

The question is if the PAHs and *n*-alkanes with such high contents can migrate into aquifer and threaten the safety of drinking water. Some references had shown that PAHs and

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n-alkanes drastically reduced from the surface to deeper soil (1–2 m) (He et al. 2009; Krauss et al. 2000; Li et al. 2011; Ma et al. 2005; Xu et al. 2016), indicating low transportability of these two groups of compounds. However, almost all the references showed a certain amount of PAHs in deeper soil, and no paper reported the occurrence and sources of PAHs and *n*-alkanes in deep soil-sediment profile (from 1 to 2 m to confined aquifer) so as to certify the possibility of migration of PAHs and *n*-alkanes from surface soil to confined aquifer. Therefore, PAH and *n*-alkane distributions, together with their sources, in a 30-m-deep soil-sediment profile, were investigated in the present paper.

Source apportionment can help us analyze the migration of PAHs and *n*-alkanes in soil-sediment profile (Yuan et al. 2016). PAHs in soil derive from natural or anthropogenic origins. Typical concentrations of endogenous PAHs in soils have reported to be in the range of 1–10 $\mu\text{g kg}^{-1}$ (Edwards 1983). The presences of PAHs in the environment results from anthropogenic processes are of two kinds: a petroleum origin (related to crude oil, its refined products, and coals) and a pyrogenic origin (related to incomplete combustion of organic matter) (Pies et al. 2008). The sources of *n*-alkanes can be either anthropogenic or natural. Anthropogenic sources of *n*-alkanes generally include fossil fuel and combustion of organic matter, while natural sources include biomass evolution products. The *n*-alkane analysis can be used to finger print spilled oils and to provide additional information on the sources of hydrocarbon contamination (Adedosu et al. 2013).

The source of PAHs and *n*-alkanes can be identified using various molecular indicators (Li et al. 2015). The PAH composition can indicate their sources (Zhang et al. 2013). Usually, PAHs from a petroleum source have more low molecular weight PAHs (LMWPAHs) (2–3 ring PAH percentage >50%), while pyrogenic sources are abundant of high molecular weight PAHs (HMWPAHs) (4–6 ring PAH percentage >50%) (Fernandes et al. 1997). The ratios of fluoranthene/(fluoranthene+pyrene) (Flt/(Flt+Pyr)) and anthracene/(anthracene+phenanthrene) (Ant/(Ant+PA)) were considered as criteria for distinguishing pyrogenic and petroleum sources. The ratio of Flt/(Flt+Pyr) <0.4 indicates petroleum sources while ratio >0.5 indicates pyrogenic source (Yunker et al. 2002). Similarly, the ratio of Ant/(Ant+PA) <0.1 is taken as an indication of petroleum source while the ratio >0.1 indicates a dominance of pyrogenic source (Budzinski et al. 1997). On the other hand, carbon preference index (CPI), as described by Eq. (1), is a measure of the relative abundance of odd versus even carbon numbered *n*-alkanes, and can be used to identify the source of *n*-alkanes (Ahmed et al. 2006).

$$\text{CPI} = \frac{(n_{23} + n_{33}) + 2(n_{25} + n_{27} + n_{29} + n_{31})}{2(n_{24} + n_{26} + n_{28} + n_{30} + n_{32})} \quad (1)$$

where n_{number} means the content of *n*-alkane with certain carbon number. The *n*-alkanes from a petroleum source have a CPI value ≈ 1 , while *n*-alkanes from biogenic sources have a CPI value >1 (Ahmed et al. 2006).

Therefore, in this paper, the compositions of PAHs and *n*-alkanes, Flt/(Flt+Pyr), Ant/(Ant+PA), and CPI values in the soil-sediment profile were determined and used for the source apportionment and discussion of migration possibility of PAHs and *n*-alkanes from surface soil to confined aquifer.

Methodology

Study site

The study area was in Jiangnan Oil Field. The texture of this field is mainly Quaternary deposits with a slope of 2°–3°, consisting of unconsolidated water-bearing sediment layers. The shallower unconsolidated sediments (10–35 m thick) from Holocene (Q_h) are mainly made up of clayey silt, sandy silt, silty clay, and interlaced clay lenses. The deeper sediment layers from Pleistocene (Q_p^3) are mainly composed of sand and gravel. The borehole (30° 26' 29.10", 112° 45' 51.77") was near an abandoned oil production well. The hydraulic conductivity coefficients are less than 100 $\text{m}^2 \text{day}^{-1}$ in upper sediment (depth < 18.7 m) from Holocene (Q_h), which is mainly aquiclude and aquitard. The hydraulic conductivity coefficients are between 350 and 2000 $\text{m}^2 \text{day}^{-1}$ in the bottom sediment (18.7 < depth \leq 30) from Pleistocene (Q_p^3), which is mainly confined aquifer.

Sampling and analysis

Totally, 41 soil and sediment samples were collected through wet drilling from the surface soil to 30-m depth sediment in June of 2015. Samples were dried and grinded at room temperature, and passed through a 60-mesh sieve and then stored in brown glass bottles prior to analysis. PAHs and *n*-alkanes were extracted from soil and sediment with dichloromethane according to EPA 3546 (EPA 1999). Identification and quantification of PAHs and *n*-alkanes were conducted using GC-MS according to EPA 8270D (EPA 2007). About 5 g dry soil and sediment samples were weighed precisely and placed into Teflon cylinders with recovery indicators (naphthalene-D8, phenanthrene-D8, benzo[a]pyrene-D10, tetradecane-D26, tetracosane-D42, triacontane-D62) and 25 mL dichloromethane. The extraction was conducted in a microwave extractor (MARS Xpress, USA). The microwave extraction was programmed as follows: raise the temperature to 80 °C for 5 min, and hold for 20 min. Microwave power was 100 W for each

sample (adjusted between the number of cylinders and the total power of instrument). The mixture was then cooled down for 10 min, and the upper extracted liquid was collected. The same extraction procedure was repeated twice by adding 10 mL dichloromethane. The three extract solutions were combined and dehydrated by adding anhydrous sodium sulfate (pre-baked), and then concentrated to about 1 mL by rotary evaporation at 35–40 °C. The concentrated extracts were cleaned up using a column (10 mm diameter, 250 mm length) consisting of 5 mm cotton wool (decontamination), 60 mm activated silica gel (decontamination and 3% deactivated with H₂O), 30 mm activated alumina (decontamination and 3% deactivated with H₂O), and capped with anhydrous sodium sulfate. The column was pre-eluted with 25 mL hexane, and the eluate was discarded. Just prior to exposure of the sodium sulfate layer to the air, the concentrated extracts were transferred into the column using an additional 2 mL mixture of dichloromethane and hexane (1:1 v/v) for repeating three times, and then eluted with 25 mL mixture of dichloromethane and hexane (1:1 v/v). Afterward, the eluate were concentrated by rotary evaporation, and blown exactly to 0.5 mL under a gentle stream of pure nitrogen, and subsequently transferred into vials with internal standard (hexamethylbenzene) for PAH and *n*-alkane analyses.

The concentrations of 16 PAHs (Table S1) containing naphthalene (Nap), acenaphthylene (AcPy), acenaphthene (AcP), fluorine (Flu), PA, Ant, Flt, 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), dibenzo[a,h]anthracene (DBA), and benzo[g,h,i]perylene (BghiP) and 20 *n*-alkanes (*n*-15 to *n*-34) in the extracts were determined by GC-MS (TRACE1310-ISQ, USA) coupled with automatic sampler (AS1310, USA). The DB-5 capillary column with 30 m × 0.25 mm i.d. × 0.25 μm film thickness was used with helium as the carrier gas. The identification of individual PAHs was based on the retention time and the mass spectrum with appropriate individual standards at full scan mode.

QA/QC measures

All data were subject to strict quality control procedures. Quantification was done using an internal calibration method. Based on a signal-to-noise ratio (S/N) of 3, the detection limits for 16 PAHs ranged from 0.27 to 3.25 ng g⁻¹ for soil and sediment samples. The detection limits for 20 *n*-alkanes ranged from 0.05 to 2.97 ng g⁻¹. The recovery efficiencies with spiked PAHs and *n*-alkanes in samples were in the range of 76.2–97.1, 85.0–110.5, 70.2–106.5, 85.7–115.5, 81.6–106.5, and 70.5–105.6% for naphthalene-D8, phenanthrene-D8, benzo[a]pyrene-D10, tetradecane-D26, tetracosane-D42, and triacontane-D62, respectively.

Results and discussion

Vertical profiles and composition of PAHs

As illustrated in Fig. 1, the surface soil was silty clay. Aquitard and aquiclude were composed of silty clay and clayey silts, and there were totally 33 samples at depth from 0.5 to 18 m in this horizon. Confined aquifer was composed of fine and medium sands, and there were totally seven samples at depth from 19 to 30 m in this horizon. Total PAH in the surface soil was 75.3 μg g⁻¹, which means severely polluted according to Maliszewska-Kordybach's classification about critical contents of soil PAH contamination (>1000 ng g⁻¹) (Maliszewska-Kordybach 1996). Total PAHs sharply reduced to 29.74–162.64 ng g⁻¹ in aquitard and aquiclude, and vertical distribution showed no clear tendency with depth and soil-sediment texture. Similarly, total PAH contents in confined aquifer sediment were from 13.02 to 156.21 ng g⁻¹.

In surface soil, 5–6 ring PAHs were dominated species, which accounted for 84.19% of total PAHs (Fig. 2). Among them, BbF, BaP, DBA, and BghiP were the most abundant (Fig. 3a). Contrastingly, in aquitard and aquiclude, 5–6 ring PAHs reduced, and 2–4 ring PAHs increased and became more than 75% of total PAHs (Fig. 2). Nap and PA were the most abundant composition in aquitard and aquiclude (Fig. 3b), but with no obvious tendency with depth and soil-sediment texture (Fig. 1). Flu, Flt, and Pyr were also of higher contents than other compositions. Aquifer sediments showed similar PAH composition with aquitard and aquiclude except that the Nap contents were much lower in aquifer sediments (8.53 ± 9.39 ng g⁻¹) than in aquitard and aquiclude (24.85 ± 13.22 ng g⁻¹).

Source apportionment of PAHs

Typical concentrations of endogenous PAHs in soils have reported to be in the range of 1–10 ng g⁻¹ (Edwards 1983). There is no standard value for the total PAH content in soil in China. However, Dutch government set 20–50 ng g⁻¹ PAHs as target value for unpolluted soil (Ma et al. 2005). In our research, total PAHs in almost all the soil-sediment samples exceeded 10 ng g⁻¹ and most of them exceeded 50 ng g⁻¹, which mean aquitard, aquiclude, and confined aquifer were polluted by PAHs.

The source of PAHs can be indicated by PAH composition (Zhang et al. 2013) and Flt/(Flt+Pyr) and Ant/(Ant+PA) ratios (Fernandes et al. 1997). Ant and PA belong to LMWPAHs, while Flt and Pyr belong to HMWPAHs. LMWPAHs and HMWPAHs may have different sources. Therefore, Ant/(Ant+PA) can indicate the source of LMWPAHs and Flt/(Flt+Pyr) can indicate the source of HMWPAHs. The high PAH contents, high percentage of HMWPAHs, and Flt/(Flt+Pyr) <0.4 in surface soil (Fig. 2) strongly suggest petroleum

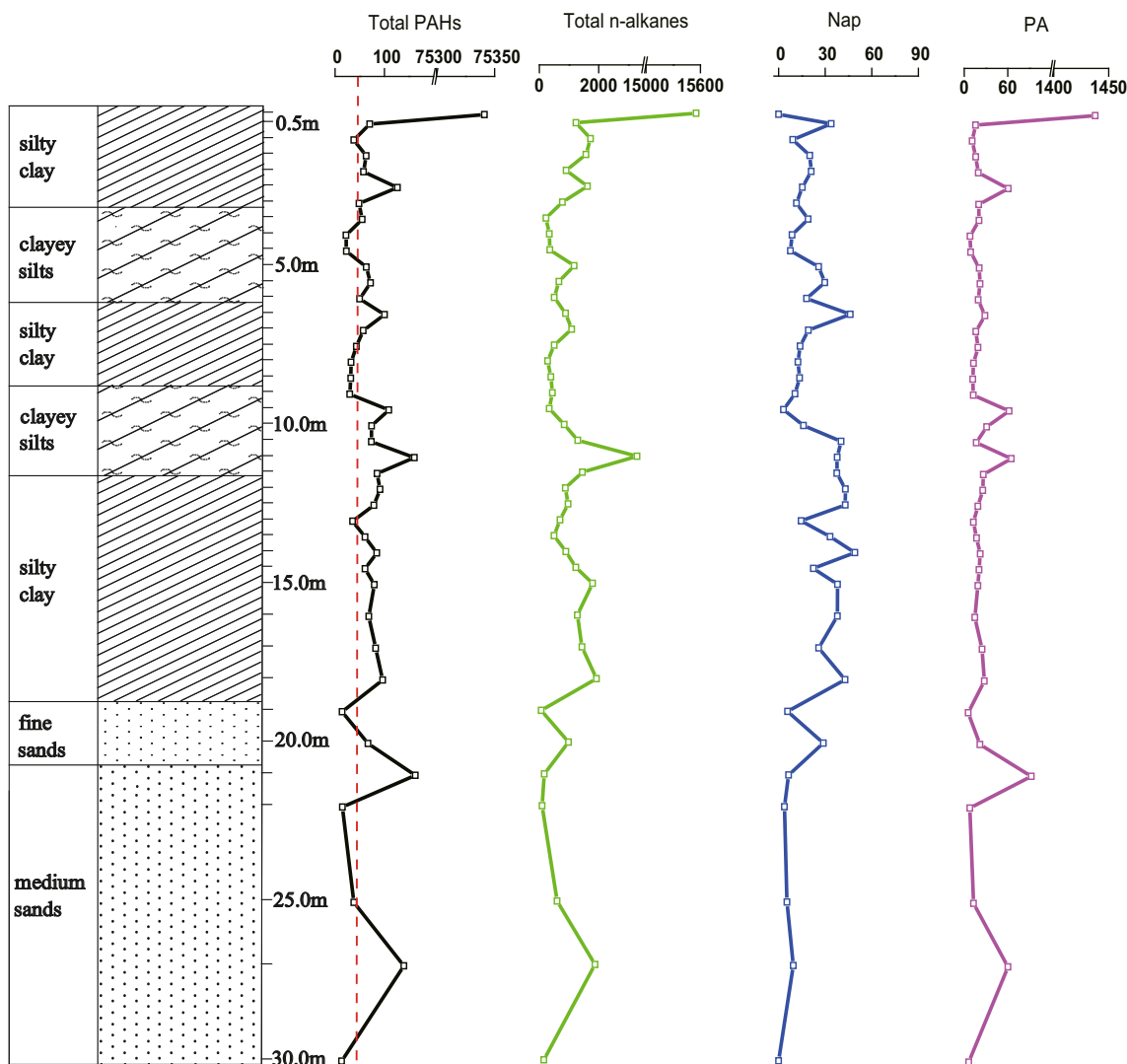


Fig. 1 Vertical texture of soil-sediment profile and contents (ng g^{-1}) of total PAHs, total *n*-alkanes, Nap, and PA

pollution. However, the LMWPAHs with $\text{Ant}/(\text{Ant}+\text{PA}) > 0.1$ indicated pyrogenic origin.

The persistence of PAHs was strongly affected by molecular weights. The aquitard and aquiclude were rich of LMWPAHs (Fig. 2), which were easier to be degraded and evaporated and with higher solubility and transportability than HMWPAHs (Jin et al. 2010), and most $\text{Ant}/(\text{Ant}+\text{PA})$ ratios were < 0.1 . These two evidences proved the presence of significant input of LMWPAHs from petroleum sources for aquitard and aquiclude. However, the HMWPAHs with most $\text{Flt}/(\text{Flt}+\text{Pyr}) > 0.5$ in aquitard and aquiclude indicated pyrogenic sources.

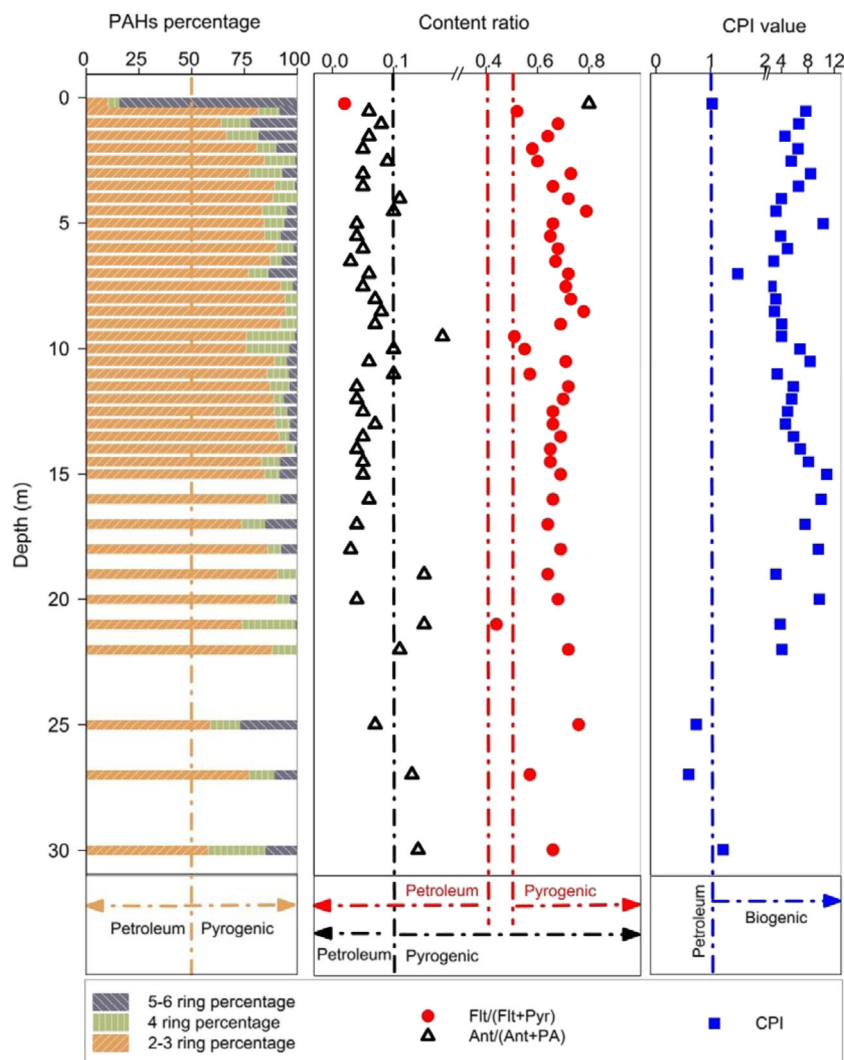
The confined aquifer also contained high percentage of LMWPAHs from 57.85 to 90.54%, but with most $\text{Ant}/(\text{Ant}+\text{PA}) > 0.1$ (Fig. 2) which suggested pyrogenic sources. Confined aquifer was of high hydraulic conductivity coefficients, and groundwater supply in the confined aquifer is more intense in the horizontal direction. High value of $\text{Ant}/(\text{Ant}+\text{PA})$ ratio may due to a new LMWPAH pollution source from groundwater

field of confined aquifer. For HMWPAHs, the same with that in aquitard and aquiclude, the confined aquifer had been polluted by pyrogenic sources due to $\text{Flt}/(\text{Flt}+\text{Pyr}) > 0.5$.

Vertical profiles and composition of *n*-alkanes

The total contents of 20 *n*-alkanes ($n\text{-}15\sim n\text{-}34$) in soil-sediment profile are shown in Fig. 1. In agreement with previous research, the total *n*-alkanes was up to $15.6 \mu\text{g g}^{-1}$ in surface soil, which indicated obvious pollution (Xu et al. 2016). Vertical distribution of total *n*-alkanes showed no clear tendency with depth. The vertical profile feature of *n*-alkanes in aquitard and aquiclude was the same with previous research (Wang et al. 2012). Total contents of *n*-alkanes ranged from 277.07 to $3291.96 \text{ ng g}^{-1}$ (average $1013.48 \text{ ng g}^{-1}$) (Fig. 1) in aquitard and aquiclude. Obviously lower than that in aquitard and aquiclude, total contents of *n*-alkanes ranged from 59.28 to $1874.28 \text{ ng g}^{-1}$ (average 560.00 ng g^{-1}) in confine aquifer.

Fig. 2 Vertical biomarkers feature in soil-sediment profile



The abundances of individual *n*-alkanes in soil-sediment profile are shown in Fig. 4. The *n*-alkane composition in surface soil tended to be long chain. There were two humps for the *n*-alkane composition in aquitard and aquiclude samples. Long-chain *n*-alkanes from *n*-27 to *n*-33 were dominated and of obvious odd carbon advantage. The shape of short chain *n*-alkanes from *n*-15 to *n*-20 was obvious hump pattern without odd carbon advantage. There also were two humps for the *n*-alkane composition in confined aquifer. However, different from aquitard and aquiclude, short-chain *n*-alkanes from *n*-15 to *n*-20 were dominant in confined aquifer. Long-chain *n*-alkanes from *n*-27 to *n*-33 were of no typical odd carbon advantage because of high *n*-28 content.

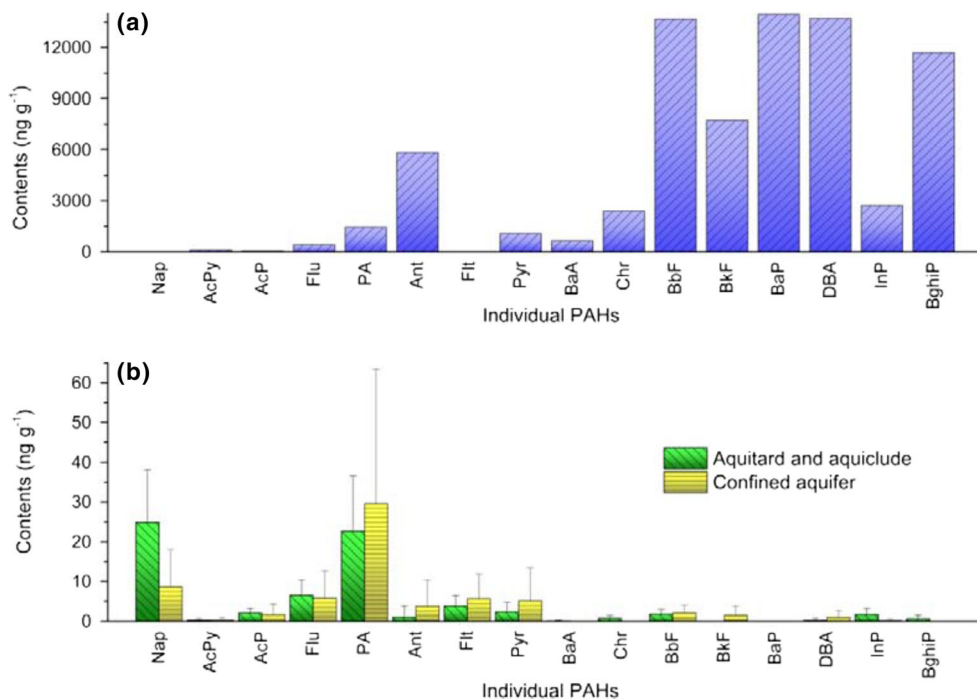
Source apportionment of *n*-alkanes

The *n*-alkanes, occupying 15–20 wt% of petroleum, were more resistant to diagenetic modifications and decomposition, and water insoluble (Chen et al. 2016). Therefore, *n*-alkane

biomarkers provide a long-lived indication of changes in sources of organic matter in sediment core (Fang et al. 2014). The *n*-alkane sources can be deduced by distribution of major composition (Wang et al. 2012). Petroleum pollution is of no even odd advantage. The hump shape of short chain *n*-alkanes from *n*-15 to *n*-20 without odd carbon advantage suggests phytoplankton sources (Wang et al. 2012). The hump shape of long-chain *n*-alkanes from *n*-27 to *n*-33 with obvious odd carbon advantage reflects land plant wax input (Rieley et al. 1991). The CPI value reflects the odd carbon advantage of long-chain *n*-alkanes (Ahmed et al. 2006).

Figure 2 shows that the CPI value of *n*-alkanes in surface soil was about 1.0, which indicated obvious petroleum pollution. Higher proportion of long chain *n*-alkanes in surface soil may be due to that *n*-alkanes with longer carbon chains were more difficult to be degraded than that with shorter carbon chains (Wentzel et al. 2007; Xu et al. 2016). For aquitard and aquiclude, there were two humps. Long-chain *n*-alkanes from *n*-28 to *n*-32 were dominant, and the CPI values were >1

Fig. 3 Composition of PAHs in surface soil (a) and aquitard, aquiclude, and confined aquifer sediments (b). Contents are expressed as means and standard deviations of each individual PAHs at depths from 0.5 to 18.0 m for aquitard and aquiclude and from 19.0 to 30.0 m for aquifer samples

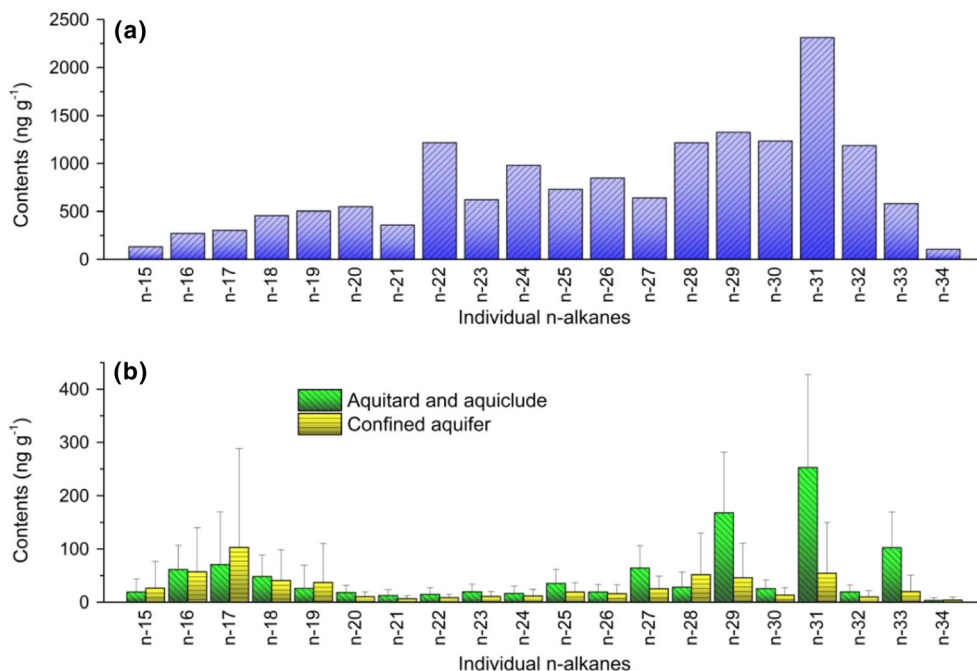


(from 1.5 to 10.9), which indicated evident biogenic higher plant wax sources instead of petroleum source. For confined aquifer sample, there also were two humps, but short-chain *n*-alkanes from *n*-15 to *n*-20 were dominating and with no odd carbon advantage, which suggested predominate phytoplankton source. Long-chain *n*-alkanes from *n*-27 to *n*-33 were of no typical odd carbon advantage, and CPI values were from 0.6 to 9.73, which indicated other sources besides biogenic origins.

Migration possibility of PAHs and *n*-alkanes from surface soil to confined aquifer

We concluded that surface soil was severely polluted by petroleum due to oil exploitation in Jiangnan Oil Field, China. It was reported that LMWPAHs were probably transported as true solutes (Krauss et al. 2000), while low solubility and strong affinity to organic matter severely limited vertical transport of HMWPAH with

Fig. 4 Composition of *n*-alkanes in surface soil (a) and aquitard, aquiclude, and confined aquifer sediments (b). Contents are expressed as means and standard deviations of each individual *n*-alkane at depths from 0.5 to 18.0 m for aquitard and aquiclude and from 19.0 to 30.0 m for aquifer samples



seepage water in deep soil (Gocht et al. 2007; He et al. 2009). Thereby, HMWPAHs were enriched in surface soil and LMWPAHs more potentially migrated to aquitard and aquiclude.

According to the source apportionment, LMWPAHs in aquitard and aquiclude had significant input from petroleum sources, which further proved the migration of LMWPAHs from surface soil to aquitard and aquiclude sediment. The confined aquifer also contained high percentage of LMWPAHs but mainly from pyrogenic sources, which suggested that the LMWPAHs in aquifer be not mainly from downward migration. The HMWPAHs with limited percentages in aquitard, aquiclude, and aquifer sediments mainly derived from pyrogenic sources. Therefore, the HMWPAHs from petroleum pollution in surface soil did not downward migrate into deep sediments.

CPI value indicated that the *n*-alkanes in surface soil were obviously from petroleum pollution. The dominant long-chain *n*-alkanes from *n*-28 to *n*-32 in aquitard and aquiclude were mainly from biogenic higher plant wax sources instead of petroleum source. The long-chain *n*-alkanes in aquifer sediments showed other sources besides biogenic origins. The dominant short-chain *n*-alkanes from *n*-15 to *n*-20 in aquitard and aquiclude together with that in aquifer sediment showed phytoplankton source. Therefore, the *n*-alkanes in surface soil did not migrate into aquitard, aquiclude, and aquifer.

Conclusions

In oil exploration area, surface soil was polluted by petroleum and with high contents of PAHs and *n*-alkanes. HMWPAHs and the *n*-alkanes with both long and short chains could not migrate into deep sediments. Even though LMWPAHs in aquifer were not mainly from aquitard and aquiclude in Jiangnan Oil Field, the high mobility of them from surface to aquitard and aquiclude warned us that the long-term risk of groundwater pollution from oil exploration should be concerned.

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