The source apportionment of polycyclic aromatic hydrocarbons (PAHs) in the topsoil in Xiaodian sewage irrigation area, North of China

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Abstract 31 topsoil samples were collected by grid method in Xiaodian sewage irrigation area, Taiyuan City, North of China. The concentrations of 16 kinds of polycyclic aromatic hydrocarbons (PAHs) were determined by gas chromatograph coupled with mass spectrum. Generally speaking, the distribution order of PAHs in the area is: those with five and six rings > those with four rings > those with two and three rings. Source apportionment shows a significant zonation of the source of PAHs: the civil coal pollution occurred in the north part, the local and far factory pollution happened in the middle area and the mixed pollution sources from coal and wood combustion, automotive emission, presented in the south area. The distribution of PAHs has a definite relationship with the sewage water flow and soil adsorption. The related coefficient between PAHs and physicochemical property showed there was a negative correlation between pH, silt, clay and PAHs while there was a positive correlation between total organic carbon, sand and PAHs.

Keywords Sewage irrigation area · Polycyclic aromatic hydrocarbons (PAHs) · Topsoil · Source apportionment

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Introduction

China is a big country with many large irrigation areas. The agricultural irrigation consumes 70 percent of the total water amount. Sewage irrigation has been the effective approach to resolve the shortage of irrigation water (Chen et al. 2004). However, the sewage contains lots of organic contaminants such as polycyclic aromatic hydrocarbons (Chen et al. 2005; Ilani et al. 2005; Song et al. 2006). Polycyclic aromatic hydrocarbons (PAHs) are ubiquitous aromatic compounds, which are included with two to eight rings and mainly derived from biomass burning, incomplete combustion of fossil fuel, petroleum leakage/evaporation, and the early diagenesis of organic matter (Khalili et al. 1995). Because of their carcinogenic and mutagenic properties, they were widely studied (Lee et al. 1981; Williams 1990; Johnsen et al. 2005; Matson et al. 2009; Clark and Di Giulio 2012). 16 kinds of PAHs have been listed as priority pollutants in wastewater and 24 PAHs in soils, sediments, hazardous solid wastes, and groundwater by U.S. Environmental Protection Agency (USEPA) (Patnaik 1997). Both gaseous and particulate PAHs can transfer into soil by wet/dry atmospheric deposition over short and long distances (Tremolada et al. 1996; Park et al. 2000).

To recognize the sources of PAHs, many various methods have been used such as molecular maker index (Simoneit 1985; Brnner et al. 1995; Budzinski et al. 1997; Yunker et al. 2002; Zhao et al. 2012), stable carbon isotope ratio (Wilcke et al. 2002; Glaser et al. 2005; Zencak et al. 2007; Guillon et al. 2013) and multivariate method (Kavouras et al. 2001; Motelay-Massei et al. 2007; Liu et al. 2009). The source of PAHs maybe mainly results from the incomplete combustion of coal, petroleum and crude oil, automobile exhausts (Xiao et al. 2008) or from highway dust or from coke oven and wood burning (Harrison et al. 1996; Li et al. 2001).

Fig. 1 Map of the sampling sites in Xiaodian sewage irrigation area
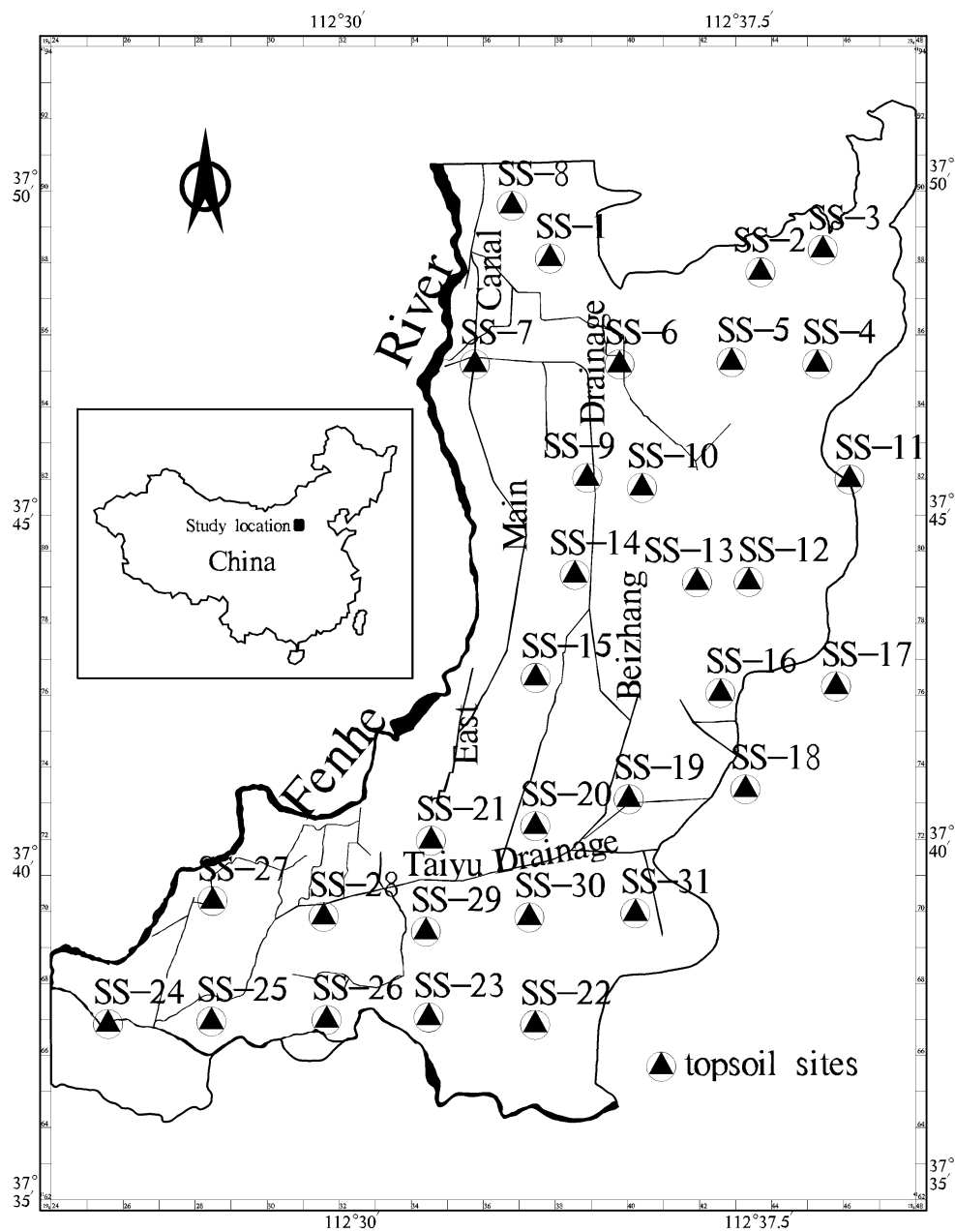
However, few studies have focused on the source apportionment of the topsoil in sewage irrigation area (Li et al. 2012; Zhang et al. 2013). This study takes Xiaodian sewage irrigation area as an example and use molecular maker index to discuss the source apportionment of PAHs in a large irrigation area, north of China Xiaodian.

Materials and methods

Study area and sampling

Our study area is Xiaodian sewage irrigation area which is located in the east part of the Taiyuan City, northern of China. Fenhe River and Xiaohe River go through the west and south of the area, respectively. There are three channels in the area: East Main Chanel, Beizhang Drainage and Taiyu Drainage. The sewage came from a sewage treatment plant lied in the northwest of the area, flowed through the area via East Main Channel, flooded all the fields and discharged to Fenhe River through Beizhang Drainage and Taiyu Drainage.

The sampling points were selected by grid method (3 km × 3 km) as shown in the Fig. 1. The topsoil samples were collected at 10–20 cm below. The sample was placed in an aluminium specimen box and sealed by parafilm and then refrigerated in freezer at 0 °C until analyses.

Experimental procedures

Extraction and clean-up

The methods of pretreatment and analysis was described in previous studies (Theodore et al. 2008; Xing et al. 2011). Briefly, topsoil samples were homogenized and freeze-dried. 20 ng of mix of deuterated PAHs standards (naphthalene-d8, acenaphthene-d10, phenanthrene-d10, chrysene-d12, and perylene-d12) as recovery surrogate standards were added to the samples prior to extraction. Samples were Soxhlet-extracted with 120 mL of Dichloromethane (DCM) for 24 h and further concentrated to about 2 mL using a rotary evaporator at 40 °C, Interfering compounds were removed by alumina/silica gel (1:2 v:v) clean-up column (8 mm i.d.) with 30 mL of dichloromethane/hexane (2:3 v:v). The samples were concentrated to about 0.2 mL under a gentle stream of nitrogen and then 1000 ng of hexamethylbenzene was added as internal standard compound prior to the GC–MS analyses.

Instrument analysis

The following 16 USEPA PAHs were analyzed: naphthalene (Nap), acenaphthylene (Acy), acenaphthene (Ace), fluorene (Flo), phenanthrene (Phe), anthracene (Ant), fluoranthene (Fla), pyrene (Pyr), benz[a]anthracene (BaA), chrysene (Chry), benzo[b]fluoranthene (BbF), benzo[k]fluoranthene (BkF), benzo[a]pyrene (BaP), indeno[1,2,3-cd]pyrene (Ind), dibenzo[a,h]anthracene (DiA), and benzo[g,h,i]perylene (BghiP). An Agilent 6890 N gas chromatography system and 5975 mass selective detector (GC–MSD) was used to determin the PAHs and the PAHs compound were separated on a HP-5 (30 m × 0.32 mm i.d. × 0.25 µm film thickness). Nitrogen was used as carrier gas at a flow rate of 2.5 mL/min in a constant flow mode. The oven temperature was programmed as follows: 60 °C for 2 min, 60–290 °C at 3 °C min^{-1}, 290 °C for 30 min.

Quality assurance (QA) and quality control (QC)

The lab blanks and field blanks showed no detected target compounds. Surrogate standards were added to each of the samples to monitor procedural performance. Mean recoveries for the surrogates were as follows: naphthalene-d8: 55 ± 15 %, acenaphthane-d10: 66 ± 12 %, phenanthrene-d10: 74 ± 15 %, chrysene-d12: 69 ± 8 %, perylene-d12: 84 ± 6 %. The concentrations of PAHs were corrected according to the recoveries of the surrogate standards.
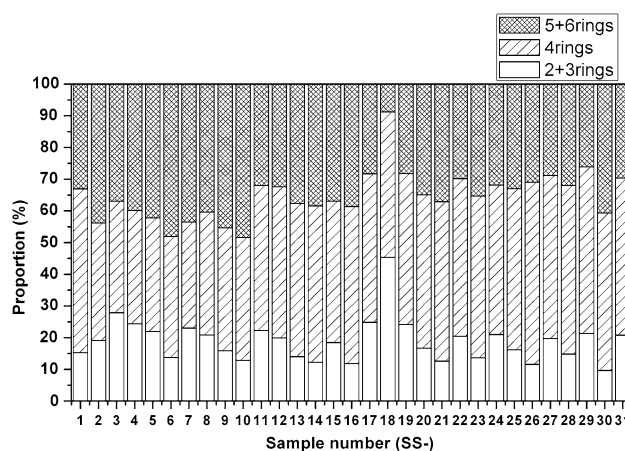

Fig. 2 Composition of PAHs in topsoils of Xiaodian sewage irrigation area

Physicochemical property analysis

5 g air-dried soil was placed in 20 mL deionized water, shaked for 1 min, stand until the pH meter (corporation: Hach, Inc., type: sension2) got stable number as the vaule of pH.

0.2 g air-dried soil was weighted, excess hydrochloric acid was placed in the soil to dislodge the inorganic carbon, placed the sample in the oven until it got dry and then the total organic carbon (TOC) was measured by total organic carbon analyser (corporation: Elementar, Inc., type: liqui-TOC, measurement range: $0 - 10^5$ mg/L C).

Grading Analysis were determined by full-automatic laser granulometer (corporation: Beckman Coulter, Inc., type: LS230, measurement range: 0.04–2000 µm).

Results and discussion

Source apportionment of PAHs

The three main common sources of PAHs are often considered such as the products from organic compounds reaction at a high temperature, the evaporation of petroleum at a low temperature, and the precipitation of geological dust. And the first one is regarded as the uppermost source (Yunker et al. 2002).

Relative abundance of PAHs ring can reflect pyrolysis and petroleum pollutions. Compared to the PAHs with a low molecular weight (two or three rings) imply petroleum pollution, those with a high molecular weight (more than four rings) come from fossil fuels combustion at a high temperature (Wang et al. 2013). Figure 2 shows the composition of PAHs in topsoil of Xiaodian sewage irrigation

area. Except soil sample SS-18, PAHs of all the rest soil samples are mainly 4 and 5+6 rings compounds, standing for over 70 % of the PAHs. It indicates PAHs here result mainly from fossil fuels combustion at a high temperature and aerosol enriched in soil by the way of dry/wet deposition.

As we known, molecular markers parameters were widely used in source apportionment of PAHs. The pollution source of PAHs with a molecular mass of 178 is considered as petroleum when Ant/(Ant+Phe) ratio is less than 0.10, otherwise it is combustion source when the ratio is greater than 0.10. Fla/(Fla+Pyr) ratio is generally considered as the transformation point to distinguish petroleum source and combustion source. When the Fla/(Fla+Pyr) ratio is less 0.50, the source of PAHs is petroleum. On the other hand, the source is coal, grass or wood combustion when its ratio is greater than 0.50 (Tsibart and Gennadiev 2013). In addition, the pollution source of PAHs is petroleum emissions when the ratio of Ind/(Ind+BghiP)is less than 0.20, while wood or coal combustion source for the ratio of Ind/(Ind+BghiP) >0.50 and petroleum combustion for between .20 to 0.50. (Yunker et al. 2002).

As can be seen from Fig. 3, the Fla/(Fla+Pyr) ratios of all the soil samples are above 0.50. It indicated that the main pollution sources of PAHs in study area are grass, coal and wood combustion, this is in agreement with another report (Wang et al. 2013). The ratios of SS-14, SS-23 and SS-30 are below 0.10, which shows that mixed pollution sources of motor vehicle exhaust, wood and coal combustion occurred in the three samples. Petroleum dischargement happened in sample SS-1. It happened in samples of SS-12, SS-19, SS-21, SS-22 and SS-27 where the Ind/(Ind+BghiP) ratio is below 0.50. These five samples just are located in the area near the cross among the East Main Channel, Beizhang drainage and Taiyu or the outlet of highroads. So, it can be referred that these areas are synthetically influenced by motor vehicles and channels.

Previous studies indicated that pollution source is traffic pollution when BaP/BghiP ratio varies from 0.3 to 0.44, the pollution source is civil coal pollution when the ratio is between 0.67 and 0.76, and that is coal pollution when the ratio range is from 0.9 to 6.6 (Yunker et al. 2002; Lin et al. 2013). Therefore, as the calculated values of BaP/BghiP ratio of all the samples, the pollution source of SS-2, SS-3, SS-4, SS-5, SS-7, SS-8, and SS-9 are civil coal pollution. The area around by these seven soil samples is in the northern Xiaodian, high ground, away from the downtown, and improbablely influenced by traffic, PAHs are mainly from local civil coal pollution. The reason why BaP/BghiP ratio of SS-6 is higher than the others is that a wool factory located in SS-6 soil sample area, its industrial coal is the pollution source.
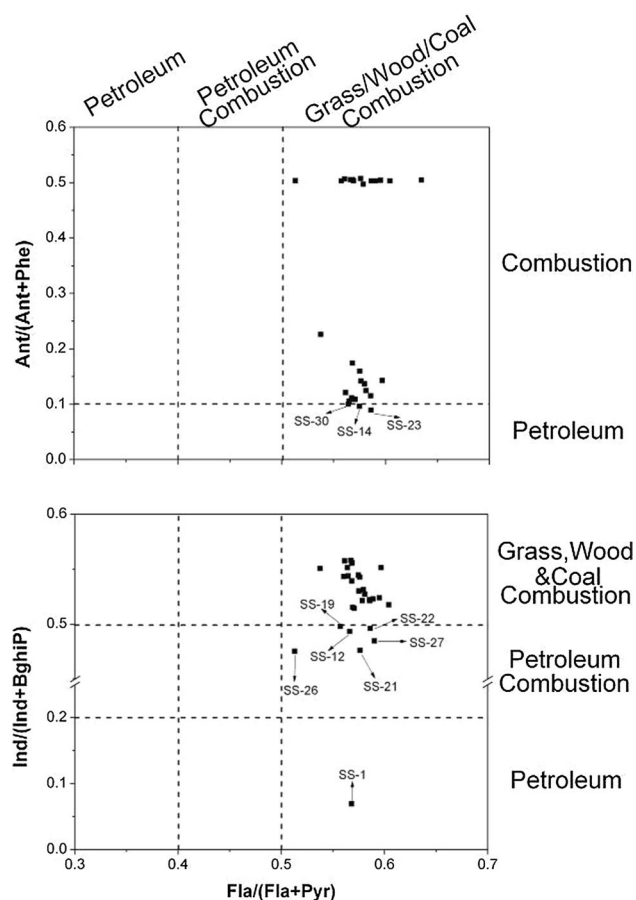

Fig. 3 Diagnostic ratios of PAHs in topsoils of Xiaodian sewage irrigation area

Assessment of PAH input pathways

As can be seen from Fig. 3, most Ant/(Ant+Phe) ratios in burden of Xiaodian sewage irrigation area are above 0.1. Combined with Fig. 1, it can be inferred that because of perennial north wind, PAHs produced by the factories located in the northwest and in the west of Xiaodian area enriched there by wet/dry deposition. This inference corresponds that the concentration of PAHs increases gradually in the direction from southeast to northwest. In addition, the concentration of PAHs has some relation with irrigation channels. As can be speculated from the phenomenon that PAHs has a low concentration in anterior east main channel, all downstream of the channels and drainage, enriched with sewage flow, the concentration of PAHs increases gradually, while as a result of soil adsorption, it decreased in downstream of the channels.

To sum up, source apportionment shows a significant zonation of the source of PAHs: the civil coal pollution happened in the north area, while the local and far factory pollution happened in the middle area and the mixed pollution, from coal and wood combustion, automotive

Table 1 Physicochemical property of topsoils of xiaodian sewage irrigation area

	pH	TOC (%)	Grading analysis			
			Clay (%) (0–2 μm)	Silt (%) (2–50 μm)	Sand (%) (50–2000 μm)	Determine
SS-1	7.65	7.87	6.05	53.02	40.92	Silt loam
SS-5	8.31	2.14	7.73	63.14	29.13	Silt loam
SS-9	8.19	1.66	12.61	86.18	1.21	Silt loam
SS-11	8.53	1.36	5.99	46.71	47.30	Sandy loam
SS-14	8.13	1.89	11.55	74.78	13.66	Silt loam
SS-15	8.47	2.66	9.26	67.80	22.94	Silt loam
SS-18	10.21	0.16	5.45	62.78	31.77	Silt loam
SS-19	8.45	0.83	9.50	79.55	10.95	Silt loam
SS-20	8.33	1.21	6.40	52.66	40.95	Silt loam
SS-21	8.16	4.35	8.78	66.39	24.83	Silt loam
SS-22	8.20	0.56	7.89	68.44	23.17	Silt loam
SS-25	7.79	1.05	8.33	71.68	19.99	Silt loam
SS-28	8.24	1.94	10.80	78.56	10.64	Silt loam

emission, happened in the south area. The distribution of PAHs has a definite relationship with the sewage water flow and soil adsorption.

In order to reduce PAHs emissions, some useful measures should be taken. Burning adequately all kinds of fuel, enhancing monitoring and control, and strictly controlling vehicle exhaust emissions are all effective methods.

Result of physicochemical property analysis

The result of physicochemical property analysis of the topsoils was showed in Table 1.

The pH of all the samples are greater than 8 except SS-1 and SS-25. All the samples are alkaline soils. Particularly, the pH of SS-18 reached 10.21, so SS-18 was caustic soil.

Table 1 shows topsoils with high concentration of TOC presented the area where lied around Street Office of Xiaodian area. The highest value of TOC occurs in SS-1 reaching 7.87 %. All the values of TOC are greater than 1 % except SS-18, SS-19 and SS-22.

It can be seen from Table 1 that all the values of silt, sand and clay have a decreasing tend in all the samples. According to USDA-SCS soil textural triangle, all of the samples are silt loam except SS-11 (sandy loam).

Influencing factor of PAHs concentration

The related coefficient between PAHs and pH, TOC, granularity was showed in Table 2. According to the result, the following was found.

There was a prominent negative correlation between pH and PAHs concentration except Ace, Flo and InP. There is a positive correlation between pH and the polarity of the soil organic matter. As the increase of pH, the polarity of the soil organic matter also enhanced to adsorb the PAHs difficultly.

TOC is an important influencing factor in the transport of PAHs in soil (Sun et al. 2011; Zhao et al. 2012). Except Ace, InP and Flo, there was a strong relation between TOC and PAHs, many previous studies have reported the similar correlation with TOC and PAHs (Wang et al. 2001; Liu et al. 2008). This due to PAHs are hydrophobic matter. Once PAHs enter into soil, they combine with organic matter.

Seeing from the Table 2, there was a negative correlation between silt, clay and PAHs while positive correlation between sand and PAHs, this is similar with a recent study (Sun and Zang 2013).

Conclusions

This study analyzed the source apportionment of PAHs, the assessment of PAH input pathways and the influencing factor of PAHs concentration in Xiaodian sewage irrigation area, northern China. The results show that the distribution order of PAHs in the area is: those with 5+6 rings > those with 4 rings > those with 2+3 rings. The civil coal pollution occurred in the north part while the local and far factory pollution happened in the middle area and the mixed pollution sources from coal and wood combustion, automotive emission, presented in the south area. The distribution of PAHs has a definite relationship with the sewage water flow and soil adsorption. There was a negative correlation between pH, silt, clay and PAHs while

Table 2 Related Coefficient between PAHs and pH, TOC, Granularity

	Nap	Acy	Ace	Flo	Phe	Ant	Fla	Pyr	BaA	Chry	BbF	BkF	BaP	InP	DiA	BghiP	∑PAHs	pH	TOC	Clay	Silt	Sand
Nap	1																					
Acy	.715**	1																				
Ace	.694**	.583*	1																			
Flo	.690**	.868**	.611*	1																		
Phe	.702**	.976**	.599*	.951**	1																	
Ant	.615*	.951**	.638*	.853**	.942**	1																
Fla	.703**	.997**	.567*	.839**	.964**	.946**	1															
Pyr	.701**	.995**	.563*	.822**	.956**	.942**	.999**	1														
BaA	.691**	.990**	.552	.790**	.937**	.934**	.996**	.998**	1													
Chry	.873**	.945**	.685**	.915**	.956**	.887**	.934**	.928**	.912**	1												
BbF	.848**	.971**	.665**	.893**	.963**	.912**	.964**	.960**	.949**	.994**	1											
BkF	.698**	.979**	.548	.752**	.914**	.922**	.988**	.992**	.998**	.900**	.940**	1										
BaP	.783**	.993**	.622**	.862**	.966**	.936**	.991**	.990**	.985**	.969**	.989**	.978**	1									
InP	.582*	.366	.477	.745**	.535	.347	.323	.298	.246	.590*	.509	.201	.398	1								
DiA	.840**	.851**	.676**	.962**	.921**	.807**	.828**	.814**	.784**	.958**	.927**	.756**	.875**	.790**	1							
BghiP	.736**	.989**	.576*	.797**	.938**	.929**	.994**	.996**	.998**	.930**	.964**	.996**	.991**	.281	.807**	1						
∑PAHs	.786**	.993**	.625**	.872**	.971**	.937**	.991**	.989**	.982**	.973**	.991**	.974**	1.000**	.418	.885**	.988**	1					
pH	-.420	-.364	-.260	-.297	-.355	-.341	-.376	-.378	-.371	-.405	-.401	-.377	-.386	-.209	-.371	-.389	-.394	1				
TOC	.895**	.923**	.677*	.803**	.896**	.843**	.924**	.925**	.918**	.957**	.966**	.917**	.954**	.458	.881**	.940**	.956**	-.493	1			
Clay	-.059	-.255	-.285	-.106	-.172	-.360	-.240	-.246	-.270	-.131	-.174	-.285	-.226	.280	-.007	-.253	-.211	-.341	-.118	1		
Silt	-.203	-.338	-.531	-.239	-.292	-.485	-.328	-.331	-.344	-.273	-.302	-.355	-.328	.065	-.188	-.341	-.321	-.103	-.289	.878**	1	
Sand	.184	.331	.501	.223	.279	.474	.320	.324	.339	.256	.287	.351	.318	-.100	.164	.333	.310	.145	.268	-.912*	-.997*	1

** means $p < .01$; * means $p < .05$

there was a positive correlation between TOC, sand and PAHs.

Conflict of interest The authors declare that they have no conflict of interest.

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