

# Quantitative assessment of polycyclic aromatic hydrocarbons in sewage sludge from wastewater treatment plants in Qingdao, China

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Received: 21 December 2009 / Accepted: 9 November 2010 / Published online: 6 January 2011  
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**Abstract** In this study, 16 polycyclic aromatic hydrocarbons (PAHs) were detected in sewage sludge samples from four wastewater treatment plants (WWTPs) in Qingdao, China. These WWTPs differ in the type of treatment used and in the origin of the wastewater. The total amounts of PAHs in digested sludges ranged from 1.9645 to 6.5752 mg/kg, which did not exceed the projected European Union cut-off limits (6 mg/kg) for sludge found in farmland, except for the Haibohe WWTP. Significant differences were observed in overall PAH values between WWTPs receiving domestic effluents and those receiving industrial effluents. The total amounts of PAHs in digested sludge from the Licunhe and Haibohe WWTPs, which mainly received industrial effluents, were

markedly higher than those of the Tuandao and Huangdao WWTPs, which received only domestic effluents. The distribution of PAH compounds in digested sludges were analysed. At the Tuandao, Huangdao and Licunhe WWTPs, 2-, 3-, 4-benzene rings were predominant, accounting for 100%, 99.8% and 99.0% of the sum concentration of 16 PAHs ( $\sum$ PAHs), respectively. At the Haibohe WWTP, a large number of high molecular weight PAHs (5-, 6-benzene rings) were observed, accounting for 30% of the  $\sum$ PAHs. The sum of seven carcinogenic PAHs ( $\sum$ PAHs-c) ranged from 0.8694 to 3.0389 mg/kg in four WWTPs. The highest value was found in the Haibohe WWTP. Moreover, the PAH concentrations in sludges from the different treatment processes in the Licunhe and Tuandao WWTPs are discussed.

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**Keywords** Sewage treatment plant · Sewage sludge · Polycyclic aromatic hydrocarbons · Quantitative assessment · Distribution

## Introduction

Sewage sludge is a by-product of wastewater treatment processes in wastewater treatment plants (WWTPs). Sludges contain a multitude of organic materials and plant nutrients (nitrogen and phosphorous), which is why sludge is a very appropriate substrate to be used in agricultural land

as a fertiliser (Abad et al. 2005). In many of the European Union member states, over 50% of the total sludge is used in agricultural land (Smith 2000). Nevertheless, most of the heavy metals (Pb, Cr, Cu, Ni, Hg, Cd), organic contaminants [e.g. polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyls (PCBs), organic chlorinated pesticides, polychlorinated dibenzo-p-dioxins and furans (PCDD/Fs), phenols] and pathogenic bacteria remain in sewage sludges produced by water treatment processes. These compounds can damage soil organisms and adversely affect the growth of crops (Harms 1996). This restricts the usefulness of sludge as a fertiliser for agricultural land. Many studies have focused on heavy metals (Abdel-Shafy et al. 1996; Dong 1997; Fernández et al. 2000; Karvelas et al. 2003; Merrington et al. 2003), but only a few have looked at organic contaminants in the past 30 years (Berset and Holzer 1999; Pérez et al. 2001; Stevens et al. 2003; Pereira and Kuch 2005; Oleszczuk 2006). In fact, many organic contaminants may be more serious environmental hazards because of their toxicity, persistence in the soil and low biodegradability.

PAHs are a group of compounds that are polymerised by two or more benzene rings in different ways, many of which have been identified to be potent carcinogens (Tsai et al. 2007; Calder and Lader 1976). There is serious concern about in the environment, especially their tendency for bioaccumulation in food chains. Certain members of the PAH class have been listed as priority pollutants by Environmental Protection Agencies of the United States, Europe and China. The PAHs often detected in sewage sludge are mainly naphthalene, phenanthrene, anthracene, fluorene, acenaphthene, acenaphthylene, 1,2-benzanthracene, benzo(*a*)pyrene, benzo(*b*)fluoranthene, benzo(*g,h,i*)perylene, benzo(*k*)fluoranthene, fluorene, indeno(1,2,3-*cd*)pyrene and pyrene. Their contents can range across very wide borders. In the European Union, the content of PAHs is approximately 1–10 mg/kg in municipal sludge. However, in China, their content exceeds 10 mg/kg (Mo et al. 2001a). The subsequent fate of these organic compounds following sewage sludge disposal is a topic of current concern. Currently, there is a need for basic information, such as the concentrations of

PAHs in sewage sludge, in order to assess the environmental impact of specific compounds in sludge (Rogers 1996). Accordingly, the objective of this study was to determine the concentrations of the 16 PAHs listed as priority pollutants by the United States Environmental Protection Agency in sewage sludge from four WWTPs in Qingdao, China. These WWTPs are found along Jiaozhou Bay and differ in the type of wastewater used and the applied treatment technology. The 16 PAHs were analyzed by gas chromatography-mass spectrometry (GC-MS).

## Materials and methods

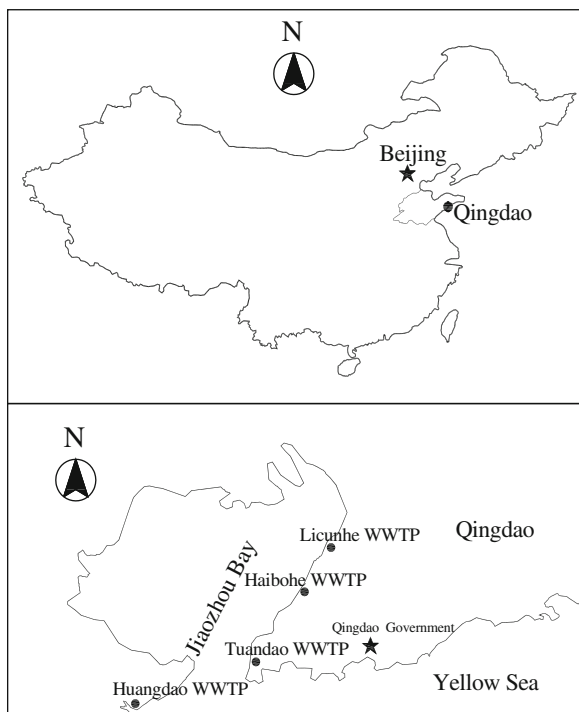
### Standard and reagents

A PAH standard solution mix was purchased from Accustandard, Inc., USA. It contained 16 PAHs including acenaphthene, acenaphthylene, anthracene, 1,2-benzanthracene, benzo(*a*)pyrene, benzo(*b*)fluoranthene, benzo(*g,h,i*)perylene, benzo(*k*)fluoranthene, chrysene, dibenz(*a,h*)anthracene, fluorene, indeno(1,2,3-*cd*)pyrene, naphthalene, phenanthrene, and pyrene.

HPLC-grade dichloromethane (DCM), acetone, and *n*-hexane were redistilled before use. Silica gel (80–100 mesh) and neutral aluminium oxide (100–200 mesh) were Soxhlet-extracted with DCM for 72 h, activated at 150°C and 400°C for 18 and 2 h, respectively, deactivated with 5% redistilled water, and kept in *n*-hexane before use. Anhydrous sodium sulphate was dried at 450°C for 5 h and stored in a sealed desiccator.

### Sampling

The sampling campaign was performed in May 2007. Given that the residence time of wastewater in these WWTPs, sampling rate allowed sufficient matching of influent and effluent samples that is a prerequisite for mass balance. Glass containers were used to collect sludge samples from four WWTPs (Licunhe, Haibohe, Tuandao, Huangdao) found along Jiaozhou Bay in Qingdao city, China (Fig. 1). The operating modes of the WWTPs are shown in Table 1. All samples were naturally dried for 5 days at room temperature in



**Fig. 1** Locations of four wastewater treatment plants (WWTPs)

a storage room, passed through a 20 mesh sieve and stored at  $-20^{\circ}\text{C}$  until analysis.

**Sample preparation**

Sludge samples were extracted by accelerated solvent extraction (ASE-100; 5 min each) for 5 min with 20 ml dichloromethane–acetone (1:1 *v/v*) at 15MPa and  $100^{\circ}\text{C}$ . The extracts was concentrated in a rotary vaporator to 1 ml and redissolved in 10 ml *n*-hexane and further reconcentrated to

1 ml. Then, the concentrated liquids were cleaned up on a Silica gel and a neutral aluminium oxide (2:1 *v/v*) column as well as on the top of a thin layer of anhydrous sodium sulphate (5 mm). The glass column (25 × 1 cm, length × I.D.), fitted with a polytetrafluoroethylene stopcock and cotton wool (Soxhlet-extracted with DCM for 72 h before use), was packed 6 cm alumina, 12 cm extracted silica, followed by 5 mm anhydrous sodium sulphate. The column was washed with 15 ml hexane in order to remove saturated alkanes and subsequently eluted with 70 ml dichloromethane-hexane (3:7 *v/v*). The analysis fraction was reconcentrated to 1 ml, carefully evaporated to dryness under a gentle stream of nitrogen and refrigerated. Before analysis it was dissolved in 1 ml hexane.

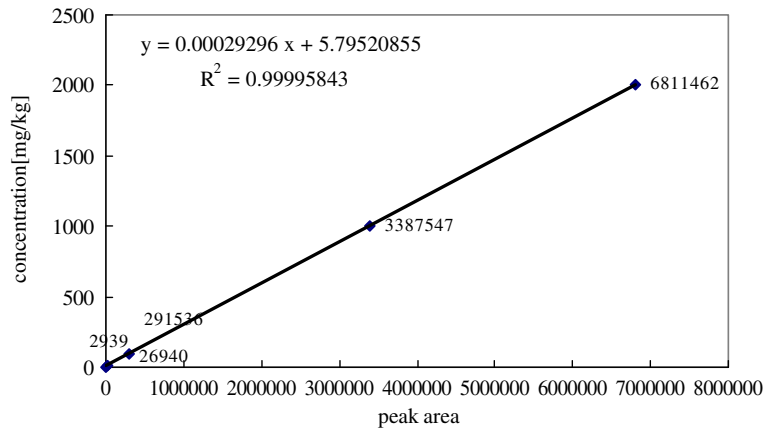
**GC-MS analysis**

GC-MS analyses were performed with an Agilent 19091S-433 Series system coupled to a mass spectrometer (Agilent 5975B). A HP-5MS column (30 m, 0.25 mm I.D., 0.25 μm film thickness) was used. The column temperature was increased from  $45^{\circ}\text{C}$  to  $200^{\circ}\text{C}$  at  $6^{\circ}\text{C}/\text{min}$ , further increased at a rate of  $8^{\circ}\text{C}/\text{min}$  until  $300^{\circ}\text{C}$  and held at that temperature for 5 min. The inlet and transfer line temperature were  $250^{\circ}\text{C}$  and  $280^{\circ}\text{C}$ , respectively. Data acquisition and analysis were performed using the selected ion monitoring (SIM) mode. Calibration curves were constructed for each PAH using a five-point calibration of mixed standard solutions ranging from 1 to 2,000 μg/l, and the correlation coefficients were  $>0.999$  for all PAH (The standard curve of benzo(*a*)pyrene showed as Fig. 2). The recoveries of real samples were

**Table 1** The sources of sludge samples and their characteristics

WWTP name	Treatment method	Number of inhabitants ( $\times 10^4$ pers)	Capacity ( $\times 10^4\text{m}^3/\text{d}$ )	Sewage source		Influents (mg/L)	
				Domestic sewage	Industrial sewage	BOD	COD
Licunhe	A/O+ VIP	25	8	25%	75%	488	933
Tuandao	A <sup>2</sup> O	26	10	90%	10%	450	900
Haibohe	AB	53	8	30%	70%	500~800	1500
Huangdao	OD	30	5.5	65~70%	30~35%	400~600	700

*A/O* anaerobic oxidisation process; *VIP* Virginia initiative plant (modified anaerobic–anoxic–oxic process); *A<sup>2</sup>O* anaerobic–anoxic–oxic process; *AB* adsorption biodegradation process; *OD* oxidising ditch process; *BOD* biochemical oxygen demand; *COD* chemical oxygen demand

**Fig. 2** The standard curve of benzo(*a*)pyrene

72.3~105.82%. The retention time, quantification ion and detection limits are provided in Table 2.

## Results and discussion

PAH concentrations in the digested sewage sludges

The PAH concentrations in the digested sewage sludges are shown in Table 3. The GC-MS chromatogram of 16 PAHs obtained from one sludge sample from the Licunhe WWTP is shown in Fig. 3. The results obtained from four WWTPs show that the sum concentration of PAHs ( $\Sigma$

PAHs) in digested sludge range from 1.9645 to 6.5752 mg/kg (Table 3). According to other reports from China,  $\Sigma$ PAHs in the sludge from Qingdao were present within a low mg/kg concentration range (Cai et al. 2007a; Dai et al. 2007). Moreover, the value of  $\Sigma$  PAHs from two WWTPs (Licunhe and Haibohe) that received industrial discharges were typically markedly higher than those (Tuandao and Huangdao) treating domestic effluents. The highest concentration was observed at the Haibohe WWTP, which is west of Qingdao. The region Haibohe WWTP serving for is a densely industrial region with many chemical plants and rubber factories, so the high concentrations of PAHs could be caused by the

**Table 2** Retention time, quantification ion and detection limits of PAH compounds in sludges

Compounds	Retention time(min)	Quantification ion(m/z)	Detection limits (ng/kg)
Naphthalene	12.85	128/102	4.08
Acenaphthene	18.83	152/76	1.35
Acenaphthylene	19.56	153/126	5.10
Fluorene	21.53	166/165	2.65
Phenanthrene	25.22	178/152	4.05
Anthracene	25.39	178/89	1.46
Fluoranthene	29.74	202/201	4.12
Pyrene	30.25	202/203	1.43
1,2-Benzanthracene	34.37	228/112	3.54
Chrysene	34.48	228/226	3.03
Benzo( <i>b</i> )fluoranthene	37.46	252/126	10.23
Benzo( <i>k</i> )fluoranthene	37.46	252/126	12.65
Benzo( <i>a</i> )pyrene	38.15	252/126	6.84
Indeno(1,2,3- <i>cd</i> )pyrene	41.43	276/138	4.76
Dibenz( <i>a,h</i> )anthracene	41.18	278/139	3.97
Benzo( <i>g,h,i</i> )perylene	41.80	276/138	3.24

**Table 3** Concentration of PAHs in digested sewage sludges from four sewage treatment plants in Qingdao, China (µg/kg)

Compounds	Ring number	Licunhe	Tuandao	Haibohe	Huangdao
Naphthalene	2	91.2	205.6	429.5	331.7
Acenaphthene	2	52.8	5.3	14.4	39.9
Acenaphthylene	2	25.2	N.D.	17.4	40.0
Fluorene	2	366.2	230.5	340.1	304.0
Phenanthrene	3	1441.2	176.0	1312.0	643.8
Anthracene	3	129.4	N.D.	109.0	43.4
Fluoranthene	3	396.6	239.5	485.0	233.7
Pyrene	4	577.0	238.2	829.0	516.7
1,2-Benzanthracene <sup>a</sup>	4	503.0	N.D.	9.8	203.2
Chrysene <sup>a</sup>	4	731.4	45.8	234.4	147.2
benzo( <i>b</i> )fluoranthene <sup>a</sup>	4	124.0	278.8	420.3	66.1
benzo( <i>k</i> )fluoranthene <sup>a</sup>	4	117.0	249.8	387.4	36.1
benzo( <i>a</i> )pyrene <sup>a</sup>	4	58.5	295.0	N.D.	10.3
indeno(1,2,3- <i>cd</i> )pyrene <sup>a</sup>	5	40.6	N.D.	1762.3	5.5
dibenz( <i>a,h</i> )anthracene <sup>a</sup>	5	N.D.	N.D.	N.D.	N.D.
benzo( <i>g,h,i</i> )perylene	6	0.1	N.D.	224.7	N.D.
∑PAHs		4654.2	1964.5	6575.2	2621.7
∑PAHs-c		1574.6	869.4	3038.9	468.5

<sup>a</sup>Carcinogenic PAH compounds

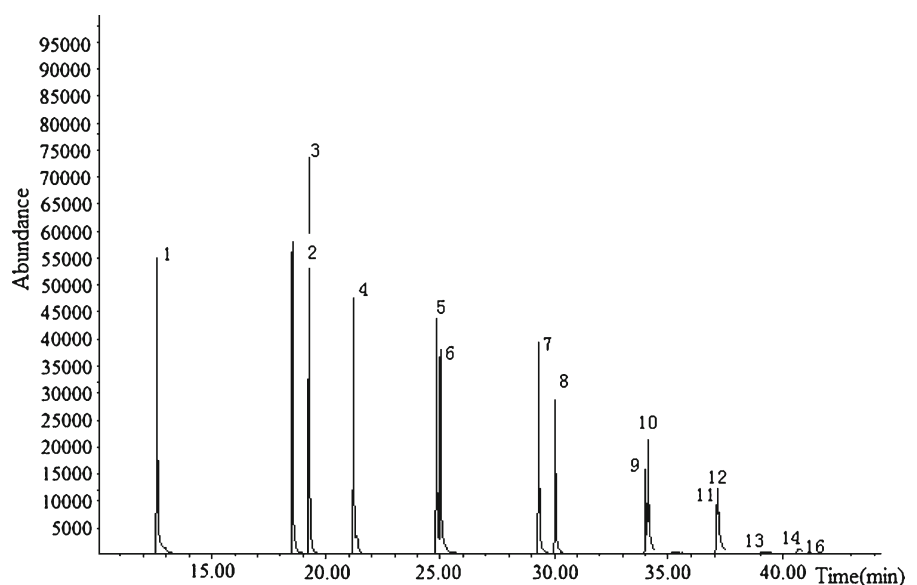
*N.D.* not detectable; ∑PAHs sum concentration of 16 PAHs; ∑PAHs-c sum concentration of carcinogenic PAHs

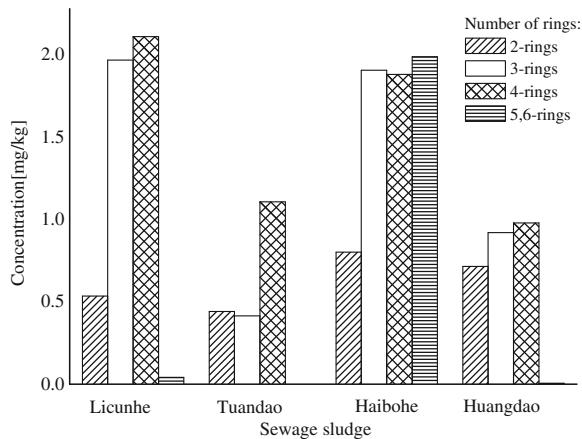
large amount of industrial wastewater. Relatively high concentrations were also found at the Licunhe WWTP, which mainly receives industrial wastewater.

Among the 16 PAHs, 1,2-benzanthracene, chrysene, benzo(*b*)fluoranthene, benzo(*k*)fluoranthene, benzo(*a*)pyrene, indeno(1,2,3-*cd*)pyrene and dibenz(*a,h*)anthracene are carcin-

ogenic (Cai et al. 2007b). The sum of the seven carcinogenic PAHs (∑PAHs-c) ranged from 0.8694 to 3.0389 mg/kg for four WWTPs. The highest value was found in the Haibohe WWTP, where the percentage was 46% of ∑PAHs. At the same time, a similar distribution between ∑PAHs-c and ∑PAHs was observed. The value of ∑PAHs-c from Licunhe and Haibohe WWTPs

**Fig. 3** The GC-MS chromatogram of 16 PAHs obtained from one sludge sample of Licunhe WWTP; Peaks: 1 naphthalene, 2 acenaphthene, 3 acenaphthylene, 4 fluorene, 5 phenanthrene, 6 anthracene, 7 fluoranthene, 8 pyrene, 9 1,2-benzanthracene, 10 chrysene, 11 benzo(*b*)fluoranthene, 12 benzo(*k*)fluoranthene, 13 benzo(*a*)pyrene, 14 indeno(1,2,3-*cd*)pyrene, 16 benzo(*g,h,i*)perylene





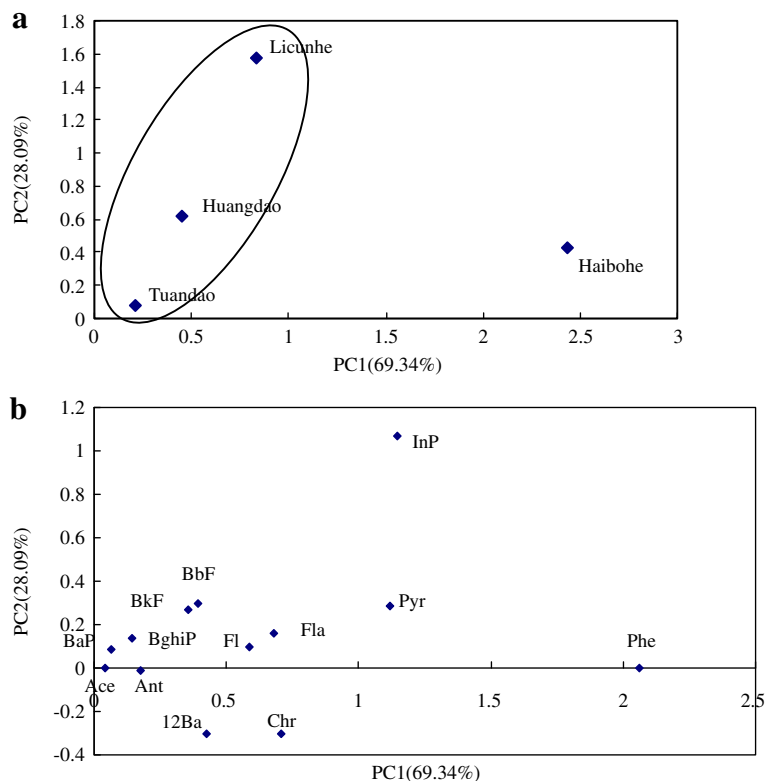
**Fig. 4** The number of rings of PAH compounds in sludges from Licunhe, Tuandao, Haibohe and Huangdao WWTPs

that received industrial discharges were typically markedly higher than Tuandao and Huangdao WWTPs treating domestic effluents.

PAHs in sludge from different WWTPs receiving different wastewater presented different dis-

tributions when grouped according to the number of benzene rings (Fig. 4). At the Tuandao, Huangdao and Licunhe WWTPs, 2-, 3-, 4-benzene rings were predominant, accounting for 100%, 99.8% and 99.0% of  $\sum$ PAHs, respectively. Of the three WWTPs, the Tuandao and Huangdao WWTPs lie in densely populated regions that mainly receive domestic wastewater. Therefore, the dominant PAH compounds were those with low molecular weight PAHs. At the same time, the Licunhe WWTP receives industrial wastewater, but the source of the wastewater is different from the Haibohe WWTP due to the many small industrial and machining companies around the Licunhe WWTP. At the Haibohe WWTP, a large number of the high molecular weight PAHs (5-, 6-benzene rings) were observed, accounting for 30% of the  $\sum$ PAHs. That result corresponded to the distribution of many chemical plants, printworks and rubber factories. The principal components analysis (PCA) revealed similar laws. The PCA results showed that the majority of the variance was contributed by two principal

**Fig. 5** Principal components analysis of PAH compounds in sludges. **a** Four wastewater treatment plants (WWTPs), **b** 16 PAHs



components and the cumulative contribution rate of them achieved 97.43%. The first principal component (PC1) contributed 69.334% of total variance. The second (PC2) was 28.09% of total variance. There are two groups on the factor loading plot. As illustrated in Fig. 5a, Group A concluded Tuandao, Huangdao and Licunhe WWTPs where 2-, 3-, 4-benzene rings were dominant, such as naphthalene, acenaphthene, acenaphthylene, fluorene, phenanthrene anthracene, fluoranthene, pyrene, chrysene, 1,2-benzanthracene, benzo(a)pyrene, benzo(b)fluoranthene, benzo(k)fluoranthene. Group B was Haibohe WWTP where 2-, 3-, 4-,5-,6-benzene rings PAHs were all observed.

PAH concentrations in different sludges

The source of wastewater, wastewater treatment methods and different treatment process affect the PAH concentrations in sludge. The PAH concentrations in sludges from the different treatment processes in the Licunhe and Tuandao WWTPs are listed in Table 4. The results showing the

PAHs concentrations varied strongly between the sludges from primary and secondary treatment processes. In two WWTPs, the  $\sum$ PAHs in the primary sludge were 1.5973 and 0.3402 mg/kg, whereas in the secondary sludge they were 8.7884 and 2.0185 mg/kg, respectively. This tendency was significantly different from the results of other correlative reports. Mocenhui and Webber each reported that the total amounts of PAHs enriched in primary sludge were between 12% and 81%, whereas in the secondary sludge the concentration accounted for only 7% (Mo et al. 2001b; Webber and Lesage 1989). This finding can be interpreted to indicate that the retention time of secondary sludge is longer than that of primary sludge, which results in an enrichment of PAHs over time. Moreover, when comparing the Licunhe WWTP to the Tuandao WWTP, the distribution of the PAHs was different for the same section of sewage sludge because the source of the sewage is different. The concentrations of phenanthrene, fluoranthene, pyrene, benzo(a)anthracene and chrysene in three sludge samples from the Licunhe WWTP were greater than those found in the Tuandao WWTP.

**Table 4** Comparison of PAHs concentrations in primary, secondary and digested sludges from Licunhe and Tuandao WWTPs (µg/kg)

Compounds	Lichuhe WWTP			Tuandao WWTP		
	Primary sludge	Secondary sludge	Digested sludge	Primary sludge	Secondary sludge	Digested sludge
Naphthalene	64.1	13.2	91.2	157.8	72.2	205.6
Acenaphthene	94.7	41.2	52.8	3.1	8.6	5.3
Acenaphthylene	21.8	2.7	25.2	N.D.	N.D.	N.D.
Fluorene	68.2	263.4	366.2	17.6	101.6	230.5
Phenanthrene	4.3	944.9	1441.2	7.9	208.1	176.0
Anthracene	10.3	N.D.	129.4	N.D.	2.1	N.D.
Fluoranthene	79.2	639.6	396.6	45.3	356.3	239.5
Pyrene	72.2	745.7	577.0	41.2	218.7	238.2
1,2-Benzanthracene	302.5	423.0	503.0	N.D.	N.D.	N.D.
Chrysene	18.1	781.2	731.4	12.3	67.5	45.8
Benzo(b)fluoranthene	30.5	212.1	124.0	17.2	386.5	278.8
Benzo(k)fluoranthene	28.2	223.1	117.0	35.1	227.1	249.8
Benzo(a)pyrene	3.4	88.2	58.5	2.7	369.8	295.0
Indeno(1,2,3-cd)pyrene	2.3	31.8	40.6	N.D.	N.D.	N.D.
Dibenz(a,h)anthracene	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
Benzo(g,h,i)perylene	N.D.	N.D.	0.1	N.D.	N.D.	N.D.
$\sum$ PAHs	1597.3	4410.1	4654.2	340.2	2018.5	1964.5

N.D. not detectable;  $\sum$ PAHs sum concentration of 16 PAHs

## Conclusion

PAHs in sewage sludge samples from four WWTPs in Qingdao, China that differed in the type of treatment and the origin of wastewater were detected. The total amounts of the PAHs were in the low mg/kg range and were found to be within the range reported by previous studies, which did not exceed the projected European Union cut-off limits (6 mg/kg) (CEC 2000) in sludge for farmland, except for the Haibohe WWTP. The PAHs in sewage sludges from the Licunhe, Tuandao and Huangdao WWTPs can be applied to soils. Significant differences in overall PAH values between WWTPs receiving domestic effluents and industrial effluents were observed. The total amounts of PAHs in digested sludge from the Licunhe and Haibohe, the WWTPs of which mainly received industrial effluents, were markedly higher than in the Tuandao and Huangdao WWTPs, which only received domestic effluents. The PAH concentrations varied strongly between the sludges from primary and secondary treatment processes. Therefore, it will be necessary in forthcoming studies to monitor the influents and sludges from different treatment processes in order to discuss the connection between PAHs in influents and various treatment sludges.

**Acknowledgements** This study has been supported by Ministry of Education Key Laboratory of Integrated Resource Development on Shallow Lakes (Project No. 2008KJ05). The authors would like to thank the four WWTPs for providing the sludge samples. We would also like to acknowledge the help of Key Laboratory of Marine Environment & Ecology, Ministry of Education for the GC-MS analysis.

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