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RESEARCH PAPER

Distribution and eco-toxicological risk characteristics of polycyclic aromatic hydrocarbons in water from Changdang Lake, China

Javid Hussain*[1], Ittehad Hussain[2], Raana Fahim[3], Jawad Ali[1], Zhang yunyun[4], Sadia Hassan Sherani[1]

[1]*Department of Environmental Sciences, Balochistan University of Information Technology, Engineering and Management Sciences, Quetta, Pakistan*

[2]*Research Center for Eco-Environmental Sciences, University of Chinese Academy of Sciences, Beijing, People's Republic of China*

[3]*Department of Environmental Science & Engineering, School of Energy and Environment, Southeast University, Nanjing, China*

[4]*Foriin Technology Company Limited, Shanghai, China*

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Key words: PAHs, Distribution, Organic matter, Eco-toxicological risk, Changdang Lake

Abstract

This study aimed to investigate characteristics of Polycyclic Aromatic Hydrocarbons in Changdang Lake, China during March (dry season), June (wet season) and September (Moderate Season). The average concentration of polycyclic aromatic hydrocarbons in March averaged 100.08ng /L (35.31~200.38ng/L), June 154.72ng/L (100.80~218.74ng/L), and in September 93.77ng/L (66.75~106.41ng/L). Among them, the residual benzo(a)pyrene was 5.90ng/L in water. By applying the redundancy analysis, results showed higher correlation between the fraction of 10-100kDa organic matter and the residues of higher rings PAHs (BbF, BkF, DahA, Bghip) and middle-rings PAHs (Ace and Chr). According to the risk classification of individual PAHs in water, the eco-toxicological risks of Ace, BaA, BbF, BaP and BkF in March, the Nap, Ace, Flu, Phe, Ant and BaA in June and Nap, Ace, Flu, Phe, Ant, BaA and BbF in September had reached moderate risk level. The lowest toxicological risk of PAHS in the Lake was found at site 2#and 3#, located near the center of the Lake. This study results also revealed that combine treatment processes of activated carbon and ultra-filtration membrane were quite efficient in removing PAHs from the water of Changdang Lake. Results obtained from this study meets the requirements of "drinking water health standards" (GB5749-2006) (PAHs <2000ng/L), However continuous monitoring and necessary actions are required to ensure the availability of safe water to the human and other living organism.

***Corresponding Author:** Javid Hussain ✉ javid.hussain@buitms.edu.pk

Introduction

Polycyclic aromatic hydrocarbon are among the most widespread persistent organic pollutants in the water environment and therefore are closely monitored in the environment (Duke, 2008; Sakuma, Leigh *et al*, 2011; Adeniji, Okoh, & Okoh, 2018). They are multiple ring structures having molecular masses in the range of 128 to 278 Dalton ((Kumar, Verma, Sharma, & Akolkar, 2015). The main sources of these contaminants in the environment include forest fire, natural petroleum seeps, and combustion of fossil fuel, coal burning, industrial and municipal waste, waste water and sewage (Santé *et al*., 2004).

PAH (which are known for their strong mutagenic, carcinogenic and toxic properties) are composed of carbon and hydrogen atoms arranged in the form of fused benzene rings (linear, cluster or angular arrangement). There are thousands of PAH compounds in the environment but in practice PAH analysis is restricted to the determination of 6 to 16 compounds. Individual PAHs differ substantially in their physical and chemical properties (Barbara Maliszewska-Kordybach, 2008)

The solubility of PAHs decreases in water with increasing molecular weight, resulting in low concentrations in the water column (Nasr, Arief, Abdel-Aleem, & Malhat, 2010; santé *et al*., 2004). Due to their hydrophobicity, the presence of PAHs in surface or ground water indicates pollution. Some PAHs occur in the environment at low concentrations due to their low biodegradability and persist with elimination difficulties. Consequently, low molecular weight (LMW) PAHs have a short residence time in the water column due to volatilization, oxidization and can quickly be eliminated (Qiu *et al*., 2009). High molecular weight (HMW) PAHs are readily adsorbed on particles in the surface water with the hydrophobic elements readily attaching to bottom sediments (Rhea *et al*., 2005). The types of PAHs present in water provide information on the derivative sources of organic contaminants. The presence of LMW PAHs such as naphthalene, fluorine and acenaphthene in environmental media indicate natural or petrogenic PAH contamination, while a prominent concentration of HMW PAHs (fluoranthene, phenanthrene and pyrene) and fewer LMW PAHs implies combustion or pyrolytic origins.

A number of them are genotoxic, mutagenic, carcinogenic, and/or teratogenic in nature with long range of transport and well implicated in endocrine system disruption at levels higher than the maximum allowable limit within a very short time (Brazkova & Krastanov, 2013; Cai, Syage, Hanold, & Balogh, 2009; Kim, Jahan, Kabir, & Brown, 2013). However, few of the nontoxic PAH congeners are found useful as synergists (Adekunle *et al*., 2017; ATSDR, 2000).

Scientists have tried to develop the best technology for the better treatment of pollutants in order to protect the human being and their environment. Number of methods such as coagulation, filtration with coagulation, ozonation, adsorption, membrane filtration, ion exchange, reverse osmosis and advanced oxidation processes have been used for the removal of organic pollutants from contaminated water (Rashed, 2013).

In recent years, the search for economical adsorbents that have pollutant-binding ability has increased. Natural materials, agricultural wastes and industrial wastes can be utilized as low-cost adsorbents. Activated carbon produced from these materials can be used as adsorbent for water and wastewater treatment (Crini, 2005).

The presence of PAHs in all these elements of the environment may create a risk not only to humans but to all living organisms. In our previous study we have studied effects of different water seasons on the residual characteristics and ecological risk of polycyclic hydrocarbons in sediments from changdang Lake, China (Hussain, J *et al*., 2016). The aim of this study was to investigate the seasonal variation of 14 PAHs, their eco-toxicological risk characterics and as well their relation with the organic matter and sampling sites in Chnagdang Lake China.

Materials and methods

Study area Description

Changdang Lake is located nine kilometers away from the south of Jintan City, China. Jintan has a maritime monsoon climate, a mild and humid climate with distinct seasons. The annual average temperature is around 17°C. Jintan is crisscrossed by numerous rivers, canals, and lakes and the water surface area occupies one-fifth of its size of the land. The natural resources mainly include salt, coal, and granite mines. Changdang. It is one of the ten largest freshwater lakes in Jiangsu province covers 8,200 hectares (32 sq mi). It has always been an important source of drinking water supply in Jintan City. Maximum depth of lake is 1.31m with average depth of 1.10m and its storage capacity is about 0.98 × 108m3. Changdang Lake receives water from the west Xuebu River, Sudou River and flows into Taihu Lake through Huangli River, Beigan River, and Zhonggan River, which lie to the east of the lake (Hussain *et al.*, 2016).

Sampling sites 1#, 2# and 3#were selected from the Xinkai River to the center of the lake, site 4# is the main outlet, sites 5# and 6# are the inlet to the Changdang lake, site 7# Qianzi Dang is the inlet to the water treatment plant, site 8# is selected after the water treatment by activated carbon process and site 9# is selected after the water treatment by ultra-filtraon membrane technology installed in the water treatment plant as shown in Fig. 1.

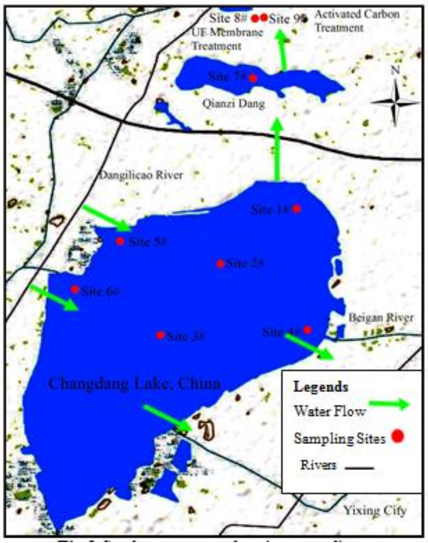

Fig. 1. Study area map showing sampling.

Sample collection and Pre-treatment

The samples of water were collected into 1L glass bottle covered by PTFE cap, screwed the caps and ensured no bubbles in the bottle. All samples were kept them in a box, transported to laboratory and stored them in refrigerator under 4°C.

Extraction and purification of samples

Water samples were filtrated through glass fiber membrane of 0.45μm, and extracted with Solid-Phase Extraction (SPE) column (1.0g of C18 in the bottom and 0.5g of copper powder in the up layer). The SPE columns were previously rinsed by 15mL of petroleum ether/acetone/methanol (v/v/v: 1/1/1) and 15mL of high performance liquid chromatography (HPLC) water. Then 1L water sample passed through the SPE columns, and control the flow velocity under 5mL/ min. After completed enrichment, using 5mL HPLC water to wash it, and then pumped for 30 minutes to evict redundant moisture. The PAHs in the SPE column was eluted with 10mL of acetone / petroleum ether (v/v=1/2), followed by 12mL of dichloromethane / n-hexane (v/v =3/2). Collected the eluent into 50mL pear-shaped bottle, concentrated it to about 1mL under 40°C by using rotary evaporator and N2 blowing, and adjusted to 1mL with n-hexane.

Experiments/Analysis Method

Polycyclic aromatic hydrocarbons in water samples were analyzed according to the USEPA Standard Test method, Filtration through a glass fiber filter, SPE method for preconcentration, Agilent1100 high performance liquid chromatography - fluorescence detection method.

Chromatographic Conditions

According to the US EPA (1979), 14 PAHs were analyzed by Agilent1100 HPLC with fluorescence and UV - adsorption detection. A 250mm × 4.6mm × 5μm reversed phase C18 column (Agilent ZORBAX Eclipse XDB-C18) served as the stationary phase. Mixed solution of acetonitrile and ultrapure water were delivered as the mobile phase in a gradient program at 0.75mL/min. The initial volume ratio of acetonitrile and water was 60: 40, and then the ratio of organic phase increased to 100:0 till 60 minutes. PAHs were quantified by using external standard solutions obtained from Ehrenstorfer (Augsburg, Germany). Further analytical detail can see (Thiele and Brűmmer, 1998). Data were statistically analyzed using the Kruskal-Wallis test at $P<0.05$.

Quality Assurance/Quality Control

For every sample, a method blank (solvent), a spiked blank (standards spiked into solvent), a matrix spike (standards spiked into pre-extracted soil and water), a sample duplicate was processed by performing the entire pretreatment procedure.

The method blanks contained no detectable amounts of the target analysts except for naphthalene. The average recoveries in matrix spikes for 14 PAHs ranged from 82% to 114% for water samples (14 components). The relative standard deviation of the repeatability was below 20% for water samples. Limits of detection ranged from 0.30 to 0.60ng/g for water samples depending on individual PAH. Results presented in this study were not blank and recovery corrected. Six-site calibration curves were constructed for the quantification, with good to excellent linearity ($r^2 > 0.99$).

Ecological risk assessment

The potential eco-toxicological risk of PAHs was assessed by the risk quotient (RQ) (Binelli. A, 2008), which was calculated as follows: RQ = CPAHs/CQV (CPAHs represents the concentration of certain PAHs in the medium and CQV represents the corresponding quality values of certain PAHs in the medium). The negligible concentrations (NCs) and the maximum permissible concentrations (MPCs) of individual PAHs in water were used as the quality values in the medium (Cao. Z, 2010; Kalf. DF, 1997).

RQNCs and RQMPCs were calculated as follows:

$$\mathrm{RQNCs} = \mathrm{CPAHs}/\ \mathrm{CQV\ (NCs)}$$

$$\mathrm{RQMPCs} = \mathrm{CPAHs}/\mathrm{CQV\ (MPCs)}$$

CQV (NCs) represents the quality values of the NCs of PAHs and CQV (MPCs) represents the quality values of the MPCs of PAHs in the medium).

In principle, RQNCs <1.0 indicates risk-free and RQMPCs = 1.0 indicates high-risk, while RQNCs = 1.0 and RQMPCs < 1.0 indicates moderate risk (Cao *et al.*, 2010).

Results and discussion

Concentration of PAHs in water

The monitoring results showed that the concentration of PAHs in March averaged 100.08ng/L (35.31~200.38ng/L, in June 154.72ng/L (100.80~218.74ng/L), and in September 93.77ng/L (66.75~106.41ng/L)

The ∑PAHs did not exceed the requirements of "drinking water health standards" (GB5749~2006) (PAHs <2000ng/L), Benzo[a]pyrene in some site exceed the requirements of "Surface Water Environmental Quality Standard" (GB3838-2002) (benzo[a]pyrene <2.8ng/L).

Despite total residue of PAHs is low in overlying water of Changdang Lake in March, but were mainly three-ring and five-ring PAHs were observed with relatively strong toxicity.

There exists a small amount of 5-ring PAHs at the site 7#, higher levels of polycyclic aromatic residues at the 8# and 9# sites, but the remaining species were mainly low toxicity of two-ring of naphthalene and three-rings Acenaphthene, and contribute to develop limits Benzo[a]pyrene is also not detected. Therefore, after the treatment with the combination of activated carbon and membrane technology, water quality was relatively safe.

In June, PAHs residual volume of the sites except site 2# was more than that in March, but its main residual toxic species were relatively of weak two-rings and three-rings and some four-rings PAHs were dominated and did not exceed. After water treatment, four-ring PAHs were significantly reduced, the residual category was mainly two-rings of naphthalene and three-ring PAHs with low toxicity.

The residual PAHs of each site in September were lower than that in June, and its composition was similar to the residual PAHs in June, toxicity of relatively weak, two-rings and three-rings and part of four-ring polycyclic aromatic hydrocarbons were not exceeded. Qingfei Zeng *et al.* investigated the distribution of PAHs in Gucheng Lake china, which showed the PAHs level in summer 184-365 ng L^{-1} and in winter 410-1160 ng L^{-1} (Zeng, Jeppesen, Gu, Mao, & Chen, 2018). Beibei Shen *et al* found the level of PAHs 368.7ng/L in water from Bosten, Lake Northwest China (Beibei Shen, Jinglu Wu, & Zhao, 2017), while Xiaoyan Yun *et al.* found the level of PAHs 10.2 to 525.1 ng L^{-1} in water from east lake, China (Xiaoyan Yun, Yuyi Yang, Minxia Liu, Miaomiao Zhang, & Wang, 2016). These studies results indicate that the present level of PAHs in water from Changdang Lake is lower the Guchneg and Bosten Lake China. The concentration of PAHs in water are shown in different months are shown in Fig. 2, Fig. 3 and Fig. 4.

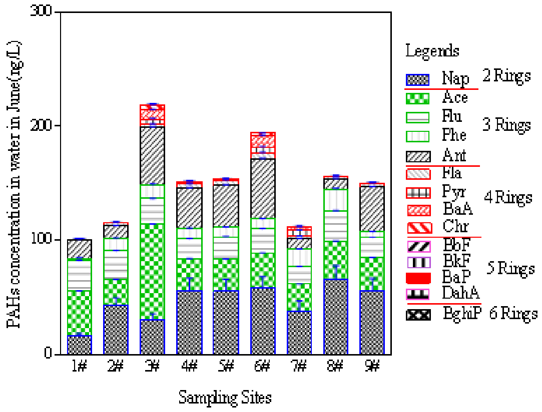

Fig. 2. PAHs concentration in water in June.

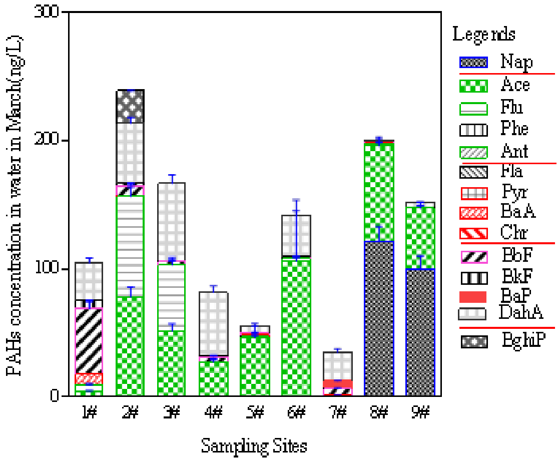

Fig. 3. PAHs concentration in water in March.

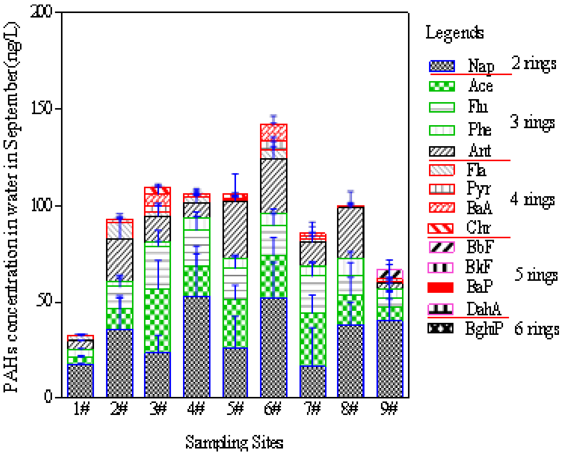

Fig. 4. PAHs concentration in water in September.

The relative percentage distribution of individual PAHs in water

Percentage contribution of individual PAHs (n=14) congeners (3-6 rings) during the study period are shown in Fig. 5. Clear dominance of low molecular weight PAHs (Ace 31%, Nap 25%, Flu 11%, Ant 14%, Phe 5% in respect to low (Pyr 1%. Chy 0%, BaA 1%, Fla 2%), and higher molecular weight (BbF 2%, BkF 1%, Bap 0%, DahA 6%, Bghip 1%) were observed. The most abundant compound of the 3-rings group is acenaphthylene and napthaene. An average, it made up to 31% and 25% of the relative individual composition in the water from the Changdang Lake as shown in Fig. 5 .

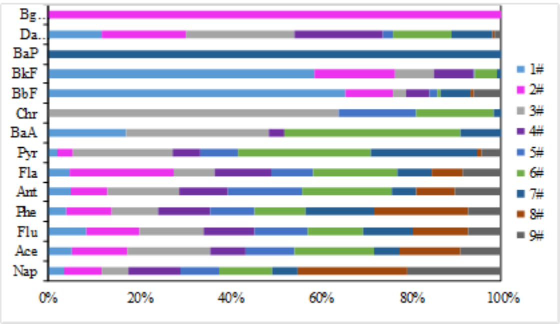

Fig. 5. The total average distribution of PAHs in water from Chnagdang Lake

Relationship between PAHs, sampling sites and organic matter

The Fig. 6 show the RDA ordination results of the sampling sites, PAHs and molecular weight of organic matter in water.

They could be divided into three groups (Ellipse lines in Fig. 6 and the components included in groups I, II, and III.

The predominant molecular weight composition and the dominant residual PAHs at the different sampling sites were determined from the length of the arrows in Fig. 6. We could find that the fractions of 10-100kDa, >100kDa and <1kDa of organic maters were the predominate components in water.

The correlation between the fraction of 10-100kDa in organic matter and the residues of high rings PAHs (BbF, BkF, DahA, Bghip) and middle rings PAHs (Ace and Chr) had positive correlation, it implied that the high- and middle -rings PAHs were mainly associated to the fraction of 10-100kDa of organic maters. Low-rings PAHs were mainly associated to the fractions of 3-10kDa and <1kDa of organic maters. The fractions of 1-3kDa and >100kDa were mainly enriched the BaA. The molecular composition of organic maters in individual sampling sites also showed obvious variance. Group I was mainly associated to the sites of 3# and 4# and the 10-100kDa fractions of organic maters. Group II was mainly associated to the sites of 2#, 5#, 6# and the fractions of 3-10kDa and <1kDa in organic maters. And Group III was mainly associated to the site 1# and the >100kDa fraction of organic maters.

The intrinsic affinity of PAHs for organic matter components can result in distribution patterns (Zhou. J, 1999). In the past, the quantity of organic matter was generally considered to be associated with the partitioning of PAH compounds between sediment and water in an aquatic environment (Evans. K, 1990). However, recent studies have shown that the quality of organic matter present is more significant (Nichols. E., 2008; Oen, 2006). Cornelissen *et al.* (2005) found the presence of black carbon altered the expected partitioning for PAH compounds between sediments and water in contaminated harbor Sediments (Cornelissen. G., 2005). The relationship between PAH compounds and organic matter constituents can be analyzed by correlation/regression coefficients based on occurrences. Tsapakis *et al.* (2003) found that occurrences of PAHs and TOC contents averaged a 0.98 R^2 value, showing almost a linear relationship (Tsapakis, 2003).

In contrast, however, Oen *et al.* (2006) (Oen, 2006) found little correlation (0.15 R^2 value) with contaminated harbor sediments, which was attributed to the majority of compounds being related to black carbon. Redundancy analysis of the relationship between PAHs, molecular weights and sampling sites are shown in Fig. 6.

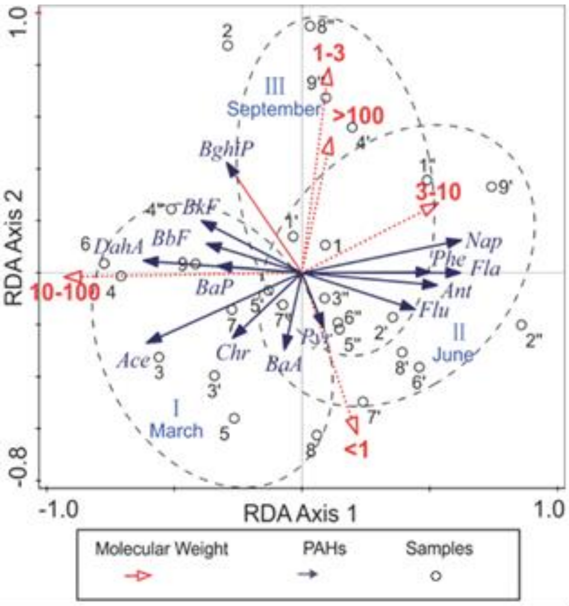

Fig. 6. Redundancy Analysis showing the Relationship between PAHs, sampling sites and molecular weights of organic matter.

Eigenvalues for RDA axis and molecular weights of organic matters correlations are shown in **Error! Reference source not found.**, while Correlation Coefficient of the RDA Ordination Axis of PAHs and molecular weights of organic matter are presented in Table 2.

Table 1. Eigenvalues for RDA axis and molecular weights of organic matters correlations.

Statistic	Axis 1	Axis 2	Axis 3	Axis 4
Eigenvalues	0.2797	0.0244	0.4913	0.0671
Explained variation (cumulative)	27.97	30.41	79.53	86.24
Pseudo-canonical correlation	0.6199	0.5982	0	0
Explained fitted	84.11	91.44	-	-

variation (cumulative)

Table 2. Correlation Coefficient of the RDA Ordination Axis of PAHs and molecular weights of organic matter.

Compounds	Resp.1	Resp.2	Resp.3	Resp.4
<1	0.1334	-0.3715	-0.0194	-0.1973
1-3	0.0654	0.4716	-0.0059	-0.1526
3-10	0.3305	0.1601	-0.0042	-0.0897
10-100	-0.5768	-0.01	0.0123	0.1512
>100	0.0704	0.3119	0.0254	0.2981
Nap	0.6248	0.125	**-0.7403**	0.0175
Ace	-0.608	-0.2702	0.2124	**-0.6062**
Flu	0.4427	-0.1412	-0.7872	-0.2261
Phe	0.5041	0.0029	**-0.7298**	-0.0845
Ant	0.5295	-0.0491	**-0.7589**	0.0729
Fla	0.6211	0	-0.3974	0.0634
Pyr	0.0852	-0.2117	-0.1771	0.3751
BaA	-0.0689	-0.2966	0.0276	**0.8337**
Chr	-0.2717	-0.2521	0.1341	0.1055
BbF	-0.374	0.1119	**0.853**	0.2002
BkF	-0.3938	0.2009	**0.8005**	0.1654
BaP	-0.3323	0.0266	0.5607	-0.3284
DahA	-0.621	0.0461	**0.7512**	-0.054
BghiP	-0.2946	0.4241	0.5696	-0.2878

Potential eco-toxicological risk of PAHs

In this study the potential eco-toxicological risk of PAHs were assessed by the risk quotient (RQ). Risk quotients (RQ) are commonly used to assess potential ecological risk of chemicals on aquatic biota (Sun, 2009; Wang. Y, 2014). The mean values of RQNCs and RQMPCs are listed in Table 4. It can be seen that the RQNCs values of Ace, BaA, BbF, BaP, and BkF were more than 1.0 and the RQMPCs values of them were less than 1.0 in March. The RQNCs values of Nap, Ace, Flu, Phe, Ant, BaA were more than 1.0 and the RQMPCs values of them were less than 1.0 in June, the RQNCs values of Nap, Ace, Flu, Phe, Ant, BaA, BbF were more than 1.0 and the RQMPCs values of them were less than 1.0 in September.

According to the risk classification of individual PAHs, the eco-toxicological risks of Ace, BaA, BbF, BaP, and BkF in March, the Nap, Ace, Flu, Phe, Ant, and BaA in June and Nap, Ace, Flu, Phe, Ant, BaA, BbF in September had reached moderate level. Therefore, measures should be put into place to prevent and control the damage of PAHs to aquatic organisms and human health. Risk classification of individual PAHs and ΣPAHs are presented in **Error! Reference source not found.**.

Table 3. Risk classification of individual PAHs and ΣPAHs.

Individual PAHs		ΣPAHs	
RQ(NCs)	RQ (MPCs)	RQ (NCs)	RQ (MPCs)
Risk-free	<1 Risk-free	0	
	Low-risk ≥1;	≥1; <800	0
Moderate-risk ≥1	<1 Moderate-risk 1	≥800	0
	Moderate-risk 1	<800	≥1
High-risk ≥1	High-risk	≥800	≥1

Table 4. Mean values of RQ (NCs) and RQ (MPCs) of PAHs in the water from Changdang Lake.

		Water (ng/L)		Mean concentration (ng/L)			March		June		September	
PAHs(rings)	TEFs	NCs	MPCs	March	June	September	$RQ_{(NCs)}$	$RQ_{(MPCs)}$	$RQ_{(NCs)}$	$RQ_{(MPCs)}$	$RQ_{(NCs)}$	$RQ_{(MPCs)}$
Nap	0.001	12	1200	0.000	46.510	33.756	0.000	0.000	**3.876**	0.039	**2.813**	0.028
Ace	0.001	0.7	70	54.177	35.157	17.954	**77.395**	0.774	**50.224**	0.502	**25.649**	0.256
Flu	0.001	0.7	70	0.646	21.479	13.242	0.922	0.009	**30.684**	0.307	**18.917**	0.189
Phe	0.001	3	300	0.183	10.146	4.956	0.061	0.001	**3.382**	0.034	**1.652**	0.017
Ant	0.001	0.7	70	0.000	28.973	16.428	0.000	0.000	**41.390**	0.414	**23.468**	0.235
Fla	0.01	3	300	0.000	2.871	2.727	0.000	0.000	0.957	0.010	0.909	0.009
Pyr	0.001	0.7	70	0.288	1.970	1.514	0.411	0.004	**2.814**	0.028	**2.163**	0.022

BaA	0.1	0.1	10	1.338	1.393	1.954	**13.378**	0.134	**13.933**	0.139	**19.544**	0.195
Chr	0.01	3.4	340	0.147	0.689	0.550	0.043	0.000	0.203	0.002	0.162	0.002
BbF	0.1	0.1	10	7.758	0.000	0.940	**77.578**	0.776	0.000	0.000	9.400	0.094
BkF	0.1	0.4	40	6.492	0.000	0.000	**16.231**	0.162	0.000	0.000	0.000	0.000
BaP	1	0.5	50	0.656	0.000	0.000	**1.311**	0.013	0.000	0.000	0.000	0.000
DahA	0.5	0.5	50	27.799	0.000	0.000	**55.598**	0.556	0.000	0.000	0.000	0.000
BghiP	0.01	0.3	30	2.788	0.000	0.000	**9.293**	0.093	0.000	0.000	0.000	0.000
Total							252.220	2.429	147.463	1.475	104.678	1.019

Conclusion

In this study the average concentration of polycyclic aromatic hydrocarbons in water from Changdang Lake averaged 100.08ng/L in March, 154.72ng/L in June, and 93.77ng/L in September. By applying the redundancy analysis, results showed higher correlation between the fraction of 10-100kDa in organic matter and the residues of higher rings PAHs (BbF, BkF, DahA, Bghip) and middle - rings PAHs (Ace and Chr. Redundancy analysis, results showed higher correlation between the fraction of 10-100kDa organic matter and the residues of higher rings PAHs (BbF, BkF, DahA, Bghip) and middle-rings PAHs (Ace and Chr). According to the risk classification of individual PAHs in water, the eco-toxicological risks of Ace, BaA, BbF, BaP and BkF in March, the Nap, Ace, Flu, Phe, Ant and BaA in June and Nap, Ace, Flu, Phe, Ant, BaA and BbF in September had reached moderate risk level.

The concentrations of polycyclic aromatic hydrocarbons got effective treatment after the combine treatment processes of activated carbon and ultra-filtration membrane technology installed in the water treatment Plant. Results obtained from this study meets the requirements of "drinking water health standards” (GB5749-2006) (PAHs <2000ng/L), However continuous monitoring is required to further improve water quality and necessary actions should be taken to ensure the availability of safe water to the human and other living organism.

Acknowledgements

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