Research Article

Polycyclic Aromatic Hydrocarbons in Surface Water from Wuhai and Lingwu Sections of the Yellow River: Concentrations, Sources, and Ecological Risk

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In this study, concentrations, sources, and ecological risk of 16 polycyclic aromatic hydrocarbons (PAHs) in 41 surface water samples collected from Wuhai and Lingwu sections of the Yellow River were investigated. The results showed that total PAH concentrations varied from 27.5 ng/L to 234 ng/L and from 135 ng/L to 265 ng/L in surface water of Wuhai and Lingwu sections, respectively. Source identification was performed by using principal component and multiple linear regression analysis. PAHs in Wuhai section of the Yellow River were mainly from coal combustion (35%) and vehicle exhausts (34%). Ecological risk of PAHs to aquatic organisms was assessed by applying the probabilistic risk assessment method. The results showed that the probabilities of exceeding the chronic toxicity for 5% of the species were 28% and 32% for PAHs in surface water from Wuhai and Lingwu sections, respectively. It has been demonstrated that there is an urgent need for environmental managers to take measures to reduce the ecological risk of PAHs in the aquatic environment in both Wuhai and Lingwu sections of the Yellow River.

1. Introduction

Polycyclic aromatic hydrocarbons (PAHs) are compounds consisting of two or more fused rings that have raised more concerns due to their persistent property and toxicity featured by considerably high direct-acting mutagenicity and carcinogenicity [1]. PAHs find their way into the environment through natural sources such as volcanoes, forest fire, and biosynthetic process. It could also be emitted from diverse anthropogenic sources such as vehicle exhausts; power plants; chemical, coke, and oil-shale industries; and urban sewage [2, 3].

It has been demonstrated that, in the process of coal mining, the treatment and storage of coal provided the basic conditions for the release of PAHs into the environment [4–9]. For example, PAHs in coal and coal waste could be leached into the mine water [10] and then enter into the aquatic environment with the discharge of mine water [11]. Therefore, the occurrence of PAHs in surface water nearby

the coal mining area has attracted lots of attention recently [5, 9, 12, 13]. However, studies on concentrations, sources, and ecological risk of PAHs in surface water of rivers located in or nearby the coal mining area are generally limited at present. Seopela et al. [9] investigated the occurrence levels and evaluated the risk of PAHs in the Loskop Dam nearby the coalmine in South Africa. They reported that the total PAH concentrations varied from 1170 ng/L to 14500 ng/L, and it was found that the occurrence of PAHs in the sediment of Loskop Dam could lead to a delay of the development and malformation of zebrafish embryo [9]. Hao et al. [12] investigated the concentrations and sources of PAHs in surface water of Fuyang River nearby the Fengfeng coal mining area. Huang et al. [5] reported that the average concentration of 16 PAHs was 427 ng/L in surface water of rivers located in Heshan coal district in Guangxi.

In the present study, we aimed to investigate the concentrations, sources, and ecological risk of PAHs in surface water of Wuhai and Lingwu sections of the Yellow River in

China. Both Wuhai and Lingwu are large coal mining bases in the northwestern China and have a long history of coal mining. Over the years, the coal mining areas in both cities have dramatically expanded. A large number of cultivated land, forest, and grassland have been occupied due to mining activities, resulting in the sharp deterioration of the local ecological environment. The Yellow River is the only surface water resource flowing through Lingwu and Wuhai. During recent years, the water resource of the Yellow River is becoming more and more stressing due to the intensive mining activities, which could also aggravate the degradation of the vulnerable ecosystem.

Therefore, we carried out a field campaign to (i) determine concentrations of PAHs in surface water collected from Wuhai and Lingwu sections of the Yellow River, (ii) identify major sources and origins of PAHs, and (iii) assess the ecological risk of PAHs to aquatic organisms by applying the probabilistic ecological risk assessment. This study would be helpful to the control of PAH risk in rivers located in or nearby coal mining areas.

2. Materials and Methods

2.1. Chemicals. The PAH mixture standard was purchased from Accustandard (USA), including naphthalene (Nap), acenaphthylene (Acy), acenaphthene (Ace), fluorene (Flu), phenanthrene (Phe), anthracene (Ant), fluoranthene (Flua), pyrene (Pyr), benzo(a)anthracene (BaA), chrysene (Chr), benzo(b)fluoranthene (BbF), benzo(k)fluoranthene (BkF), dibenzo(a, h)anthracene (DBA), benzo(a)pyrene (BaP), indeno(1,2,3-cd)pyrene (Ind), and benzo(g, h, i) perylene (BghiP). The injection standard (hexamethylbenzene, HMB) was provided by Dr. Ehrenstorfer (Germany). Phe-d10 and Chr-d12 were used as recovery standards and obtained from Accustandard (USA). All solvents, including methanol, dichloromethane, and *n*-hexane, were of HPLC grade and obtained from Fisher (USA). C18 cartridges (500 mg/6 cc) used for solid phase extraction (SPE) were purchased from Waters (USA).

2.2. Sample Collection and Analytical Procedure. A total of 30 sampling sites at Wuhai section were set up along the mainstream of the Yellow River, while 11 sampling sites were selected at Lingwu section (Figure 1). At each sampling site, approximately four liters of surface water were collected and transported to the lab immediately. All water samples were stored at 4°C and analyzed in 24 h. The sampling events were conducted in July, 2019.

The sample pretreatment method for surface water was adapted from Bu et al. [14]. In brief, approximately 2 L surface water samples (in triplicate) were filtered using a glass fiber filter (0.45 μm, Millipore, USA) prior to SPE. The sample was enriched by SPE (Supelco, USA) with the C18 cartridge that had been preconditioned with 10 mL of dichloromethane, 10 mL of methanol, and then 10 mL of ultrapure water. After enrichment, the cartridge was eluted with 10 mL of dichloromethane. The eluents were collected in a K-D concentrator and concentrated to approximately

0.5 mL under a gentle nitrogen stream. Thereafter, 20 μL of injection standards (10 μg/mL HMB) were added, and the final volume was made up to 1 mL in dichloromethane for instrumental analysis.

2.3. Instrument Analysis. Samples were analyzed by using a GC2010 gas chromatograph coupled with a QP2010 mass spectrometer (GC-MS, Shimadzu, Japan). Aliquots of sample extracts (1 μL) were introduced by the splitless injection at 280°C. Chromatographic separation of PAHs was achieved on an Rtx-5MS capillary column (length 30.0 m, i.d. 0.25 mm, film 0.25 μm, Shimadzu, Japan) over 57 min using a triple ramp oven programme (initial temperature 60°C; 20°C/min to 160°C; 3°C/min to 280°C, held for 6 min; 20°C/min to 300°C, held for 5 min). A constant flow (1 mL/min) of ultrapure helium carrier gas (99.999%) was maintained. Mass spectra were collected over the range m/z 50 to 500. The temperatures of the transfer line and ion source were 220°C and 250°C. Ionization of analyte was performed using the electron ionization at 70 eV. A full scan of the mixture standard containing each compound (1 μg/mL) allowed the identification of the compound peaks according to their retention times and mass spectra. Retention time locking with an internal standard was applied to ensure the reproducibility of retention time of each target compound. This was followed by data acquisition in the selective ion monitoring mode that entailed the identification of the most intense and distinct fragment ions for each compound, within a well-defined time window.

2.4. Quality Control and Assurance. Matrices-spiked control experiments were used to evaluate the method performance in parallel with each batch of samples. In brief, two liters of the contaminant-free ultrapure water were spiked with 10 μL of the PAH mixture standards (each PAH congener at 10 μg/mL), and the recovery rates of the 16 PAHs were 47%–116% ($n = 5$). Procedure blanks were run with each batch of samples to evaluate possible contamination during analysis, and levels of PAHs were below the limit of quantification (LOQ) in all cases. The LOQs of PAHs were estimated as the concentration when the signal-to-noise ratio was 10:1 (S/N = 10). The method detection limit (MDL) was 0.049–5.57 ng/L, assuming a final extract volume of 1.0 mL and surface water samples of 2 L. Recovery rates were also evaluated by adding the PAH mixture standard (each PAH congener at 10 μg/mL) into 2 L real surface water samples ($n = 6$) collected from Qinghe River in Beijing. The results showed that recovery rates of PAHs were 50%–104%. The average recoveries of Phe-d10 and Chr-d12 were 114% ± 4.5% and 71% ± 3.8%, respectively.

2.5. Probabilistic Ecological Risk Assessment for PAH Mixture. In this study, the probabilistic ecological risk assessment method was used to assess the hazard of PAHs to aquatic organisms in the Yellow River. Specifically, a joint probability curve (JPC) was generated through combining exposure and toxicity data [15]. The specific point on the JPC
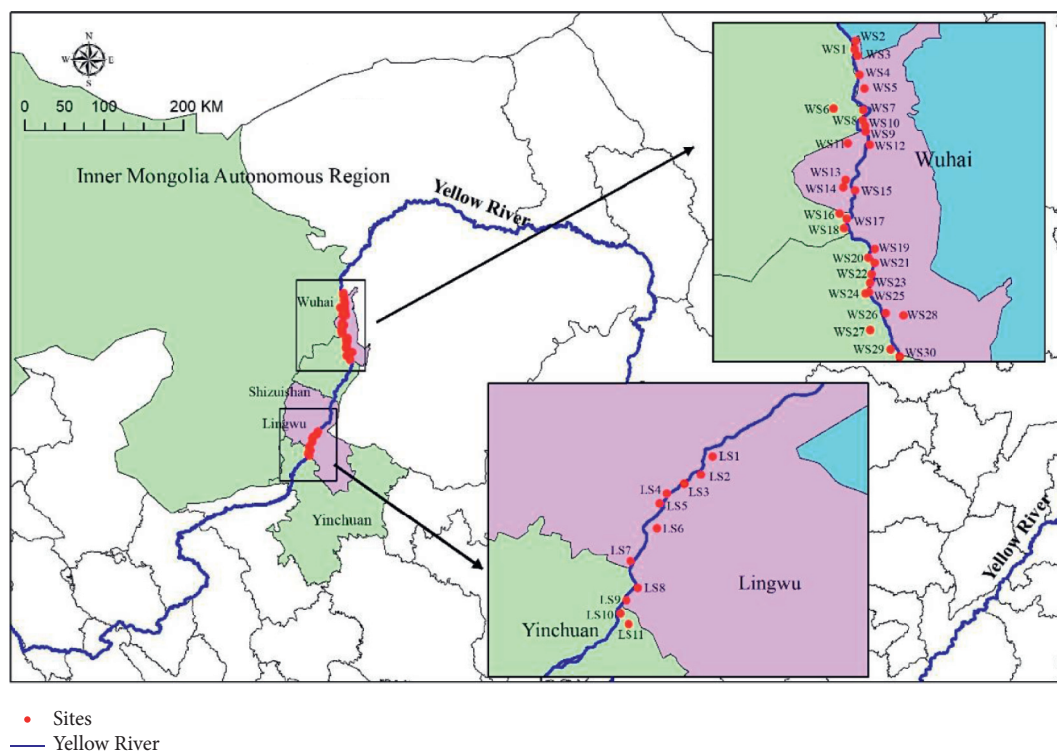
FIGURE 1: Schematic diagram showing the locations of the sampling sites in the Yellow River.

represents both the probability that the chosen fraction of species would be in danger, and the frequency with which the level of effect would be exceeded.

To reflect the additive effect of the 16 PAHs, BaP equivalency (BaPeq) of PAHs was calculated based on the toxic equivalency factors (TEFs) for PAHs [16]. Then, the ecological risk of PAH mixture was evaluated. Ecotoxicology data for BaP were collected from the United States Environmental Protection Agency ECOTOXicology Database (http://www.epa.gov/ecotox/). All toxicity data were the no observed effect concentration (*NOEC*) obtained from tests conducted in a freshwater system and the data selection procedure followed principles of accuracy, relevance, and reliability [17].

3. Results and Discussion

3.1. Concentrations and Distribution. Concentrations of the 16 PAHs in surface water from Wuhai and Lingwu sections of the Yellow River are presented in Figure 2. Total concentrations of PAHs ranged from 27.5 ng/L (WS16) to 233 ng/L (WS19) in the Wuhai section of the Yellow River with a mean concentration of 162 ng/L, while PAH concentrations range from 135 ng/L (LS10) to 265 ng/L (LS6) in the Lingwu section with a mean concentration of 197 ng/L. The concentration of Nap was the highest among different congeners, accounting for 12% and 14% of the total PAH concentrations on average for Wuhai and Lingwu sections, respectively. Average concentrations of most congeners in surface water from Lingwu section were slightly higher than that from Wuhai section, except for Phe, BaA, BkF, DBA,
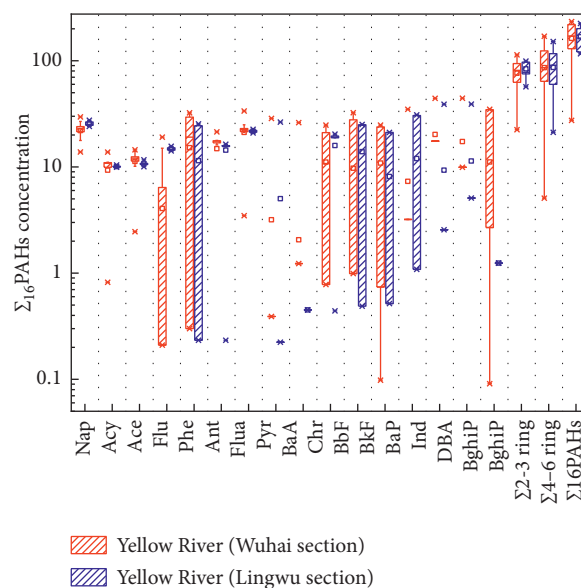

FIGURE 2: Concentrations of PAHs in surface water from Wuhai and Lingwu sections of the Yellow River.

and BghiP. In general, the compositional pattern of PAHs was in the following order for both studied areas: 3 rings > 4 rings > 5 rings > 2 rings > 6 rings (Figure 2).

We collected the available data on PAH concentrations in surface water surrounding coal mining areas from previously published literature. Through the comparison among different studies, it can be found that PAH concentrations in the present study were significantly lower than

those in the Loskop Dam (South Africa, 1170–14500 ng/L) [9], surface water of rivers in the Shilong coalmine (China, 68–8377 ng/L) [13], and rivers in the Heshan coal district (China, 199–1351 ng/L) [5]. However, PAH concentrations in surface water of rivers in the Fengfeng coalmines (China, 1.35–2.92 ng/L) [12] were significantly lower than that in our study.

Table 1 shows a comparison of PAH concentrations in surface water samples collected from rivers around China during the past decade. When compared to other studies conducted in the Yellow River watershed, our results were in the same order of magnitude to that reported for PAHs in surface water collected from Henan reach [18] and Xi'an section [19] of the Yellow River but were significantly lower than that from the Yellow River Estuary [20]. A comparison was also made to studies conducted in regions other than the Yellow River watershed (Table 1). It can be found that the mean values of the total concentration of PAHs observed in the present study were comparable to that from the Songhua River [23], the Yangtze River Delta [26], the Poyang Lake [28], Xiamen Coastal Area [30], and the Chaohu Lake [32], slightly lower than that from the Daliao River Estuary [22], the Songhua River basin [21], and the Jiangsu section of the Yangtze River [25], but significantly lower than that from the Yangtze Estuary and coastal areas [29], the Liaohe River basin [21], the Taihu Lake [27], the Huaihe River basin [21], and the Weishan Lake [31]. Moreover, our results were significantly higher than that from the Baiyangdian Lake [24], the Fujiang River [23], the Pear River [33], the Dongjiang River [33], and the Pearl River Estuary [33, 34]. Overall, PAH concentrations in surface water collected from Wuhai and Lingwu sections of the Yellow River were at moderate levels in China.

3.2. Source Apportionment

3.2.1. Sources of PAHs in Wuhai Section of the Yellow River.
Principal component analysis (PCA) was conducted to evaluate the contribution of different PAH sources, and the results are presented in Table 2, which shows the variable loadings describing the major contamination patterns and their explained variance. Note that Pyr, BaA, BaP, and Ind were not considered because of their low detection rates. Five principal components accounting for 81% of the total variance were extracted through PCA.

The first component (PC1) explained 38% of the total variance and was predominately loaded with Flua, Ace, Nap, Acy, and Ant. It has been reported that Flua and Ant were related to coal combustion-generated PAHs [2, 35–37]. Loadings of Acy and Nap have been associated with vaporization or spill of petroleum-related products [38]. Ace was one of the dominant PAHs from the coke industry and could enter into the Yellow River via industrial wastewater and atmospheric fallout [39]. Therefore, PC1 explained the compounds originating from coal combustion, spill of petroleum-related products, and the coke industry. The second component (PC2) contributed 15% to the total variance and was heavily weighted with Flu and DBA. Flu was another one

Table 1: Summary of concentrations (ng/L) of PAHs in surface water samples collected from rivers in China during the past decade.

Locations	n	Min	Max	Mean	References
Yellow River (Wuhai section)	30	27.5	234	162	This study
Yellow River (Lingwu section)	11	135	265	197	This study
Yellow River (Henan reach)	26	144	2361	662	[18]
Yellow River (Xi'an region)	6	357	2017	824	[19]
Yellow River Estuary (flood season)	8	474	1190	729	[20]
Yellow River Estuary (dry season)	8	1682	6014	2944	[20]
Liaohe River Basin	15	209	33930	4021	[21]
Daliao River Estuary	12	139	1718	486	[22]
Songhua River	7	88.6	140	—	[23]
Songhua River Basin	15	7.12	9131	759	[21]
Baiyangdian Lake	6	40.1	74	51	[24]
Fujiang River	8	43.9	81	—	[23]
Yangtze River (Jiangsu section)	15	12	3576	925	[25]
Yangtze River Delta	14	12.9	638	201	[26]
Taihu Lake	5	11000	34000	—	[27]
Poyang Lake	40	5.56	266	—	[28]
Yangtze Estuary and coastal areas	18	478	6273	1858	[29]
Xiamen coastal area	13	62	342	151	[30]
Huaihe River Basin	—	1700	7770	4386	[21]
Weishan Lake	9	5350	12970	8572	[31]
Chaohu Lake	15	95.6	370	171	[32]
Dongjiang River	8	10.9	42.3	24.9	[33]
Pearl River	8	15.4	48.1	27.1	[33]
Pearl River Estuary	20	12.9	182	45.4	[34]
Pearl River Estuary	3	11.7	18.6	15.1	[33]

n, numbers of collected samples.

Table 2: Rotated component matrix of studied PAHs in the Yellow River (Wuhai section).

PAHs	Components				
	1	2	3	4	5
Nap	**0.912**	0.195	0.014	0.158	0.086
Acy	**0.884**	−0.156	0.093	−0.168	−0.032
Ace	**0.921**	0.038	0.184	0.198	0.148
Flu	−0.055	**0.861**	−0.004	−0.073	0.042
Phe	0.154	−0.336	**0.589**	0.158	**0.41**
Ant	**0.623**	−0.038	0.5	−0.057	**0.408**
Flua	**0.907**	0.105	0.187	0.122	0.225
Chr	0.173	0.086	**0.855**	0.128	−0.21
BbF	0.248	0.059	0.296	**0.729**	−0.155
BkF	0.229	−0.047	−0.077	0.002	**0.9**
DBA	0.154	**0.781**	−0.025	0.004	−0.12
BghiP	−0.044	−0.128	−0.022	**0.885**	0.131
Eigenvalues	4.587	1.777	1.477	1.033	0.866
% variance	38.226	14.809	12.309	8.606	7.216
Cum. percentage	38.226	53.035	65.344	73.95	81.166

dominant PAHs from the coking process [39], while a relatively higher abundance of DBA has been identified as a marker of gasoline vehicle emissions. Hence, PC2 was

reflective of PAHs from the coking process and vehicle exhausts. The third component (PC3) characterized for 12% of the total variance. This component was heavily loaded with Chr and Phe. Chr and Phe had been identified as typical tracers of coal combustion [36]. Hence, PC3 was reflective of PAHs derived from coal combustion. The fourth component (PC4) contributed 8.6% to the total variance and was dominated by BghiP and BbF. Dominance of BghiP and BbF over other PAHs has been attributed to diesel combustion [40, 41]. Thus, PC4 was selected to represent diesel combustion source of PAHs in surface water of the Yellow River. The fifth component (PC5) was responsible for 7.2% of the total variance and weighted by BkF, Ant, and Phe. BkF has been identified as an indicator of diesel combustion [40, 41], while it has been reported that Ant and Phe were related to coal combustion [2, 36, 42]. Hence, PC5 was reflective of PAHs from coal and diesel combustion.

Using the multivariate linear regression (MLR) from PCA factor scores, the equation was calculated as follows:

$$\sum_{16} \text{PAHs} = 0.901 \times \text{PC1} + 0.408 \times \text{PC2} + 0.454 \times \text{PC3}$$
$$+ 0.457 \times \text{PC4} + 0.529 \times \text{PC5} \left(R^2 = 0.6 \right). \tag{1}$$

Therefore, PAHs in Wuhai section of the Yellow River were originated from four major sources, and the quantitative contributions were 35% from coal combustion, 34% from vehicle exhausts, 21% from coke industry, and 11% from spill of petroleum-related products.

3.2.2. Sources of PAHs in Lingwu Section of the Yellow River. Note that BaA, BghiP, Pyr, Ind, and DBA were not considered in the PCA because of their low detection rates. According to the results presented in Table 3, PAHs were separated into four major categories. Over 85% of the total variance of the data could be interpreted by four eigenvectors.

The first component (PC1) explained 38% of the total variance and was predominately loaded with Acy, Ace, and Flu. It has been reported that Ace and Flu were related to coke industry [39], while Acy has been related to vaporization or spill of petroleum-related products [38]. Therefore, PC1 explained the compounds originating from coking industry and spill of petroleum-related products. The second component (PC2) contributed 24% to the total variance and was heavily weighted with BbF and Flua. BbF has been recognized as an indicator of diesel combustion [40, 41], while Flua has been linked to coal combustion [2, 35–37]. Hence, PC2 was a reflection of PAHs from diesel exhaust and coal combustion. The third component (PC3) contributed 15% to the total variance and was dominated by Chr and Ant. Coal combustion could be sources of Chr and Ant [36]. Thus, PC3 was selected to represent coal combustion originated PAHs. The fourth component (PC4) was responsible for 11% of total variance and weighted by BkF and NaP. It has been pointed out that a relatively higher

TABLE 3: Rotated component matrix of studied PAHs in the Yellow River (Lingwu section).

PAHs	Components			
	1	2	3	4
Nap	0.521	−0.417	0.294	**0.623**
Acy	**0.953**	0.038	0.036	0.234
Ace	**0.904**	−0.026	0.295	−0.144
Flu	**0.92**	0.193	0.23	−0.038
Phe	−0.436	−0.786	0.191	−0.176
Ant	0.309	−0.247	**0.837**	−0.222
Flua	0.326	**0.753**	0.389	0.024
Chr	0.275	0.101	**0.829**	−0.031
BbF	−0.206	**0.916**	−0.019	0.046
BkF	−0.036	0.257	−0.166	**0.926**
BaP	−0.168	0.455	0.599	0.298
Eigenvalues	4.127	2.623	1.67	1.232
% variance	37.521	23.841	15.186	11.202
Cum. percentage	37.521	61.362	76.548	87.75

abundance of BkF was the marker of diesel vehicle emission [40, 41]. In the meantime, Nap was associated with vaporization or spill of petroleum-related products [38]. Hence, PC4 was representative of PAHs from petroleum-related products and diesel combustion.

PCA-MLR was performed, and the following equation was established:

$$\sum_{16} \text{PAHs} = 0.175 \times \text{PC1} + 0.043 \times \text{PC2} + 0.417 \times \text{PC3}$$
$$+ 0.230 \times \text{PC4} \left(R^2 = 0.4 \right). \tag{2}$$

Therefore, PAHs in Lingwu section of the Yellow River were originated from four major sources and the quantitative contributions were 34% from coal combustion, 27% from spill of petroleum-related products, 24% from coke industry, and 16% from vehicle exhausts.

It should be noted that the quantitative results for source contributions should be explained with caution due to the small R^2 value of the regression, which could be caused by the limited number of data points used in the source identification. The other reasons could be the complex sources of PAHs in Lingwu section of the Yellow River, as it is well known that Lingwu was close to Yinchuan, and as well, it is one of the most important bases of coal chemical industry in the Northwestern China. More data should be collected for further analysis.

3.3. Ecological Risk Assessment. BaP$_{eq}$s of PAHs ranged from 0.06 ng/L to 264 ng/L in surface water in Wuhai section of the Yellow River. As for Lingwu section, BaP$_{eq}$s of PAHs ranged from 0.59 ng/L to 257 ng/L among different sampling sites, which was similar to that in surface water of Wuhai section. As shown in Figure 3, probabilities of exceeding the NOEC for 5% of the species were 28% and 32% for PAHs in surface water in Wuhai and Lingwu sections of the Yellow River, respectively. This indicates that ecological risks of PAHs to aquatic organisms were high in both study areas. It
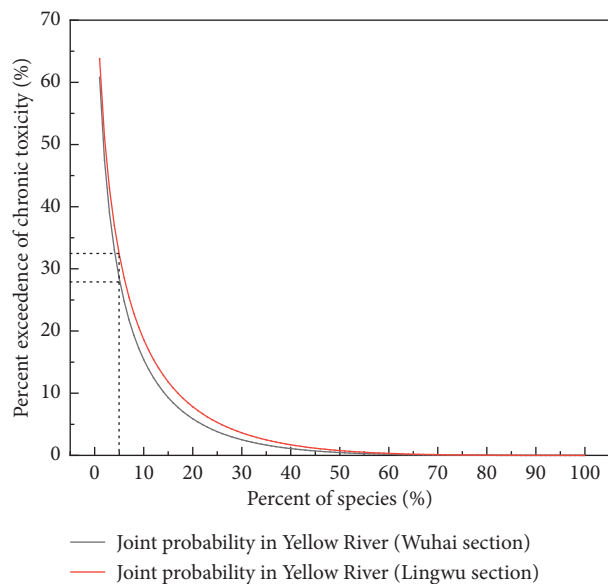
Figure 3: JPC of PAHs in surface water of the Yellow River.

has been demonstrated that there is an urgent need for taking measures to reduce the ecological risk of PAHs in the aquatic environment in the study areas.

However, there could be some uncertainties in the above assessing results. Other concomitants that were not taken into account may cause additive and synergistic effects, which could cause the underestimation of toxic effects [43]. For example, Wang et al. [44] reported that the joint toxic effect was observed when exposing the marine microalga to the mixture of Ant with profenofos. Moreover, the bioaccumulation and metabolism of PAHs in aquatic organisms were not being considered. In addition, collected toxic data and exposure data were limited. As emission continues, concentrations of PAHs in the aquatic ecosystem could increase.

4. Conclusions

The results showed that total PAH concentrations varied from 27.5 ng/L to 234 ng/L and 135 ng/L to 265 ng/L in surface water of Wuhai and Lingwu sections, respectively. PAHs in Wuhai section of the Yellow River were mainly from coal combustion (35%) and vehicle exhausts (34%). Results of ERA showed that the probabilities of exceeding the chronic toxicity for 5% of the species were 28% and 32% for PAHs in surface water from Wuhai and Lingwu sections, respectively. It has been demonstrated that there is an urgent need for taking measures to reduce the ecological risk of PAHs in the aquatic environment in our study areas.

Data Availability

The numerical data used to support the findings of this study are included within the article.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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