

Probabilistic Ecological Risk Assessment and Source Apportionment of Polycyclic Aromatic Hydrocarbons in Surface Sediments from Yellow Sea

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Abstract Based on the concentrations of polycyclic aromatic hydrocarbons (PAHs) in 12 surface sediment samples from Yellow sea, the relative risk of 9 PAHs was investigated using joint risk probability distribution curves and overlapping area, which were generated based on the distributions of exposure and acute toxicity data (LC_{50}), and the sources of PAHs were apportioned using principal component analysis. It was found that joint probability curve and overlapping area indicated the acceptable ecological risk of individual PAHs, only a small fraction of the benthic organisms was affected. Among the nine PAHs studied, the overall risk of pyrene was the highest, with that of naphthalene the lowest. For lower exposure levels at which the percentage of species affected was less than 10%, the risk associated with phenanthrene and fluorene were clearly higher than that of the other seven PAHs. It was indicated that PAHs in surface sediments mainly originated from vehicular emissions, coal combustion sources, coke oven emission and wood combustion, petroleum origin made little influence on sources of PAHs by PCA.

Keywords Surface sediment · PAHs · Probabilistic risk assessment · Source apportionment

Polycyclic aromatic hydrocarbons (PAHs) may exhibit a wide range of hazardous effects to aquatic organisms including acute toxicity, developmental and reproductive toxicity, photo-induced toxicity, mutagenicity and carcinogenicity. Sediment-associated PAHs are known to exhibit narcotic effects in benthic organisms, but also have been implicated in the development of tumors in bottom feeding fish and in the induction malformation, loss of fertility or immune deficiency in many organisms including oysters. Due to their lipophilicity, PAHs are known to accumulate in sediments as well as in mussels and other aquatic invertebrates. By accumulating in invertebrates, PAHs can enter the aquatic food webs and pose a risk to human health via consumption of seafood. It has been reported that consumption of shellfish polluted by PAHs may cause lung cancer in humans (Baumard et al. 1998; Gewurtz et al. 2000). Therefore, analyzing ecological risk of PAHs is crucial for protecting of human health and marine environment security. Ecological risk effects of chemicals in sediment can be assessed using different methods, such as sediment quality guidelines (SQGs) and hazard quotient (HQ) (Burton 2002; Solomon and Sibley 2002). The probabilistic risk assessments (PRAs) approach has been used in sediments with single chemicals such as pesticides, organotin by comparing distributions of all available species toxicity response data (or species sensitivity distributions, SSDs) with actual environment exposure concentrations (AEC) (Solomon et al. 2000; Hall and Gardinali 2004; Carriger et al. 2006; Kenneth et al. 2006). Bioaccumulation and variability of data are not considered in the development of SQGs (Burton 2002). As the HQ is based on a ratio of point estimates, it is not proportional to the risk, its use assumes that the conditions of the HQ exist on every occasion and in every location. Probabilistic approaches to ecological risk assessment have been

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recommended for later tiers in the ERA process. The PRAs allows variability of exposure concentrations and distributions of species sensitivity in the risk assessment process. It can better describe the likelihood of exceedences of effect thresholds and the risk of adverse effects (Solomon and Sibley 2002; Yang et al. 2004). The major advantage of PRAs is that they use all relevant single-species toxicity data, when combined with exposure distributions, allows quantitative estimations of risk. One of the most widely applied PRA is to calculate the overlapping area between the exposure and the effect distributions. The joint probability curve (JPC) is considered to be widely applicable. The two approaches can fully address the uncertainty and stochastic properties of the exposure and the effects (Solomon et al. 2000). The overlapping area assumes that the extent of overlap between exposure and toxicity distribution curves is proportional to the magnitude of potential risk for a contaminant or the probability that a certain percentage of species may be adversely affected. However, it can not predict the probability that the environmental exposure concentrations that exceed species acute (e.g., LC_{50} s) effect concentrations. The potential probability that a certain percentage of the environmental exposures (e.g., in sediment, water) that exceed species acute (e.g., LC_{50} s) effect concentrations can be estimated using joint probability curves (JPCs) (Carriger et al. 2006). The JPC illustrates the relationship between the proportion of species affected and the likelihood that their response concentration will be exceeded as a simple vector on a diagram (Solomon et al. 2000; Carriger et al. 2006). A point on the JPC represents the probability (in percent) of environmental exposure concentrations exceeding the endpoint (e.g., LC_{50}) for $x\%$ of the species in a SSD. Combined ecological risk of PAHs in sediments from Yellow Sea have been analyzed using the organiccarbon-normalized and mean effects range-median quotient (MERM-Q) (Liu et al. 2008). However, there have no scholars in China forecasting ecological risk of individual PAHs in surface sediments from Yellow Sea using the PRAs.

The PAH origin can be assessed by different methods, such as PAH ratios and multivariate analysis (Lang et al. 2008; Luo et al. 2008). Principal component analysis (PCA) can identify and quantify factors contributing to pollution data sets without prior knowledge of source profiles or source characteristics. This approach has been used successfully for source apportionment of PAHs in sediments (Lin et al. 2007; Xu et al. 2007).

The primary objectives of this study were twofolds: (1) to integrate sediment monitoring data and toxicity response data to evaluate ecological risk of 9 PAHs in Yellow sea sediment; and (2) to establish primary pollution source of 15 PAHs using PCA.

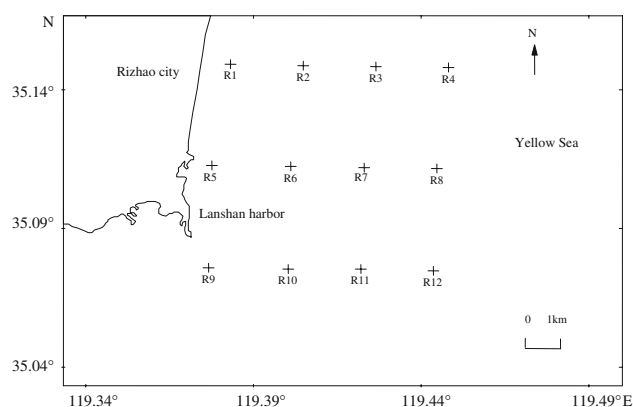


Fig. 1 The coastal sediment sampling locations of Yellow Sea

Materials and Methods

The 12 surface sediment samples (top 0–3 cm) were collected from Yellow Sea in February 2007 (Fig. 1) by a stainless steel grab sampler and stored in the pre-combusted amber glass jar. The samples were transported on ice to the laboratory and stored at -20°C condition until being analyzed.

The sediment samples were freezing dried and sieved with 100 mesh sieve. Approximately 2.0 g of samples were mixed with 0.5 g of activated copper powder to desulfurize and loaded into a 100 ml colorimetric tube, where 2.0 g sodium sulfate was added to dry the sample. The samples were then repeatedly extracted three times with 30 mL of (1:1 v/v) hexane/dichloromethane for 20 min using sonication. The extracts were concentrated to 1 mL using a rotary evaporator. The concentrated liquid was cleaned up through the fully activated silica gel column chromatography with 50 mL of (7:3 v/v) hexane/dichloromethane, followed by concentration with rotary vacuum evaporation down to about 2.0 mL. The extract was concentrated to 1.0 mL by blowing a gentle stream of nitrogen through a pasteur pipette across the top of the extract.

A GC (6890-N) with 5975B Mass Selective Detector were used for all analyses. The GC column was an HP-5MS 30 m \times 0.25 mm I.D. (film thickness 0.25 μm) fused-silica capillary column (Hewlett-Packard). Helium was the carrier gas and a flow-rate of 1.0 mL/min was used for column elution. Sample injection was carried out in the splitless mode with an injection volume of 1 μL . The GC oven temperature was programmed first from 45°C for 1 min, $6^{\circ}\text{C}/\text{min}$ to 200°C , then to 300°C at a rate of $8^{\circ}\text{C}/\text{min}$, and finally held constant for 5 min. The temperatures of the injection port and the interface to the MS system were set at 250 and 280°C , respectively. Peak quantification was carried out in selected ion monitoring (SIM) mode.

Method blanks, laboratory method control samples, and matrix duplicated samples were analyzed as quality control measures. Control samples purchased from Hengyuanqitian Chemical Technology Research Institute of Beijing were processed in a manner identical to actual samples. The recoveries of surrogates were as followed: naphthalene-d8 (68.9% ± 11.4%), acenaphthylene-d10 (76.5% ± 10.1%), phenanthrene-d10 (81.4% ± 9.2%), chrysene-d12 (91.2% ± 12.4%). Two segments in each long core and one in each short core were analyzed in duplicates. The average relative standard deviations (RSD) ranged from 9.6% to 24.1% among the 16 PAHs, with an average of 15.4%. The method detection limits (MDL) were in the range (4.98–21.70 ng/g).

All available acute (LC₅₀) laboratory toxicity data for Acute species sensitivity distributions (SSDs) were constructed to model the sensitivity of all aquatic species in water to PAHs. Toxicity data were obtained from the U.S. EPA AQUIRE database (<http://www.epa.gov/ecotox/>) and regulatory reviews such as the water quality criteria documents. All available acute (LC₅₀) laboratory toxicity data for aquatic organisms were used in the preparation of the SSDs and included fish, crustaceans, mollusks, noncrustacean invertebrates, and amphibians. Nine PAH compounds were selected for this study based on the availability of toxic data. The compounds included Nap, Ace, Acy, Fle, Phe, Ant, Fla, Pyr and Chr.

Results and Discussion

The Kolmogorov–Smirnov test was applied to test the normal distribution for both raw and log-transformed data from the exposure concentrations and acute toxicity data. The results indicated that all the log-transformed exposure data and most of the log-transformed LC₅₀ were log-normally distributed. For the risk analysis, log-transformations were conducted for all of the data utilized, test results are listed in Table 1. The log-transformed concentrations may vary from infinitely small to infinitely great, although for

each particular area they will vary within a certain range. As such, the monitored values for the exposure and toxicity data were used to represent the current exposure conditions and the extent of known effects (Wang et al. 2002). The means and standard deviations shown in Table 1 were used to construct the log-normal probability distribution curves for risk analysis.

$$f(x) = \frac{1}{\sigma\sqrt{2\pi}x} e^{-\frac{1}{2}\left(\frac{\ln x - \mu}{\sigma}\right)^2}$$

where $f(x)$ is the distribution function of variable x which represent the exposure concentration or LC₅₀, μ is geometric mean of $\ln x$, σ is SD of the log-transformed data.

Two probabilistic risk assessment (PRA) approaches, including (a) overlapping area of the exposure probability density distribution curves and LC₅₀ probability density distribution curves and (b) joint probability function, were applied in this study to assess the ecological toxicity of individual PAHs studied in sediments.

The risk assessor can estimate potential risk in terms of the overlap between the exposure and effect curves and predict the probability that a certain level of protection can be achieved (Wang et al. 2002). There was some degree of overlap between the two distributions for each of the nine chemicals studied. The calculated overlapping areas of nine PAHs for all aquatic species were tabulated in Table 2. The risks shown in Table 2 were relatively small with the highest overlapping area around 6% for Pyr, which happens to be the most toxic compounds to benthic organisms among the nine PAHs. Although the concentrations of Nap was higher in the list (Table 2), ecological risk of Nap was the lowest.

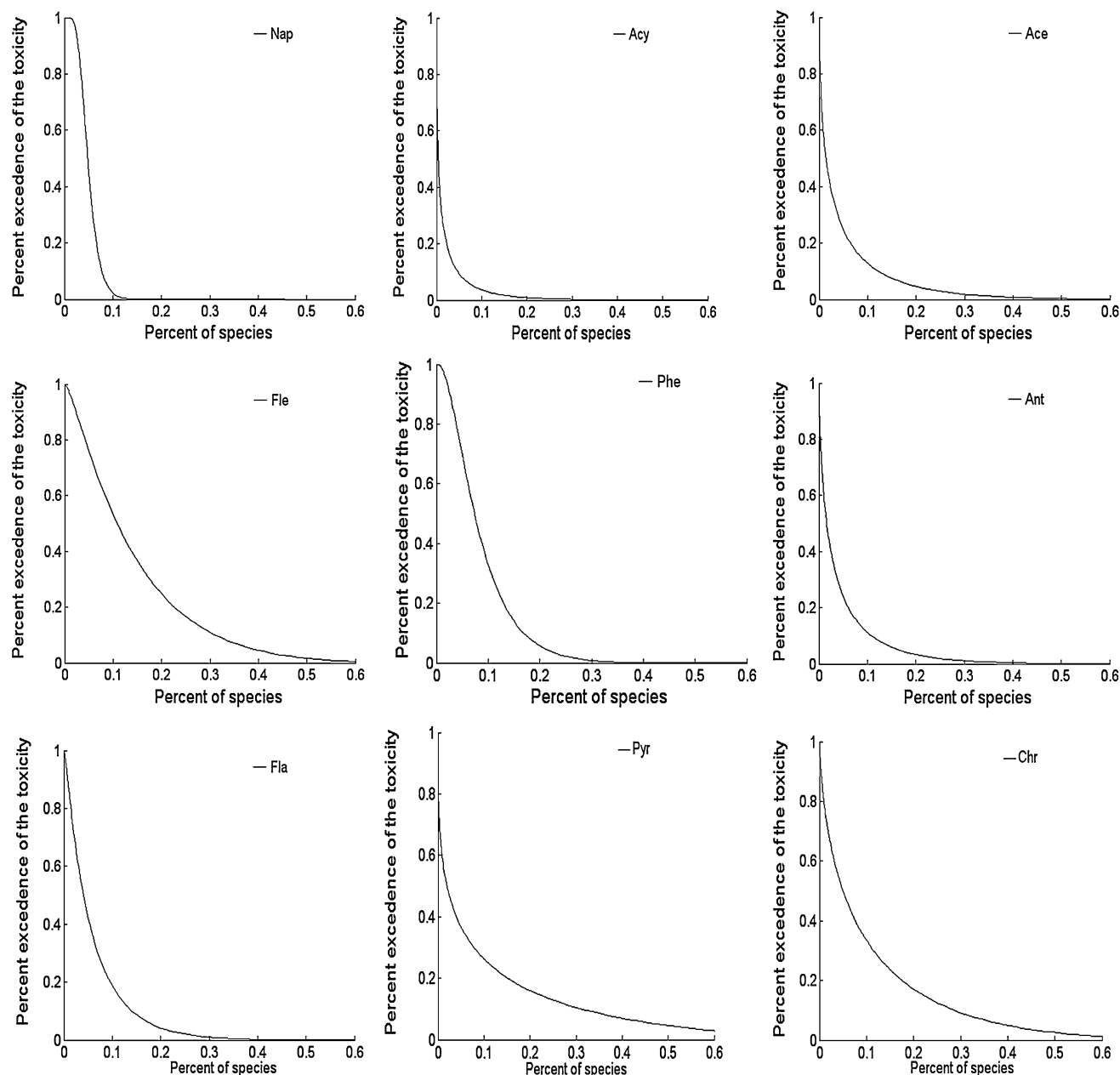
The risk of individual PAHs can be illustrated in a more explicit way by calculation of joint probability functions (Shi et al. 2004). The joint risk probability curves were used to describe the probability of exceeding an exposure concentration associated with the specific probability of effects for a given chemical and to compare the relative risks for nine pollutants of concern in different

Table 1 Distribution parameters for log-transformed exposure concentrations and toxicity data of the nine PAHs in surface sediment from Yellow Sea (Rizhao city) (μg/g)

PAHs	Log-transformed toxicity data		Log-transformed exposure concentrations	
	Mean (μg/g)	SD (μg/g)	Mean (μg/g)	SD (μg/g)
NaP	3.707	4.009	−2.949	0.751
Acy	2.650	3.561	−6.786	2.719
Ace	−0.170	2.735	−5.822	2.193
Fle	−0.211	2.132	−2.841	1.231
Phe	2.687	3.549	−2.456	1.356
Ant	1.007	3.098	−5.626	2.193
Fla	1.105	3.238	−4.539	1.684
Pyr	−1.108	3.077	−4.825	3.088
Chr	0.350	3.098	−4.743	2.608

Table 2 The calculated overlapping areas of the distribution functions of the toxicity to benthic organism and exposure concentrations of individual PAH compounds in surface sediment from Yellow Sea (Rizhao city)

PAHs	NaP	Acy	Ace	Fle	Phe	Ant	Fla	Pyr	Chr
Overlapping area	1.2×10^{-4}	6.8×10^{-3}	4.5×10^{-3}	1.39×10^{-2}	4.14×10^{-2}	5.8×10^{-3}	4.3×10^{-3}	6.07×10^{-2}	3.62×10^{-2}

**Fig. 2** Joint probability curves of individual PAHs compounds in surface sediment from Yellow Sea

concentration ranges (Wang et al. 2002). The joint probability curve of the nine PAHs were presented in Fig. 2. It was found that in the relatively low concentration range or low percentage of affected species (approximately less than 10%) the difference in exposure risk was relatively insignificant, with the risk associated with Phe and Fle

exceeding that of the other seven chemicals. Whereas in the high concentration range the exposure risk for Pyr was considerably higher than that of the other eight chemicals, that of Phe and Fle were relatively low. It is possible to rank the overall exposure risks as $\text{Pyr} > \text{Chr} > \text{Fle} > \text{Phe}$, and ecological risk of Nap was the lowest.

However, the order of the risk demonstrated by the joint probability functions shown in Fig. 2 was not identical to that from the overlapping area calculation. For instance, the overlapping area of Phe was higher than that of Fle in Table 2. Instead, Phe showed a lower ecological risk in comparison with Fle in Fig. 2. It appeared that although the results of overlapping areas and joint probability curves showed similar results for the risk of the nine PAHs in surface sediments from Yellow Sea, they were not identical in terms of detailed order of the risk for individual compounds.

All the results of described above indicated the acceptable ecological risk of individual PAHs with Pyr was the most hazardous one in surface sediments from Yellow Sea. Both overlapping areas and joint probability curves suggested that only a small fraction of the benthic organisms was affected. The fractions were from 6% for Pyr to negligible for most PAH compounds under investigation. Pyr imposed the relatively higher risk than the others did. For lower exposure levels at which the percentage of species affected was less than 10%, the risk associated with Phe and Fle were clearly higher than that of the other seven PAHs.

Although PRA approaches are useful for characterizing variability in conducting risk assessment process, uncertainty, variability, and other factors may coexist within the data available. A potentially important uncertainty in the risk assessment process is selection of toxicity data. In this study, the acute effect concentrations (LC_{50}) was selected as toxicity reference value, this may underestimated the ecological risk. In the future, as greater amounts of information on chronic exposure and effect and the role of the organism in ecosystem structure and function are fully considered, a more complete evaluation of the ecological risks of chemicals to populations, communities, and ecosystems will be possible (Bartell et al. 2000; Wang et al. 2002).

An attempt was made to identify the sources of PAH in Yellow Sea using the well-known principal component analysis (PCA). The analysis was carried out using SPSS 13.0 software. The results of the PCA analysis were presented in Tables 3 and 4, where the factors along with their factor loadings for individual PAHs were given. The principal components with eigenvalues >1 have been

Table 3 Accumulated variance and eigenvalues of individual PAH components

PC	Eigenvalues	Variance%	Cumulative%
1	10.067	62.919	62.919
2	2.831	17.694	80.613
3	1.318	8.235	88.848

Table 4 Factor loading of individual PAH components

PAHs	PC ₁	PC ₂	PC ₃
NaP	0.454	−0.257	0.800
Acy	0.697	−0.381	−0.059
Ace	0.691	0.523	0.084
Fle	0.577	0.722	−0.175
Phe	0.714	0.577	−0.254
Ant	0.777	0.527	0.076
Fla	0.916	0.200	0.088
Pyr	0.936	0.177	0.193
BaA	0.874	−0.291	−0.239
Chr	0.940	−0.140	0.204
BbF	0.872	−0.293	0.227
BkF	0.874	−0.357	−0.043
IND	0.807	−0.498	−0.210
DBahA	0.606	−0.489	−0.563
BghiP	0.896	−0.350	0.027

selected. PC₁ and PC₂ accounted for 62.92% and 17.69% of the variability in the data, respectively, while a third significant component (PC₃) accounted for 8.24% of the variability in the data (Table 3).

The first factor was responsible for 62.92% of the total variance. Fla, IND, BaA, Chr, BbF, BkF, Pyr and BghiP were highest loaded on the factor. BghiP has been identified as tracers of gasoline emissions. Diesel emissions may have a more significant contribution from BbF, BkF, BaA and IND (Homdutt et al. 2007). There was also a moderate loading from Phe, Acy, Ace, Fle and Ant. The compounds of Phe, Acy, Ace, Fle and Ant are indicative of coal combustion sources (Chen et al. 2005; Duran and Beyhan 2005). Therefore, factor 1 was selected to represent vehicular emissions and coal combustion sources.

The second factor was responsible for 17.69% of the total variance. This factor was predominately weighted in Fle. Emissions from coal combustion exhibit higher relative amounts of alkyl PAHs than other combustion sources (Matt et al. 1999). This component was also highly loaded on Ace, Phe and Ant, all of which are predominant in coal combustion signals (Mastral et al. 1996). Phe and Ant are associated with wood combustion (Luo et al. 2006). Uncombusted petroleum was also high in alkyl PAHs (Matt et al. 1999). However, the high loadings on the higher molecular weight PAHs such as BbF, BkF, BghiP which are either very low in abundance or absent in uncombusted petroleum suggest that factor 2 was not representative of uncombusted petroleum. Ace and Fle were indicative of coke oven emission of PAHs (Shen et al. 2007). Based on the above information, the second factor was selected to represent coal combustion origin, wood combustion and coke oven emission.

Factor 3 was heavily weighted by NaP, which was associated with unburned fossil derived PAHs (Yunker et al. 1996; Marko et al. 2001). Consequently, this factor was selected to represent petroleum origin.

The source identification of PAHs in surface sediment from Yellow sea has been studied using CMB model and diagnostic Ratios. It was indicated that coal combustion, vehicular emissions (diesel and gasoline engines exhaust), coke oven emission and wood combustion were the principal sources of PAHs in surface sediment from Yellow sea, petroleum origin made little influence on sources of PAHs (Ling et al. 2008; Xue et al. 2008), and was basically in accordance with our study. To summarize, it can be inferred from the above analysis that the major source of PAHs was vehicular emissions, coal combustion sources, coke oven emission and wood combustion, petroleum origin made little influence on sources of PAHs.

In conclusion, joint probability curve and overlapping area indicated the acceptable ecological risk of individual PAHs. Only a small fraction of the benthic organisms was affected. The fractions were from 6% for Pyr to negligible for most PAH compounds under investigation. Among the nine PAHs studied, the overall risk of Pyr was the highest, with that of NaP the lowest. For lower exposure levels at which the percentage of species affected was less than 10%, the risk associated with Phe and Fle were higher than that of the other seven PAHs.

PCA indicated that PAHs in surface sediments mainly originated from vehicular emissions, coal combustion sources, coke oven emission and wood combustion, petroleum origin made little influence on sources of PAHs.

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