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Contamination and Ecotoxicology risks of Polycyclic Aromatic Hydrocarbons in Shantou Coastal Waters, China

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Abstract Nine locations in Shantou coastal waters were chosen for the study on contamination and ecotoxicology risks posed by polycyclic aromatic hydrocarbons (PAHs). Sediment samples were collected to investigate PAH distribution behaviour, sources and understand their origin, which is fundamental in predicting their subsequent behaviour. Many approaches and methods were applied to accomplish these objectives and study purpose. The results found revealed the critical importance of improving our understanding of PAH equilibrium relationships. The serious environmental and health concern, imposed by the high concentrations of PAHs in the area, were widely discussed. Furthermore, the location of Shantou within the town and vicinity of Guiyu, which is a booming E-waste processing centre in China, might explain the significance of atmospheric transportation source of PAHs and enhance the occurrence of air–water exchange.

Keywords Polycyclic aromatic hydrocarbons · Shantou coastal waters · Sediment

PAHs are an important class of persistent organic pollutants (POPs) prevalent in the environment. Formed of two or more fused benzene rings, they are found in the environment in the form of parent compounds and a variety of alkylated homologues arising from both combustion and oil-derived sources (Woodhead et al. 1999). In this paper, the concentrations and behaviour of 16 parent compounds in the marine environment have been studied. The 16 compounds have designated as priority pollutants by the US Environment Agency (USEPA) for their potential carcinogenic and toxic effects.

PAHs can be produced and then released into the marine environment via a range of processes, the nature of which has a strong influence on the subsequent fate of the compounds (Hartmann et al. 2004). The mechanisms by which PAHs form and enter the environment are summarized here. Firstly low temperature processes, including the thermal alteration of organic matter such as in the formation of fossil fuels, are known as petrogenic sources. These are characterized by 2- and 3-ring PAHs, a high proportion of alkyl substituted PAHs and the preferential formation of isomers with higher thermodynamic stability. Secondly, high temperature combustion, or pyrolytic, processes lead to PAH distributions dominated by high molecular weight compounds, minimal alkylation products and the dominant isomers are those that form most rapidly and so are kinetically, not necessarily thermodynamically, favoured (Simpson et al. 1996, Readman et al. 2002).

Certain industrial processes often produce PAHs with characteristic distribution patterns close to their origins (Simpson et al. 1996) including the metallurgical industry (Naes et al. 1998), and the steel and iron industries (Yang et al. 2002). Gaseous stack emissions from twelve steel and iron plants in Taiwan, for example, were found to contain elevated concentrations of low molecular mass PAHs, particulate emissions contained increasing levels of the heavier PAHs; the dominant compound in both phases was observed to be naphthalene (Yang et al. 2002). The majority of PAHs derive from anthropogenic sources with

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the highest concentrations typically found in urban centres. There are a variety of routes by which they can enter the marine environment, including the discharge of industrial effluents, urban run-off, atmospheric deposition, and the spillage and disposal of oil and petroleum products. Petroleum products are largely introduced through road run-off, industrial seepage, storm water drains, shipping activities and spillage. Combustion related inputs arises from bush fires, agricultural burn-off and vehicle exhaust (Readman et al. 2002, Woodhead et al. 1999). The importance of run-off as source of PAHs to the aqueous environment has been shown by the observation of elevated concentrations of PAHs in estuarine sediments near road bridges (Rogers 2002).

The aim of this study were initially to investigate of the PAH distribution in sediments collected from Shantou coastal waters. Secondly, assess the sources of the compounds to sediments in the system. And finally, understand of the compounds origin which is fundamental to predicting their subsequent behaviour and ecotoxicology risks.

Materials and Methods

Shantou, one of the Special Economic Zones, is located on the eastern coast of Guangdong Province, Shantou prefectures to South China Sea, borders Fujian to the north and faces Taiwan. Shantou is in an advantageous position, the climate here is temperate, warm without bitter cold days in winter, and cool without hot day in summer the average temperature within a year is 21.3°C.

This city has established itself as the key distribution centre for the plastics raw materials that are vital components of manufacturing in southern China. Shantou is well connected by road and rail to Guangdong, Fujian and other provinces. An expressway runs from Shantou to Shenzhen. A rail link connects Shantou to the arterial route linking Hong Kong with Beijing, and to Guangzhou. These connections make Shantou an ideal centre for long-haul traffic.

The site and allocation of sampling are shown in Fig. 1. Samples of surface sediment were collected from nine sites using a grab sampler. The toxic fraction removed using a stainless steel spoon and transferred to a solvent washed glass jar. On return to the laboratory, sediment samples were stored at −20°C until extraction.

Wet sediment (20 g) was accurately weighed into conical flasks. Copper granules (3 g) were added to remove sulphurous compounds and pre-combusted sodium sulphate (6 g) added to remove water. Sediments were spiked with 1 μg (10 μl) of the PAH internal standard and 50 ml dichloromethane (DCM) was added to the sediment and samples were subjected to a further 30 min ultrasonication. Extraction of PAHs by ultrasonication with DCM has been previously to be a suitable analytical technique (e.g. Mitra et al. 1999, Song et al. 2002, Wang et al. 2001). The extracts were then combined and concentrated using rotary evaporation to approximately 1 mL and purified in a silica gel column (4-mm i.d. × 90 mm). The column was then eluted by dichloromethane (3.5 mL), (Zhou et al. 2000; Zhou and Maskaoui 2003; Maskaoui et al. 2002). All the extracts were concentrated under a stream gentle of N_2 to

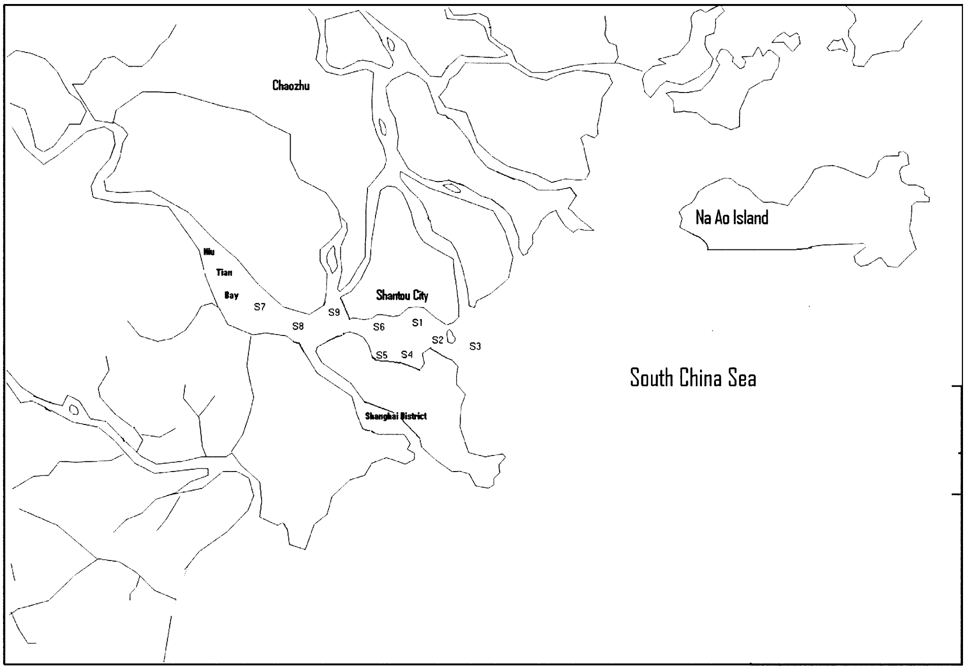

Fig. 1 Map of Shantou showing the sampling locations

about 100 µL. Good recovery was obtained using this procedure as shown by Zhou et al.

A Hewlett-Packard 5890 GC with a Flame Ionisation Detector (FID) was used, an autosampler, and Chemstation software was used for determining the levels of PAHs in sediment samples. An HP-5 (5% diphenyl and 95% dimethylsiloxane) capillary column of 30 m × 0.32 mm i.d. (0.25 mm film) was used. The column temperature for analyses was programmed from 60°C (initial time, 2 min) to 120°C at a rate of 10°C/min, 120–325°C at a rate of 3°C/min, and held at 325°C for 5 min.

Before analysis, relevant standards were run to check column performance, peak height and resolution, and the limits of detection (LOD). With each set of samples, a solvent blank, a standard mixture and a procedural blank were run in sequence to check for contamination, peak of identification and quantification. Compounds were identified mainly by their retention times. Selected samples were analyzed by full scan GC/MS for confirmation. All results for the samples were reported on a day-weight basis.

All analytical data were subject to strict quality control. Spiked sediment samples were determined with good precision, and recoveries ranged from 60 ± 8% to 94 ± 10% for the samples (Zhou et al. 2000; Maskaoui et al. 2002, 2006; Zhou and Maskaoui 2003). In addition, the errors involved in sampling were assessed by carrying out triplicate sampling of sediment at the same site and the analysis of sample extracts. Results showed good reproducibility of the sampling process.

Results and Discussion

The results obtained from this survey are presented in the following sections. Initially the discussion of the concentrations of the 16 parent PAHs in the samples collected. This followed by molecular indices and analysis of chromatographic data, potential compound source are then apportioned.

All sediment PAH concentrations discussed here are reported on a dry weight basis. Sum of the total concentration of 16 parent PAH compounds analysed in this study was denoted as total PAH. The pollution levels are assigned Baumard et al. (1998) as low (L) = 0–100 ng/g, moderate (M) = >100–1000 ng/g, high (H) = >1000–5000 ng/g, very high (VH) = >5000 ng/g. The classification of sediments based on total PAHs contamination levels presented in Table 1, show the majority of the samples were classified as highly contaminated.

Table 1 Pollution levels of sediments from different sampling sites

Site	S1	S2	S3	S4	S5	S6	S7	S8	S9
Level	H	VH	H	VH	M	H	H	M	VH

The results of PAH concentration from different stations are given in Table 2. The total PAH concentration in sediments was found to vary between 884.1 (site 8) and 13183 ng/g (Site 4) with a mean total PAH concentration of 3413.6 ng/g. PAH concentrations were found to fluctuate quite widely between sampling locations as shown in Fig. 2. All of the compounds of interest, with the exception of dibenzo(a,h)anthracene, were present in sediment samples. The PAH load was dominated by 3- and 4-ring compounds. Pyrene, fluoranthene, phenanthrene and fluorene were typically present at high concentrations although the naphthalene was only detected at two locations S8 and S9 at concentrations 4.652 and 6.890 ng/g, respectively. In contrast to other sites, these stations were located at the mouth of a river, which discharge waters and sewage from the most industrialized areas in Chaozhu and Shantou. The high molecular mass compounds containing 5- and 6-rings except Dibenzo(a,h) anthracene were also detected in sediment samples at different low concentrations. Benzo(g,h,i)perylene were present at almost half of the sampling stations.

Once again compounds were well distributed between the sampling sites, the highest sediment PAH concentrations were measured at site 4 where the total PAH concentration of 13183 ng/g was observed. Interestingly this site also contained the highest concentration of pyrene (for site 4, pyrene concentration represents 90% of total PAHs). Three replicate sediments from sites 1, 4 and 9 were realized while only two replicate for the sites 5 and 7 in order to confirm the accuracy and recovery of the results. The high contamination of the site 4 (as shown Fig. 2) may be linked to its location close to the Shantou Electrical Power Station near or Taiwan Bridge.

It is important to note that the site 2 located just between Shantou harbour and the open Sea and close to a small Island is one the most polluted site with the total PAH concentration of 7896 ng/g. High concentrations of PAHs were also detected at station 9 (6325 ng/g), which is situated at the mouth of the river with huge of the industrial discharge effluents and especially sewage (with a bad strong odour observed during sampling). This river comes from a long way linked to the different industrials areas located in Chaozhou and Shantou. Consequently, it represents the major point source of polluted waters discharged in Shantou harbour.

Aromatic compound distributions differ according to the production sources and depend on the chemical composition and combustion temperature of the organic matter (Neff 1979). The molecular PAH patterns generated by each source are like fingerprints and it is possible to determine their origin using molecular indices based on

Table 2 Concentration of PAHs (ng/g dry weight) in Shantou marine sediment

	S1	S2	S3	S4	S5	S6	S7	S8	S9
Naphthalene	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	4.652	6.89
Acenaphthylene	<LOD	51.14	17.54	<LOD	<LOD	<LOD	<LOD	1.495	<LOD
Acenaphthene	47.59	53.65	19.9	48.79	30.7	39.15	14.72	1.644	4.407
Fuorene	330.1	822	296.2	557.6	373.8	513.7	384	145.5	444.6
Phenanthrene	280.8	363.4	164.2	296.1	103.7	205.1	451.1	269.5	486.1
Anthracene	5.251	4.027	1.787	3.677	1.193	2.377	6.376	3.114	1.465
Fluoranthene	166.3	168.3	79.01	132.9	291.5	98.06	737.7	171.2	356.8
Pyrene	434.7	6147	2116	11923	118.3	267	2502	265	4886
Benzo(a) anthracene	8.015	261.3	96.86	188	2.453	4.43	34.73	7.11	103
Chrysene	4.8	3.752	1.401	3.122	0.548	0.905	6.887	3.449	4.406
Benzo(b) fluoranthene	5.599	7.01	2.757	17.73	2.158	4.087	27.79	5.808	8.783
Benzo(k) fluoranthene	8.394	7.896	1.188	4.942	3.527	5.411	42.13	2.267	18.18
Benzo(a)pyrene	112.8	4.598	2.085	3.57	0.372	0.405	7.154	0.913	1.596
Indeno(1,2,3-cd) pyrene	<LOD	2.1	0	0.072	0.227	0.457	2.554	0.475	2.33
Dibenzo(a,h) anthracene	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
Benzo(g,h,i) perylene	<LOD	<LOD	<LOD	<LOD	2.659	<LOD	27.91	1.977	0.233
Total PAHs	1404	7896	2799	13183	931.2	1141	4245	884.1	6325

LOD < 0.001

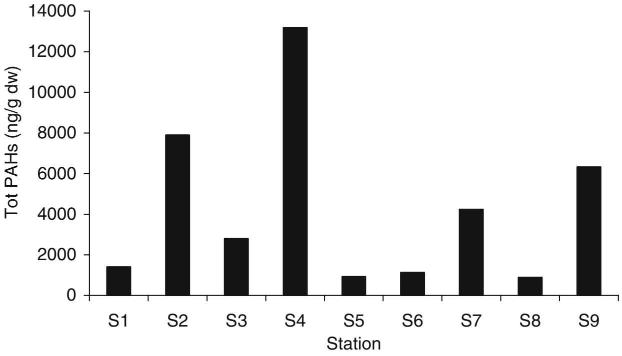

Fig. 2 Distribution of total PAHs in sediments from Shantou coastal waters

Table 3 Molecular indices for Shantou marine sediments

Site	Phe/ Ant	Fluor/ Pyr	BaA/ Chr	LMW/ HMW	BaP/ BghiP
S1	53	0.4	1.7	9	*
S2	90	0.03	70	27	*
S3	92	0.04	69	26	*
S4	81	0.01	60	60	*
S5	87	2.5	4.5	77	*
S6	86	0.37	4.9	72	*
S7	71	0.30	5	27	*
S8	87	0.65	2.1	39	0.46
S9	332	0.07	23	45	6.85

* One of the analytes below LOD (0.001)

ratios of selected PAH concentrations (Soclo et al. 2000). To estimate the origin of the pollution in this area, the LMW/HMW ratio (sum of the low molecular weight PAH concentrations/sum of higher molecular weight PAH concentration) was used (Table 3). The theory of this origin index was based on the fact that the petrogenic contamination was characterized by the predominance of the lower molecular weight PAHs (tri- and tetra-aromatics) (Neff 1979; Wise et al. 1988; Berner et al. 1990), while the higher molecular weight PAHs dominate in pyrolytic PAH contamination (Muel and Saguem 1985). Examination of Table 3, shows that the LMW/HMW are higher than 1 (between 9 and 77) indicating the influence of petrogenic contaminants. The highest petrogenic PAH origin were observed at the sites (4, 5 and 6) located near ports used by the Chinese navy and for import/export container shipping activities, while site 4 is located close to the Electrical power plant of Shantou explaining the high petrogenic sources from its effluents discharges (Fig. 1). However the high petrogenic origin in other sites may be explained by the fact that Shantou harbour is a semi enclosed body of water located in the most industrialized areas in Guangdong province, has been subjected to pollution surrounding domestic and industrial discharges during the last 25 years. The bathymetric properties of this area may be another factor in the distribution of PAHs by its shallow characteristics. The depths of all stations (4–10 m) may play a crucial role in the suspension of sediments and the LMW contents become much more predominant.

To characterize PAH distribution better, the molecular indices based on the ratios of various selected PAHs have been widely used to characterize their origins (e.g. Baumard et al. 1998; Readman et al. 2002). These ratios are based on the differences in physico-chemical behaviour of various structural isomers. For example, phenanthrene and anthracene are isomers but phenanthrene is the more thermodynamically stable of the two compounds- hence the ratio of phenanthrene to anthracene is typically high in samples contaminated by petrogenic pollution but lower when contamination arises from pyrolytic sources (e.g. Soclo et al. 2000). In general, values of phenanthrene/anthracene >15 indicate petrogenic inputs while values <10 suggest pyrolytic sources. The ratio of phenanthrene to anthracene, in crude oils is normally around 50 (Webster et al. 2000). From the Table 3, it was clear to confirm that the petrogenic sources are the major source of PAH pollution in Shantou waters since the ratio of phenanthrene to anthracene for all samples were >15. Moreover the fact that most of the ratio values are close or even superior to the ratio 50, which is related to crude oils, it is important to note that there has been an immense oil spill in this area.

Also, another commonly used source apportionment ratio is that of fluoranthene to pyrene, with values above unity typically suggesting pyrolytic origins and values below unity typical of petrogenic sources which is the case for this study area as shown in Table 3, the ratio fluoranthene to pyrene are below unity except for one sample at site 5. However, when contamination arises from a combination of both petrogenic and pyrolytic processes, it has been clearly observed that the ratios of phenanthrene to anthracene and fluoranthene to pyrene will typically indicate pyrolytic processes as being the dominant source, thus making specific apportionment more complex (Webster et al. 2000).

Further isomer ratios include benzo(a)anthracene/chrysene, where value >0.9 are associated with pyrolytic origin and values ≤0.4 petrogenic sources (Gschwend and Hites 1981), and other characteristic isomer ratios that arise from specific processes. The value of the ratio benzo(a)anthracene to Chrysene for Shantou sediment samples, which range from 1.7 to 70, give confirmation at the pyrolytic origins of PAHs in this study area. On the other hand, a ratio of benzo(a)pyrene/benzo(g,h.i)perylene in the range 1–5 may suggest sediments contaminated through wood and coal burning (Maher and Aislabie 1992). The results shown here clearly suggest there is no evidence of any sediment contamination through wood and coal burning since the ratio are either below the limit of detection and or in excess of 5 for the site 9.

PAHs can be persistent pollutants in the marine environment and distribute between the various phases including overlying seawater, surficial sediment, buried sediments and sediment interstitial waters. Knowledge of phase distribution is fundamental in understanding the environmental fate and behaviour of PAHs. Some PAH compounds are known to be human carcinogens as classified by the International Agency for Research on Cancer (IARC 2002).

IARC Group 2 A “ probably carcinogenic to humans”	IARC Group 2 B “possibly carcinogenic to humans”	IARC Group 2 B “not classifiable”
Benzo(a)pyrene, Benzo(a)anthracene, Dibenzo(a,h) anthracene	Benzo(b)fluoranthene, Benzo(k)fluoranthene, Indeno(1,2,3c-d)pyrene	Benzo(g,h,i) perylene, Chrysene, Phenanthrene, Fluorene, Anthracene, Pyrene, Fluoranthene

It has been shown that men exposed occupationally to high concentrations of PAH mixtures have an increased incidence of tumours of the lung, skin and possibly bladder (DEFRA 2002). In a study by Vyskocil et al. (2000) ingestion in food was found to be the main source of pyrene and total PAH in children, even in areas where reasonably high concentrations were measured in air samples. In terms of the potential ingestion of PAHs in food, of much concern is the potential of ingestion of the compounds through seafood caught in areas contaminated by PAHs.

The lifestyle of an organism has a determining influence on the exposure it receives to PAHs in the dissolved, particulate associated and sedimentary phases. For example, fish can be exposed to sedimentary PAHs via direct contact with sediment, respiration of interstitial water and ingestion of contaminated sediments and invertebrates (Woodhead et al. 1999). While fish do not necessarily bioaccumulate these compounds, as they possess active metabolizing and excretion mechanisms, biological effects can be seen as a result of the carcinogenic effects of some PAHs and their metabolites. Filter-feeding bivalves are exposed to concentrations dissolved in overlaying water as well as associated with sediment and sediment interstitial water. Burrowing organisms can be exposed to concentrations dissolved in overlaying and interstitial water (Chapman et al. 2002).

Another important factor influencing exposure is the life style of the organism in question related with the direct consumption of particulates that can contribute a significant fraction of total PAHs. However, the major concern with respect to the impact of PAH on fish in aquatic sediment systems in the carcinogenicity of some congeners. Six of the PAH which are known carcinogenic to mammals were found in this area namely; benzo(a)anthracene (Baa), Chrysene (Cry), benzo(b)fluoranthene (Bbf), benzo(k)fluoranthne (Bkf) and benzo(a)pyrene (Bap) except for Dibenzo(a,h)anthracene (Dba) which was below limit of detection (LOD) in all the stations. Others such as anthracene (Ant), fluoranthene (Fla) and pyrene (Pyr) may be acutely toxic at high concentrations were also found in the sediment samples with the highest concentrations at station 4.

A variety of approaches have been developed to use the available ecotoxicology data on PAH to set numerical sediment quality guidelines (SQGs) (Chapman 1989; Macdonald et al. 2000). Selection of the most appropriate SQGs for specific applications can be a daunting task for sediment assessors. In addition, the SQG selection process is further complicated due to uncertainties regarding the bioavailability of sediment-associated contaminants, the effects of covarying chemicals and chemical mixtures and ecological relevance of the guidelines (MacDonald et al. 2000). In this study, the Threshold Effect Level (TEL) and Probable Effect Level (PEL) weight of evidence approach was used as SQGs (based on Macdonald et al. 1996; CCME 1999) in order to evaluate PAH contamination of Shantou coastal waters. TEL represents the concentration below which adverse effects are expected to occur only rarely. PEL represents the concentration above which adverse effects are expected to occur frequently (MacDonald et al. 2000).

Table 4 Comparison of the LMW individual PAH contents with (a) PEL values (b) TEL values

	Ace	Flu	Phe	Ant	Fla	Pyr	Baa	Cry	Total LMW
(a) PEL values									
PEL	89	144	544	245	1494	1398	693	846	1442
S1	−	+	−	−	−	−	−	−	−
S2	−	+	−	−	−	+	−	−	+
S3	−	+	−	−	−	+	−	−	+
S4	−	+	−	−	−	+	−	−	+
S5	−	+	−	−	−	−	−	−	−
S6	−	+	−	−	−	−	−	−	−
S7	−	+	−	−	−	+	−	−	+
S8	−	+	−	−	−	−	−	−	−
S9	−	+	−	−	−	+	−	−	+
(b) TEL values									
TEL	6.7	21.2	86.7	46.9	113	153	74.8	108	312
S1	+	+	+	−	+	+	−	−	+
S2	+	+	+	−	+	+	+	−	+
S3	+	+	+	−	−	+	+	−	+
S4	+	+	+	−	+	+	+	−	+
S5	+	+	+	−	+	+	−	−	+
S6	+	+	+	−	−	+	−	−	+
S7	+	+	+	−	+	+	−	−	+
S8	−	+	+	−	+	+	−	−	+
S9	+	+	+	−	+	+	+	−	+

(+) Exceed standards, (−) below standards

Considering the results given in Table 2, individual PAH contamination values, which exceed the PEL and TEL were tabulated in Table 4a and b. Most of the PAH value at station 4 exceeded TEL however only the PAH compounds Fluorene and Pyrene did exceed the EPL in almost for all the stations.

Considering the values exceeding only PEL, as shown in the Table 4a and b, Total LMW PAH values exceeded the PEL at five stations 2, 3, 4, 7 and 9. However, tough the remaining stations did not exceed the PEL, their values were close. It is important to note that Tot LMW PAH values exceed the TEL at the nine stations.

The TEL/PEL analysis suggests that the Shantou sediments were likely to be contaminated by acutely toxic PAH compounds. These results raise an alarm about the risk posed for the water quality and the ecology of the area. A pilot study in urgently needed to assess bioaccumulation of these pollutants in marine organisms and to investigate the distribution of PAHs in water column and pore-water.

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