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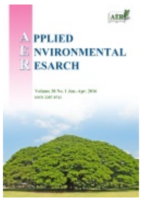

Source and Distribution of Polycyclic Aromatic Hydrocarbons (PAHs) in Surface Sediment from Chalong Bay, Phuket, Thailand

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Abstract

This study aimed to investigate the spatial distribution and apportionment of potential sources of the 16 US-EPA priority PAHs in surface sediments of Chalong Bay, Phuket, Thailand. A total 28 of sediment samples were collected from areas with high maritime activity in Chalong Bay, subjected to conventional high recovery Soxhlet extraction, purified with SiO_2 column chromatography, and quantified by Gas Chromatography-Mass Spectrometry (GC/MS). Total concentration of PAHs (Σ16PAHs) ranged from 31.15 to 1696 ng g^{-1} dry weight, with an average of 198.2 ± 318.5 ng g^{-1} dry weight. Binary diagnostic ratios plots were used to distinguish between petrogenic and pyrogenic sources. Most of the PAHs in sediments originated from pyrolytic sources. PCA analysis explained 82.3% of the variance by only 4 predominant components. The first principal component (PC1) (26.7%) was mainly contributed to urban street runoff and municipal wastewater discharge. PC2 (25.8%) represented vehicular combustion sources, PC3 (19.1%) was attributed to petrogenic sources, and PC4 (10.7%) was only associated with Naphthalene. The sources of PAHs distributed around Chalong Bay included oil spills, combustion of fossil fuels by shipping, urban street runoff, and municipal wastewater discharge. Each type of sources affected different locations along shores of Chalong Bay.

Keywords: PAHs; Spatial distribution; Coastal sediment; Source apportionment; Pyrolytic source; Petrogenic source

Introduction

Polycyclic aromatic hydrocarbons (PAHs) are hazardous organic pollutants in the marine environment, as they are mutagenic and carcinogenic, and toxic to marine organisms and, also with concerns about risks to humans through food consumption [1–3]. Therefore, the US Envi-

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ronmental Protection Agency (US-EPA) has listed 16 PAHs as priority pollutants [4]. The PAHs have two main potential sources. First, petrogenic sources (2–3 ring PAHs) originate from crude and refined oil spills, while pyrogenic sources (4–6 ring PAHs) involve petroleum combustion, biomass burning, and urban stormwater runoff [5–8]. The number of aromatic rings can be used to identify sources of PAHs [9]. Each source has a signature fingerprint in PAHs composition, so they can be apportioned to the sources [10]. In a marine environment, the most common sources of PAHs are petroleum spills, combustion of fossil fuels, and non-point sources such as municipal wastewater discharge, urban street runoff, and atmospheric fall-out [11–15].

Analysis of PAHs in sediment matrices requires sophisticated procedures. Commonly, extraction methods used in PAH determinations from sediment samples include Soxhlet extraction [16–19], accelerated solvent extraction (ASE) [20–21], ultrasound assisted extraction [22], and solid phase microextraction (SPME) [23]. However, these techniques have limitations related to simplicity of operation, cost, sample matrix effects, and the greater the sample preparation the lower the throughput [24]. Regarding the quantitative analytical methods for PAHs in sediment samples, the most frequently used methods are GC/MS, and HPLC coupled with UV/ Fluorescence detectors. The considerations for choosing a suitable instrumental technique include sensitivity, selectivity, polarity of target compounds, and ability to isolate PAH isomers [25]. Diagnostic ratios of PAHs, such as the anthracene / (anthracene + phenanthrene) ratio, the benzo [a]anthracene / benzo[a]anthracene + chrysene) ratio, and the fluoranthene / (fluoranthene + pyrene) ratio, have been used to identify the likely parent sources of PAHs in a number of studies [6–8, 26].

Chalong Bay, located on the eastern shoreline of Phuket Island, Thailand, is a muddy beach that serves as a center of intense boating activity and is a commercial port. A parking area, restaurants, hotel, shopping center, tourist information kiosks and open-air waiting areas have been built to serve the many visitors. Therefore, potential sources of anthropogenic PAHs in Chalong Bay include municipal wastewater discharges, oil spills, and incomplete vehicular combustion [27–29].

In this study, we aimed to investigate the spatial distribution and apportion to potential sources of the 16 EPA priority PAHs in Chalong Bay sediments, by using fundamental analytical chemistry to determine trace level PAHs in the sediment medium. The diagnostic ratios in PAH composition were subjected to a multivariate statistical technique for identifying signatures of various sources in the study area, to properly attribute the emissions.

Materials and methods

1) Reagents and chemicals

All solvents were HPLC grade, purchased from RCI Lab Scan limited. A 16 US-EPA priority mixed standard solution was purchased from Sigma Aldrich (St. Louis, MO, CAS no. 36991) containing the diagnostic compounds Naphthalene (Nap), acenaphthene, (Ace), acenaphthylene (Acy), phenanthrene (Phe), anthracene (An), fluoranthene (Fluo), pyrene (Pyr), benz[a]anthracene (B[a]A), chrysene (Chry), benzo[b]fluoranthene (B[b]F), benzo[k] fluoranthene (B[k]F), benzo[a]pyrene (B[a]P), benzo[e]pyrene (B[e]P), indeno[1,2,3-cd]pyrene (Ind), dibenz[a,h]anthracene (D[a,h]A), and benzo [g,h,i] perylene (B[g,h,i]P)]. The categorized of 2–3 ring PAHs were Nap, Ace, Acy, Phe and An, for 4–6 ring PAHs consist of Fluo, Pyr, B[a]A, Chry, B[b]F, B[k]F, B[a]P, B[e]P, Ind, D[a,h]A and B[g,h,i]P). Deuterated internal standard (IS) solutions of d12-perylene (d12-Per) and d10-fluorene (d10-Fl) were from Chiron AS (Stiklestadveine 1, N-7041 Trondheim, Norway). Stock solutions of the deuterated and native PAHs were prepared in nonane. Working standards from the nonane solutions were diluted in n-cyclohexane.

2) Study area and sampling methods

Chalong Bay was chosen as the study area because it has intensive maritime activities depositing PAHs into the sediments. The intensive traffic ferry port, residential areas, travel agency, parking lot for one day trips, hotels, restaurants and shopping center are all potential sources of PAHs. Furthermore, water discharge from Mudong canal also contributed to the non-point sources of PAHs in this area.

The van-Veen Grab sampler was used to collect 28 samples of surface sediments during 22–23 September, 2016. The sampling sites are shown in Figure 1. Site allocations were as close as possible to a simple grid except in areas of the bay that were not readily accessible. The sediment samples were wrapped in aluminum foil, then placed in glass bottles, and stored at -20 °C. For measurements the sediment samples were freeze dried, then ground to a uniform powder, then kept in a refrigerator at 4 °C until they were analyzed.

3) Analytical procedures

The dried sediment sample (exactly 5.00 g) was extracted by conventional Soxhlet extraction for 6 h using dichloromethane. The recovery internal standard mixture (d10-Fl and d12-Per) was spiked into sediment samples prior to the extraction. Fractionation was carried out by using the activated silica gel chromatography column [16–19, 30]. The target PAHs were trapped into the silica gel column, then eluted by using 40/60 (volume/volume) toluene/hexane as the solvent. The pre-concentration was performed by gently purging the eluate with N_2, then changing the solvent to cyclohexane and purging again until the final volume was 100 µL.

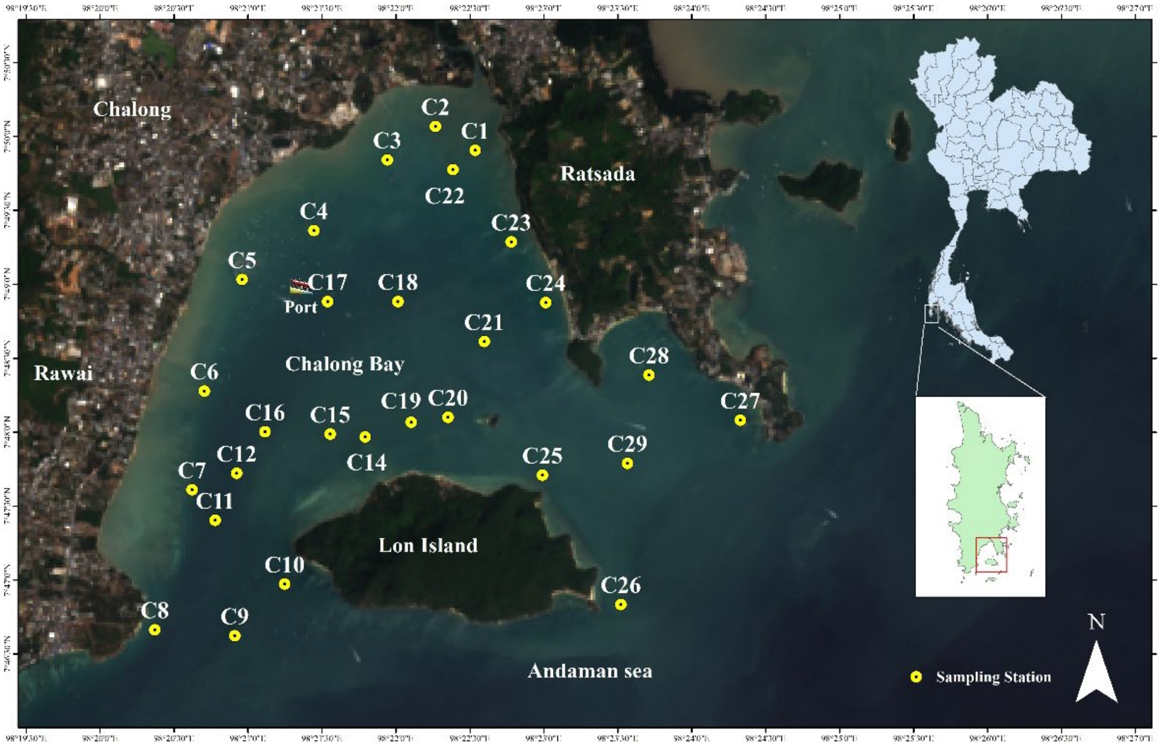

Figure 1 The 28 of sampling stations of surface sediment in the Chalong Bay.

The LECO Pegasus Time of Flight Mass Spectrometer system (TOF/MS) equipped with a 30 m DB-5MS capillary column (0.25 mm *i.d.* x 0.25 µm film thickness) was used to quantify the target PAH compounds. Helium was used as the carrier gas at 1 mL min^{-1} flow rate, 250 °C inlet temperature, 300 ºC ion source temperature, and 320 °C transfer line temperature. The temperature program maintained 40 °C for 1 min, heated at 8 °C min^{-1} to 300 ºC, and maintained this temperature for 45 min. All samples (1 µL) were injected through an injector in the splitless mode. ChromaTOF workstation software collected and interpreted all the data.

4) Quality control

The recovery of extraction and analysis procedure was evaluated using the Standard Reference Material (SRM 1941b). Mean recovery (n=8) was in the range 76–102 %. The precision of the procedure was better than ±15% when calculated as relative standard deviation of duplicate samples. Standardized relative response factor (RRF) between batch runs were calculated. The limit of quantification for PAHs analysis was 0.005 ng g^{-1}.

5) Data visualization and statistical analysis

The spatial distributions were created by using Arc GIS version 10.5. Principal Component Analysis (PCA) was done using IBM SPSS Statistics version 20.0. The data for PCA were arranged in a matrix, with the columns representing concentrations of 16 PAHs in sediment and the rows representing the sampling stations. The variance-normalized data matrix was used to identify the predominant components for implicating the potential PAHs sources. Varimax rotation with Kaiser Normalization was used in the PCA.

Results and discussion

1) Distribution of PAHs in Chalong Bay sediment

The total PAHs concentration ($\sum_{15}$PAHs) is defined as the sum of the compounds in the 15 US-EPA priority standard in ng g^{-1} dry weight. Figure 2 provides the pattern of the $\sum_{15}$PAHs distribution in the Chalong Bay sediment. According to the results, hotspots of PAHs were found at the following sampling stations: the mooring points (C1 and C8), the canal mouth (C2), the municipal discharge pipeline outlet and the hotel/restaurant area (C5, C6 and C7) and the sea port (C10).

The concentration of Nap was excluded from a $\sum_{15}$PAHs calculation because it had the highest concentration among the PAHs [31–32] and so needed to be considered separately. The results showed that Nap in the sediments was only found along the shoreline of Chalong Bay where were the municipal discharge pipeline (C5, C6 and C7) due to its very poor solubility in water. In Thailand, Nap has been widely used in solid block deodorizers in toilets and locker cabinets, so it directly enters the municipal wastewater discharge.

$\sum_{16}$PAHs were in the range from 31.15 to 1696 ng g^{-1} dry weight, with an average of 198.2 ± 318.5 ng g^{-1} dry weight. The level of PAHs contamination in Chalong Bay is comparable to those found previously in other warm water coastal areas, as shown in Table 1. However, the results show that the level of contamination in Chalong Bay was not higher than in other locations that are under strong influences of human activities, such as the Gulf of Thailand [33], the Malaysian riverine and coast areas [34], the Mahakam River in Indonesia [28], the Bhavnagar Coast in Gujarat, India, and the Bohai Sea in China [35]. It can be claimed that the level of contamination is still at safe levels because it is below the Effects Range Low (ERL) for $\sum_{16}$ PAHs (4022 ng g^{-1} dry weight) [36].

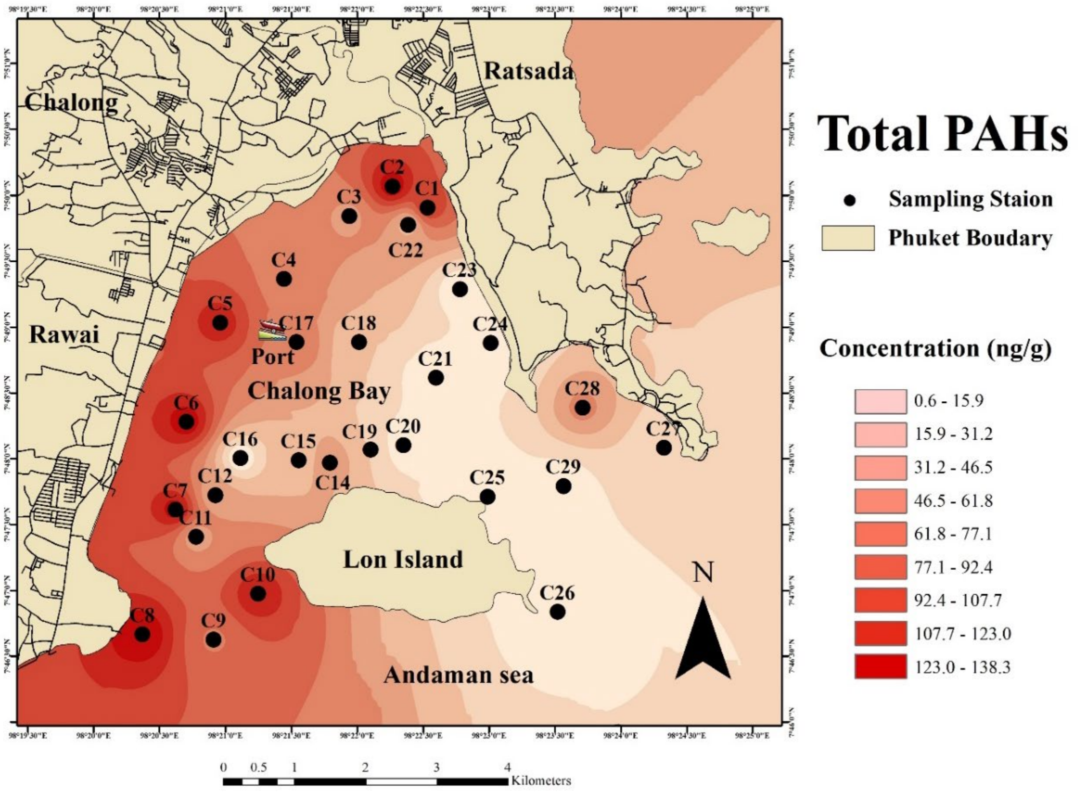

Figure 2 The spatial distribution of $\sum_{15}$PAHs in surface sediment of the Chalong Bay.

Table 1 Comparisons of $\sum_{16}$PAHs in surface sediment of various warm water coastal areas (ng g^{-1} dry weight)

Location	Range	Mean	Reference
Chalong Bay, Phuket	31.15-1696	198.7	This study
Phang Nga Coast	12.6–272	64.5	[19]
Gulf of Thailand	11.05–1992	506.2	[33]
Malaysian Riverine and coast	4.00–924	139	[34]
Mahakam River, Indonesia	54.70–2256	611.1	[28]
Bhavnagar Coast Gujarat, India	5.02–981	345	[29]
Bohai Sea, China	149.2–1211	481.2	[35]

Hong and coworker [9] reported that the numbers of aromatic rings can help identify sources of PAHs. Figure 3 shows the distribution patterns of low molecular weight PAHs (2–3 rings) and of high molecular weight PAHs (4–6 rings). For low molecular weight PAHs, the results show hot spots located at the mooring point for fisherman's boats (C1, C7, and C10), ferry sea port (C5 and C6) and fisherman village (C8). The potential sources from these sampling stations are petrogenic, from leakage of fuel and lubricating oil from maritime vessel activities [37–40]. Otherwise, pyrogenic sources (4–6 rings PAHs) were indicated along the coastal line (C6 and C7), at mooring points for fisherman's boats (C10), at the ferry port (C17) and the canal mouth (C2 and C28), indicating that the sedimentary PAHs came from the combustion of fossil fuels (including ship and automobile exhausts), and from non-point sources such as municipal wastewater discharge, urban street runoff, river, and atmospheric fall-out [18–19].

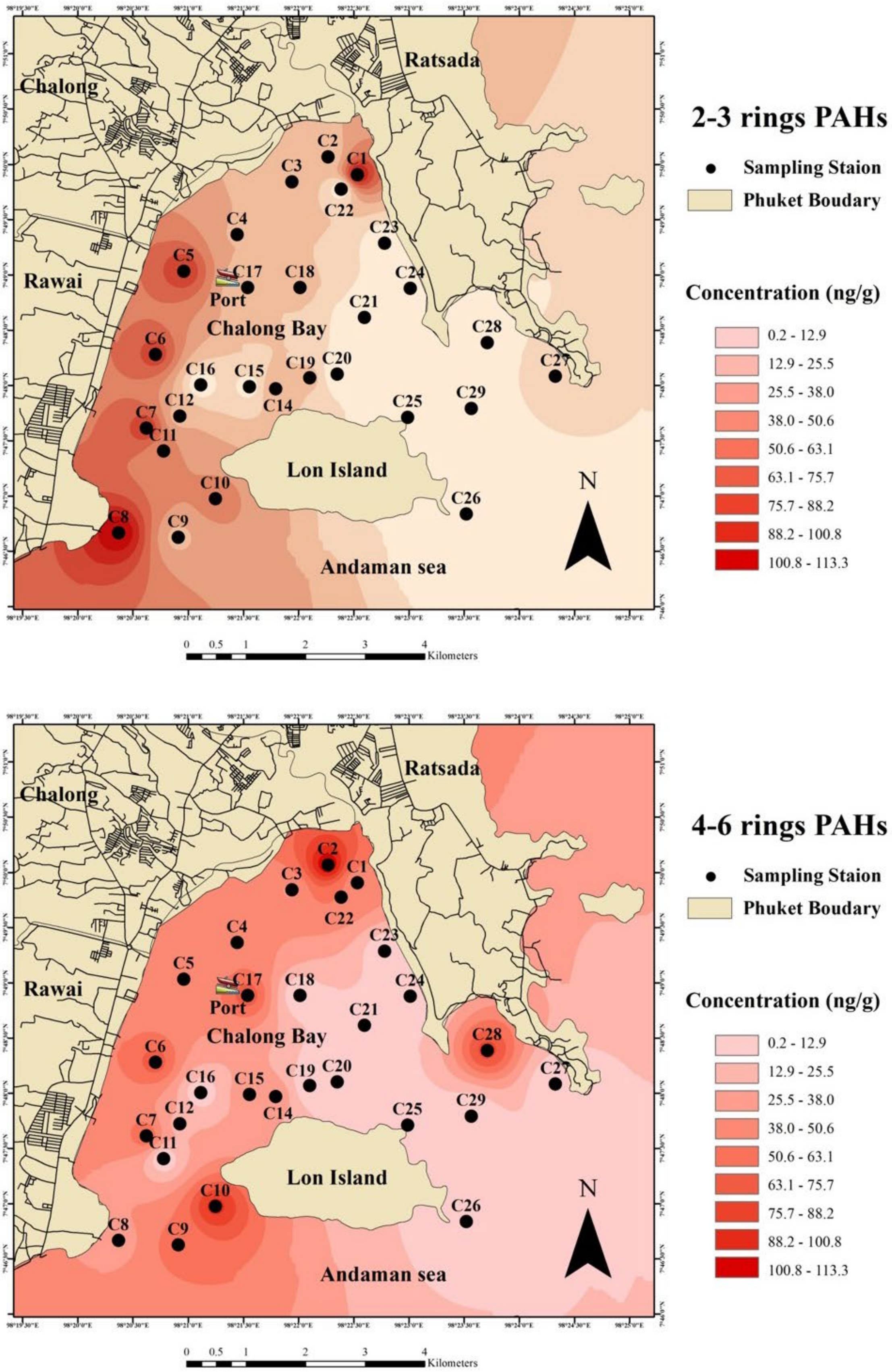

Figure 3 The spatial distributions of 2–3 ring PAHs and 4–6 ring PAHs in Chalong Bay sediment. Sites C2 and C28 are close to canal discharges.

2) Identification of sources by diagnostic binary PAH isomer ratios

Cross-plots of the binary ratios An/(An+Phe) and Fluo/(Fluo+Pyr) and Ind/(Ind+B[g,h,i]P and Fluo/(Fluo+Pyr) were used to identify the potential sources of PAHs in the Chalong Bay sediment. Based on differences in PAH isomer ratios, that are generated by combustion at various temperatures, these can be used to identify the potential sources for PAHs in sediment [5–8]. Figure 4 shows that the PAHs in sediment samples in this study were from mixed sources, and can be attributed to three types of potential sources, namely petrogenic, vehicular combustion, and pyrogenic sources. Figure 4a indicates petrogenic sources for the stations C1, C15, C16 C17, C24, C25, and C29, related to the shipping activities in Chalong Bay. In Figure 4b, a similar assessment indicates that some contamination originated from petrogenic sources (C1, C15, C16 C17, C25, and C29), while vehicular combustion sources were implicated at C9, C10, C11, C18, C19, C20, C22, C26 and C27. Finally, pyrogenic sources were implicated at C2, C3, C4, C5, C6 and C7, which are located along the northern part of Chalong Bay and Mudong canal mouth. These pyrogenic sources feed municipal wastewater discharge, urban street runoff, river, and petroleum combustion, as discussed in the previous section.

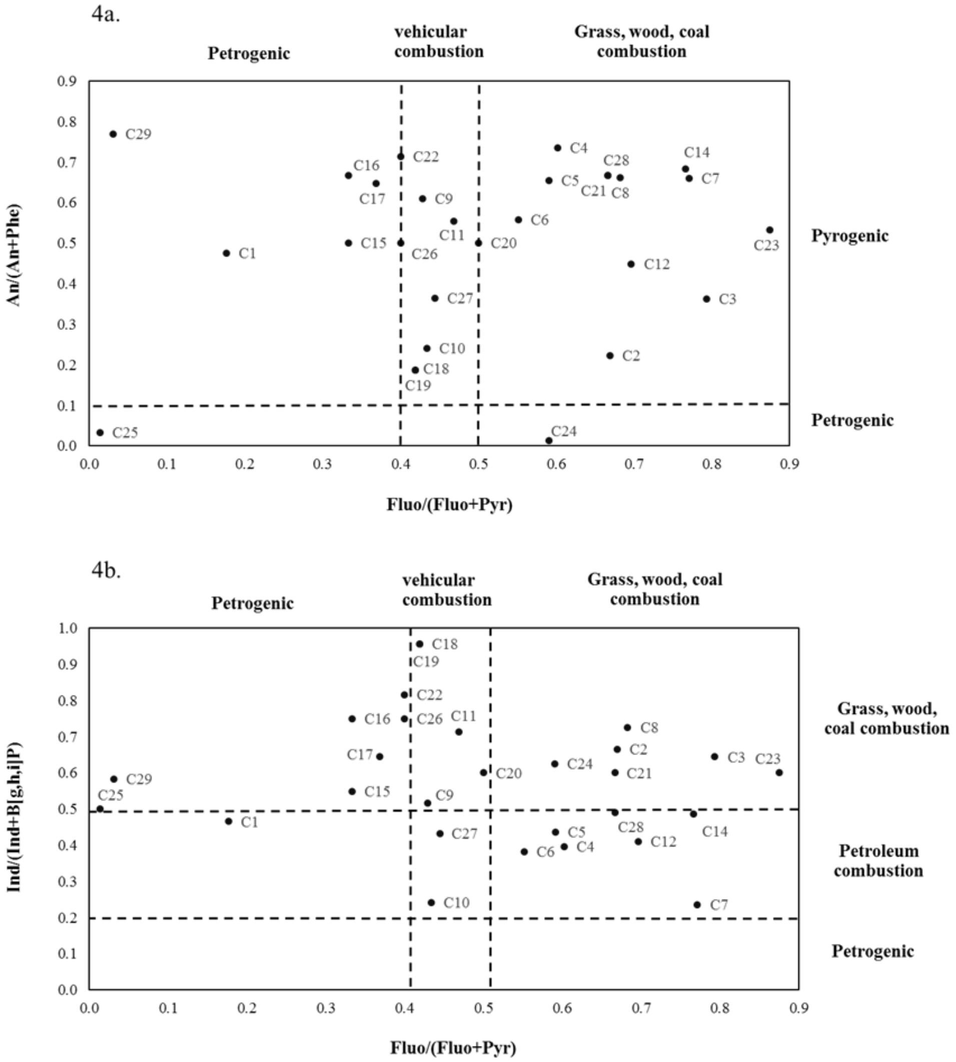

Figure 4 Cross-plots of the PAHs ratios: a) Fluo/(Fluo + Pyr) and An/(An + Phe), and b) Fluo/(Fluo + Pyr) and Ind/(Ind + B[g,h,i]P).

However, once the potential sources of PAH originated from the combustion process, then emitted into the air, they are bound with particles and aerosols and fall down into the water column and then sink down to settle in the sediment. Almost all of the PAHs came from non-point sources such as street run-off and they have the potential to mix with point sources such as petroleum leaking. Hence, these PAHs ratios are not authoritative enough to be exact pin-pointers of source; they only provide a rough idea for source-distinction. More techniques like PCA would be needed to more properly distinguish sources.

3) Using principal component analysis (PCA) to identify the relative importance of different PAH sources in Chalong Bay sediment

PCA is widely used to reduce large data sets to a few components, and here it makes it easier to understand the potential sources of PAHs in Chalong Bay. In our PCA the concentrations of 16 US EPA priority PAHs were the active variables and the 28 sampling stations were the (study) sites. Four principal components in the normalized data (PC1, 2, 3 and 4) explained the majority of the variance (82.3%): PC1 explained 26.7% of the total variance, PC2 25.8%, PC3 19.1%, and PC4 accounted for 10.7%. PC4 was heavily weighted by Nap, and the unique pattern of Nap distribution in the sediment is discussed in the section on "Distribution of PAHs in Chalong Bay sediment". From the total variances the potential sources of PAHs fall into three types, with similar amounts of sampling stations (PC1: 26.7%, PC2: 25.8%, PC3: 19.1%). These results clarify the distribution of PAHs better than the diagnostic binary PAH isomer ratio results. Table 2 shows the rotated component matrix of 16 PAHs from Chalong Bay sediments for three dominant principal components. Figure 5 shows the 3-dimensional rotated loading plot of the composition of 16 PAHs, which can distinguish the potential sources of PAHs into 3 groups.

Table 2 Rotated component matrix of 16 PAHs from Chalong Bay sediments[a]

Component PAHs	**Principal components**		
	PC1	**PC2**	**PC3**
Nap	0.30	0.03	-0.16
Acy	0.05	-0.04	**0.90**
Ace	0.20	0.02	**0.95**
Fl	0.33	0.14	**0.83**
Phe	**0.85**	0.07	0.05
An	**0.76**	0.13	0.30
Fluo	**0.83**	0.06	0.28
Pyr	**0.65**	0.25	0.36
B[a]A	**0.95**	0.15	0.01
Chry	**0.77**	0.25	0.19
B[b]F	0.13	**0.88**	-0.05
B[k]F	0.28	0.60	0.42
B[a]P	0.19	**0.90**	-0.03
Ind	0.08	**0.88**	0.15
D[a,h]A	0.10	**0.82**	-0.08
B[g,h,i]P	0.04	**0.73**	0.25
Variance (%)	26.7	25.8	19.1
Estimated Sources	Urban street run off / wastewater discharge	Vehicular combustion	Petrogenic

Remark: [a] Rotation Method: Varimax with Kaiser normalization

[b] Bold loading >0.65

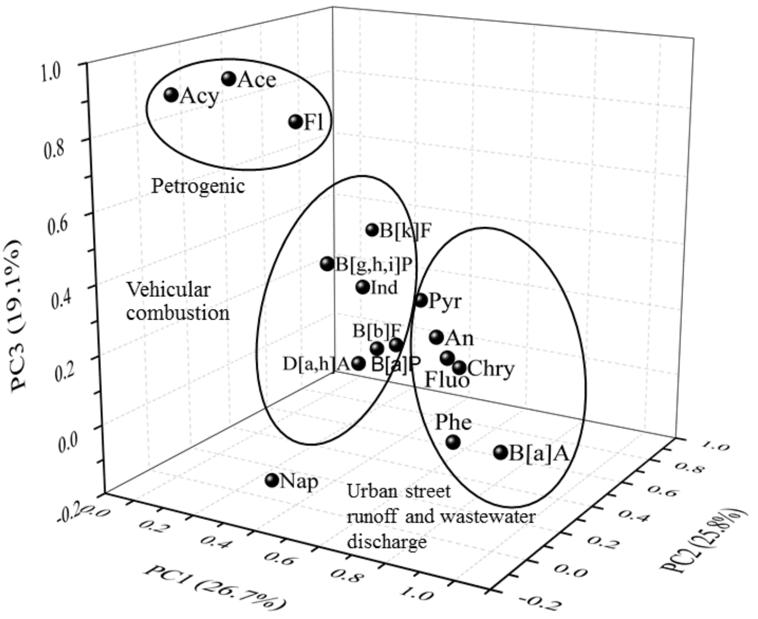

Figure 5 The 3D loadings plot of sedimentary PAHs in Chalong Bay.

PC1 was heavily weighted by Phe, An, Fluo, Pyr, B[a]A and Chry, which are 3–4 ring PAHs. Some studies have found that high loadings of B[a]A and Chry originated from diesel combustion [41–42]. High loadings of Pyr and Fluo result from the incomplete combustion of fossil fuel [43]. Wang and other researchers [44–45] also found that high loading of Phe was a proxy for petroleum contaminated wastewater and street runoff. Additionally, Adeniji et al. [25] state that high loadings of Fluo and Chyr were generated from stormwater runoff and domestic wastewater discharge. In summary, PC1 was mainly associated with urban street runoff and municipal wastewater discharges.

PC2 had heavy weightings on B[b]F, B[a]P, Ind, D[a,h]A and B[g,h,i]P (5–6 ring PAHs). Harrison et al. [46] and Larsen and Baker [47] have concluded that high loadings of B[b]F/B[a]P and Ind/B[g,h,i]P originated from automobile exhaust (both gasoline and diesel engines). Hao et al. [48] reported that high molecular weight PAHs are generated from gasoline combustion. Thus, PC2 implicated vehicular combustion sources.

Finally, PC3 was only associated with low-molecular-weight PAHs (2–3 ring) namely Acy, Ace, and Fl, and this PAHs group implicates leakage of fuel and lubricating oil from maritime vessel activities (non-combustion) in the Chalong Bay. [39–40, 49–50]

Moreover, Figure 6 presents the three-dimensional scores plot of Chalong Bay sedimentary PAHs. It confirms that the potential sources of PAHs in Chalong Bay were pyrogenic and petrogenic sources, and mixed pattern sources. In Figure 5, the PAH sources fall into 3 clusters. The location of each type of PAHs sources was used to implicate the distribution of sedimentary PAHs at each of sampling station of the study area. On comparing the groups in loading plot and score plot, it is found that C2, C4, C5, C6 and C7 located along the upper shoreline of Chalong Bay were most affected by urban street runoff and municipal wastewater discharges (PC1). At stations C1, C8, C10, C15 C18, C19 and C29 there were heavy contributions from petrogenic sources (PC3). Finally the center group of stations was dominated by vehicular combustion sources, especially from maritime vessel activities (PC2). These findings also agree with the diagnostic binary ratio results.

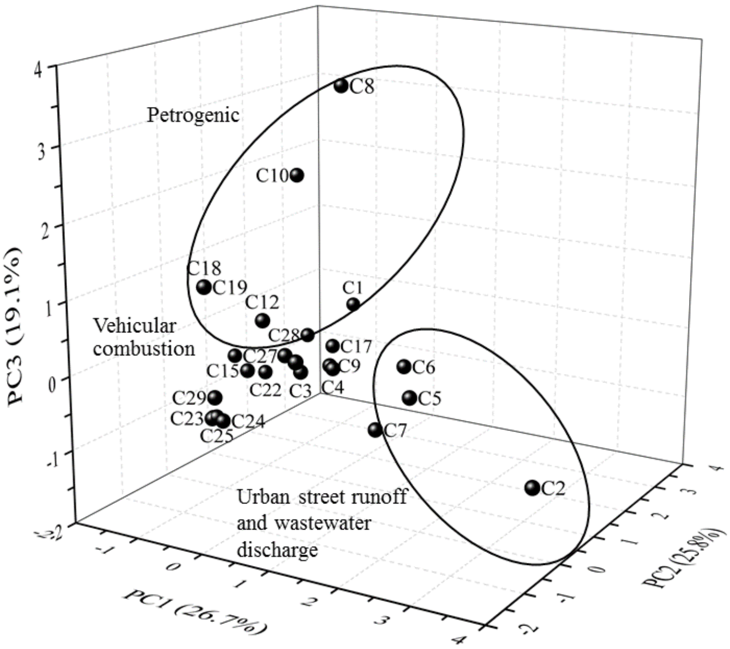

Figure 6 The 3-D scores plot of sedimentary PAHs in Chalong Bay.

Conclusions

The 16 US-EPA priority PAHs were quantitatively analyzed in Chalong Bay sediment. The level of $\sum_{16}$PAHs in sediment ranged from 31.15 to 1696 ng g^{-1} dry weight, these levels of contamination are still at safe levels because even the highest figures are still below the Effects Range Low (ERL). The distribution patterns of low molecular weight PAHs (2–3 rings) show hot spots located at the mooring point for fisherman's boats, the ferry sea port and the fisherman's village which originated from leakage of fuel and lubricating oil from maritime vessel activities. Otherwise, the high molecular weight PAHs (4–6 rings) were indicated along the coastal line at mooring points for fisherman's boats and at the canal mouth, indicating that PAHs came from the combustion of ship and automobile combustion, and from non-point sources such as municipal wastewater discharge, urban street runoff and river pollution. The diagnostic ratios of PAH isomer pairs also provided the information that the most of sampling stations released from potential mixed sources. Moreover, PCA was used to confirm the potential sources of PAHs, which fell into three main types (excluding Nap) namely petrogenic, vehicular combustion, and urban street runoff/municipal wastewater discharge. From results, the source and distribution of PAHs can be explained in terms of the anthropogenic activities would be expected to release PAHs into the study area. These experimental results will be useful for effective management of the maritime activities in Chalong Bay to improve the marine environment.

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