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*Full Length Research Paper*

# Distribution and source identification of polycyclic aromatic hydrocarbons in sediments of the Ambarlı Port area, Sea of Marmara, Turkey

S. Ünlü<sup>1\*</sup>, E. Sari<sup>1</sup>, R. Apak<sup>1</sup>, N. Balcı<sup>2</sup> and B. Koldemir<sup>3</sup>

<sup>1</sup>Istanbul University, Institute of Marine Sciences and Management, 34116 Istanbul, Turkey

<sup>2</sup>Department of Geological Engineering, İstanbul Technical University, Ayazağa, Turkey

<sup>3</sup>Maritime Transportation and Management Department, İstanbul University, Avcılar, Turkey

\*Corresponding Author E-mail: [su@istanbul.edu.tr](mailto:su@istanbul.edu.tr); [vsunlu@gmail.com](mailto:vsunlu@gmail.com); Tel: +90(212)5282539; Fax: +90(212)5268433

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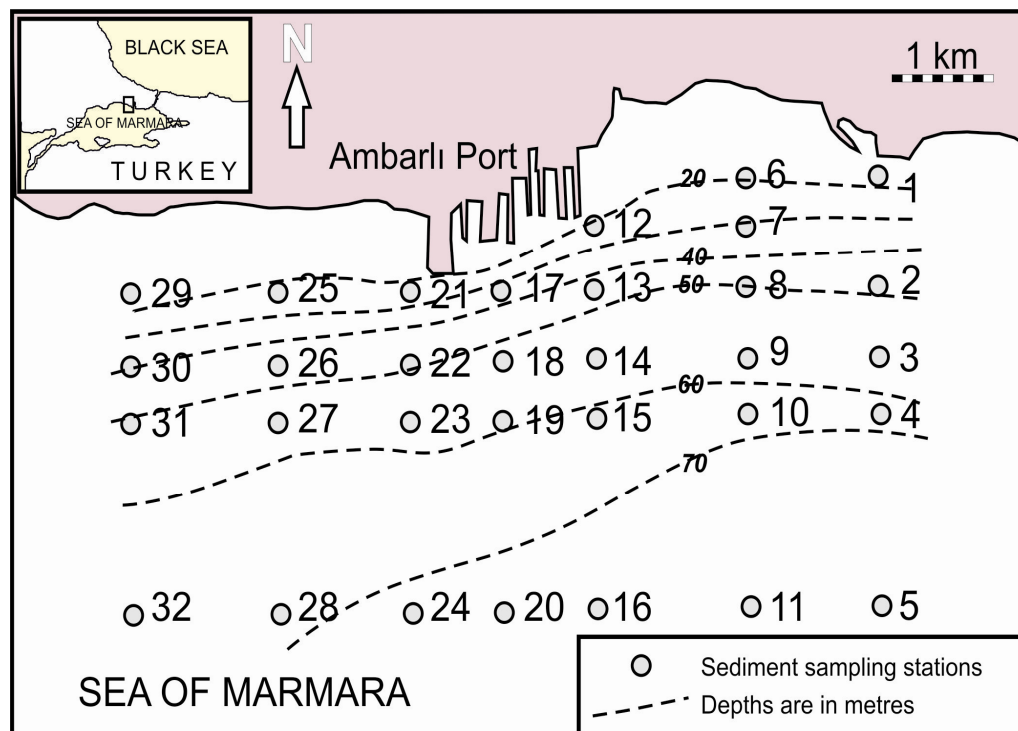
Surface sediments were collected from 32 sites in the Ambarlı Port located in the northern coast of the Marmara Sea and analysed for 16 polycyclic aromatic hydrocarbons (PAHs) listed as the priority pollutants by the US EPA. Total concentrations of the PAHs ranged from 56 to 3685 ng/g, dry weight, and the highest values were recorded at the coast locations, and the anchoring areas. Source analysis using the isomer ratios suggested that the PAHs in the Ambarlı Port area sediments were derived to a mixture of the pyrolytic input coming from anthropogenic combustion processes such as industry, vehicles and heating processes. Further, the carcinogenic PAH levels at the near shore and anchoring areas around the Ambarlı Port were exceeded the effect range low (ERL), and could cause the acute biological impairments when compared to the sediment quality guidelines.

**Keywords:** Polycyclic aromatic hydrocarbons, Surficial marine sediment, Ambarlı Port, Diagnostic ratio, Sediment Quality Guidelines.

## INTRODUCTION

Polycyclic aromatic hydrocarbons (PAHs) as one of the typical persistent organic compounds (POPs) have been featured in the regional and global cycling. PAHs are emitted mainly into the atmosphere, and they have been detected at long distances from their source (McVeety and Hites, 1988). Because of their low vapor pressure, compounds composed of five or more aromatic rings are mainly adsorbed to airborne particulate matter, such as fly ash and soot (Blumer, 1975; Sicre et al., 1987). PAH compounds are strongly sorbed onto the surface of particles associated with the organic compounds of solid

phase matrix and can be deposited to the underlying sediments (Kim et al., 1999). Therefore, the investigation of PAH concentrations in aquatic environments is needed to provide important information on anthropogenic impact on the environment and serve as an indicator of contaminant loading. Anthropogenic input has been regarded as the main source for the elevated concentration of PAHs in the environmental matrix. There are two types of anthropogenic sources of PAHs: petrogenic and pyrogenic. Petrogenic sources include crude oil and petroleum products. Pyrogenic sources



**Figure 1.** Location map of the studied samples from the Ambarlı Port Area.

result from the incomplete combustion of organic matter in industrial operation, riverine discharge, shipping activities, garbage incinerators, power plants, vehicle engines and forest fires (Azevedo et al., 2002; Yunker et al., 2002; Mostafa et al., 2003). The following: important sea pollutants originated from ship and seaport are as oil-products, crew-disposals (i.e., shower, sink and toilet), bilge water, ballast, washing water for ballast, liquid-, and solid type poisonous materials carried by the ships and garbage of the ships.

The study area, Ambarlı Port, is situated on the European side of Istanbul City located in the northern coast of the Marmara Sea, which has an oceanographical link between the Black and Aegean Sea via Istanbul (Bosphorus) and Çanakkale (Dardanelles) Straits (Figure 1). It has also an important position as a seaport for the city of Istanbul. The Port handles a large portion of Turkey's foreign trade, holds 38% of the container movements in Turkey. It serves to the ships such as container-, cargo-, RoRo- and tanker types. The usage ratio of the seaport has been reported as the totally 4959 ships in 2007 (Sarı et al., 2011). The Ambarlı Port area has the fuel oil storage tank areas, a diversified number of industries, heavy vehicles traffic and an intense agricultural activity in its surroundings. Industrial harbor areas are well known for their high environmental potential risk because of high flow of dangerous and potentially harmful materials. Future development and continued operation of Ambarlı Port is of great economic

importance to the Marmara region, but these activities may also impact its ecological functioning.

The objectives of this study were (1) to determine the distribution of PAHs in sediments, (2) to identify potential sources of PAH pollution, (3) to assess the toxicological significance on the marine ecosystem of these contaminants, and (4) to generate the baseline information on the PAH pollution status of Ambarlı Port and its nearby open-coast area.

## MATERIAL AND METHODS

### Sampling

Sediment samples were collected from the selected locations in the Ambarlı Port and its nearby open-coast area using a van Veen type grab sampler in May 2009 (Figure 1). All samples were frozen to  $-20^{\circ}\text{C}$  immediately after collection till analysis.

### Analytical methodology

Collected sediments were homogenized and subsampled for the determination of water content, grain size analysis and extraction of organic compounds. Extraction of samples with a Soxhlet apparatus, cleanup procedure and analyses by gas chromatography coupled to mass

spectrometry (GC/MS) were given in detail by Ünlü and Alpar (2006).

The extracted compounds were determined by a Gas Chromatography/Mass Spectrometry (GC/MS, Finnigan (Thermo) Trace DSQ) with a HP- 5MS capillary column (50m x 0.32mm i.d: 0.25 µm of film thickness). The GC oven temperature program was maintained at 50°C for 1 min, from 50 to 320°C at 10°C/min and then held at 320°C for 5 min. The carrier gas was helium at a constant flow rate of 1.0 mL/min. The GC/MS was calibrated for the 16 priority pollutant PAHs using the internal standard calibration procedure described in US EPA method 8000 (PAH calibration mixture purchased from Dr. Ehrenstorfer, Augsburg, Germany). The following 16 USEPA priority PAHs were analyzed: naphthalene (Na), Acenaphthene (Ace), Acenaphthylene (Acy), phenanthrene (Phe), anthracene (Ant), Fluorene (Fl), fluoranthene (Flu), pyrene (Py), benz[a]anthracene (B[a]A), chrysene(Ch), benzo[b]fluoranthene (B[b]F), benzo[k]fluoranthene (B[k]F), benzo[e]pyrene (B[e]P), benzo[a]pyrene (B[a]P), benzo[ghi]perylene (B[ghi]P) and indeno[1,2,3-cd]pyrene (IP).

Quality control for the PAHs analyses was carried out by monitoring the recovery of surrogate standards (Ultra Scientific) (anthracene-d<sub>10</sub>, phenanthrene-d<sub>10</sub>, chrysene-d<sub>12</sub> and perylene-d<sub>12</sub>). Concentrations of all analytes were corrected for the surrogate recoveries. Recoveries of PAHs in the analytical procedure were between 80% and 105%. Reproducibility of analyses was tested by two replicate analyses of sediment extracts. The relative standard deviation of replicates ranged from 2.4% and 15. Method detection limits for individual PAHs were between 1 and 5 ng/g, dry wt.

The grain size analysis of the fraction <63µm was performed using a Micromeritics Sedigraph 5120 instrument (Molinarioli et al., 2000). Wet sediment samples were placed in an oven 105°C and heated to a constant weight. The water content of sediments was calculated by the weight difference before and after heating. The organic carbon content of sediment samples were measured by a Carlo Erba NC2500 model CHN analyzer after removing the inorganic carbonate fractions.

## RESULTS AND DISCUSSION

The concentrations of 16 individual PAHs, sum of 16 PAHs, TOC(%) and fine fraction in sediments from the Ambarlı Port area are summarized in Table 1. The sediment PAHs in the port area increased slightly with organic carbon content fraction (0.4-3.8%, average 1.4%), while the distribution of total PAHs was not consistently associated with a specific silt/clay ratio (0.3-1.0%, average 0.6%). The distribution of PAHs in the sediments has been affected by chemical composition of the sediments such as organic matter and clay content. For instance, sediments with more fines and high total

organic carbon (TOC) generally have higher contaminant concentrations than sandy and low TOC sediments (Horowitz, 1991). In the present study the regression analysis was performed to investigate the relationship between the concentration of ΣPAHs and the percentage of organic matter and fine fraction. Only there is a slight relationship between the elevated level of PAHs and corresponding organic carbon content. The distribution of sediment PAHs in the port area was not consistently associated with sediment texture, while a meaningful correlation was existed with silt/clay ratio ( $r^2=0.47$   $p>0.06$ ).

Each sample was analyzed for its content of 16 PAHs and the sum of 6 low molecular weight LMW PAHs (i.e., Na, Ace, Acy, Phe, Ant and Fl) and 10 high molecular weight HMW PAHs (i.e., Flu, Py, B[a]A, Ch, B[b]F, B[k]F, B[e]P, B[a]P, B[ghi]P and IP). The concentrations of Σ<sub>16</sub> PAHs were in range of 56-3685 ng/g, dry weight, in sediments from the Ambarlı Port area (Table 1). As given in Table 1, the higher concentrations (>1000-5000 ng/g) were also observed in coast and inner Port Area samples (i.e., stations 1, 6, 8, 12, 17, 21 and 27). Highly polluted sediments coincide to the nearshore areas with high anthropogenic input coming from the marinas, the local fuel storage terminal and anchoring areas scattered in and around the Ambarlı Port. The comparison with the ΣPAH levels reported from different harbours in the world is given in Table 2. On the basis of classification adapted by Baumard et al. (1998), the sediments from the Ambarlı Port area can be considered low to high polluted with PAHs. Only two stations (e.g., 11 and 19) had values of ΣPAHs <100 ng/g indicating of low pollution, whereas the values higher than 1000 ngg<sup>-1</sup> were reported for the samples corresponding to chronically polluted industrialized areas and harbors (Table 2). In the Port Area sediments, the dominant PAH compounds were B[b]F>Ch>B[e]P>Phe >B[k]F>Fl; totally accounted for 68% of the grand total (Table 2). B[b]F, Ch, B[e]P and B[k]F mainly originated from high temperature combustion such as vehicular emission, atmospheric particles and urban aerosols (Muel and Saquem, 1985; Sicre et al., 1987; Mastral and Callen, 2000). The total contribution of high molecular weight PAHs (4–6 ring) was 68%; as 4- and 5-ring PAHs contributed 25% and 39%, respectively (Table 3). This might indicate a consistent source, predominantly pyrogenic (combustion derived) PAH inputs into the study area. However, it should be noted that volatilization or transformation of PAHs in sediment might change the source profiles significantly. For instance, LMW<sub>PAHs</sub> were predominant in diesel fuel (Mi et al., 2000), because of their high volatilization, their composition might have been reduced in sediments. Indeed, Naphthalene, the simplest low molecular weight PAH, accounted for 5% of the total PAHs in the present study. The sum of 2- and 3-ring aromatic compounds varied from 2 to 415 ng/g with an average of 55.2 ng/g while the sum of 4- to 6-ring

**Table 1.** Depth, grain size distribution, total organic carbon (TOC) and total polycyclicaromatic hydrocarbon ( $\Sigma$ PAH) content (ng/g, dry wt) for the sediments from the Ambarlı Port Area

Site	Depth (m)	Sand (%)	Silt (%)	Clay (%)	TOC (%)	$\Sigma_{16}$ PAHs
1	11	50.2	15.2	18.8	0.9	3638
2	50	2.1	39.3	58.7	1.3	742
3	58	3.4	41.4	55.1	1.6	538
4	64	17.1	32.7	49.7	1.8	494
5	78	2.9	38.5	58.6	2.1	879
6	18	5.7	33.8	60.5	2.3	2309
7	38	2.8	43.3	53.8	1.7	106
8	50	4.3	41.7	53.8	1.0	3685
9	57	41.2	20.4	35.1	0.8	334
0	71	3.6	36.5	59.8	0.4	239
11	77	3.0	36.3	60.7	1.0	56
12	27	0.4	49	50.6	2.0	3022
13	46	0.5	35.2	64.2	2.8	247
14	57	13.1	27.2	59.5	1.4	375
15	63	28.0	22.1	49.0	1.4	626
16	76	2.7	40.1	57.0	2.7	595
17	36	4.9	40.2	54.3	1.8	2565
18	55	15.7	30.2	54.1	0.7	870
19	61	26.5	15.2	57.9	1.2	83
20	74	13.4	33.2	52.8	1.0	142
21	29	0.9	33.2	65.9	1.6	1013
22	50	4.1	40.4	55.1	1.1	356
23	60	15.3	27.1	52.9	2.5	700
24	73	6.2	38.6	55.3	1.2	257
25	18	75.6	5.2	8.1	1.2	205
26	46	1.9	35.9	62.2	3.8	422
27	57	2.3	38.1	59.6	1.2	1418
28	69	18	31.7	50.0	0.7	223
29	10	73.4	3.9	7.7	0.5	291
30	42	6.2	39.7	53.7	1.3	267
31	55	4.1	38.9	57.0	3.5	243
32	64	22.3	27.5	49.5	1.1	259

$\Sigma_{16}$ PAHs: Sum of the EPA's 16 PAHs

**Table 2.** Comparison of the total PAH concentrations (ng/g, dry wt) in the sediments from the harbor areas in the world

Location	$\Sigma$ PAH <sup>a</sup>	Contamination level <sup>b</sup>	Reference
Xiamen Harbor, China	2900 – 61,000	High to very high	Hong et al. (1995)
Baltimore Harbor, USA	2944 – 29,590	High to very high	Pereira et al. (1999)
Western Harbor, Egypt	8 – 131,150	Low to very high	Mostafa et al. (2003)
Norwegian Harbor, Norway	2000 – 76,000	High to very high	Oen et al. (2006)
Kaohsiung Harbor, Taiwan	472 – 16,201	Moderate to very high	Chen and Chen (2011)
Ambarlı Port Area, Turkey	56– 3685	Low to high	This study

<sup>a</sup> Concentrations are the reported sums of the measured PAH, which vary for each study

<sup>b</sup> Scale of level of contamination: low: 0–100; moderate: >100–1000; high : >1000–5000; very high:>5000 (cited from Baumard et al. (1998))

**Table 3.** Concentrations of the PAHs in the sediment matrix (ng/g, dry wt)

PAHs	Range		Mean	% of $\Sigma$ PAHs
	Min	Max		
Na	9.4	268.3	42.6	5
Acy	2.0	313	51.7	4
Ace	3.0	163	39.9	3.1
Fl	9.0	415	83.1	6.4
Phe	2.6	343.3	82.2	9.7
Ant	4.9	95.1	34.7	4.1
Flu	1.7	247.9	41.2	4.9
Py	2.1	184	38.3	4.5
B[a]A	0.4	58.5	8.5	1
Ch	1.2	2571.8	132.7	14.6
B[b]F	0.1	1310	244.7	18.9
B[k]F	0.1	542	96.1	7.4
B[e]P	0.1	785	148.1	11.4
B[a]P	3.8	168.5	41.1	1.7
B[ghi]P	13.3	95.6	36.1	0.8
IP	2.9	155.5	35	2.6
$\Sigma$ PAHs	55.9	3684.9	849.8	100
LMW <sub>PAHs</sub>	2.0	415	55.2	32.3
HMW <sub>PAHs</sub>	0.1	2572	82	67.7

LMW<sub>PAHs</sub>:  $\Sigma$  of 2-3 ring PAHsHMW<sub>PAHs</sub>:  $\Sigma$  of > 4ring PAHs

hydrocarbons ranged from 0.1 to 2572 ng/g with an average of 82 ng/g (Table 2).

The sources of pollution are always not unique or with a same pattern. It can be a contribution of different sources such as natural and anthropogenic. It is essential to identify the origin and potential sources of PAHs in the environment for the assessment of the environmental risks. Based on the characteristics of PAH composition and the distribution pattern, the sources of anthropogenic PAHs, which are formed mainly by the combustion processes and released of the uncombusted petroleum products, can be distinguished by the ratios of individual PAH compounds. Six specific PAH ratios were calculated for the studied samples as the following: LMW<sub>PAH</sub>/HMW<sub>PAH</sub>, Phe/Ant and Flu/Py, Ant/(Ant+Phe), Flu/(Flu+Py) and (B[a]A/(B[a]A+Ch)). Budzinski et al. (1997) used the abundance ratios of LMW<sub>PAH</sub> to HMW<sub>PAH</sub> to distinguish between petrogenic and pyrolytic sources of PAHs.

As given in Table 1, the ratios of LMW<sub>PAH</sub>/HMW<sub>PAH</sub> were lower than 1.0 at the most polluted spots (i.e. 1, 2, 3, 5, 6, 8, 12, 15-18, 21 and 27) of the Ambarlı Port area, this indicated that the major input of pyrogenic PAHs was from various combustion-related sources, such as industry, vehicles and heating process, rather than fossil fuel spillages (Laflamme and Hites, 1978). PAHs produced by the combustion, were characterized by the predominance of Flu over Py or Phe over Ant. The ratios of Phe/Ant and Flu/Py in the most sediment samples

were lower than 10 and the sediments from the stations of 2, 4, 6, 9, 13, 19, 20-23, 28, 30 and 32 with Phe/Ant < 10 and Flu/Py < 1, were the characteristics of a mixed pattern of pyrolytic and petrogenic contamination (Budzinski et al., 1997). Also, the results in Table 4 show that the values of ratios Ant/(Ant+Phe) are in the range between 0.08 and 0.74. This presents a dominance of combustion since the ratio values below 0.10 usually indicate the low temperature sources, e.g. petroleum (Budzinski et al., 1997; Yunker et al., 2002; Zhang et al., 2004). The ratios of Flu/(Flu+Py) are in the range between 0.35 and 0.57 with an average of 0.49.

These findings from the stations of 2, 4, 6, 8-10, 13, 16, 20-23, 28, 30 and 32 (Table 4) conform with the results in the range from 0.4 and 0.5 reported by Budzinski et al (1997); Yunker et al (2002) and Zhang et al (2004), and point out the petroleum contaminated samples, the existence of gasoline, diesel and fuel oil combustion. The other stations having ratios of Flu/(Flu+Py) above 0.5 were originated from the coal and biomass (wood, soot, grass, dried dung and forest) combustion. The values of ratios B[a]A/(B[a]A+Ch) ranged from 0.19 to 1.00 with an average of 0.31. Depending on the criteria given by Yunker et al. (2002), which suggested the source might be petroleum (<0.20), petroleum or combustion (>0.20 and <0.35) or combustion (>0.35), our samples show the variable sources, mostly mixed type. In Ambarlı port area, the calculated mass contributions were compared with the source emission depending on the amount of

**Table 4.** Diagnostic ratios for the surface sediments from the Ambarlı Port Area

Site	$\frac{\sum \text{LMW}_{\text{PAH}}}{\sum \text{HMW}_{\text{PAH}}}$	Phe/Ant	Flu/Py	Ant/(Ant+Phe)	Flu/(Flu+Py)	B[a]A/(B[a]A+Ch)
1	0.42	2.27	1.05	0.31	0.51	0.30
2	0.47	2.74	0.84	0.27	0.46	0.28
3	0.82	3.70	1.15	0.21	0.53	0.29
4	1.39	2.53	0.78	0.28	0.44	0.32
5	0.57	2.10	1.17	0.32	0.54	0.30
6	0.38	0.35	0.95	0.74	0.49	0.39
7	1.67	2.01	1.04	0.33	0.51	0.19
8	0.18	6.23	1.01	0.14	0.50	*
9	7.00	2.13	0.88	0.32	0.47	0.30
10	1.88	11.37	0.93	0.08	0.48	0.20
11	1.47	0.87	1.07	0.54	0.52	0.30
12	0.17	0.66	1.17	0.60	0.54	0.26
13	3.18	3.40	0.86	0.23	0.46	0.32
14	3.01	0.93	1.05	0.52	0.51	0.20
15	0.33	0.49	1.18	0.67	0.54	0.36
16	0.60	2.25	1.01	0.31	0.50	0.30
17	0.22	1.28	1.03	0.44	0.51	0.25
18	0.12	3.29	1.03	0.23	0.51	*
19	3.52	0.35	0.55	0.74	0.35	0.29
20	2.03	9.41	0.96	0.10	0.49	0.29
21	0.77	0.46	0.68	0.69	0.40	*
22	1.32	2.60	0.91	0.28	0.48	0.29
23	1.09	1.87	0.82	0.35	0.45	0.33
24	1.18	1.79	1.08	0.36	0.52	0.33
25	9.09	0.66	1.16	0.60	0.54	0.30
26	2.17	2.61	1.06	0.28	0.51	0.28
27	0.27	3.29	1.35	0.23	0.57	0.23
28	10.24	0.63	0.86	0.61	0.46	1.00
29	2.33	5.81	1.22	0.15	0.55	0.24
30	3.10	3.18	1.00	0.24	0.50	0.30
31	2.31	2.08	1.08	0.33	0.52	0.31
32	1.62	1.08	0.89	0.48	0.47	0.32

\*:One of the analytes below (0.001)

LMW/HMW: low molecular weight to high molecular weight, Phe/Ant: phenanthrene to anthracene

Flu/Py: fluoranthene to pyrene, Ant/(Ant+Phe): anthracene to anthracene + phenanthrene

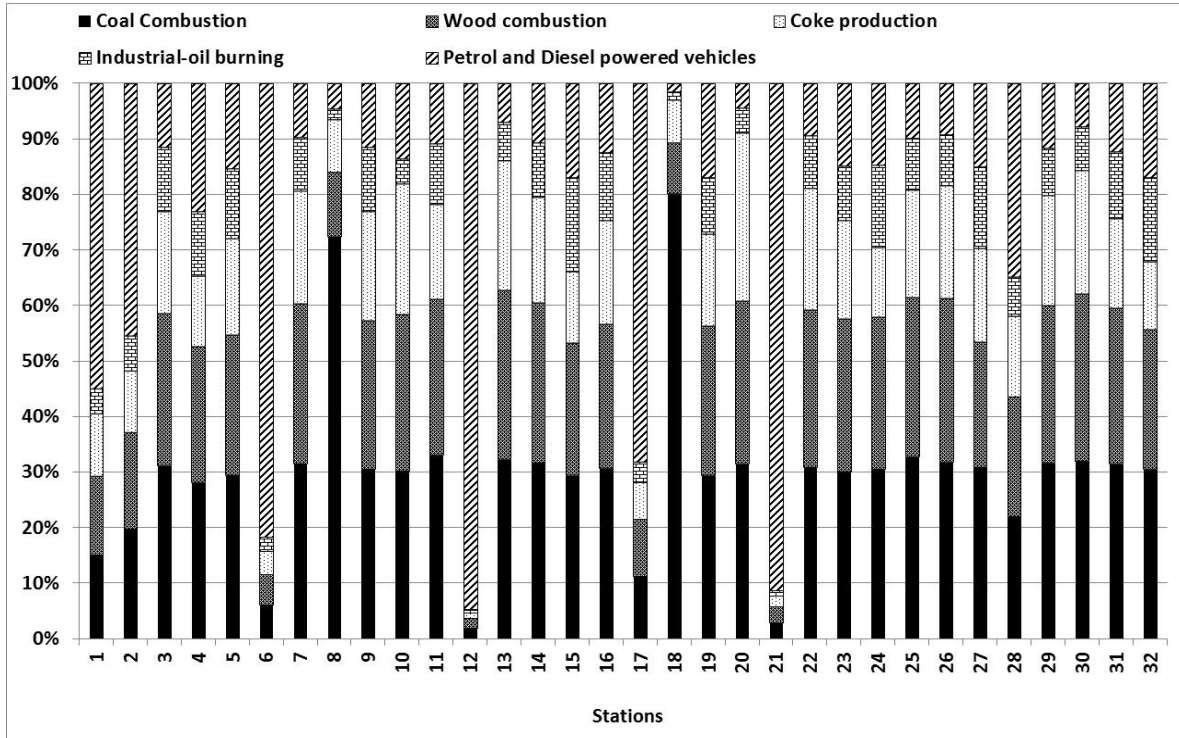
B[a]A/(B[a]A+Ch: benzo(a)anthracene to benzo(a)anthracene + chrysene

emission factors from the literature (Khalili et al.,1995). The distributions of the types of emission sources for PAHs in each sediment sample are shown in Figure 2.

Results in Figure 2 showed that the dominant sources of PAHs in the Ambarlı port area were coal combustion and vehicular emission with a composite contribution of 60% of the total sources. Other sources might be included wood combustion, coke production and industrial-oil burning, with source contribution of 19%, 13% and 8%, respectively. Contributions of burned petroleum sources, heavy traffic, navigation activities in the shipping route in the Port area resulted in the

increase of PAH loadings by vehicle emission. The emission of atmospheric particles enriched with the combustion-derived PAHs might be transported and deposited into coastal sediments.

In summary, the dominant sources of PAHs in the Ambarlı port area were derived to a mixture of the pyrolytic input coming from anthropogenic combustion processes such as industry, vehicles and heating processes rather than from direct petroleum input. To assess sediment quality, PAH levels in the Ambarlı Port sediments were compared with effects-based sediment guideline values (Long et al., 1995). The effect range low



**Figure 2.** Contributions of PAHs sources in the sediments from the Ambarlı Port Area

**Table 5.** Comparison of the measured PAH concentrations with the ERL and the ERM guideline values of selected PAH components in the sediment matrix (ng /g,dry wt).

Compound	This study					Stations (S)	
	ERL <sup>a</sup>	ERM <sup>a</sup>	Mean	Max	n of <ERL	S of ERL-ERM	S of >ERM
Na	160	2100	42.6	268.3	31	9	0
Acy	44	640	51.7	313	26	1, 6,12, 17,21,28	0
Ace	16	500	39.9	163	19	1, 4, 5,6, 8,10,12,17,21,23,28,31, 32	0
Fl	19	540	83.1	415	17	1,2,5,6,8,10,12,14,17,21,23,24,25,28,29	0
Phe	240	1500	82.2	343.3	31	8	0
Ant	85	1100	34.7	95.1	30	1,23	0
Flu	600	5100	41.2	247.9	32	0	0
Py	665	2600	38.3	184	32	0	0
B[a]A	261	1600	8.5	58.5	32	0	0
Ch	384	2800	133	2571.8	30	8,18	0
B[a]P	430	1600	41.1	168.5	32	0	0
Total PAH	4022	44,792	596.3	4828.4	29	1, 8,12	0

a: Long et al.(1995) n: number of sites  
 ERL: Effect range low; dry weight  
 ERM: Effect range median; dry weight

(ERL), and the effect range median (ERM) values of some PAH compounds are listed in Table 5. No samples contained PAHs whose the concentrations exceeded the ERM or frequently pose biological impairments; however,

there was at least one PAH in a site that exceeded the ERL (especially those in the near coast and anchoring area of the Ambarlı Port). The PAHs with potential impacts were naphthalene, acenaphthene,

acenaphthylene, phenanthrene, anthracene, fluorene and chrysene, respectively. An evaluation of ecotoxicological risk according to the ERL/ERM values indicated that 59 % of the analyzed-sediments had the total PAH concentrations between the ERL and the ERM values that could occasionally pose biological impairment. These findings also might be useful to design the future strategies for the environmental protection and management in the Ambarlı Port Area.

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