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Polycyclic Aromatic Hydrocarbons (PAHs) in the Surficial Sediments from Lake Iznik (Turkey): Spatial Distributions and Sources

Selma Ünlü · Bedri Alpar · Kurultay Öztürk · Denizhan Vardar

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Abstract The concentrations of 12 polycyclic aromatic hydrocarbons (PAHs) were determined from 28 sediment samples taken from the Lake Iznik located in the northwest area in Turkey. Total concentration of the PAHs was observed as in the range of 17–835 ng g^{-1} dry weight, with the highest values recorded offshore the cities of Iznik and Orhangazi, and the Sölöz creek. According to the molecular indices, contamination of the PAHs in the lake was a mixture of the atmospheric input of high temperature pyrolytic processes and the petrogenic sources transported by the creeks. Further, the higher proportion of high molecular-weight PAHs (>85%) suggests the domination of combustion-related sources. Compared to the consensus-based sediment quality guidelines for PAHs, there are no harmful biological effects on the short term to aquatic life.

Keywords Contaminated sediment · Pyrolytic · Petrogenic · Pollution sources · Lake Iznik

Lakes are important habitats for many species and play significant roles in the bioaccumulation and biomagnification of organic contaminants such as polycyclic aromatic hydrocarbons (PAHs), organo chlorine pesticide residues and other compounds. After entering the organic contaminants to the environment, the PAHs can accumulate into the lakes through the direct discharges, river inputs, and surface run-off, as by products from the commercial or domestic uses, atmospheric transport and deposition (e.g. from the burning of fossil fuels) in the aquatic sediments. The compositional patterns and diagnostic ratios of PAHs mixtures can provide useful information regarding their sources and transport pathways. There are two types of anthropogenic sources of the PAHs; petrogenic and pyrogenic sources. The petrogenic PAHs are introduced to the aquatic environments through the accidental oil spills, discharge from routine tanker operations, municipal and urban runoff. The combustion of fossil fuel (coal and petroleum) and biomass also produce the pyrolytic PAHs which are released into the environment in the form of exhaust and solid residues (Yunker et al. 2002).

The Lake Iznik, the largest freshwater lake in the southern Marmara region in Turkey, occupies about 313 km^2 area on an active branch of the North Anatolian fault zone (Fig. 1). The water quality of the lake tends towards the eutrophic stage from the mesotrophic stage parallel to the land use profile (Akkoyunlu 2003).

The Lake Iznik is 31.8 km long and 12.0 km wide, and holds about 12 km^3 of water with annual inflow of 132–273 million m^3 by small streams (e.g. Sölöz, Oluk, Karadere and Kiran). The Karsak River discharges its water into the Gulf of Gemlik in the west (Fig. 1). The lake is an important recreational area for swimming, boating and fishing, as well as it supplies water for the industry and irrigation. The historical city of Iznik (Nicaea) is located at the eastern shore of the lake and has been the capital of the Byzantine Empire, the Seljuk Turks and then the Ottoman Empire. The other most important settlements are Boyalıca and Çakırca located in the north, and Narlıca is in the

S. Ünlü (✉) · B. Alpar · K. Öztürk · D. Vardar
Institute of Marine Sciences and Management,
Istanbul University, 34116 Istanbul, Turkey
e-mail: su@istanbul.edu.tr

B. Alpar
e-mail: bedrialpar@gmail.com

K. Öztürk
e-mail: kurultay@istanbul.edu.tr

D. Vardar
e-mail: vardar@istanbul.edu.tr

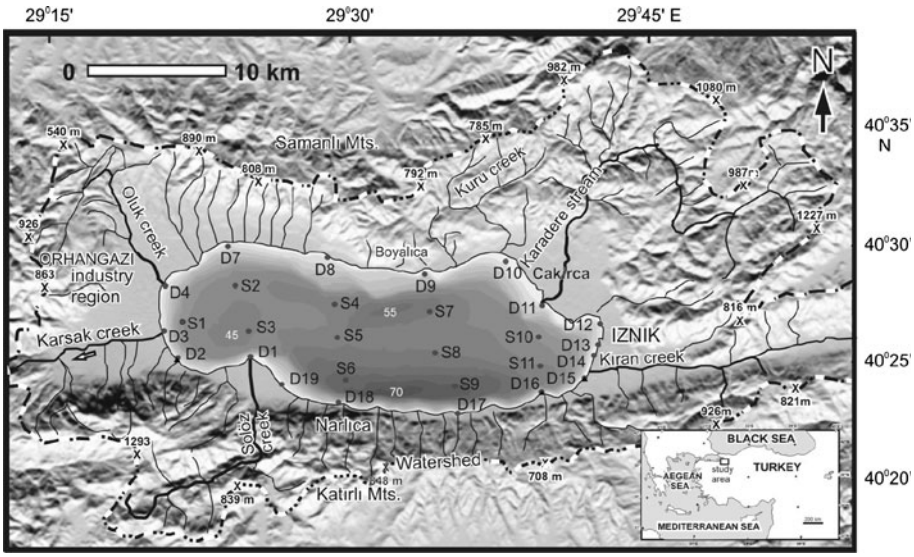

Fig. 1 Map of the bathymetry and sampling stations. Depth contour interval is 10 m. Rivers drain an area of 920 km^2 drawn by the *dashed watershed*

south. More than 24 small-to-moderate scale industrial plants around the town Orhangazi in the west forms the biggest and organized industrial region.

Lake sediments, which are potential sinks for several organic contaminants, have been the subject of numerous scientific researches mostly on geology, geochemistry, paleolimnology, organochlorine pesticide and heavy metal contamination (Budakoğlu 2000; Akkoyunlu 2003; Franz et al. 2007; Ülgen et al. 2007). The PAHs are also of concern due to their toxicity; many of them have carcinogenic properties. Furthermore, the lack of PAH measurements prevents an evaluation of the levels of these pollutants, and status of their transportation and deposition. In the present study, we report the levels of 12 parent PAHs in the bottom sediments of Lake Iznik, providing a baseline data on both the environmental state of Lake Iznik, and spatial distribution and possible origins of the PAHs.

Materials and Methods

A total of 28 surface sediment samples were recovered throughout the lake using a snapper. Extraction of frozen 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). Priority PAH compounds were analyzed by a Gas Chromatography/Mass Spectrometry (GC/MS, Pelkin Elmer, Turbo Mass Spectrometer) with a HP-5MS capillary column (50 m × 0.32 mm 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^{-1} 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 12 priority pollutant PAHs using the internal standard calibration procedure described in US EPA method 8000 (PAH calibration mixture purchased from Supelco, Sigma–Aldrich Corporation). The following 12 USEPA priority PAHs were analyzed: phenanthrene (Phe), anthracene (Ant), fluoranthene (Flu), pyrene (Py), benz[a]anthracene (B[a]A), chrysene + triphenylene (Ch = Chry + Triph), 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-d10, phenanthrene-d10, chrysene-d12 and perylene-d12) prior to solvent extraction. Concentrations of all analytes were corrected for surrogate recoveries. Recoveries of PAHs in the analytical procedure were between 70% and 105%. Reproducibility of analyses was tested by two replicate analyses of sediment extracts. The relative standard deviation of replicates ranged from 2.7% to 17%. The method detection limits for the individual PAHs were between 1 and 5 ng g^{-1} dry wt.

Sediment grain size analysis was performed using a method adapted from Folk (1974) as described by GERG SOP-8908. The organic carbon content of sediment samples were measured by a Thermo Finnigan FLASH EA 1112 model CHN analyzer at the Advanced Analyses Laboratories, Istanbul, after removing the inorganic carbonate fractions, and were replicated within runs and over time with a confidence interval of 0.1%. The analytical precision of analysis was better than ±4% at 95% significance level from five replicates.

Results and Discussion

The minimum and maximum concentration of Σ12 PAHs in the sediments of Lake Iznik were 17 and 835 ng g^{-1} dry weight, respectively (Table 1). The highest values of total PAHs were found in the samples of D11 (835 ng g^{-1}) off the northern part of the city of Iznik, D1 (798 ng g^{-1}) near the Sölöz creek and D4 (691 ng g^{-1}) off the city of Orhangazi and the Oluk creek (Fig. 2). These stations were followed by D2 (301 ng g^{-1}) near the mouth of the Gölyatagi creek and D12 (275 ng g^{-1}) off the central part of the city of Iznik. Besides these highly contaminated locations, other areas were weakly contaminated. On the basis of pollution levels assigned by Baumard et al. (1998), the ΣPAH concentrations in the sediments of Lake Iznik are low to moderate, which are also lower compared to the levels found in sediments from some of the lakes in the United States and China (Table 2).

Variation in the long-term sediment ΣPAH concentration trends may be influenced by sediment characteristics. For instance, the sediments with more fines and high total organic carbon (TOC) generally have higher contaminant concentrations than those of sandy and low TOC sediments (Horowitz 1991). The TOC contents of surface sediments in the Lake Iznik show a range between 0.52% and 5.54% with an average of 2.26% (Table 1). The lowest values were distributed along the eastern coasts as well as between the western and eastern deep basins (<2.5%). The most elevated values were observed at the stations S1, S10, S11, D18, D8, D1 and S2. There was no meaningful correlation between the level of ΣPAHs and sediment organic carbon content.

The changes in moisture and grain size fractions influence the chemical compositions of the sediments. The water content varies between 20% and 68% throughout the lake sediments. The total aromatic hydrocarbon concentrations,

Table 1 Sediment texture, TOC%, sediment water content (SWC%), concentrations of ΣPAHs (ng g^{-1} dry wt), and ratios of LMWPAHs over HMWPAHs in the sediments of Lake Iznik

Station	Depth (m)	Sediment texture	TOC (%)	SWC (%)	ΣPAH (ng g^{-1}dw)	LMW_{PAH}/HMW_{PAH}
S1	39.5	Silt	5.54	63.8	131	0.13
S2	43	Silt	2.80	65.2	76	0.24
S3	46	Silt	2.10	56.1	211	0.02
S4	47	Sandy silt	2.30	60.5	150	0.23
S5	48	Sandy silt	2.37	62.3	112	0.40
S6	63	Silt	1.88	62.2	117	0.18
S7	52	Silt	2.16	63.4	169	0.42
S8	50	Silt	2.60	64.1	96	0.18
S9	68	Sandy silt	2.04	67.9	94	0.12
S10	39	Sandy silt	4.44	59.4	131	0.11
S11	44.5	Sandy silt	4.44	51.4	151	0.19
D1	25	Slightly gravelly sand	3.11	40.0	798	0.40
D2	13	Silty sand	2.20	51.9	301	0.07
D3	6.5	Sand	1.48	35.3	205	0.04
D4	2	Gravelly sand	1.73	31.9	691	0.64
D7	1	Slightly gravelly muddy sand	1.79	61.4	160	0.02
D8	2	Gravelly sand	4.23	27.5	17	0.10
D9	5.5	Gravelly sand	0.72	22.3	36	0.11
D10	4	Sandy silt	1.67	47.4	166	0.25
D11	3	Slightly gravelly muddy sand	1.52	32.9	835	0.40
D12	6.5	Silty sand	1.32	48.9	275	0.18
D13	4.5	Sand	0.74	24.3	24	0.49
D14	3.5	Muddy sandy gravel	0.98	26.5	47	0.21
D15	8.5	Slightly gravelly sand	0.52	25.4	152	0.02
D16	32.5	Silty sand	1.37	42.4	148	0.06
D17	7	Slightly gravelly sand	1.95	28.0	39	0.06
D18	8.5	Silty sand	4.42	55.6	207	0.05
D19	2.5	Gravelly sand	0.57	20.1	73	0.05

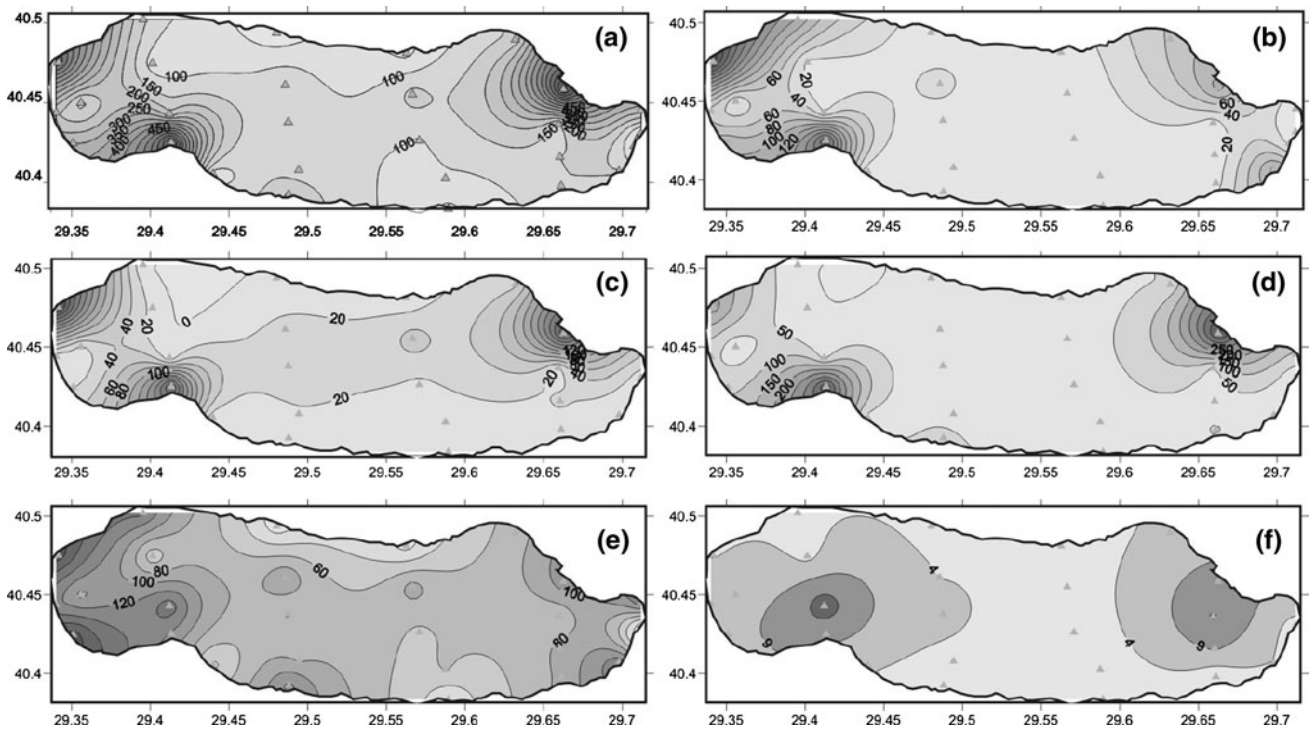

Fig. 2 Spatial distribution of **a** Σ12 PAHs **b** silt/clay ratio **c** 2- 3-ring PAHs **d** 4-ring PAHs **e** 5-ring PAHs **f** 6-ring PAHs

Table 2 ΣPAH (ng g^{-1} dry wt) in lacustrine sediments from Lake Iznik and other locations for comparison

Lakes	n	ΣPAH[a]	Contamination level[b]	References
Michigan, USA	12	538–6,099	Moderate to very high	Christensen and Zhang (1993)
White Rock, USA	19	135–3,440	Moderate to high	Van Meter et al. (2000)
Erie, USA	16	224–5,304	Moderate to very high	Simirnov et al. (1998)
Yuan Dan, China	15	982–1,377	Moderate to high	Ou et al. (2004)
Taihu, China	19	13–963	Low to moderate	Peng et al. (2005)
Laja, Chile	15	77–398	Low to moderate	Quiroz et al. (2005)
Iznik, Turkey	12	17–835	Low to moderate	The present study

[a] Concentrations are the reported sums of the measured PAH, which vary for each study

[b] Scale of level of contamination: low: 0–100; moderate: >100–1,000; high: >1,000–5,000; very high: >5,000 (Baumard et al. 1998). n: Number of PAH compounds analyzed in each study

excluding the highest three samples, slightly increase with the water content ($r^2 = 0.16$). The bottom samples collected in this study exhibit that sand is dominant at the coastal areas (<35 m deep) and especially at the mouth of creeks while the mud (mainly silt) is dominant at all over the lake. The annual sedimentation rate, in fact, is 3.3 mm in the central and northern rim of the lake (Ülgen et al. 2007). The mud is the most reactive fraction of the total sediment for the hydrocarbon adsorption and/or organic–inorganic complex formation. The silt ratios increase up to 75%–90% in the deepest parts as being higher in the western basin, while the clay ratios increase up to 17% in the center of the eastern basin. The silt/clay ratio was high (>20) at the western and eastern rims of the lake (Fig. 2). A significant correlation was found between ΣPAHs and silt/clay ratio ($r^2 = 0.71$) while there was not meaningful relationship for the correlation of ΣPAHs with sediment texture.

The major PAH compounds were as the following: Phe with the low molecular weight (LMW) PAH range (2- and 3-ring), and Ch, B[b]F, B[k]F, B[e]P, B[a]P and B[ghi]P with the high molecular weight (HMW) PAH range. Phe was the dominant compound with the largest range of 1.0–218.0 ng g^{-1} in the sediment samples. IP exhibited the smallest range of 0.3–17.2 ng g^{-1}. The total contribution of HMWPAHs (4–6 rings) was 5.2%; as 4- and 5-ring PAHs contributed as 53.4% and 28.0%, respectively. The average ratio of LMWPAHs/HMWPAHs was approximately 1/5. The total combined contents of

Table 3 Comparison of the measured PAH concentrations with the important guideline values of selected PAH components for sediment matrix (ng g^{-1} dw)

Compound	Mean	SD	Min	Max	ERL[a]	ERM[b]	Consensus-based TEC[c]	Consesus-based PEC[d]
Phe	30.3	57.5	1.0	218.0	225	1,380	204	1,170
Ant	9.6	16.9	0.5	66.7	85	960	57.2	845
Flu	21.9	38.3	1.0	146.7	600	3,600	423	2,230
Py	18.5	42.5	0.4	168.0	350	2,200	195	1,520
B[a]A	10.8	15.8	0.8	56.0	230	1,600	108	1,050
Ch	18.3	25.9	2.4	120.0	400	2,800	166	1,290
B[a]P	15.0	10.4	1.1	37.8	400	2,500	150	1,450
Total PAH	124.4	207.3	7.2	813.2	4,000	35,000	1,610	22,800

[a] Effect range low

[b] Effect range median (Long et al. 1995)

[c] Threshold effect concentrations (below which the harmful effects are unlikely to be observed)

[d] Probable effect concentrations (above which the harmful effects are likely to be observed) (MacDonald et al. 2000)

LMWPAHs varied from 2 to 270 ng g^{-1} with an average of 40 ng g^{-1} while the values of for HMWPAHs were in the range of 15–595 ng g^{-1} with an average of 162 ng g^{-1}. The individual PAH components and ΣPAH concentrations in the sediment samples were compared with the effects range approach (Long et al. 1995) and the consensus-based effect concentration approach (MacDonald et al. 2000) which provide a reliable basis for assessing the sediment quality conditions in freshwater ecosystems. Our results were mostly below the effect range low values and the consensus-based threshold effect concentrations, implying harmful effects were unlikely to be observed (Table 3).

In general, the non-combusted sources such as oil seeps and petroleum spills contain predominantly 2- and 3-ring PAH compounds or petrogenic PAHs, whereas the combustion sources (e.g., vehicle exhaust, domestic heating with coal, forest fires) or pyrolytic PAH compounds contain predominantly more of the HMWPAH species (Irwin et al. 1997). The HMWPAHs are less water soluble, less volatile, and are more persistent than the LMWPAHs. In the study area, the LMWPAHs were relatively dominant at the most polluted spots (i.e. D1, D4, and D11) and, on the W-E trending ridge in the middle of the eastern basin separating the northern and southern troughs (stations S4, S5 and S7). The relative ratios of the 4-ringed PAHs were higher at the stations D1 and D9, while they were low in the western basin (D7, S1-S7 and S9) and also at D16. Five-ring PAHs were dominant at deep stations (S1–S5 and S8–S11) and at two coastal stations (D7 and D15), but not at the most polluted zones. Six-ring PAHs were not dominant throughout the lake except the deep stations of S3, S5, S10 and S11. The increment of carcinogenic PAH concentrations (e.g. B[a]P, B[ghi]P and IP) towards the deep basin might be attributed to atmospheric deposition mostly coming from various combustion sources around the region.

Although the PAH species might be attributed to the specific sources; such as coal combustion, vehicular emission, coking, petroleum emission including oil leakage and the spill during production and transportation, the PAH isomer pair ratios, such as Ant/(Ant + Phe), Flu/(Flu + Py), B[a]A/(B[a]A + Ch) and IP/(IP + B[ghi]P), were applied widely as diagnostic indicators to identify possible PAH sources (Wang et al. 2009). PAHs produced by combustion, for example, were characterized by the predominance of Flu over Py or Phe over Ant. Depending on the criteria suggested by Budzinski et al. (1997), the ratios of Phe/Ant plotted against Flu/Pyr indicated that the sediments with PAHs originated from the pyrolytic origin were dominant throughout the Lake Iznik with some mixed petrogenic input for some coastal stations which were close to creeks around the lake (Fig. 3). Sediments from the stations of D1, D7, D11 and D15 with Phe/Ant<10 and Flu/Pyr<1 were characteristic of a mixed pattern of pyrolytic and petrogenic contamination.

The values of ratios Ant/(Ant + Phe) were in the range of 0.13–0.48 with an average of 0.28, indicating a dominance of combustion due to the fact that the ratio values below 0.10 usually pointed out the low temperature sources, e.g. petroleum (Yunker et al. 2002).

The values of ratios Flu/(Flu + Py) were in the range of 0.29–0.86 with an average of 0.63. The values below 0.50 mostly pointed out the petroleum contaminated samples and gasoline, diesel and fuel oil combustion (Yunker et al. 2002), such as the stations of D1, D7, D11 and D15 (Fig. 3). Depending on this criterion, the remaining samples mostly revealed a contamination due to the combustion of coal, wood and grass. The values of ratios B[a]A/(B[a]A + Ch) were in the range of 0.10–0.75 with an

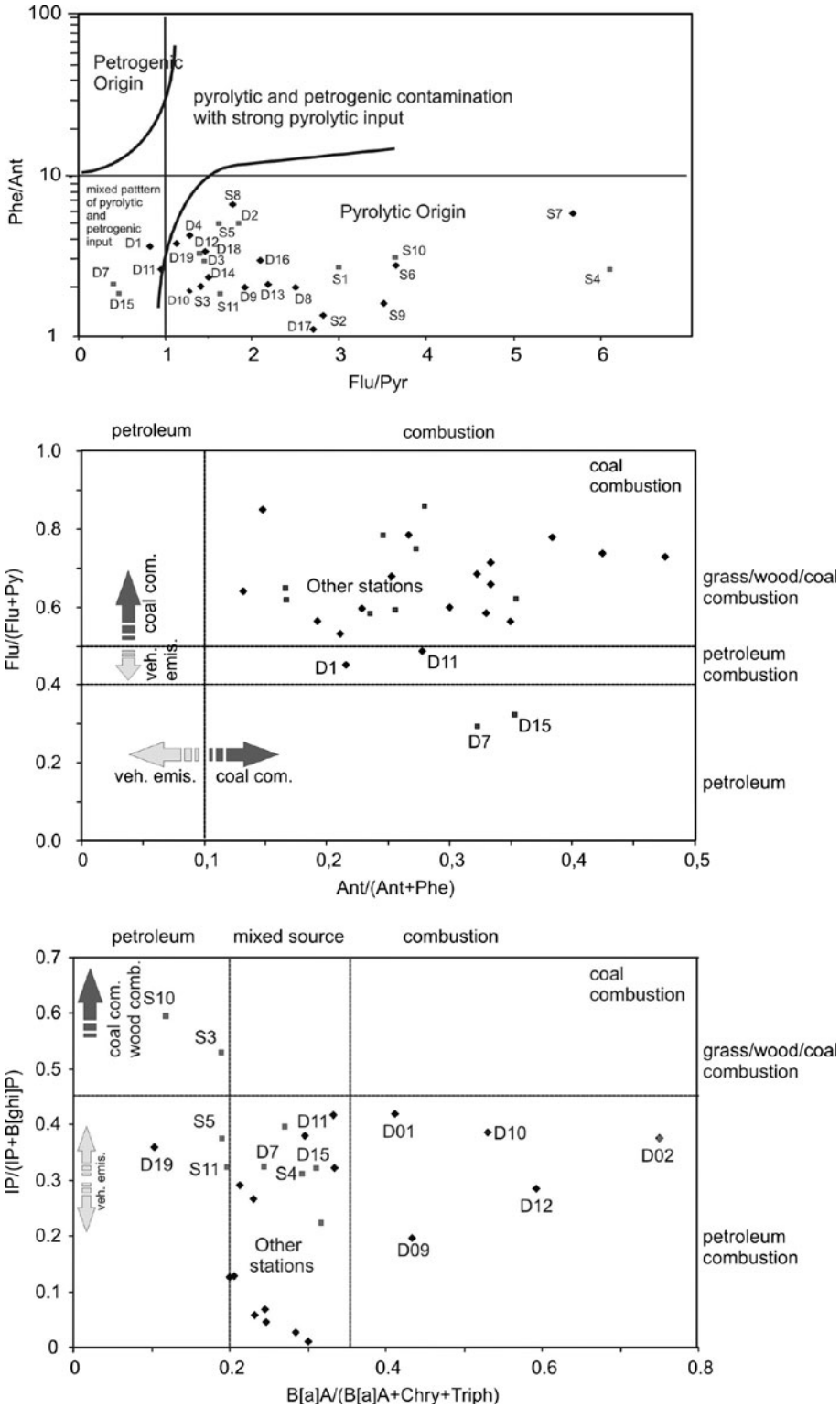

Fig. 3 Relations between some of the ratios of PAH species and indexes used in the estimation of PAH sources for the sediments of the Lake Iznik

average of 0.30. According to the criteria given by Yunker et al. (2002), which described that the source might be petroleum (<0.20), petroleum or combustion (>0.20 and <0.35) or combustion (>0.35), our samples exhibited the variable sources, and mostly mixed type (Fig. 3). The ratios of IP/(IP + B[ghi]P) were in the range of 0.01–0.59 with an average value of 0.28. The ratios lower than 0.50 were observed in the most of studied samples except for S3

and S10. According to the literature data (e.g. Yunker et al. 2002), these samples implied the contamination due to the petroleum combustion and petroleum (Fig. 3).

Even the individual ratios can hardly be used as diagnostic criteria; the applied PAH isomer pair ratios as the diagnostic indicators show a combination of the different sources which change their characteristics place to place. The overall results confirmed that the all of the identified PAHs in the lake sediment samples were originated from the combination of three main sources; (a) unburned petroleum (petrogenic and pyrolitic mixed pattern), (b) atmospheric inputs due to the burned petroleum and liquid fossil fuel oil combustion (vehicular engine emission, heating with coal, petroleum coke particles, gasoline, and diesel), and finally c) biomass burning from various sources (wood, soot, grass, dried dung and forest).

Contribution of the first PAHs source was highly correlated with the main riverine input sources (Sölöz, Oluk and Karadere), and as well as the deepest part of the lake. There were no petrochemical plants in this area. The PAH from unburned petroleum was entered into the water column primarily through urban runoff discharges. Another high anthropogenic concentration was close to a waterfall carrying thermal spring water into the lake (station D8). The secondary source of PAHs contributions was dominant between the western and eastern margins where the industrial zones and big towns were located. Atmospheric particles should act as an input pathway for the PAHs throughout the lake system in E-W direction. This source can be correlated with the petroleum and fossil fuel oil combustions (the coal and coking emission). It covers incineration of municipal and industrial wastes of the towns of Orhangazi and Iznik. The industrial zone in Orhangazi also contributes this source. Pollution from the coal combustion for heating in winter is high in this area. Currently, the coal burned boilers are widely used to provide steam, hot water and heating in factories in the Orhangazi industrial region in the west. Meanwhile, the vehicular emissions around the lake and the activities of fishing and power boats, primarily those with two stroke motors, can also be considered as the possible sources of this main contribution class. Spatial distribution of the third source of PAHs contribution class exhibited higher elevation levels in the southeastern part of the lake and attributed to biomass (wood or grass) burning.

It is believed that the aspect of the present results are related to the environmental management issues; therefore, it can be helpful for the public administrators as well as the scientists in diverse areas.

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