



# Distributions and Sources of PAHs and OCPs in Surficial Sediments of Edremit Bay (Aegean Sea)

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Received: 12 December 2018 / Accepted: 18 March 2019 / Published online: 27 March 2019  
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## Abstract

In this study, levels, distributions, and sources of polycyclic aromatic hydrocarbons (18 compounds) and organochlorine pesticides (19 compounds) in surface sediments of 14 stations from Edremit Bay (Aegean Sea) were investigated in April and November 2015. ΣPAH concentrations (0.65–175 ng/g) in Edremit Bay sediments indicated low pollution. ΣPAH levels were decreased in the order of inner (81.1 ± 47.0 ng/g), northern (48.4 ± 15.9 ng/g), and southern (19.0 ± 15.8 ng/g) bays. *p,p'*-DDE was the only organochlorine pesticide detected in Edremit Bay sediments and found between nd to 1.16 ng/g dw. According to sediment quality guidelines, PAHs and *p,p'*-DDE levels in Edremit Bay were below the threshold effect level, effect range low, and threshold effect concentration limits and the sediments have no potential ecological risks. Two- to 3-ring PAHs were found at higher levels than 4- to 5-ring PAHs. Molecular PAHs ratios and Principal Component Analysis-Multiple Linear Regression analyses indicated combustion of wood-coal and vehicle emissions might have contributed to PAH levels in the bay.

Polycyclic aromatic hydrocarbons (PAHs) and organochlorine pesticides (OCPs) are classes of persistent organic pollutants. PAHs have been added to the priority pollutant lists of the USEPA and EU (USEPA 1993; Nieva-Cano et al. 2001). Benz[a]anthracene (BaA), chrysene (Chr), benzo[b]fluoranthene (BbF), benzo[k]fluoranthene (BkF), benzo[a]pyrene (Bap), and benzo[ghi]perylene (BghiP) are known as potential human carcinogens and show widespread distribution in the environment (IARC 1983; Menzie et al. 1992; Shaw and Connell 1994; Yu 2002, Nadal et al. 2004; Anyakora et al. 2005).

PAHs can be generated from emissions of land and marine vehicles, incomplete combustion of organic materials, combustion of wood and coal, natural combustion processes (i.e., volcanic eruptions, forest fires), anthropogenic

activities, such as transportation and incineration of wastes and sewage sludges, industrial production, petroleum and oil transportation, oil spills and timber operations (Baek et al. 1991; Lorber et al. 1994; Nwachukwu 2000; Nwachukwu et al. 2001; Grova et al. 2002; UNEP 2002; Lourenço et al. 2013; Nguyen et al. 2014; Schintu et al. 2015). OCPs are one of the widespread contaminants introduced mainly from rain run-offs, river discharges, and untreated effluents from agricultural, industrial, and municipal activities. PAHs and OCPs are among the organic pollutants with low water solubility in natural environments, poor or non-biodegradability, high persistence, toxicity, bioaccumulation, and biomagnification properties in aquatic ecosystems (Bastami et al. 2013; Nguyen et al. 2014; Li et al. 2015; Migani et al. 2015; Akhbarizadeh et al. 2016).

Due to their persistence and hydrophobicity, organic pollutants have tendency for adsorption onto particles and accumulate in sediments following their introduction to the marine environment (Chiou et al. 1998; Li et al. 2014; Duan et al. 2015; Schintu et al. 2015). Some factors controlling the availability and deposition of organic contaminants in sediments are textural, chemical, and biological properties of marine sediments, physicochemical properties of pollutants (e.g., solubility, vapor pressure, and lipophilicity), and environmental conditions (e.g., pH and  $E_h$ ) (Zhou et al. 1998; Chakraborty et al. 2015).

**Electronic supplementary material** The online version of this article (<https://doi.org/10.1007/s00244-019-00619-8>) contains supplementary material, which is available to authorized users.

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Edremit Bay (Northwestern Turkey) is one of the important natural bays in the Aegean Sea. The water depth of the bay is decreased from its western part (110 m) to eastern part. North part of the bay is surrounded by mountains with very high slope. Same slope continues into the bay leading to deeper water depths at the north. Main urban settlement around the bay is the city of Edremit and there are many little towns, such as Ayvalık, Gömeç, Burhaniye, Akçay, Güre, Altınoluk, and Küçükkuyu. In this area, touristic activities and human population are increased especially in summer. There are many streams (Karınca, Havran, Kadıncık, Edremit, Zeytinli, Kızılteçili, Manastır, Şahin, Mihli, and Küçükkuyu) with high sediment load capacity around the bay (Yücesoy-Eryılmaz et al. 2005). Edremit and surrounding towns are among the most important olive oil production areas and host olive-based industries in Turkey. In addition, there are some cement and lime factories in the area.

According to our knowledge, no studies have been performed on the distributions, sources, and ecological risks of molecular PAHs and OCPs in Edremit Bay sediments. In the present study, distributions and levels of 18 PAHs and 19 OCPs in surface sediments from Edremit Bay have been investigated to determine the sources of PAHs using multiple linear regression following principal component analysis (PCA–MLR) and to determine ecological risks of individual PAHs and OCPs using sediment quality guidelines (SQGs).

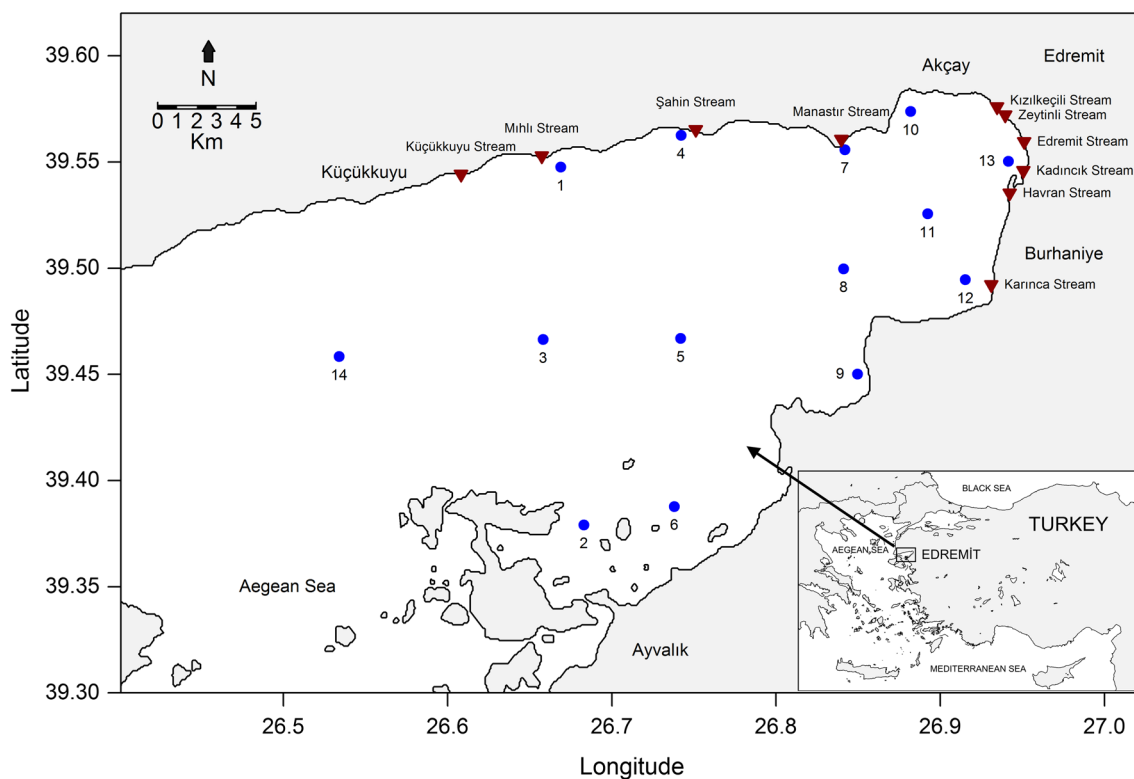
The results obtained in this study can be used as a reference providing information on sources and ecological risks of PAHs and OCPs in Edremit Bay for future studies.

## Materials and Methods

### Study Area and Sampling

Samplings were performed at wet (spring) and dry (autumn) seasons. High precipitation ratio at spring, presence of many streams with high sediment load capacities, and high sedimentation rates in the bay were reported previously (Yücesoy-Eryılmaz et al. 2005). Northern part of the bay is surrounded by mountains with high slopes that continue into the bay. These geographic properties might have influence on transfer of terrestrial materials to the bay.

Surface sediments ( $n=28$ ) were collected using a box-corer (50 × 50 cm) from 14 stations in April and November 2015 from the Edremit Bay (Fig. 1). The stations were selected based on bathymetric (shallow or deep parts) and coastal properties (presence of streams) of the bay. The stations can be grouped according to their positions at northern (Sta. 1, 4, 7), inner (Sta. 10, 11, 12, 13), and southern (Sta. 2, 3, 5, 6, 8, 9, 14) parts of the bay. Samplings were performed with R/V K. Piri Reis within the framework of



**Fig. 1** Sampling stations at Edremit Bay, Aegean Sea

the TUBITAK-113Y447 Project. An internationally well-established guideline was followed in the samplings (UNEP-IOC-IAEA 1992). The samples were kept in aluminum foils and stored frozen until analysis at  $-20\text{ }^{\circ}\text{C}$ .

### Sediment Grain Size and Organic Carbon Analyses

Sieve analysis and hydrometer tests were used for determination of grain size distributions (sand, silt and clay) in surface sediment samples. Grain size analyses were performed according to ASTM D2487-83, Wagner (1957) and British Soil Classification System for Engineering Purposes (BSCS) criteria (Dumbleton 1981). Distributions of fine and coarse grain sizes of sediments (greater than No. 200 sieve) as well as the total amount of clay and silt in the samples were determined quantitatively with sieve analysis. Distributions and percentages of the grades in sizes smaller than the No. 200 sieve have been determined with hydrometer analysis.

Organic carbon levels were determined using sulphochromic oxidation method spectrophotometrically (Hach 1988). This method has accuracy of  $\pm 0.017\%$  for organic matter.

### Sample Preparation

The samples were freeze-dried and passed from a  $250\text{ }\mu\text{m}$  sieve. In all steps, chromatographic grade chemicals and solvents were used.  $\text{Na}_2\text{SO}_4$  was pre-treated with hexane and methanol by Soxhlet extraction. Then, it is pre-combusted in an ash furnace at  $400\text{ }^{\circ}\text{C}$  for 6 h. Prior to extraction, column packing materials (alumina, florisil and silica gel) were refluxed with methanol and then with n-hexane for 8 h in a Soxhlet apparatus. The solvents were evaporated, and the packing materials dried in an oven. Alumina and silica gel were activated in an oven for 4 h at  $200\text{ }^{\circ}\text{C}$  and deactivated with water partially (5% w/w for silica, 5% w/w for alumina). Florisil was activated in an oven for 12 h at  $130\text{ }^{\circ}\text{C}$  and deactivated with water (0.5% w/w for florisil) (UNEP-IOC-IAEA 1992).

### Extraction, Cleanup, and GC–MS Analysis

In this study, 18 PAH compounds (16 priority PAHs defined by USEPA and methyl naphthalenes) and 19 OCPs were analyzed. Studied PAHs were naphthalene (Nap), 1-methyl naphthalene (1-M Nap), 2-methyl naphthalene (2-M Nap), acenaphthylene (Acy), acenaphthene (Ace), fluorene (Flu), phenanthrene (Phe), anthracene (Ant), fluoranthene (Flt), pyrene (Pyr), indeno[1,2,3-cd]pyrene (IcdP), dibenzo[a,h]anthracene (DahA), BaA, Chr, BbF, BkF, BaP, and BghiP. Instrument detection limits (ng/g) of PAH compounds were as follows: Nap:1.3, 1-M Nap:1.1, 2-M Nap:1.1, Ace:0.9, Acy:1.0, Flu:0.9, Phe:0.9, Ant:1.0, Flt:0.8, Pyr:0.8, BaA:0.9, Chr:0.9, BaP:1.4, BbF:1.2, BkF:1.1, IcdP:1.5, DahA:1.4,

BghiP:1.3. Studied OCPs and their instrument detection limits (ng/g) were as follows: Aldrin (1.0),  $\alpha$ -BHC (0.8),  $\beta$ -BHC (0.8),  $\gamma$ -BHC (0.7),  $\delta$ -BHC (0.9), a-chlordane (1.1), g-chlordane (1.1), *p,p'*-DDD (0.6), *p,p'*-DDE (0.5), *p,p'*-DDT (0.7), dieldrin (1.2), endosulfan I (1.1), endosulfan II (1.3), endosulfan sulfate (1.5), endrin (1.3), endrin aldehyde (1.3), heptachlor (1.2), heptachlor epoxide (1.2), and methoxychlor (1.4).

1,4-dichlorobenzene-d4, naphthalene-d8, acenaphthene-d10, phenanthrene-d10, chrysene-d12, and perylene-d12 were added to the sediments as internal standards for PAHs analyses. PCB 18, PCB 28, and PCB52 were added to the sediments as internal standards for OCPs analyses. Quality control procedures for PAHs and OCPs were applied in this study. Recoveries of internal standards were found between 78–105% and 88–102% for PAHs and OCPs, respectively. Blank samples were free of all target compounds. PAHs and OCPs standards were obtained from Dr. Ehrenstorfer and AccuStandard, respectively.

The analytical procedure used for the PAHs and OCPs in the marine sediments was based on the United Nations Environment Programme method (UNEP/IOC/IAEA 1992).  $\sim 15\text{ g}$  (mean:  $14.9\text{ g}$ ) of freeze-dried sediments, internal standards and 40 ml of dichloromethane/hexane (1:1) mixture were placed into glass extraction vessels for microwave extraction. Sulphur was removed using activated copper. For cleanup and elution of PAHs and OCPs, the extracts were applied onto silica-alumina and florisil columns, respectively. Solvents were evaporated to 15 ml using rotary evaporator and to 1 ml with  $\text{N}_2$  stream. Samples were analyzed with an GC–MS system (Agilent 6850 GC, Agilent 5975c MS, DB-5MS column:  $30\text{ m}\times 0.25\text{ }\mu\text{m}$ ) using electron impact (EI) ionization source. Target ions were identified using selected ion monitoring (SIM) mode. Helium was used as a carrier gas (99.9995%) at constant flow rate ( $1.5\text{ ml/min}$ ). The temperature programme was set from  $60\text{ }^{\circ}\text{C}$  (1 min) at  $3\text{ }^{\circ}\text{C/min}$  to  $300\text{ }^{\circ}\text{C}$  (10 min). The mass scan rate was 50–550 Da per second and electron voltage was 70 eV. Injection was made using splitless mode with  $1\text{ }\mu\text{L}$  sample volume at  $280\text{ }^{\circ}\text{C}$ .

### Sediment Quality and Ecological Risks

SQGs have been used to evaluate pollutant levels in aquatic environments (Long and MacDonald 1998; Long et al. 1998). Contamination levels were evaluated by comparing with SQG values of effect range low and median (ERL, ERM), threshold and probable effect levels (TEL, PEL), and threshold and probable effect concentrations (TEC, PEC) (Long et al. 1995; Macdonald et al. 1996; Weinstein et al. 2010; Li et al. 2015; Akhbarizadeh et al. 2016; Hong et al. 2016).

## Statistical Analyses

Statistical analyses were performed with R Statistical Computing Software, v3.5.1 (R CoreTeam 2018). In all statistical tests, significance level was  $\alpha = 0.05$ . Seasonal (spring and autumn) changes of contaminant and OC levels among Northern, Southern, and Inner Bays were investigated with nonparametric one-way Kruskal–Wallis tests. Pearson's correlation test used for assessment of statistical relationships between PAHs, OCPs, sediment grain sizes, and OC. Principal components analysis (PCA) were performed on molecular PAH concentrations and PAH levels in stations. PCA was used for reduction of dimensions in the data and for discrimination of sources. Multiple linear regression (MLR) was applied using PCA loadings of PAH levels in stations and  $\Sigma$ PAH levels as independent and dependent variables, respectively. By using MLR, it is possible to determine contribution percentages of each sources (Li et al. 2015). MLR and mean percentage contributions of each sources were calculated according to methods given in previous studies (Li et al. 2015; Ma et al. 2017).

## Results and Discussion

### Sediment Grain Size and Organic Carbon

Sediment grain size distributions (sand, silt, and clay contents) were given in Fig. S1. Sand content ranged from 1.4 to 77.0%. Highest and lowest sand percentages found in Sta. 14 and Sta. 2, respectively. Silt contents were between 18.1 and 75.5%. Highest and lowest silt percentages determined in Sta. 2 and Sta. 13, respectively. Clay content ranged from 4.9 to 24.9%. Highest and lowest clay percentages recorded in Sta. 2 and Sta. 14, respectively. Grain sizes of sediments in Edremit Bay were characterized as silty sand at Stas. 2, 5, 6, 8, and 9, sandy silt at Stas. 1, 3, 4, 7, and 12, and silt at Stas. 10, 11, 13, and 14. While northern and inner bay sediments have high silt (> 50%) content, southern bay has high sand contents (> 50%). Organic carbon contents changed between 8.3–30.5 and 8.9–24.1 mg/g in spring and autumn, respectively. Highest organic carbon level found at Sta. 2 in both seasons. Lowest organic carbon contents observed in Stas. 1 and 14 in spring and autumn, respectively. Mean OC levels were decreased in the order of inner, southern, and northern bay. Higher OC levels at inner and southern bays might be related with anthropogenic inputs and detrital materials.

### PAH Levels in the Bay

Concentrations and compositions of the studied PAHs and pesticides in Edremit Bay sediments were given in Table 1 and Fig. 2. Predominant PAH compounds were Flt and Pyr.

PAHs concentrations (ng/g dw) in sediments ranged between 0.65 to 21.2 for Nap; 1.2–8.5 for 1-M Nap; 0.16–11.6 for 2-M Nap; 1.9–7.0 for Flu; 2.4–38 for Phe; 2.0–30 for Ant; 1.5–40 for Flt; 1.6–30 for Pyr; nd–6.4 for BaA; nd–4.1 for Chr; nd–2.8 for BaP. BaA, Chr and BaP were only found at Sta. 10 in autumn. On the other hand, Acy, Ace, BbF, BkF, IcdP, DahA, and BghiP were below detection limits in this study. Maximum PAH concentrations were observed at inner bay stations. PAH levels in autumn were higher than that of spring. OCPs other than *p,p'*-DDE were below detection limits in this study. *p,p'*-DDE levels were found between nd–1.16 and nd–1.14 ng/g dw in spring and autumn, respectively. *p,p'*-DDE levels in inner and northern bays were higher than southern bay. OCPs might be below detection limits due to the bans of their uses between 1971 to 1989 in Turkey, which was in parallel to other countries. *p,p'*-DDE is one of the degradation products of DDT, and it is stable in the environment for many years compared with others.

$\Sigma$ PAH levels in sediments ranged between 12.9 to 91.4 and 0.6 to 175 ng/g in spring and autumn, respectively.  $\Sigma$ PAH levels were decreased in the order of inner, northern and southern bay. Minimum  $\Sigma$ PAH level was found at Sta. 6 in spring and autumn. Maximum  $\Sigma$ PAH levels were observed at Stas. 12 and 10 in spring and autumn, respectively. Pollution levels for  $\Sigma$ PAH can be classified as low (0–100 ng/g), moderate (100–1000 ng/g), high (1000–5000 ng/g), and very high (> 5000 ng/g) (Baumard et al. 1998a). Based on this classification,  $\Sigma$ PAH concentrations in Edremit Bay sediments indicated low pollution except moderate pollution observed at Sta. 10 in autumn. According to SQGs, all PAHs and *p,p'*-DDE levels were found below the TEL, ERL, and TEC limits (Table 2). Therefore, PAHs and *p,p'*-DDE levels in Edremit Bay sediments have no potential ecological risks.

Seasonal and spatial variabilities in PAHs, *p,p'*-DDE and OC data were investigated with one-way nonparametric Kruskal–Wallis analysis. 2-M Nap and Ant were found statistically higher in spring than in autumn ( $p < 0.05$ ). Phe, Flt, Pyr,  $\Sigma$ PAH, and *p,p'*-DDE were observed statistically higher at northern and inner bays than southern bay ( $p < 0.05$ ). Nap, Flu, and OC were found statistically higher at inner bay than northern and southern bays ( $p < 0.05$ ). 2-M Nap and Ant levels were decreased significantly in the order of inner, northern, and southern bays ( $p < 0.05$ ). There were no significant seasonal or spatial differences for 1-M Nap, BaA, Chr, and BaP levels ( $p < 0.05$ ).

Correlations between individual PAHs, *p,p'*-DDE, OC, and sediment grain size were determined for Edremit Bay sediments (Table S1). Highly strong positive correlations ( $r > 0.75$ ) found between Nap and 1-M Nap; Nap and 2-M Nap; 1-M Nap and 2-M Nap; Flu–Phe; Flu–Ant; Phe–Ant; Phe–Flt; Flt–Pyr and Silt–Clay. Strong positive correlations ( $0.5 < r < 0.75$ ) observed between Nap–Flu, Nap–Ant, Nap–Silt, Nap–Clay, 2-M Nap–Flu, 2-M Nap–Ant; 1-M Nap–Flu,

**Table 1** PAHs and OCPs (ng/g, dry weight) and OC (mg/g, dry weight) concentrations in surface sediments of Edremit Bay

	Spring	Autumn	Inner Bay	Northern Bay	Southern Bay
Nap	1.0–12.8 (5.6±3.5)	0.6–21.2 (5.5±5.3)	1.0–21.2 (9.4±5.8) <sup>a</sup>	1.5–7.1 (4.4±2.0) <sup>b</sup>	0.6–8.1 (3.8±2.6) <sup>b</sup>
1-M Nap	1.2–3.7 (2.7±0.8)	2.7–8.5 (5.0±3.1)	2.0–8.5 (4.3±2.9)	2.7–3.7 (3.2±0.5)	1.2–2.8 (2.3±0.9)
2-M Nap	0.3–3.7 (1.9±1.2) <sup>†</sup>	0.2–11.6 (2.5±3.6) <sup>‡</sup>	0.3–11.6 (3.2±3.6) <sup>a</sup>	0.2–3.6 (1.7±1.4) <sup>ab</sup>	0.3–3.0 (1.5±0.9) <sup>b</sup>
Flu	1.9–5.5 (3.4±1.5)	2.7–7.0 (4.7±2.1)	2.7–7.0 (4.6±1.7) <sup>a</sup>	nd	1.9–2.9 (2.4±0.6) <sup>b</sup>
Phe	2.4–31.7 (11.7±7.6)	6.0–38.0 (13.1±10.0)	6.9–38.0 (18.6±10.9) <sup>a</sup>	6.0–15.8 (11.4±3.7) <sup>a</sup>	2.4–14.1 (7.2±3.8) <sup>b</sup>
Ant	3.0–28.0 (11.5±6.7) <sup>†</sup>	2.0–30.0 (7.8±9.5) <sup>‡</sup>	3.9–30.0 (15.1±10.2) <sup>a</sup>	2.9–16.4 (8.3±6.0) <sup>ab</sup>	2.0–14.0 (6.7±4.3) <sup>b</sup>
Flt	1.5–22.4 (8.0±6.2)	5.1–40.0 (14.3±11.1)	6.2–40.0 (17.2±11.7) <sup>a</sup>	8.5–13.6 (11.0±1.8) <sup>a</sup>	1.5–6.6 (4.0±1.9) <sup>b</sup>
Pyr	1.6–26.1 (8.3±6.9)	3.7–30.0 (10.8±8.4)	5.3–30.0 (14.9±9.9) <sup>a</sup>	8.3–12.9 (9.9±1.8) <sup>a</sup>	1.6–7.0 (3.9±2.0) <sup>b</sup>
BaA	nd	6.4	6.4	nd	nd
Chr	nd	4.1	4.1	nd	nd
BaP	nd	2.8	2.8	nd	nd
∑PAH	12.9–91.4 (48.2±26.9)	0.6–174.9 (37.9±47.8)	19.1–175 (81.1±47.0) <sup>a</sup>	30.7–70.0 (48.4±15.9) <sup>a</sup>	0.6–47.3 (19.0±15.8) <sup>b</sup>
Phe/Ant	0.78–1.13 (0.98±0.13)	0.96–3.25 (2.38±0.83)	1.0–3.1 (1.5±0.8)	1.0–3.2 (1.9±1.1)	0.8–3.1 (1.3±0.8)
Flt/Pyr	0.86–1.24 (0.99±0.10)	1.07–2.12 (1.35±0.33)	0.9–2.1 (1.2±0.4)	1.0–1.2 (1.1±0.1)	0.9–1.4 (1.1±0.2)
Ant/(Ant + Phe)	0.47–0.56 (0.51±0.03)	0.24–0.51 (0.32±0.10)	0.2–0.5 (0.4±0.1)	0.2–0.5 (0.4±0.1)	0.2–0.6 (0.5±0.1)
Flt/(Flt + Pyr)	0.46–0.55 (0.50±0.02)	0.52–0.68 (0.57±0.05)	0.5–0.7 (0.5±0.07)	0.5–0.6 (0.5±0.03)	0.5–0.6 (0.5±0.04)
∑2–3 rings/∑4–6 rings	0.70–5.55 (2.60±1.36)	0.17–4.75 (1.38±1.53)	0.9–5.6 (3.0±2.0)	0.2–3.8 (2.5±1.7)	0.7–4.1 (1.7±1.0)
<i>p,p'</i> -DDE	nd–1.16 (0.43±0.36)	nd–1.14 (0.39±0.34)	0.3–1.1 (0.6±0.3) <sup>a</sup>	0.2–1.2 (0.7±0.3) <sup>a</sup>	nd–0.8 (0.2±0.2) <sup>b</sup>
OC	8.3–30.5 (15.6±5.9)	8.9–24.1 (15.3±3.9)	14.7–23.4 (18.2±2.8) <sup>a</sup>	8.3–17.2 (13.5±2.9) <sup>b</sup>	8.4–30.5 (14.8±6.0) <sup>b</sup>

Acy, Ace, BbF, BkF, IcdP, DahA, and BghiP were below detection limits

†,‡,a,ab,b Indicate statistically significant differences,  $p < 0.05$

1-M Nap-Phe, 1-M Nap-Ant, Flu-Flt, Phe-Pyr, Phe- *p,p'*-DDE, Phe-Silt, Phe-Clay, Ant-Flt, Ant-Pyr, Ant- *p,p'*-DDE, Flt- *p,p'*-DDE, Flt-Silt, Pyr- *p,p'*-DDE, Pyr-Silt and *p,p'*-DDE-Silt. Highly strong negative correlations found between Sand-Silt and Sand-Clay. Strong negative correlations observed between Nap-Sand, Phe-Sand, Flt-Sand, Pyr-Sand, and *p,p'*-DDE-Sand. Strong positive correlations found between PAH compounds. Also, PAHs and *p,p'*-DDE compounds strongly correlated with silt and/or clay content in sediments. There were no correlations between OC and other variables.

Comparison of ∑PAH levels in Edremit Bay sediments with other representative coastal sediments were given in Table 3. Concentrations of ∑PAH in sediment samples from Edremit Bay were close to those found in the Amazon River Estuary, Brazil (dos Santos Rodrigues et al. 2018) and in the Hainan Island, China (Xiang et al. 2018). ∑PAH values in Edremit Bay were lower than Southeast Mediterranean, Egypt (Barakat et al. 2011), Mar Piccolo, Ionian Sea, Italy (Cardellicchio et al. 2007), Barcelona Harbour, Spain (Martínez-Lladó et al. 2007), Saronikos Gulf, Thessaloniki Bay, Gulf of Corinth, North Evoikos Gulf, Greece (Botsou

and Hatzianestis, 2012), coastal zones of Athens, Greece (Kapsimalis et al. 2014), Çanakkale Strait and Istanbul Strait, Turkey (Balcioglu et al. 2018). Edremit bay has lower ∑PAH levels compared to Çanakkale and İstanbul straits from Turkey that are heavily used in maritime activities. In this study, *p,p'*-DDE levels (n.d.–1.16 ng/g) were generally below than that of Bizerte Lagoon, Tunisia (1–4 ng/g; Ben Salem et al. 2017), Rosetta Nile branch estuary, Egypt (0.75–2.41 ng/g; Abbassy 2018), Gulfs of Naples and Salerno, Italy (n.d.–4.03 ng/g, Qu et al. 2018), Mediterranean coasts of Egypt (n.d.–11.2 ng/g, Barakat et al. 2013), coastal zone of Athens, Greece (14.9–152.2 ng/g, Kapsimalis et al. 2014), and Cadiz Bay, Spain (mean: 5.1 ng/g, Pintado-Herrera et al. 2017).

### Identification of PAH Sources

The source identification of PAHs in this study was performed using molecular indices, ring composition, and PCA. Molecular indices of PAHs have been widely applied to identify their sources at different environments (Readman et al. 1987; Budzinski et al. 1997; Baumard et al. 1998a, b;

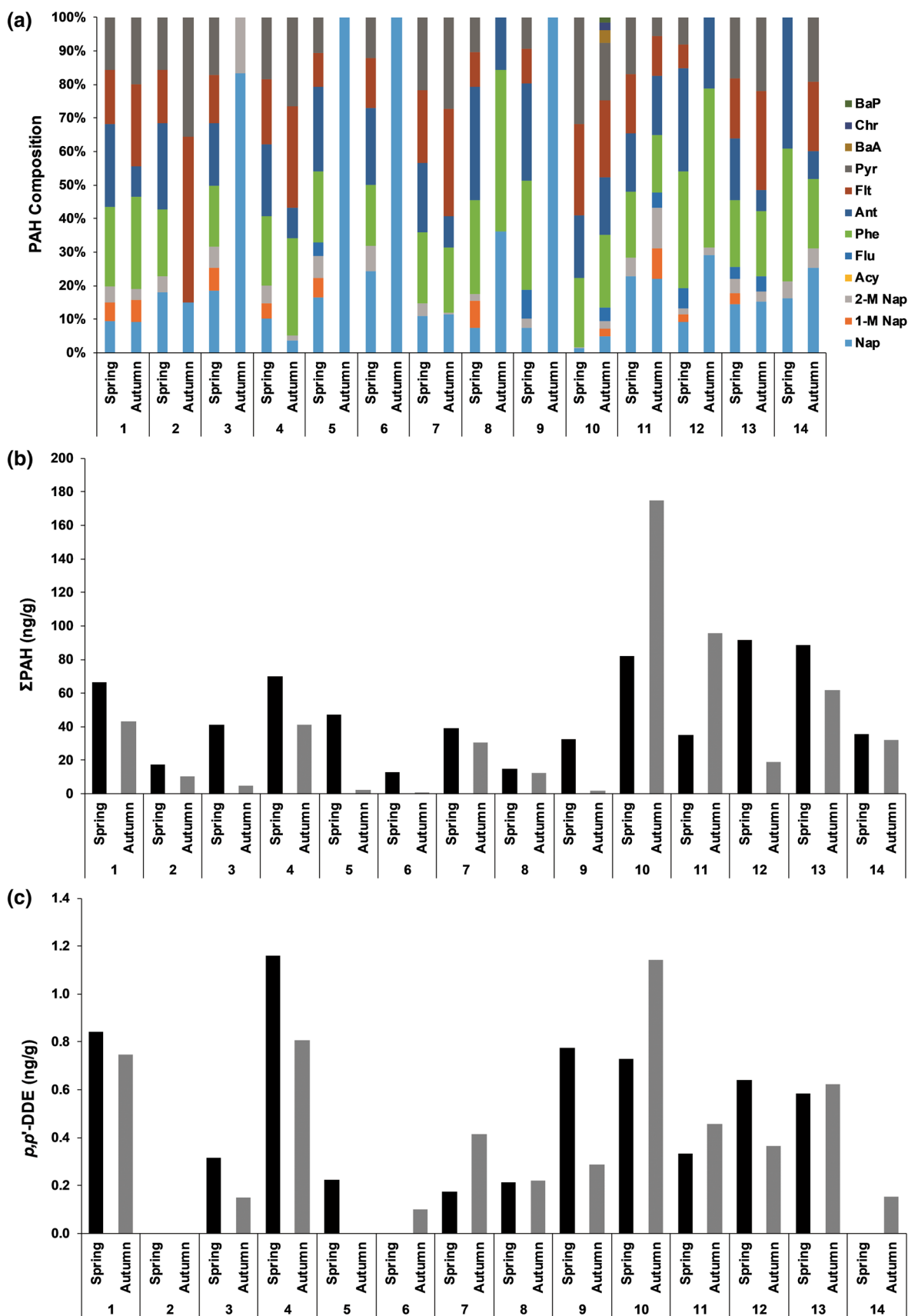


Fig. 2 Molecular PAHs levels in sampling stations: a PAH Composition, b ΣPAH, and c p,p'-DDE

**Table 2** Comparison of PAHs and OCPs levels (ng/g) with sediment quality criteria

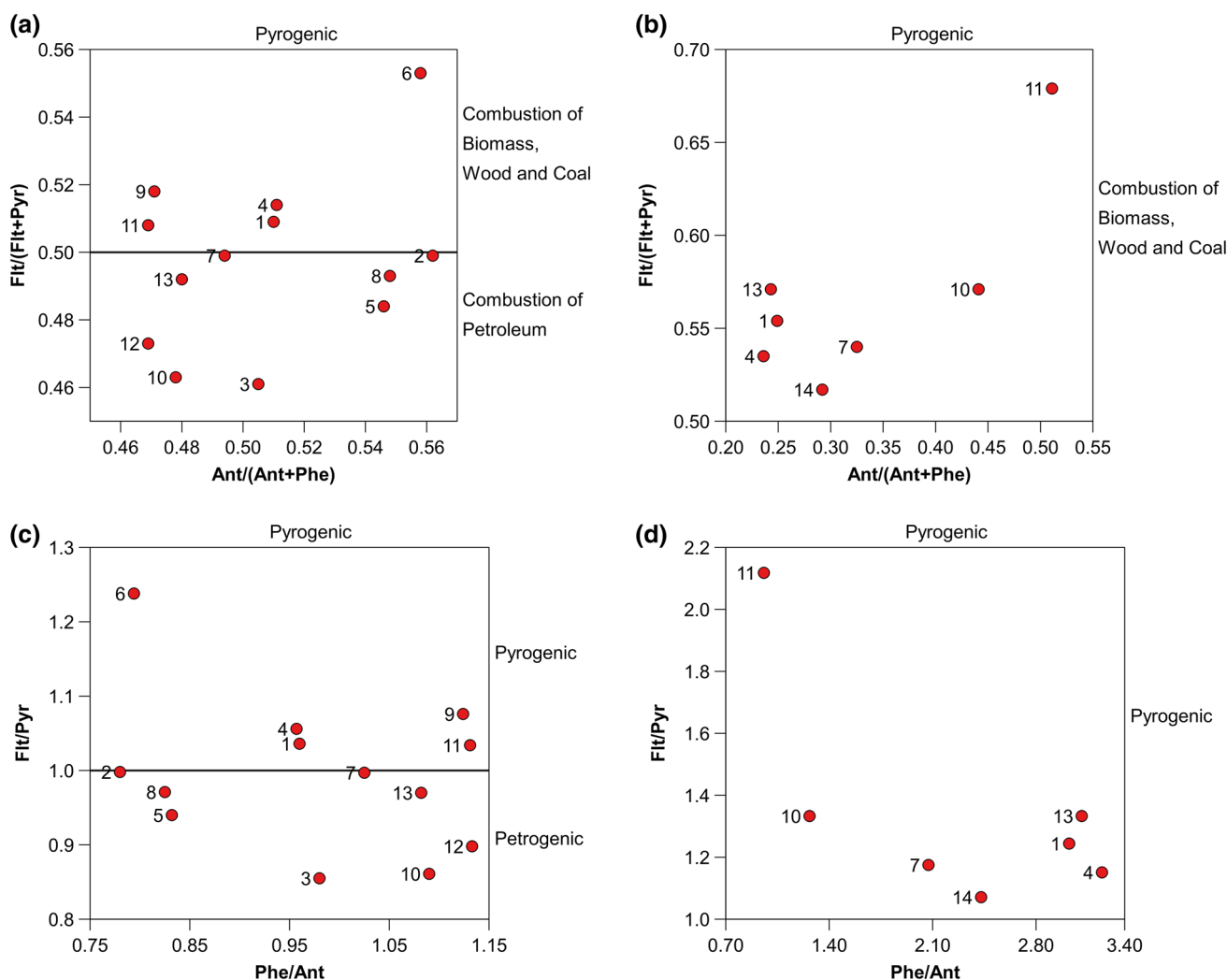
	Range <sup>a</sup>	Mean <sup>a</sup>	TEL <sup>b</sup>	PEL <sup>b</sup>	ERL <sup>b</sup>	ERM <sup>b</sup>	TEC <sup>b</sup>	PEC <sup>b</sup>
Nap	0.65–21.2	5.55	34.6	391	160	2100	176	561
1-M Nap	1.21–8.5	3.38	–	–	85	800	–	–
2-M Nap	0.16–11.6	2.13	20	201	70	670	–	–
Ace	–	–	5.87	128	44	640	6.71	88.9
Acy	–	–	6.71	88.9	16	500	5.87	128
Flu	1.95–7.0	3.96	21.2	144	19	540	423	2230
Phe	2.36–38.0	12.2	86.7	544	240	1500	204	1170
Ant	1.97–30.0	10.1	46.9	245	85.3	1100	57.2	845
Flt	1.53–40.0	10.4	113	1494	600	5100	77.4	536
Pyr	1.56–30.0	9.29	153	1398	665	2600	195	1520
BaA	6.4	6.4	75	693	261	1600	108	1050
Chr	4.1	4.1	108	846	384	2800	166	1290
BaP	2.8	2.8	88.8	763	430	1600	150	1450
BbF	–	–	–	–	320	1800	–	–
BkF	–	–	–	–	280	1620	240	13,400
IcdP	–	–	–	–	240	–	200	3200
DahA	–	–	6.22	135	60	260	33	135
BghiP	–	–	–	–	85	1600	170	3200
ΣPAH	0.65–174.9	43.1	1684	16,770	4022	44,792	1610	22,800
<i>p,p'</i> -DDE	nd–1.16	0.41	2.07	374	2.2	27	–	–

<sup>a</sup>This study<sup>b</sup>Long et al. (1995), 1998, Macdonald et al. (1996), Long and MacDonald (1998), Weinstein et al. (2010), Li et al. (2015), Akhbarizadeh et al. (2016) and Hong et al. (2016)**Table 3** ΣPAH (ng/g, dry weight) concentrations in surface sediments of Edremit Bay and in other representative coastal sediments

Study area	ΣPAH (ng/g)	No. of PAHs	No. of stations	References
Cochin Estuary, India	194–14,149	16	15	Ramzi et al. (2017)
Amazon River Estuary, Brazil	15.7–158.9	16	16	dos Santos Rodrigues et al. (2018)
Hainan Island, China	67.3–197.0	16	3	Xiang et al. (2018)
Southeast Mediterranean, Egypt	13.5–21,600	16	26	Barakat et al. (2011)
Mar Piccolo, Ionian Sea, Italy	380–12,750	16	9	Cardellicchio et al. (2007)
Barcelona Harbour, Spain	300–10,320	16	20	Martínez-Lladó et al. (2007)
Saronikos Gulf, Greece	109–8417	21	15	Botsou and Hatzianestis (2012)
Thessaloniki Bay, Greece	145–943	21	8	Botsou and Hatzianestis (2012)
Gulf of Corinth, Greece	207–26,632	21	8	Botsou and Hatzianestis (2012)
North Evoikos Gulf, Greece	133–7453	21	10	Botsou and Hatzianestis (2012)
Coastal zones of Athens, Greece	929–18,907	26	12	Kapsimalis et al. (2014)
Çanakkale Strait, Turkey	335–2912	17	7	Balcioğlu et al. (2018)
Istanbul Strait, Turkey	120–2199	17	7	Balcioğlu et al. (2018)
Edremit Bay, Aegean Sea	0.65–175	18	14	This study

Gogou et al. 1998; Soclo et al. 2000; Yunker et al. 2002; Li et al. 2006; Wang et al. 2006; Yan et al. 2009). Molecular compositions of PAHs show variations depending on their sources; petrogenic PAHs are abundant in lower molecular weight components, and pyrogenic PAHs are abundant in high molecular weight components (Aichner et al. 2007; Peng et al. 2011). Molecular indices of Phe/Ant, Flt/Pyr,

Flt/(Flt + Pyr) and Ant/(Ant + Phe) were used to determine potential PAHs sources in this study. These indices are used in identification of sources mainly based on thermodynamic stabilities of PAHs (Readman et al. 1987; Budzinski et al. 1997; Baumard et al. 1998a, b, c; Gogou et al. 1998; Yunker et al. 2002; Tsapakis et al. 2003; Yan et al. 2009). According to Table S2 and Fig. 3, Ant/(Ant + Phe) ratios for Edremit



**Fig. 3** Scatter plots of molecular PAHs ratios in stations for determination of their sources: Flt/(Flt + Pyr) versus Ant/(Ant + Phe) in **a** spring, **b** autumn; Flt/Pyr versus Phe/Ant in **c** spring, **d** autumn

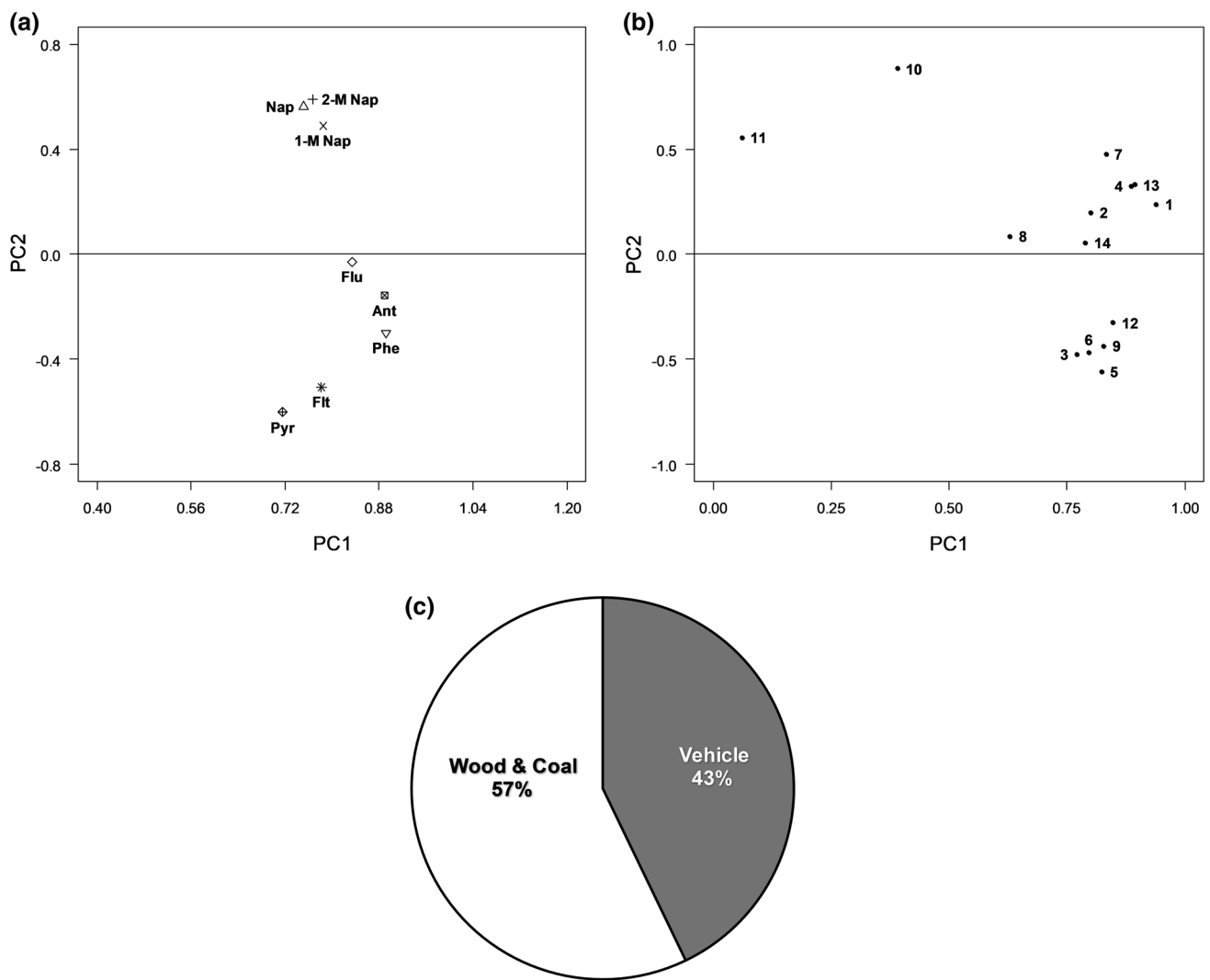
Bay sediments pointed pyrogenic sources. Flt/(Flt + Pyr) ratios indicated influences of coal (or biomass/wood) and petroleum combustion in spring, while these ratios indicated only coal (or biomass/wood) combustion in autumn. Flt/Pyr ratios suggested petrogenic origin in Stas. 3, 5, 8, 10, 12, and 13 in spring and other stations were of pyrogenic origin. On the other hand, Flt/Pyr ratios pointed pyrogenic origin in autumn. Molecular ratios indicated that PAHs might be mainly originated from combustion of biomass, wood and coal. In this study, 5- and 6-ring PAHs were not found in sediments, and therefore, other molecular indices (such as IcdP/BghiP, IcdP/(IcdP + BghiP), BbF/BaP, BkF/BaP) were not used.

PAH compounds can be classified according to their ring numbers: 2 rings (Nap, 1-M Nap, 2-M Nap), 3 rings (Acy, Ace, Flu, Phe, and Ant), 4 rings (Flt, Pyr, BaA, and Chr), 5 rings (BbF, BkF, BaP, DahA), and 6 rings (IcdP

and BghiP). Seasonal and spatial compositional patterns of PAHs based on their ring numbers were given in Fig. S2. PAHs with 2–3 rings (15–100%) were the most abundant in spring and autumn. On the other hand, 4-ring PAHs were found between 0 to 85%. Four-ring PAHs were higher than 2–3 rings at Stas. 1, 2, 4, 7, 10, and 13 in autumn and Sta. 2 in spring. Five-ring PAHs were only found at Sta. 10 in autumn. These stations are close to coastal areas and they might be influenced from urban settlements, vehicle emissions, and streams.

According to PCA analysis of PAH compounds, two components were statistically sufficient to model the variations in data (Fig. 4). PC1 and PC2 explained 65% and 21% of the variability in the data, respectively. All PAH compounds (Nap, 1-M Nap, 2-M Nap, Flu, Phe, Ant, Flt, and Pyr) were explained by PC1. While Nap, 1-M Nap, and 2-M Nap identified positively, Flt, Pyr, Phe, Ant, and Flu identified





**Fig. 4** Results of PCA and MLR analyses: **a** PCA between molecular PAHs levels, **b** PCA between PAHs levels in stations, **c** contribution percentages for each sources of PAHs

negatively by PC2. Positively explained Nap, 1-M Nap, and 2-M Nap indicated that these PAHs might be originated from petroleum related sources (Yunker et al. 1996; Larsen and Baker 2003; Ma et al. 2017). Negatively explained Flt, Pyr, Phe, Ant, and Flu suggested that these PAHs might be originated from incomplete combustion of wood, coal, and biomass at low to medium temperatures (Harrison et al. 1996; Simcik et al. 1999; Ma et al. 2017).

According to PCA analysis of PAH levels in stations, two components were statistically sufficient to model the variations in data. PC1 and PC2 explained 59% and 19% of the variability in the data, respectively. While most of the stations were explained by PC1, Stas. 10 and 11 identified by PC2. Stations were clustered into 2 groups as Group 1: 3, 5, 6, 9, and 12; Group 2: 1, 2, 4, 7, 8, 10, 11, 13, and 14. Whereas southern bay stations were included in group 1,

northern and inner bay stations were found in group 2. This PCA analysis indicated two sources might have contributed to PAH levels and their distributions in the bay. The bay is surrounded by little towns and cities that have a constant population throughout the year, but overall population is increased in summer due to tourism activities especially at the north and inner coasts. Mainly wood and coal combustion, and followingly, vehicle emissions (cars and ships) might have contributed to PAHs levels. Also, agriculture-based industries (i.e., olive oil), cement, and lime factories use coal as an energy source in the area.

Percentage contributions of these sources were determined with multiple linear regression analysis using PCA loadings and  $\Sigma$ PAH levels in stations as independent and dependent variables, respectively ( $\Sigma$ PAH = 48.6 PC1 + 64.8 PC2,  $r^2 = 0.671$ ). According to Fig. 4c, combustion of wood

and coal was the most significant source (57%), followed by vehicle emissions (43%).

## Conclusions

Concentrations, distributions, and sources of PAHs and OCPs in Edremit Bay sediments were investigated in this study. Acy, Ace, BbF, BkF, IcdP, DahA, BghiP, and OCPs other than *p,p'*-DDE levels were below detection limits. Only BaA and Chr from carcinogenic PAHs were detected at Sta. 10 in autumn.  $\Sigma$ PAH concentrations in Edremit Bay sediments indicated low pollution levels. Highest  $\Sigma$ PAH levels were found in northern and inner bays. Lowest levels were observed at southern bay. PAHs and *p,p'*-DDE levels were found below the TEL, ERL, and TEC limits; therefore, no potential ecological risks were observed for Edremit Bay sediments.  $\Sigma$ PAH and *p,p'*-DDE concentrations in Edremit Bay sediments were lower than most of the coastal sediments at other polluted sites. Abundances of PAHs were decreased in the order of 2–3, 4, and 5 rings. OCPs results pointed out absence of newly used pesticides in the area. Molecular ratios indicated that PAHs might be mainly originated from combustion of biomass, wood, and coal. PCA and MLR analyses indicated that combustion of biomass, wood, and coal and vehicle emissions might have contributed to PAH levels and distributions in the bay.

**Acknowledgements** This work was supported by the Turkish Scientific and Technological Research Council (TUBITAK) (Grant Number 113Y447) with project “Determination of Organic Matter and Pollutant Levels in Water, Sediment and Organisms from Edremit Bay”. Authors thank to Gamze Kordaci Uzkuç for sediment grain size analysis.

## Compliance with Ethical Standards

**Conflict of interest** The authors declare that they have no conflict of interest.

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