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Baseline

Evaluation of sediment contamination by monoaromatic hydrocarbons in the coastal lagoons of Gulf of Saros, NE Aegean Sea

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ABSTRACT

The concentrations and distribution of monoaromatic hydrocarbons (benzene, toluene, ethyl benzene and the sum of *m*-, *p*- and *o*-, xylenes) were determined in the sediments of coastal lagoons of the Gulf of Saros, using a static headspace GC–MS. The total concentrations of BTEX compounds ranged from 368.5 to below detection limit 0.6 μg kg^{-1} dw, with a mean value of 61.5 μg kg^{-1} dw. The light aromatic fraction of *m*-, *p*-xylene was the most abundant compound (57.1% in average), and followed by toluene (38.1%) > ethylbenzene (4.1%) > o-xylene (2.5%) > benzene (1.1%). The factor analysis indicated that the levels and distribution of BTEX compounds depend on the type of contaminant source (mobile/point), absorbance of compounds in sediment, and mobility of benzene compound and degradation processes. Point sources are mainly related to agricultural facilities and port activities while the dispersion of compounds are related with their solubility, volatility and effect of sea/saline waters on lagoons.

Monoaromatic hydrocarbons represent a significant part of volatile organic compounds, a collective name for benzene, toluene, ethyl benzene, and xylenes (BTEX compounds). Monoaromatic hydrocarbons are the most mobile and toxic hydrocarbon constituents and present various aquatic environments (Bianchi et al., 1991). These compounds are often found in discharges and petroleum products such as gasoline, and other common environmental contaminants and acutely toxic to aquatic organisms if contact is maintained (Wang and Fingas, 1998). They may be yielded by uncompleted burning of organic matter and their major sources to the environment include vehicle exhaust (mobile source), coal/waste burning, oil refining processes, etc. (point sources). In addition, monoaromatic hydrocarbons are used extensively in manufacturing processes, production of synthetic materials and consumer products, such as plastics, insecticides and paints. They are included in the US Environmental Protection Agency (EPA) purgeable priority pollutants list (USEPA, 1989).

The coastal areas of the Gulf of Saros, NW Turkey, are important places in daily life, providing contributions to regional and national economy in terms of conservation biological diversity, particularly fisheries, livestock, salt production, reed cutting and recreational activities. These areas are mostly characterized by coastal lagoons (Fig. 1), which form ecotones between freshwater, marine and terrestrial biotopes under the control of coastal geomorphological processes. These lagoons have been partially affected over time by anthropogenic pollution; mainly related with the increasing population and recreational activities around them. Only a few researches have addressed the role of the anthropogenic impacts in some of these lagoons around the Gulf of Saros (e.g. Yılmaz and Serbest, 2005; Yaşar, 2010; Barut et al., 2015). No published papers or available data was found on the monoaromatic hydrocarbons in these lagoons. Therefore, the present study aims to assess the concentrations of monoaromatic hydrocarbons, their distribution and possible sources in the bottom sediment of these coastal lagoons.

Beginning from the border with Greece and down to the western coasts of the Gelibolu (Gallipoli) Peninsula, many small coastal lagoons are situated at the coastal regions (Fig. 1). All these lagoons, usually located on places where coastal and shelf areas are relatively wider, are characterized by shallow coastal salt lakes, partially or completely separated from the sea by some barriers. Their salinity may vary depending on balance between precipitation and evaporation and feeding watercourses.

One of the most important wetlands of the world is located on the Enez-Evros delta and represents key environments for the sustainable development of the economy with abandoned channel mouths, dunes, marshlands, lagoons and saltpans (Alpar, 2001). This was an important settlement region during the Hellenic, Roman, Byzantine and Ottoman periods, and an important port dating back to the 12th century BC. Shifting riverbed of Maritza and siltation turned the ancient estuary into adjacent large and small lakes and lagoons (Fig. 1a); namely Enez (S_1), Işık/Bücürmene (S_2 and S_3), Kuvalak (S_4), Küçükgöl (S_5), Dalyan ($S_{6–10}$) and Taşaltı ($S_{11–12}$).

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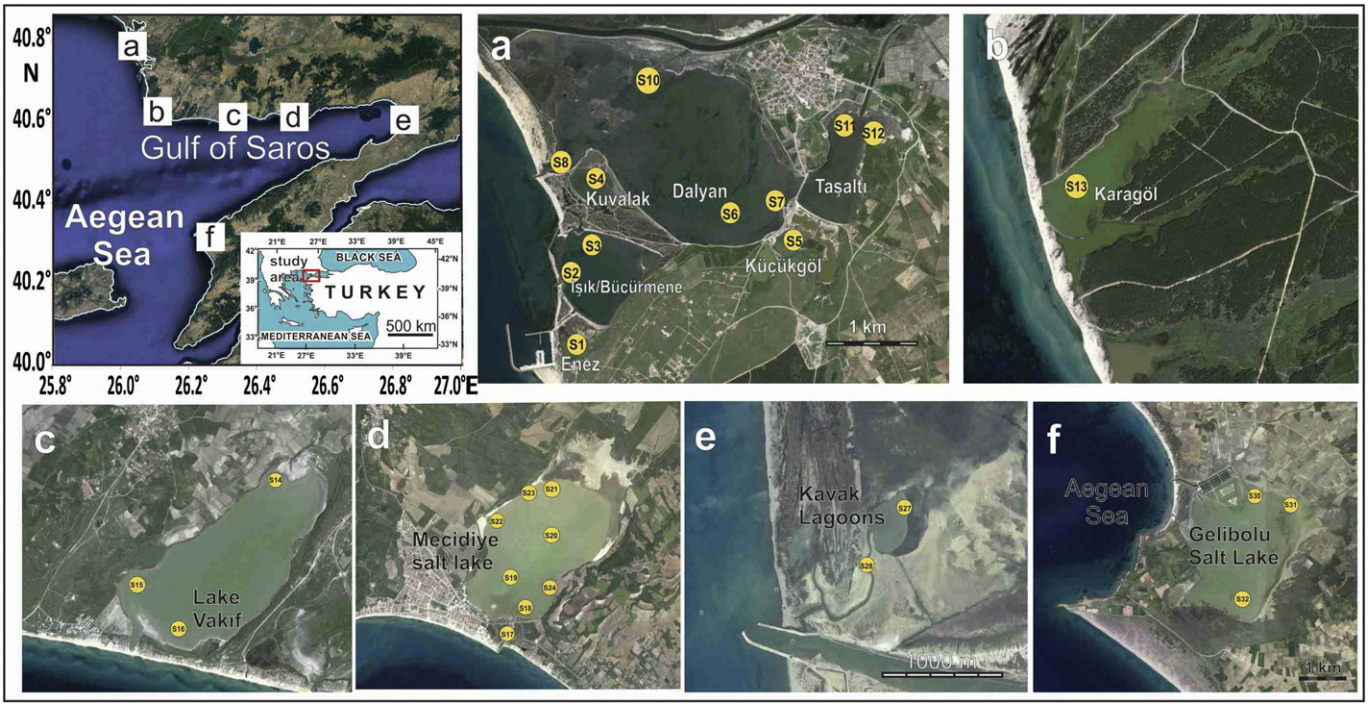

Fig. 1. Locations of coastal lagoons with sampling sites; a: Enez (S_1), Işık/Bücürmene (S_{2-3}), Kuvalak (S_4), Küçükgöl (S_5), Dalyan (S_{6-10}) and Taşaltı (S_{11-12}), b: Karagöl (S_{13}), c: Lake Vakıf (S_{14-16}), d: Mecidiye (Erikli) salt lake (S_{17}–S_{24}), e: Kavak Lagoons (S_{27-28}) and f: Gelibolu salt lake (S_{30-32}).

Farther to the east and south, the geomorphologic units around the Gulf of Saros represent different stages of development such as sandy beaches, mudflats, marshes, shallow lagoons, small lakes and channels. The Karagöl (S_{13}) is a very small lagoon with a surface area of 0.1–0.3 km^2 depending on the seasons (Fig. 1b). Along the northern coasts of the Gulf of Saros, the salt lakes of Vakıf (S_{14-16}) and Mecidiye-Erikli (S_{17-24}) are the most important lagoons located at the river mouths and marsh areas (Fig. 1c and d). Their waters are pungent and salty. The Lake Vakıf (S_{14-16}) is small (1.8 km^2). The salt lake on Mecidiye-Erikli coastal beach zone (2.5–3.3 km^2) was mentioned in Ottoman archives as the source of salt to meet Istanbul's needs. The Kavak Lagoons (S_{27-28}) at the easternmost part of the Gulf of Saros are rather small (<0.1 km^2) (Fig. 1e). The Gelibolu salt lake (S_{30-32}) is located at the western tip of the Gallipoli Peninsula (2.5 km^2) (Fig. 1f). This salt lake has greatly influenced by a fish farm that has been opened to operation in recent years.

The samples were collected from 26 stations in the coastal lakes around the Gulf of Saros on October 2004 (Fig. 1). The samples were removed manually with PVC pipes from 10 to 20 cm depth below the bottom surface. The sampling vessels were capped and stored on a dry-ice bed inside the collection box. The samples were frozen immediately after sampling and analysed within 48 h of collection.

The concentrations in the sediment samples were determined at MERLAB Central Research Laboratory of Istanbul University. A static headspace autosampler (Thermo Finnigan model HS 2000) equipped with standard glass vials of an internal volume 10 ml was employed. HS-GC–MS reference procedure set was in accordance with the description of a literature (Esteve-Turrillas et al., 2007). Standard solutions containing benzene (99.99%), toluene (99.5%), ethyl benzene (99.97%), *m*-xylene (99.8%), *p*-xylene (99.9%) and *o*-xylene (99.3%) were purchased from Merck (Darmstadt, Germany). For qualitative and quantitative identification of the BTEX compounds in the sediment samples, standard curves have been generated for different concentration ranges using benzene, toluene, ethyl benzene, *m*-, *p*- and *o*-xylene standards in hexane. Detection limits and recoveries were obtained from analysis of three replicates standard solutions at concentrations of 1.5, 3.12, 6.25, 12.5, 25 and 50 μg/l, respectively.

One gram of sediment sample was weighted in a 10 ml standard glass vial. The sample heated in headspace autosampler at 90 °C for 10 min with shaking. The syringe temperature was selected at 100 °C. The oven temperature program started from 40 °C held for 10 min, increased at a rate of 20 °C/min, up to 200 °C and finally held for 2 min. Electron impact ionization (EI) was used at 70 eV and helium flow is 1 ml/min. Transfer line temperature were fixed was held at 250 °C. The detector temperature was set to 230 °C. A Hewlett Packard HP 5MS column (Palo Alto, CA, USA) (30 m × 0.32 mm i.d., 0.25 μm film thickness) was used to obtain the reference data by chromatography.

For instrumental control and intensity measurements, it was employed using vendor software Xcalibur from Thermo (Waltham, MA, USA). Calibration data were developed with the help of TurboQuant Analyst 6.0 software (Thermo Nicolet Corp. Madison, USA).

The mass spectra were obtained at a mass-to charge ratio (*m*/*z*) scan range from 75 to 200. The specific ions generated at *m*/*z* 77 and 78 for benzene, *m*/*z* 91 and 92 for toluene and *m*/*z* 91 and 106 for ethylbenzene and xylenes. The recoveries of compounds were found to be between 70 and 130%. The mean level of relative percentage difference (RPD) duplicate sample was <15%. The limit of detection (LOD) was between 0.25 and 0.5 μg kg^{-1} dw for each component.

Pearson's correlation coefficients (r) calculated the strength of relationships between the monoaromatic hydrocarbon concentrations, and principal component analysis (PCA) quantified spatial/temporal variability of BTEX sources for lagoon sediment samples ($n = 26$). As a whole, the first few components explain the inherent variation of the data to the maximum possible extent (Varmuza and Filzmoser, 2008). In the present study, PCA was conducted with varimax rotation. The first three eigenvalues retained were 3.2, 1.3 and 0.8.

The granulometric analysis was performed using petrographic procedures adapted from Folk (1980), as described by GERG SOP-8908. The bottom sediments in the lakes are under the control of material supplied with river alluvium, surficial water flows. They are composed of mainly sand and mud mixtures depending on their locality (Table 1). The contents of water in the samples range between 15.6% and 67.1%, with an average of 31.3%.

Table 1
Sediment texture (%), sediment water content (swc) and concentrations of light aromatic BTEX fraction (μg kg^{-1} dw) in the sediment of the coastal lagoons.

Region	St.	Pebble	Sand	Mud	swc	B	T	E	*m*-, *p*-X	*o*-X	∑BTEX
Enez	S_1	4.7	49.7	45.6	25.4	4.3 ± 1.2	32.1 ± 8.1	4.9 ± 2.4	28.3 ± 13.5	BC	69.7
Işık	S_2	31.2	66.6	2.3	18.6	BC	15.5 ± 2.5	2.4 ± 0.7	27.5 ± 6.4	9.9 ± 3.0	55.3
	S_3	0.0	3.8	96.2	47.5	1.8 ± 0.2	18.2 ± 2.1	BC	BC	BC	20.0
Kuvalak	S_4	17.2	78.0	4.8	21.0	BC	15.1 ± 2.1	3.5 ± 0.3	36.0 ± 4.9	13.3 ± 1.6	68.0
Küçükgöl	S_5	1.3	22.4	76.3	67.1	BC	9.1 ± 1.4	0.8 ± 0.2	4.9 ± 1.2	BC	14.7
Dalyan	S_6	14.4	64.9	20.7	22.9	BC	2.4 ± 0.7	1.7 ± 0.3	10.7 ± 0.5	BC	14.8
	S_7	13.5	70.3	16.3	19.2	BC	2.2 ± 0.4	BC	BC	BC	2.2
	S_8	5.0	93.0	2.0	17.6	BC	4.7 ± 0.4	BC	BC	BC	4.7
	S_{10}	9.6	84.4	6.1	42.8	BC	4.1 ± 0.5	BC	8.8 ± 0.8	BC	12.9
Taşaltı	S_{11}	0.5	65.3	34.2	40.8	BC	11.1 ± 3.2	BC	7.5 ± 5.0	BC	18.6
	S_{12}	1.6	6.9	91.5	64.1	1.6 ± 0.3	18.8 ± 1.1	5.0 ± 0.7	31.5 ± 2.7	BC	56.9
Karagöl	S_{13}	20.9	70.0	9.2	22.8	2.6 ± 1.3	52.1 ± 17	11.0 ± 5.0	80.7 ± 8.7	17.1 ± 4.8	163.4
Vakıf Salt	S_{14}	0.4	14.9	84.7	46.0	1.5 ± 0.2	95.2 ± 12	27.1 ± 3.5	244.6 ± 24.2	BC	368.5
Lake	S_{15}	1.2	23.6	75.2	39.7	1.7 ± 0.6	8.5 ± 1.8	2.4 ± 0.6	12.9 ± 1.6	BC	25.5
	S_{16}	16.0	75.2	8.8	20.6	0.9 ± 0.3	14.8 ± 4.6	2.1 ± 0.6	13.3 ± 2.6	BC	31.1
Mecidiye	S_{17}	0.0	79.8	20.2	21.2	BC	13.2 ± 2.6	BC	BC	BC	13.2
(Erikli)	S_{18}	12.4	85.3	2.4	15.6	BC	0.6 ± 0.2	BC	BC	BC	0.6
Salt Lake	S_{20}	0.0	6.4	93.6	29.9	BC	17.7 ± 1.0	2.9 ± 0.3	27.2 ± 2.7	BC	47.9
	S_{22}	0.0	14.5	85.5	30.5	BC	12.5 ± 0.7	2.6 ± 1.9	16.2 ± 9.3	BC	31.2
	S_{23}	0.0	2.7	97.3	27.4	BC	24.5 ± 3.8	3.7 ± 0.4	26.7 ± 1.4	BC	54.8
	S_{24}	0.0	8.3	91.7	35.7	BC	32.4 ± 1.8	3.1 ± 0.2	15.4 ± 0.6	BC	50.8
Kavak	S_{27}	0.0	15.1	84.9	24.0	BC	46.3 ± 2.1	8.6 ± 0.5	78.4 ± 2.9	BC	133.3
Lagoons	S_{28}	0.0	91.8	8.2	23.8	BC	40.3 ± 5.2	5.2 ± 0.9	40.5 ± 2.4	BC	86.0
Gelibolu	S_{30}	2.8	82.8	14.4	28.9	BC	29.6 ± 1.9	4.4 ± 1.0	33.6 ± 5.0	BC	67.6
Salt Lake	S_{31}	61.5	37.2	1.3	25.6	BC	21.2 ± 8.4	2.2 ± 0.7	14.5 ± 3.1	BC	37.8
	S_{32}	16.5	57.0	26.5	35.6	3.5 ± 0.6	67.3 ± 7.0	11.6 ± 2.3	68.3 ± 22.7	BC	150.7
Minimum		0.0	2.7	1.3	15.6	0.0	0.6	0.0	0.0	0.0	0.6
Maximum		61.5	93.0	97.3	67.1	4.3	95.2	27.1	244.6	17.1	368.5
Mean		8.9	48.8	42.3	31.3	0.7	23.4	4.1	31.9	1.5	61.5

BC: not calculated due to concentrations below the detection limits. B: Benzene, T: Toluene, E: Etylbenzene, *m*-, *p*-X: *meta*, *para*-Xylene, *o*-X: *ortho*-Xylene.

Results indicated that total concentrations of BTEX compounds in sediment ranged from 368.5 μg kg^{-1} to 0.6 μg kg^{-1}, with a mean value of 61.5 μg kg^{-1} (Table 1). The highest concentration of total BTEX was observed at S_{14} in the salt lake Vakıf (368.5 μg kg^{-1}), followed by the stations of S_{13} (Karagöl), S_{32} (Gelibolu Salt Lake), $S_{27–28}$ (Kavak lagoons). Other stations contain low level of total BTEX (0.6–69.7 μg kg^{-1}). The variability of ∑BTEX level may depend on various causes such as distance to the hot points, depth, sediment texture, total petroleum hydrocarbon distribution, variability of biodegradation processes, etc.

The present study has shown that toluene (51.5%) and then *m*-, *p*-xylene (40.3%) are the dominant congeners (Fig. 2). Toluene was

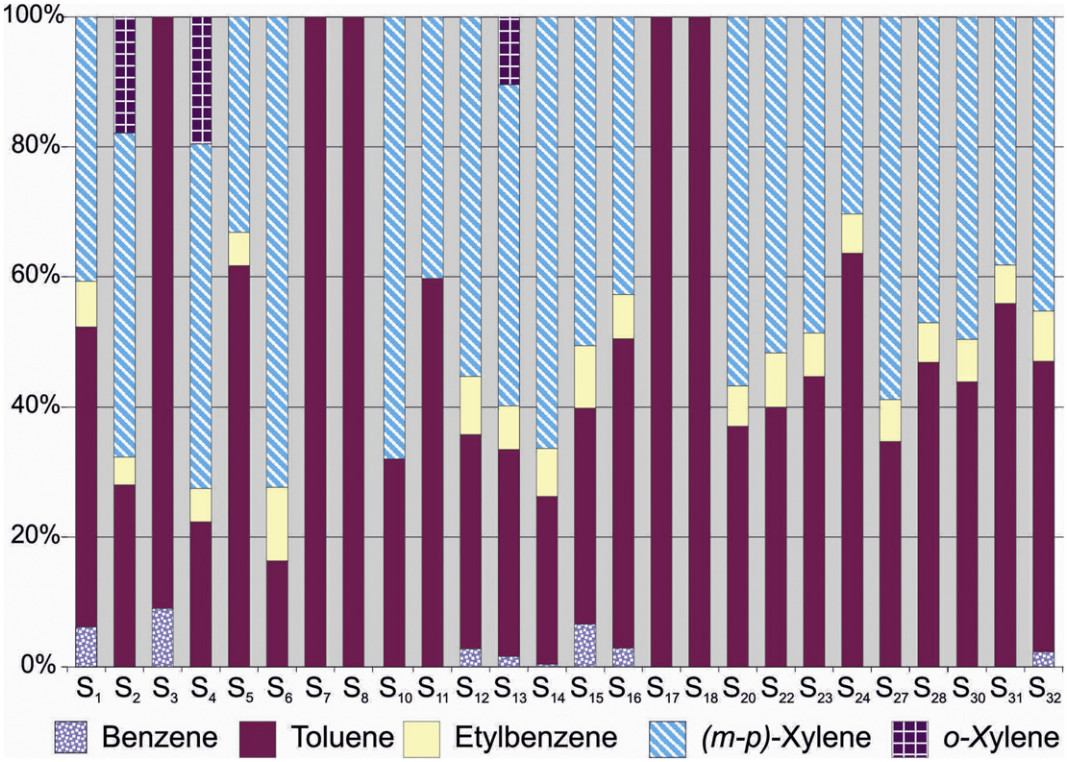

Fig. 2. Contributions of BTEX levels in the sediments from the coastal lagoons around the Gulf of Saros.

Table 2
Comparison of the sedimentary records of maximum BTEX concentrations (μg kg^{-1} dw) in coastal lake sediments of the Gulf of Saros with other regions in the world.

Location	B	T	E	*m*-, *p*-X	*o*-X	Reference
Gatuna Lagoon, SE New Mexico	460	1140	750	1080	690	Bristol (1998)
Lane Salt Lake, SE New Mexico	128	27	281	205	136	
Quatro Lagoon, SE New Mexico	650	3290	2250	4640	2940	
Tres Lagoon, SE New Mexico	200	330	740	1130	1050	
Uno Lagoon, SE New Mexico	90	110	90	310	110	
Waldon Lagoon, New Mexico	130	220	120	470	170	
Sava and Bosna River, Croatia	<54	1977	412	1652		Medunić and Šmit (2015)
Karavasta Lagoon, Albania	480	380	550	1640	45	Dukaj et al., (2015)
Coastal lagoons, Gulf of Saros	4.3	95.2	27.1	244.6	17.1	This study

ND: not detected. B: Benzene, T: Toluene, E: Etylbenzene, *m*-, *p*-X: *m*-*p*-Xylene, *o*-X: *o*-Xylene.

generally most eminent congener at all stations compared to the other light aromatics; possibly because of more extensive diversity of its natural and fabricated sources (Bianchi et al., 1991). Only small amount (<4.3 μg kg^{-1} dw) of benzene concentrations were found only in the stations of S_1, S_3, S_{12-16} and S_{32}, implying that volatilization, dissolution, photochemical activity, degradation by microorganisms and sorption of benzene were significant.

The maximum contributions of individual BTEX to the total are in the following order: *m*-, *p*-xylene (244.6 μg kg^{-1} dw) > toluene (95.3 μg kg^{-1} dw) > ethylbenzene (27.1 μg kg^{-1} dw) > *o*-xylene (17.1 μg kg^{-1} dw) > benzene (4.3 μg kg^{-1} dw). These contaminant levels are usually smaller if compared to those obtained from similar lagoons at other parts of the world (Table 2).

In the present study, the regression analysis was completed to investigate the relationship between the concentration of individual BTEX compounds and the percentage of mud fraction and sediment water content (Table 3). The linear regressions between these parameters show no significant correlation of BTEX compounds while correlation matrix exhibit good results between *m*-, *p*-xylene, ethylbenzene and toluene ($0.890 > r > 0.983$; $p < 0.01$).

Principle component analysis (PCA) technique reveals if relevant relationships were existed between the cases, that is, the concentration of the BTEX congeners in the lagoon sediments. Three principal components were retrieved for the lagoon sediments. The variance loading of the first three factors is 53.4, 22.3 and 13.1% of the total variability respectively (accumulative variance 88.8%). As the three factors were used to identify the source categories and the loadings of different BTEX congeners are shown in Fig. 3a and b.

The first factor exhibits higher loadings for ethylbenzene, toluene and *m*-, *p*-xylene; all used extensively as solvents and as raw materials in the synthesis of a variety of chemicals. Ethylbenzene is also present in xylene (up to 20%), and this mixture is used as a diluent in the paint industry, in agricultural sprays for insecticides and in gasoline blends (USEPA, 1980). This factor separates point sources, which show either agricultural facilities (e.g. use of nitrate and phosphate, riverine input, and small amount of unrefined domestic inputs) or port activities (use of paints and solvents), from mobile sources, which are mainly atmospheric and include vehicle exhaust emissions (gasoline/diesel). The most significant samples showing point sources include S_{14} (river mouth), S_{32} (agriculture and fish farming), S_{13} (vehicular activity in dry seasons), S_{27-28} (agriculture), S_1 (port activities) and S_{12} (river mouth, rice fields). On the other hand, the sediment samples of S_2 (0:15.5:2.4:27.5:9.9) and S_4 (0:15.1:3.5:36.0:13.3) near the Enez port show a similar source or input pathways, which indicate pollution due to urban and vehicle exhaust emissions.

Table 3
Pearson-coefficient correlation matrix (r) between concentrations of BTEX compounds, sediment texture and water content in the sediment of coastal lagoons around the Gulf of Saros (n = 26).

	Mud	swc	B	T	E	*m*-, *p*-X	*o*-X
swc	0.588**						
B	0.118	0.184					
T	0.219	0.134	0.488*				
E	0.226	0.157	0.426*	0.936**			
m-, *p*-X	0.216	0.146	0.309	0.890**	0.983**		
o-X	−0.340	−0.269	0.125	0.122	0.148	0.154	
∑BTEX	0.201	0.129	0.394*	0.946**	0.992**	0.987**	0.206

** Correlation is significant at the 0.01 level (2-tailed).
* Correlation is significant at the 0.05 level (2-tailed).

The second factor predominantly composed by *o*-xylene and mud percentage (Fig. 3b). This factor separates sandy and muddy sediment, as mud adsorbed more of the BTEX than sand due to large surface area and high hydraulic conductivity, while sand desorbed more of the BTEX than mud.

The third factor controls mobility of benzene and degradation level. Benzene and partly toluene are highly mobile (biodegrade more easily) in the environment if they compared to that of xylene and ethylbenzene. The aerobic/anaerobic biodegradation of BTEX group, in particular benzene, is resulted from photo-degradation, microbial activity and salinization (Sei and Fathepure, 2009). The salinization is high than sea water (>38 ppm) at the samples from the lagoons of Enez, Vakıf, Mecidiye and Gelibolu (Barut et al., 2015). In addition, BTEX compounds can be degraded during denitrification, if large amounts of fertilizers leach from agricultural fields (nitrate reducing conditions) (Reinhard et al., 1997; Farhadian et al., 2008).

In an overall interpretation, the right upward diagonal of the factor plain 1 × 2 indicate more recent pollution exposure mainly at the river mouths behind the lagoons, near the Enez harbour and aquaculture centre in Gelibolu salt lake, which is arising from agricultural, portal and fish farming activities (Fig. 3b).

Multivariate factor analyses revealed that the type of contaminant source (mobile/point), absorbance of BTEX in sediment, and mobility of benzene compound and degradation processes were the most dominant factors in the explanation of accumulation, evaporation and distribution of monoaromatic hydrocarbons in these coastal lagoons. Point sources are mainly related to fertilizers and pesticides used in agricultural facilities and mosquito fighting. As there is almost no industrial facilities in the surrounding region, the gulf is one of the pristine marine environments in the world. However, the coastal lagoons in the region face the risk of pollution lately because of the excessive use of chemical fertilizers in the surrounding region. Farmers apply more fertilizers and pesticides to increase agricultural production and maintain rice productivity, while the residue is being carried to the coastal lagoons by the water drainage systems, creeks and rivers. The nitrogenous fertilizers increase the salinity and nitrate pollution in underground waters.

The concentration levels and fate of the different BTEX components include discriminatory removal of certain BTEX components along the transport path, their solubility, dispersion and volatility. Sea or saline water is also important in removal of organic pollutants. The BTEX assemblages in such kind of stations (e.g. S_{7-8} and S_{17-18}) located where the seawater may intrude, were comprised of toluene.

In conclusion, the lagoons have been affected over time by anthropogenic pollution and need urgent and particular actions. The monoaromatic hydrocarbon concentrations obtained in this study

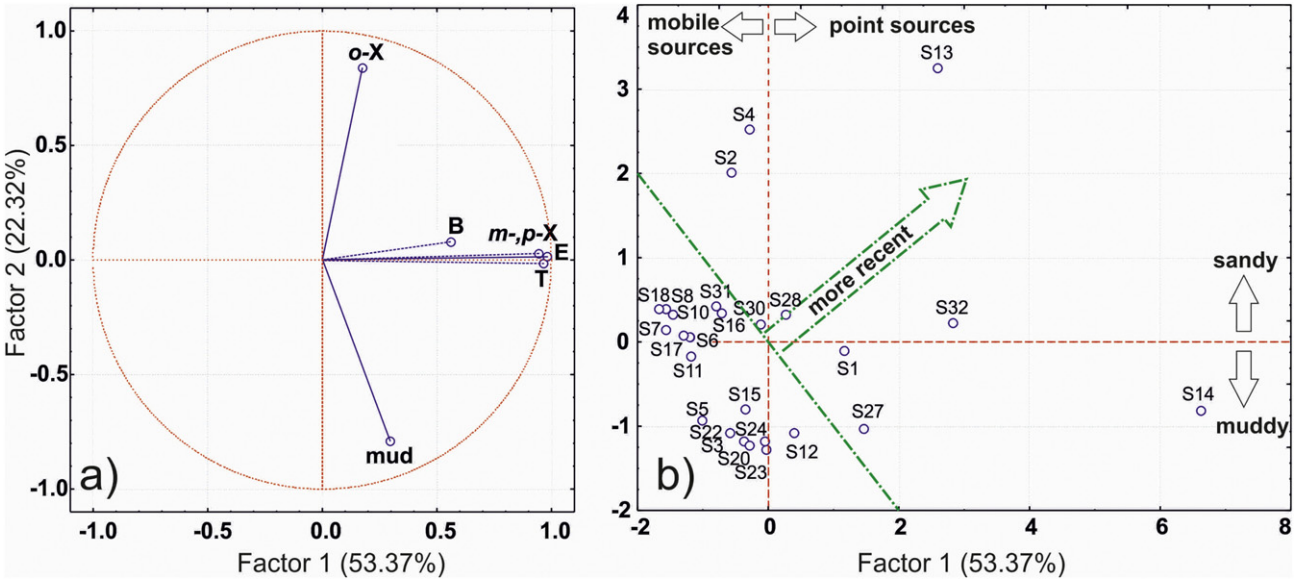

Fig. 3. The projection of the a) variables and b) cases on the factor plain 1 × 2.

would serve as a baseline to the administrative authority for preparing their rational environmental strategies needed, since there is not any national guideline for sediment quality to compare. Local authorities must encourage the citizens, farmers and private industries to decrease the level of contaminants that pollute the lagoons, which are important places in daily life, providing contributions to regional and national economy.

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