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**Selma Ünlü and Bedri Alpar**

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## Spatial distribution and sources of BTEX and TPH contamination in freshwater sediments from Lake İznik, NW Turkey

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### Abstract

Iznik Lake is an important water supply deteriorating gradually due to anthropogenic pollution. Concentration and distribution of monoaromatic hydrocarbons (benzene, toluene, ethylbenzene and three xylene isomers; BTEX) were detected in the lake sediments using a static headspace GC-MS.  $\Sigma$ BTEX concentrations varied between 10.6 and 272.3  $\mu\text{g kg}^{-1}$  dry weight (dw), with an average of 88.3  $\mu\text{g kg}^{-1}$  dw. The light aromatic fraction of m-,p-Xylene was the most abundant compound (50.0% in average), followed by toluene (31.2%), o-xylene (12.2%), ethylbenzene (5.3%) and benzene (1.3%). Multivariate statistical analyses indicated that the BTEX levels and their distributions were controlled mainly by morphological and textural features of the sediment; anthropogenic inputs transported by the surrounding rivers, mainly influenced by agricultural facilities; absorbance of BTEX compounds in sediment; and biodegradation processes. Due to lack of any national sediment quality guideline regarding BTEX, the results will establish a significant baseline that will shed light on the administrative authorities for formulating their rational environmental strategies.

**Keywords:** Monoaromatic Hydrocarbons; Contaminated Sediment; Pollution Sources; Lake; Multivariate Analysis

### Introduction

The monoaromatic hydrocarbons of the BTEX group (benzene, toluene, ethyl benzene and the sum of meta-, para- and ortho-xylene) are volatile aromatic compounds of the gasoline range organics. They are found in crude oil, coal tar, and petroleum products (oil refining and manufacturing processes) and are widely used as industrial solvents for organic synthesis. Contrary to other petroleum hydrocarbons (e.g. PAHs), the BTEX compounds are relatively mobile in the environment, usually evaporate quickly into the atmosphere. However, BTEX compounds may be introduced into water and dissolve in water; especially near natural oil and gas fields and at the contaminated sites because of spills of oil and/or petroleum products, industrial effluents and atmospheric pollution. BTEX congeners at

high concentrations have serious adverse effects on aquatic and terrestrial organisms (Kang et al., 2006; Chen et al., 2011; Wang and Fingas, 1998) and therefore included in the purgeable priority pollutants list issued by US Environmental Protection Agency and the European Agency of Chemicals.

The Lake İznik is a freshwater lake with a surface area of approximately 313 km<sup>2</sup> (Figure 1). It contributes to regional and national economy in terms of irrigational and industrial water supply, fisheries and recreational activities (Budakoğlu, 2000). It is a mesotrophic lake with medium level nutrient enrichment, possibly due to intensive land use around the lake (Akkoyunlu, 2003). The lake is fed by various short streams, mostly Sölöz, Oluk, Karadere and Kiran, having a total annual flow rate between 132 and 273 million m<sup>3</sup>. As

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it is 80-90 m above the mean sea level, it also discharges its water into the Gulf of Gemlik via the Karsak River at the west (Figure 1).

There are a few scientific researches about the bottom morphology, sediment texture, geochemical and paleolimnological properties of the bottom sediment of the Lake İznik. The Lake İznik is situated in an active depression field created by faults developed in relation to the still-active North Anatolian Fault (Öztürk et al., 2009). The lake is divided into three separate basins (Figure 1); the deepest 75 m. The meltwater inflow into the lake was started 18,000 years ago, causing carbonate deposition depending on the water level (Roeser et al., 2012). The sub-bottom sediments reflecting the last 100 years are consisted from allochthonous siliciclastic and autochthonous material deposited with different sedimentation rates

(Franz et al. 2006). The most striking finding is the significant changes of physicochemical parameters (e.g. enrichment of TOC, TN, TP, Pb, Cu, Zn and increasing diatom and cladoceran contents) which was interpreted as a serious anthropogenic shift in 1980, causing eutrophication (Franz et al. 2007) and trace element pollution (Ünlü et al. 2016). Serious changes of land types and intense farming with enhanced use of fertilizers and irrigation were also reported especially for the northern farmlands. Furthermore, the polycyclic aromatic hydrocarbon concentrations in the lake sediments reflect a mixture of atmospheric input of high temperature pyrolytic processes and the petrogenic sources transported by the creeks Öztürk et al. (2007) and Ünlü et al. (2010).

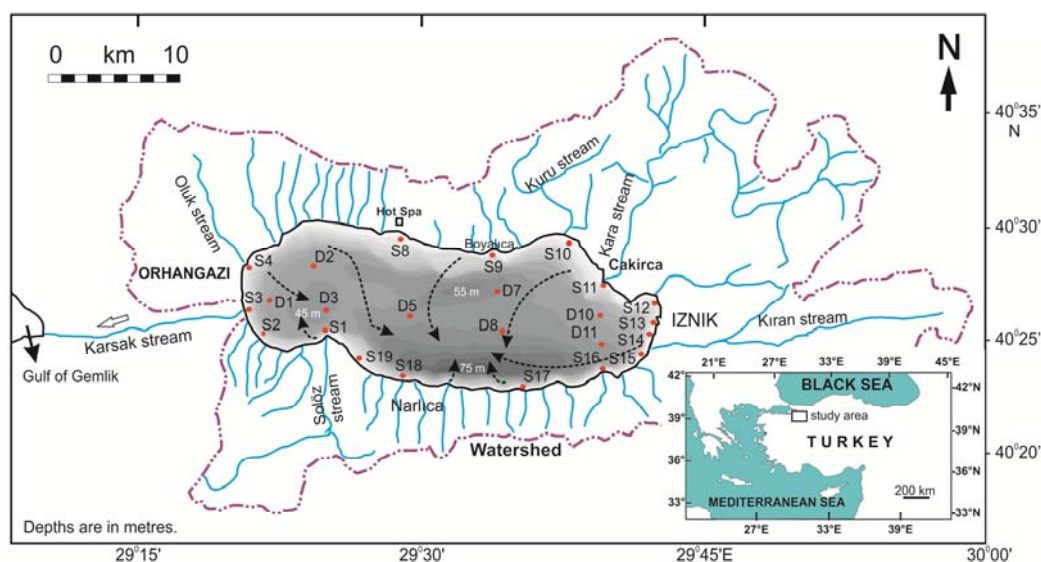


Figure 1. Sediment sampling stations superimposed on the bathymetry map of Lake İznik which is made up of three separate sub-basins (after Öztürk et al., 2009). Dashed arrows show the sediment transportation pathways (Ünlü and Alpar, 2016). Inset shows location.

The highest values were reported offshore İznik and Orhangazi, and the Sölöz creek. The authors also reported that there were no harmful biological effects on the short term to aquatic life. The present study provides the first baseline data on the levels, spatial distribution and possible sources of the BTEX compounds from re-refined petroleum products in the bottom sediments of Lake İznik.

## Materials and Methods

### Sampling procedure

The sediment samples collected from 24 sampling points using a van Veen type grab sampler were frozen until laboratory analyses. The depths of the samples were between 2 and

52 metres. The samples starting with “D” represent deep stations whilst the sample names starting with “S” show shallow stations (Figure 1). The topmost 3 cm parts of the grab samples were removed carefully using clean stainless steel spatula. The sampling vessels were capped and stored on a dry-ice bed inside the collection box. The samples were frozen to -20°C immediately after sampling and analyzed within 48h of collection.

#### **Elemental analysis**

The carbon contents were measured by a Thermo Finnigan FLASH EA1112 (Bremen, GmbH) model CHN analyzer at the MERLAB Central Research Laboratory of Istanbul University. The precision was better than  $\pm 4\%$  at 95% significance level from five replicates.

#### **Chromatographic analysis**

The qualitative and quantitative identification of BTEX compounds 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. The details of HS-GC-MS procedure used in this study are given by Ünlü and Alpar (2017). One gram of sediment sample was weighted in a 10 ml standard glass vial. The sample heated in headspace auto sampler 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<sup>-1</sup>, up to 200°C and finally held for 2 min. Electron impact ionization (EI) was used at 70eV 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) (30m x0.32mm i.d., 0.25µm film thickness) was used to obtain the reference data by chromatography.

#### **Analytical quality assurance**

The 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). Detection limits and recoveries were obtained from analysis of

three replicates standard solutions at concentrations between 1.5 and 50µg L<sup>-1</sup>. The recoveries, relative percentage difference and limit of detection (LOD) were 70-130%, <15%, and 0.25-0.5 µg kg<sup>-1</sup> (dw), respectively. 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 Thermo Scientific Xcalibur software (Waltham, MA, USA) was utilized for instrumental control and intensity measurements. Calibration data were developed using TurboQuant Analyst 6.0 software (Thermo Nicolet Corp. Madison, USA).

#### **Statistical Analyses**

Pearson's correlation coefficients (*r*) were calculated as statistical measures of the strength of linear relationships among the monoaromatic hydrocarbon concentrations, water depth, sediment grain size, TOC content, TPH and ΣPAHs. The principal component analysis (PCA), with varimax rotation, was used in order to quantify spatial/temporal variability of BTEX sources for the sediment samples. The first three eigenvalues (>1) retained were 4.5, 3.0 and 1.3 (n=24)

### **Results and Discussions**

#### **Textural characteristics and organic carbon content of sediment**

In this study, coarse and fine-grained sediments were analyzed using a dry sieve and the hydrometer methods, as described by GERG SOP-8908. Sediment particle size and organic matter content are the most important physical factors affecting the sediment distribution of organic, natural, and anthropogenic compounds. Table 1 summarizes the results for particle size and TOC percent in sediment samples collected from Lake Iznik. The silt fractions were highest at the deep basins of the Lake İznik, between 75 and 93%, whilst the coarse-grained sediments were common along the coastal regions, with water depth less than 35 m. This general appearance in texture changes slightly at river mouth regions (Figure 2). The content of organic carbon in the samples from deep (D1-D11) and shallow stations (S1-S19) ranged from 2.1 to 5.5 %,

with a mean value of 3.3% and from 0.5 to 4.4 %, with a mean value of 1.8 %, respectively (Table 1).

The elevated levels were observed at the stations D1, D10, D11, S18, S8, S1 and S2 (Figure 2). On the other hand, the low-level values were distributed along the eastern coasts and between the deep basins. The mean organic carbon content of the deep samples (>10 m) was 1.8 times higher than that for the shallow samples. The proportion of total organic carbon to total nitrogen (C/N ratio) of the sediment samples were between 2.4 and 24.9 (mean 9.3) for shallow stations, while they were between 6.6 and 27.8 (mean 11.6) for deep stations (Figure 2). High ratios of C/N ratio (>20) in lakes usually show some terrestrial sources or

unproductivity which may be the case for oligotrophic lakes (Meyers and Ishiwatari, 1995). Another possibility is the organic matter derived from dissolved sources of phytoplankton in the lake (Hecky et al., 1993). Considering the spatial characteristics of the C/N ratio (Figure 2), a mixture distribution of terrestrial and lake origin sources may be considered. In addition, at some shallow stations (S9, S13, S15 and S19) the C/N ratio was found to be less than 4 (Figure 2). Such a decrease in C/N ratio of organic matter is generally indicative of carbon mineralization and microbial immobilization of nitrogen, in other words, planktonic and/or microbial activity in organic matter (Gomes and Azevedo, 2003).

Table 1. Grain size, total organic carbon (TOC) and ratio of carbon and nitrogen for sediment samples from Lake İznik

Sampling Stations	Range	Depth (m)	Grain size fractions (%)			TOC (%)	C/N
			Sand	Silt	Clay		
Deep Stations (8 samples)	Min.	39.0	3.28	74.5	4.3	2.1	6.6
	Max.	52.0	12.9	92.4	17.7	5.5	27.8
	Mean	45.3	7.9	81.4	10.7	3.3	12.2
Shallow Stations (16 samples)	Min.	2.0	12.0	0.4	0.0	0.5	2.4
	Max.	32.5	95.5	77.6	10.4	4.4	24.9
	Mean	8.4	78.8	13.2	1.5	1.8	9.2

**Distribution of the BTEX compounds in surface sediments**

The total concentrations of BTEX compounds in the sediment of Lake İznik varied between 272.3 and 10.6 µg kg-1 dw, with a mean value of 79.4 µg kg-1 dw (Table 2). Elevated levels of ΣBTEX (>100 µg kg-1) were observed at the coastal stations (mainly S10, S2, S1, S4, and S11) and the deep stations (D2, D3, D8 and D10) in general (Figure 2). Even the highest levels were observed at some coastal stations, the mean concentration of ΣBTEX for the “D” station sediments (106.1 µg kg-1 dw) was higher than “S” station sediments (79.4 µg kg-1 dw). The maximum contributions of individual BTEX to the total are in the following order: m-, p-xylene (129.1 µg kg-1, dw) > toluene (99.1 µg kg-1, dw) > o-xylene (25.4 µg kg-1, dw) > ethylbenzene (18.7 µg kg-1, dw) > benzene (4.3 µg kg-1, dw).

There are only a few studies in literature that have been performed on BTEX distribution and sources in lake sediments. A comparison show that the maximum contributions of BTEX in Lake İznik were lower than that from the lakes Lane and Vakif; but higher than that from the lakes Mecidiye and Gelibolu (Table 3). The most important factors affecting the spatial distribution of BTEX compounds are sediment texture, distance to the pollutant sources (depth), soil erosion, the higher proportion of high-molecular weight PAHs and various biodegradation processes such as photodecomposition, microbial activity, and denitrification, etc.

Results indicated that benzene concentrations were dominant only in the deeper stations (>30 m; all D stations and S16) which were between 1.1 and 4.3µg kg-1, dw (Figure 2). It disappears above -30 m water depth, due to the combine

effects of volatilization, dissolution, photochemical activity, degradation by microorganisms and sorption. Contrary to this overall appearance, the benzene concentration measured at the station S4 must be related with high level of  $\Sigma$ PAHs at this coastal station (Figure 2). The concentration of TPH in the sediments of Lake İznik are low to moderate

(Öztürk et al., 2007). An acceptable upper limit is  $50 \mu\text{g g}^{-1}$  for clean sediments (Gomes and Azevedo, 2003) and concentrations higher than  $100 \mu\text{g g}^{-1}$  dry weight are mainly related with terrestrial (riverine) inputs, based on classification adapted by Readman et al., (2002).

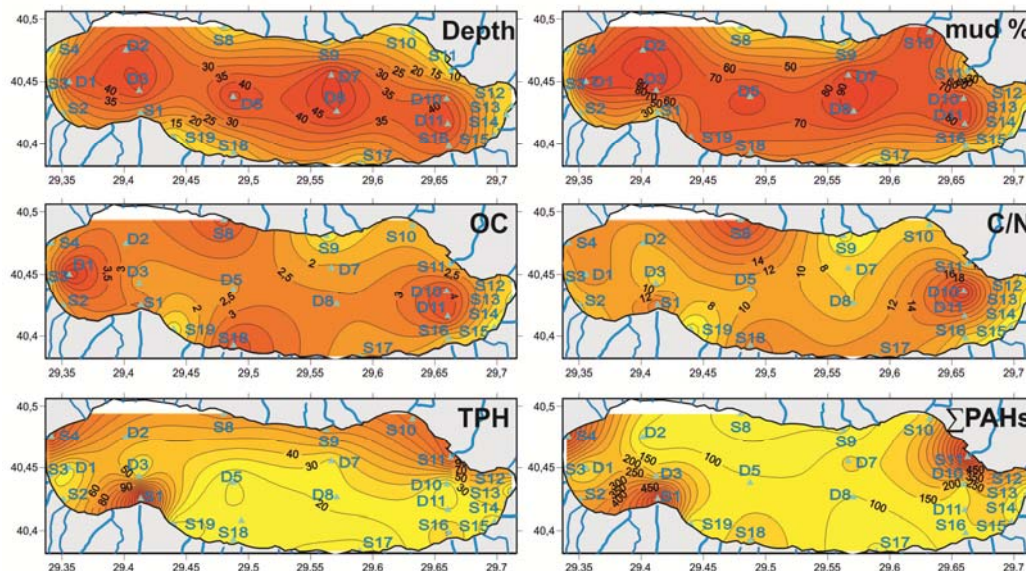


Figure 2. Spatial distribution of the depth (m), mud (%), organic carbon (%), C/N ratio, TPH ( $\mu\text{g kg}^{-1}$ , dw) and TPAHs ( $\text{ng g}^{-1}$ , dw) in the sediment of the Lake İznik. TPH and  $\Sigma$ PAH data are from Öztürk et al. (2007) and Ünlü et al. (2010).

Table 2. Concentrations of sediment samples from Lake İznik ( $\mu\text{g kg}^{-1}$ , dw).

Stations	Mono aromatic fractions	Range		Mean	% of $\Sigma$ BTEXs
		Min	Max		
Deep Stations	B	1.1	4.3	2.8	2.6
	T	18.1	34.1	25.9	24.4
	EB	3.4	8.5	6.0	5.7
	<i>m-,p-X</i>	34.4	97.9	57.9	54.6
	<i>o-X</i>	0.2	20.2	13.5	12.7
	$\Sigma$ BTEX	69.9	143.3	106.1	100
Shallow Stations	B	<DL	2.3	0.4	0.4
	T	10.6	99.1	28.4	35.8
	EB	<DL	18.7	4.0	5.0
	<i>m-,p-X</i>	<DL	129.1	37.3	47
	<i>o-X</i>	<DL	25.4	9.3	11.7
	$\Sigma$ BTEX	10.6	272.3	79.4	100

Only the sediment samples were higher than 100µg g<sup>-1</sup>, dw (Figure 2), which confirm the existence of a chronic oil pollution due to rivers. In addition, these sampling points are located close to the mouths of the rivers Kara, Sölöz and Oluk (Figure 1). According to Ünlü et al., (2010), the concentrations of ΣPAHs in the sediments of Lake Iznik are low to moderate and the contamination of 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 domination of combustion-related sources. The <0.5 values of ratios Flu/(Flu+Py), herein the stations S1, S7, S11 and S15, mostly show petroleum contaminated samples, and gasoline, diesel and fuel oil combustion (Yunker et al., 2002).

Table 3. Comparison of maximum values for light aromatic BTEX fractions (µg kg<sup>-1</sup>, dw) in sediments from different regions.

Location	B	T	E	m-, p-X	o-X	References
Lake Lane, SE New Mexico	128	27	281	205	136	Bristol, 1998
Lake Vakıf, NE Aegean Sea	1.7	95.2	27.1	244.6	<DL	Ünlü and Alpar, 2017
Lake Mecidiye, NE Aegean Sea	<DL	32.4	3.7	27.2	<DL	Ünlü and Alpar, 2017
Lake Gelibolu, NE Aegean Sea	3.5	67.3	11.6	68.3	<DL	Ünlü and Alpar, 2017
Lake Iznik, Marmara Region	4.3	99.1	18.7	129.1	25.4	This Study

In this study, the spatial distribution of and toluene, ethylbenzene and m-,p-xylene, concentrations are similar (except at the samples S12, S13 and S14) (Figure 3). In particular, the total contribution of TEX congeners in deep (24.4:5.7:54.6:12.7) and shallow (35.8:5.0:47.0:12.0) sediment samples show that the pollution sources were not unique or having a similar same distribution pattern. As a result, the present data has shown that m-, p-xylene was the dominant congener (50.0 % in average) in the lake sediment samples, and followed by toluene (31.2%), o-xylene (12.2%), ethylbenzene (5.3%) and benzene (1.3%) (Figure 4). According to the U.S. Department of Health and Human Services, the commercial xylene usually contains 40-65% m-xylene and up to 20% each of o- and p-xylene and ethylbenzene. Xylene mixtures are distributed throughout the environment, especially where they are produced and used as industrial solvents. In addition to accidental gasoline spills, release of toluene may occur in association with disposal of municipal sludge, domestic manufacturing, processing facilities and pesticide applications. Sorption of toluene depends on particles size and in certain conditions occurs with diffusion and adsorption in macro and micropores (Arocha et al., 1996). The concentrations of o-xylene were also

detected high at the samples S1, S2 and D3, possibly influenced by the transported material of the Sölöz creek.

In the present study, the linear regressions among the concentrations of individual BTEX compounds, depth, grain size fractions (silt and clay), organic carbon content, levels of TPH and ΣPAHs show moderate correlations (Table 4). The correlation matrix exhibits relatively consistent results between the concentration levels of m-,p-xylene, ethyl benzene and toluene (0.75>r> 0.95; p<0.01).

**Sources of BTEX in Lake Sediment**

Principle component analysis (PCA) technique reveals if relevant relationships were existed between the cases, that is, the concentration of the BTEX congeners, sediment texture, organic carbon and ΣPAH in our case. Three principal components (eigenvalues >1) were calculated using a covariance matrix of the pollutants in question and additional data needed for normalizations. Their variance loadings were 40.9, 27.2 and 11.6% of the total variability, and therefore with an accumulative variance of 79.7%. As these factors were used to identify the source categories and the loadings of different BTEX congeners are shown in Figure 5.

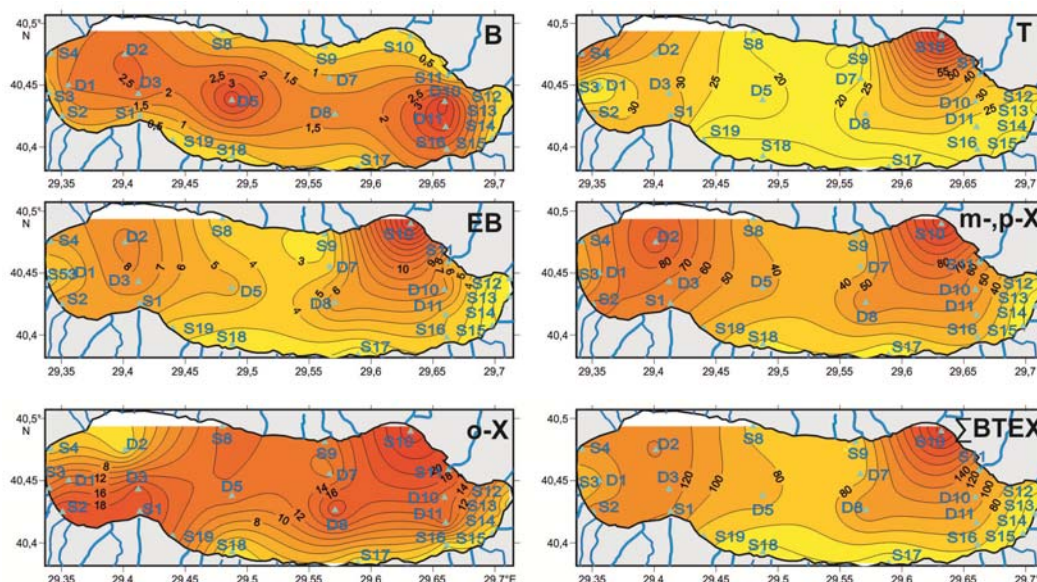


Figure 3. Spatial distribution of BTEX and their fractions ( $\mu\text{g kg}^{-1}$ , dw) in the sediment of the Lake İznik.

The first factor exhibits higher loadings for depth, fine-grained sediments, TOC and mobility of benzene (Figure 5). 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 aerobic/anaerobic biodegradation of benzene is resulted from photo-degradation and microbial activity (Sei and Fathepure, 2009).

Table 4. Pearson-coefficient correlation matrix (r) between depth (m), silt (%), clay (%), TOC (%), TPH ( $\mu\text{g g}^{-1}$ ),  $\Sigma\text{PAHs}$  ( $\text{ng g}^{-1}$ ) and the levels of BTEX compounds ( $\mu\text{g kg}^{-1}$ , dw) in the sediment of Lake İznik (n=24).

	Depth	Silt	Clay	TOC	TPH	$\Sigma\text{PAH}$	B	T	EB	<i>m-, p-X</i>
Silt	0.810**									
Clay	0.713**	0.783**								
TOC	0.458*	0.549**	0.351							
TPH	-0.230	-0.325	-0.338	-0.017						
$\Sigma\text{PAH}$	-0.157	-0.223	-0.218	-0.016	0.809**					
B	0.822**	0.701**	0.518**	0.495*	-0.224	-0.112				
T	-0.087	0.247	0.153	-0.070	0.350	0.352	0.023			
EB	0.229	0.527**	0.327	0.164	0.151	0.186	0.273	0.849**		
<i>m-, p-X</i>	0.309	0.563**	0.293	0.252	0.219	0.206	0.316	0.746**	0.946**	
<i>o-X</i>	0.213	0.326	0.302	0.215	0.176	0.235	0.167	0.413*	0.628**	0.636**

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



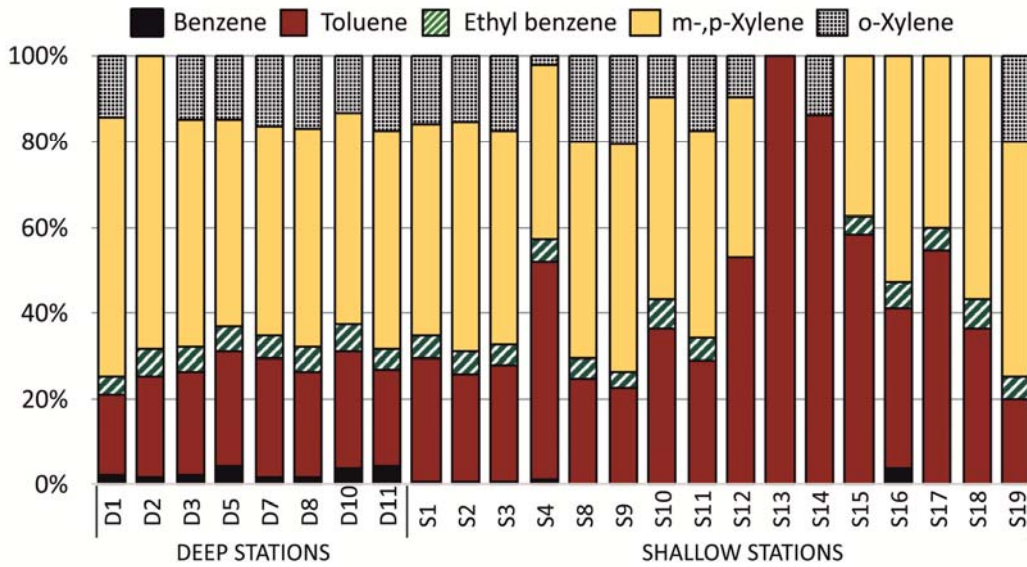


Figure 4. Contributions of BTEX levels in the sediments from the Lake İznik.

The second factor predominantly composed by TEX congeners (m-,p-xylene, ethylbenzene, o-xylene, and toluene); all used extensively as solvents and as raw materials in the synthesis of a variety of chemicals. This factor separates dominant point sources, which may indicate agricultural facilities (e.g. use of nitrate and phosphate), unrefined domestic inputs, industrial activities (use of paints and solvents) increased riverine input and slightly

atmospheric deposition. The most significant samples showing point sources include S10 (Kuru stream), S1, S2 and S4 (Sölöz and Oluk streams), and S11 (Kara stream). TEX 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).

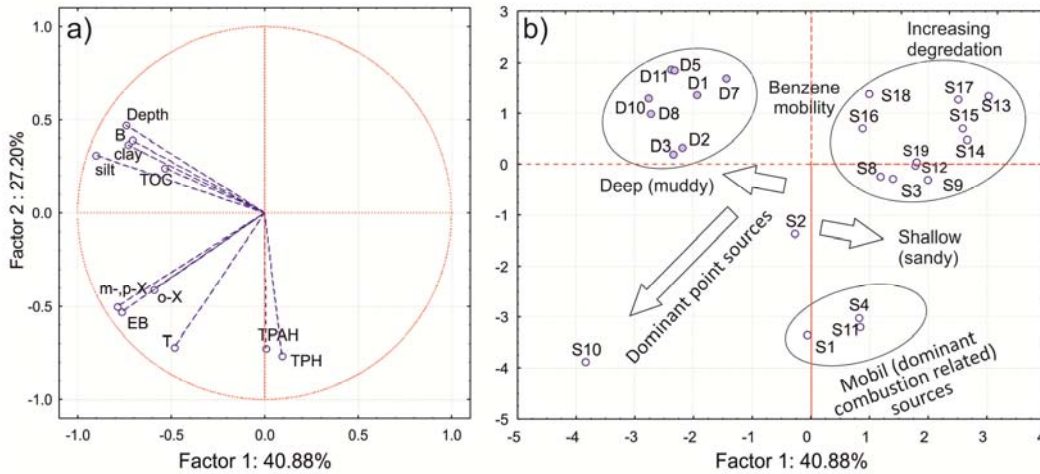


Figure 5. The projection of variables (a) and cases (b) on the factor plain 1x2.

The third factor exhibits local pollution effects of  $\Sigma$ PAH and partly TPH. This factor mainly controls the stations S1, S4 and S11. Indeed, the PAHs in the Lake sediments were derived to a mixture of the pyrolytic input coming from anthropogenic combustion processes such as industry, vehicles and heating processes

### Conclusion

The spatial distribution and potential sources of the BTEX compounds were investigated in the sediments of Lake İznik, a freshwater lake located at an altitude of 85 m and under threat due to anthropogenic pollution. Multivariate factor analyses were used to investigate the potential source apportionment of BTEX compounds. Clear spatial distributions were observed, mainly depending on the water depth, texture, TOC content and mobility of benzene. Land-based pollution sources, mainly riverine activity, agricultural facilities, unrefined domestic inputs, and industrial activities, were the most dominant factors in the explanation of such an accumulation, evaporation and distribution of monoaromatic hydrocarbons in the sediment. Riverine sources are mainly related to excessive use of chemical fertilizers (e.g. nitrate and phosphate) and pesticides in the surrounding region.

Considering these points, lakes are important places in daily life and provide contributions to regional and national economy. The concentration levels and distribution of the monoaromatic hydrocarbon concentrations in the sediment of the Lake İznik serve an important baseline data. The results may enlighten the administrative authorities for preparing their rational environmental strategies, since there is not any national guideline for sediment quality to compare. As the Lake İznik is one of the most fragile lakes due to increasing anthropogenic pollution, the citizens, farmers and private industries must be encouraged to decrease the level of contaminants that pollute the lake.

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