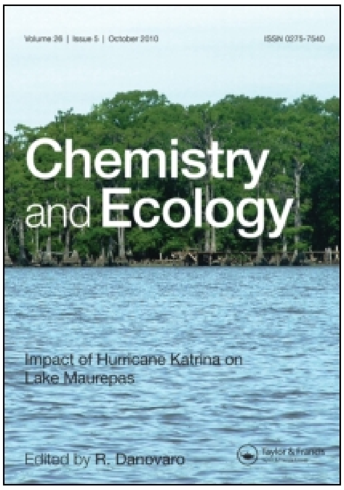

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Distribution of polychlorinated dibenzo-*p*-dioxins, polychlorinated dibenzofurans, dioxin-like polychlorinated biphenyl and polycyclic aromatic hydrocarbons in the sediment of Temsah lake, Suez Canal, Egypt

Pietro Tundo[a]; Stefano Raccanelli[b]; Laila A. Reda[c]; Mohamed Tawic Ahmed[c]
[a] Dipartimento Scienze Ambientali - Universit Ca' Foscari di Venezia, Venezia, Italy [b] Consorzio Interuniversitario Nazionale La Chimica per l'Ambiente, INCA, Via della Liberta' 5/12, Marghera, Italy [c] Faculty of Agriculture, Suez Canal University, Ismailia, Egypt

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DISTRIBUTION OF POLYCHLORINATED DIBENZO-*P*-DIOXINS, POLYCHLORINATED DIBENZOFURANS, DIOXIN-LIKE POLYCHLORINATED BIPHENYL AND POLYCYCLIC AROMATIC HYDROCARBONS IN THE SEDIMENT OF TEMSAH LAKE, SUEZ CANAL, EGYPT

PIETRO TUNDO[a], STEFANO RACCANELLI[b], LAILA A. REDA[c] and
MOHAMED TAWIC AHMED[c,*]

[a]*Dipartimento Scienze Ambientali - Università Ca' Foscari di Venezia, Dorsoduro 2137, 30123 Venezia, Italy;* [b]*Consorzio Interuniversitario Nazionale La Chimica per l'Ambiente, INCA, Via della Liberta' 5/12, 0175 Marghera, Italy;* [c]*Faculty of Agriculture, Suez Canal University, Ismailia, 41522, Egypt*

(In final form 2 March 2004)

Lake Temsah is one of the main wetlands in the Suez Canal region, and the main source for fish for the area. The lake is the end-point of several wastewater effluents. In the present study, residues of polychlorinated dibenzo-*p*-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs), dioxin-like polychlorinated biphenyl (PCB) and polycyclic aromatic hydrocarbons (PAH) were monitored in the sediment of the lake. Samples were collected from six different sampling stations around the lake using a box-corer sampler, then kept frozen. Samples were extracted and cleaned up before residue determination was conducted using an HRGC/HRMS. An HP 6890 plus gas chromatograph was coupled to a Micromass Autospec Ultima mass spectrometer operating in EI mode at 35 eV and with a resolution of 10.000 (5% valley). PCDDs and PCDFs were detected in all sediment samples collected from various sampling stations. Results showed some progressive increase in PCDDs concentrations relevant to increase in chlorination. In the PCDD group of congeners, 1, 2, 3, 4, 6, 7, 8, 9 octa-CDD had the highest detected concentrations in all samples, while 2, 3, 7, 8 tetra-CDD showed the lowest concentrations. The World Health Organization toxicity equivalents ranged from 0.387 to 11.20 ng kg^{-1} d.w. For PCDD homologues, hexa-CDD was the most dominant homologue in all sediment samples analysed. Regarding dioxin-like PCBs, results showed that IUPAC No. 118 congener, 2, 3, 4, 4, 5, pentachlorobiphenyl was the most concentrated of all detected congeners, with concentrations ranging between 0.039 and 43.201 $\mu g\,kg^{-1}$. For polycyclic aromatic hydrocarbons, benzo(b + k + j)fluoranthene had the highest concentrations in almost all sampling stations. However, fluorene was the smallest detected concentration in almost all stations. This result would indicate that PAH contamination of the lake seems to be coming from one main source in all sampling stations. The present work is the first record of PCCDs, PCCFs, and dioxin-like PCBs in the Temsah lake. The concentrations of the contaminants monitored in this study, especially those of PAHs, are rather alarming. Efforts should be made to stop point sources that contaminate the lake.

Keywords: Dioxins; Lake Temsah; Dioxin-like PCBs; PAH; Sediment

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[*] Corresponding author. E-mail: motawfic@tedata.net.eg

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1 INTRODUCTION

Before the construction of the Suez Canal in the 19th century, the Bitter Lakes were relatively small hyper-saline inland lakes surrounded by salt-encrusted sabkha. After the lakes were connected with both the Mediterranean and the Red Sea by the Suez Canal, they became a single marine body; their size increased, and their salinity decreased, reaching between 43 and 46 g l^{-1} (Bahaa El Dien, 1999). The northern wider end of the water body is known as the Great Bitter Lake, while the southern narrower part is know as the Little Bitter Lake. The bottom is sandy and scantily covered with vegetation. Agricultural land, tourist development and occasional areas of salt marsh border the lakes on the western side, while the eastern side is mostly sandy desert.

Lake Temsah, one of the small lakes that constitute the Bitter Lakes and located on the north of the Suez Canal, is a land-engulfed embayment with a total area of 15 km^2. The lake is the end-point of various outlets that discharge large volumes of agricultural, municipal and industrial wastewater. The lake is bounded by Ismailia, the main city of the region that discharges parts of its raw and partially treated municipal waste into the lake. The lake is the main wet dock of the city, a small port that also harbours a variety of marine works, including the maintenance work of the Suez Canal Authority and its allied maritime works. The lake is a repository site for a variety of pollutants, including pesticides and heavy metals originating from farming and industrial activities in the vicinity of the lake. Some of the drains discharging into the lake are leaden with a variety of industrial pollutants originating from industrial zones in Cairo.

The lake is also the end-sink of aliphatic and aromatic hydrocarbons that originate from shipping activities, ballasting water, maintenance and maritime works in the several docks around. The lake supports fishing and tourism industries that employ a large number of local citizens and provide a significant portion of the district revenues.

Several studies were conducted to monitor persistent organic pollutants in the different component of the lake, including different marine organisms (Tawfic Ahmed and Ismail, 1991; Tawfic Ahmed *et al.*, 2001). Chlorinated hydrocarbon pesticides, polycyclic aromatic hydrocarbons (PAHs) and polychlorinated biphenyl (PCB) were the compounds mapped out in these studies.

Polychlorinated dibenzo-*p*-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs) are ubiquitous contaminants with a long persistence that tend to accumulate in sink areas such as sediment, the main repository site of persistent organic pollutants in the lake ecosystem. Contaminated sediment could be a pathway for the redistribution of PCDDs and PCDFs and their introduction into the food chain. PCDDs and PCDFs also tend to accumulate in adipose tissues of exposed organisms, causing a variety of deleterious effects on biological systems.

PCDDs and PCDFs are produced from various combustion sources and manufacturing processes, such as combustion (Baker and Hites, 2000), incinerators (Schuhmacher *et al.*, 1997), and vehicle exhausts (Miyabara *et al.*, 1999). These contaminants are mainly transported to aquatic systems through the atmospheric deposition or directly via rivers (Bruzuzy and Hites, 1996).

PAHs are among the most toxic environmental contaminants. Various reports have documented their carcinogenic and mutagenic effects (endocrine disturbers). Some reports have shown that PAHs could be naturally occurring (Preziosi, 1998). Nevertheless, a typical and important source of PAHs emission into the environment is the incomplete combustion of fossil fuel and organic materials from incinerators, residential heating, power generators and industrial facilities.

2 MATERIALS AND METHODS

2.1 Chemicals

All solvents (*n*-hexane, dichloromethane, acetone, toluene, ethylacetate) were of Picograde® reagent grade (Pomochem GmbH, Wesel, D). Native and deuterated PAH standards were purchased from Supelco (Belfonte, PA); native and $^{13}C_{12}$-labelled PCDD/F, PCB, HCB and acenaphtilene-D8, benzo[e]pyrene-D12 standards were purchased from Cambridge Isotope Laboratories (Woburn, MA); and the hydroscopic sample dispersing agent (Spe-ed Matrix) was purchased from Applied Separation (Allenton, PA).

All glassware was washed with basic detergent, rinsed with distilled water, treated with a solution of ammonium persulphate (350 g l^{-1}) in sulphuric acid (98%) and rinsed twice with distilled water and acetone. Subsequently, the cleaned glassware was treated with 5% dimethyldichlorosilane in toluene, rinsed twice with distilled water and acetone, heated to 300 °C for 3 h and covered with aluminium foil.

2.2 Sediment Sampling

Surface sediments were sampled at six sites of the lake Temsah during December 2002 (Fig. 1). The sampling sites were selected from evidence of pollutant accumulation, based on previous studies (Tawfic Ahmed and Ismail, 1991; Tawfic Ahmed *et al.*, 2001) and based on outlets of effluents discharged into the lake. Sites were also selected to include potential polluters such as dry docks and ships and tankers passing through the Suez Canal. Accessibility and spatial distribution were also considered. Samples were collected

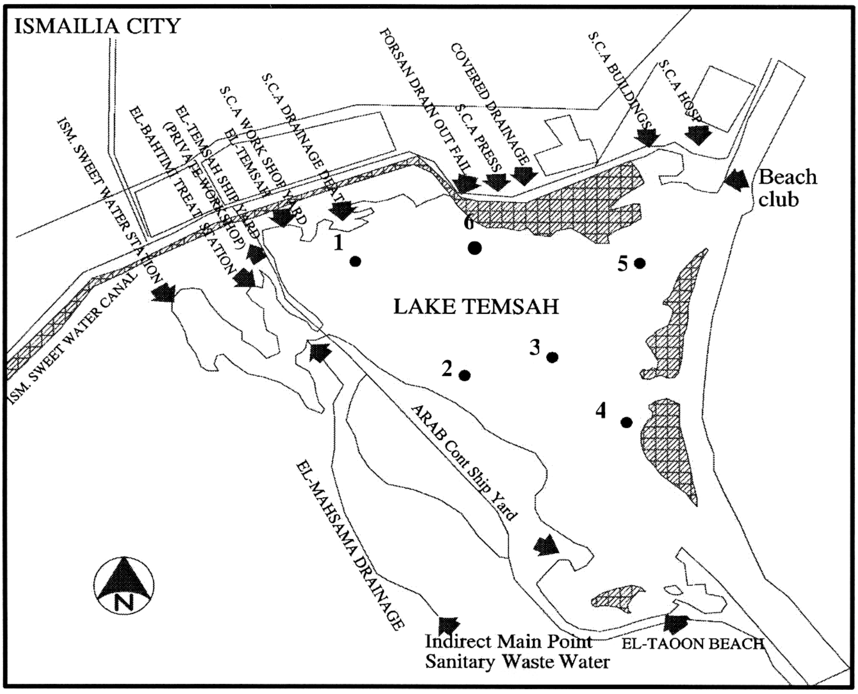

FIGURE 1 Lake Temsah and sampling stations.

from a depth ranging from 2.7 to 3.2 m. Samples were collected with a box-corer sampler, and about 2 kg from the middle of each sample was placed into a polyethylene bag and then kept frozen.

The sediment samples were transported (while kept in frozen storage) back to the laboratory and stored at 4 °C before analysis. In the laboratory, the sediment samples were thoroughly mixed with a stainless-steel spatula, and 10.0 ± 0.1 g sub-samples were transferred to a glass container for analysis.

2.3 Extraction

The samples were first spiked with a series of 15 $^{13}C_{12}$-labelled 2, 3, 7, 8 PCDD/F (EDF8999) with a series of 12 $^{13}C_{12}$-labelled PCB (EC4937), with $^{13}C_{12}$-HCB (CLM351) substituted isomers, with a series of five deuterated PAH (Acenaphtene-D10, Chrysene-D12, Naphtalene-D8, Perylene-D12, Phenanthrene-D10) as internal standards, mixed with Spe-ed Matrix. Extraction by ASE 200 (DIONEX Sunnyvale, CA) was performed with 50 ml of *n*-hexane/dichloromethane 50/05 at 150 °C, 1500 psi, 7 min heat-up and two cycles of 10 min static time. The extracts were transferred to hexane before clean-up.

2.4 Clean-up

The sample extracts were first spiked with 37C4-labelled 2, 3, 7, 8 PCDD (EDF6999) and with three $^{13}C_{12}$-labelled PCB (EC4978) and then cleaned up using the automatic system, Dioxin Prep (Fluid Management System). Pre-packed disposable columns containing multilayer silica and sodium sulphate were used for PAH clean-up. After PAH analysis, the extracts were treated with sulphuric acid (98%) and potassium hydroxide (20%) in a 100 ml separatory funnel and then cleaned up using the automatic three column system with pre-packed disposable columns containing multilayer silica, alumina and carbon for PCDD/PCDF, PCB and HCB.

2.5 High-Resolution Gas Chromatography Mass Spectroscopy Analysis

The HRGC/HRMS analyses were conducted using an HP 6890 plus gas chromatograph coupled to a Micromass Autospec Ultima mass spectrometer operating in EI mode at 35 eV and with a resolution of 10.000 (5% valley).

2.6 Dioxins and Furans

PCDD/F sample injections were performed in the splitless mode on a 60 m DB5 MS column (J&W 0.25 mm ID, 0.25 μm film) and on a 60 m Rtx 200 (Restek 0.25 mm ID, 0.25 μm film) for verification.

The quantitative determination of PCDDs/PCDFs was performed by an isotope dilution method using relative response factors previously obtained from five standard solutions injections (EDF 9999 Cambridge Isotope Laboratories, Woburn, MA).

At the beginning of each day during which analyses are performed, the GC/MS system performance and calibration were verified for all CDDs/CDFs and labelled compounds with the CS3 calibration verification standard and the isomer specificity test standard. Two $^{13}C_{12}$-labelled PCDDs (EDF5999) were added to the extract before injection for recovery calculations. The recovery always ranged between 55% and 105%. The reproducibility was 15% for a lower value or better.

Sample injections were performed in the splitless mode on a 30 m Rtx 5 MS column (Restek 0.25 mm ID, 0.25 μm film) with the column held at 60 °C for 1 min, then heated to 140 °C at 15 °C min^{-1}, then heated to 300 °C at 6 °C min^{-1}, then heated to 325 °C at 4 °C min^{-1} and held at 325 °C for 4 min. The carrier gas was helium (1.0 ml min^{-1}). The injector and transfer-line temperatures were 290 °C and 300 °C, respectively. Mass spectral data were acquired in the selected ion monitoring mode.

2.7 Polycyclic Aromatic Hydrocarbons

The quantitative determination of PAHs was performed using an isotope dilution method with relative response factors previously obtained from three standard solutions injections.

Two deuterated PAHs (acenaphthilene-D8, benzo[e]pyrene-D12) were added to the extract before injection for recovery calculations. At the beginning of each day during which analyses were performed, the GC/MS system performance and calibration were verified for all PAHs and deuterated compounds using the ST2 calibration-verification standard.

The corrected response for an analysis varied by more than 15% from the calibration standard of the analysis sequence. The recovery always ranged from 40% to 110%. The reproducibility was 20% for a lower value or better. The laboratory blanch tests were performed twice a week to make sure that their values were 10% lower than the least detected concentrations.

3 RESULTS AND DISCUSSION

PCDDs and PCDFs were detected in all sediment samples collected from various sampling stations of Lake Temsah. The spectrum of congeners detected and their concentrations at different sampling stations are shown in Table I.

Tetra-CDD congeners were detected in all samples alike, with concentrations ranging between 1.1 and 5.43 ng g^{-1}, where the highest concentration was detected in station 4.

TABLE I Residues of PCCDs and PCCFs detected in sediment samples collected from Lake Temsah, Ismailia (ng kg^{-1} liquid).

Detected compounds	*Sampling stations*					
	1	*2*	*3*	*4*	*5*	*6*
TCDDs	2.85	3.94	2.98	5.43	1.1	1.00
PeCDDs	5.03	4.8	2.91	5.54	1.55	1.28
HxCDDs	15.46	10.06	6.51	5.91	3.04	2.63
HpCDD	40.99	37.48	11.87	11.14	7.87	3.95
OCDDs	143.75	164.43	30.57	42.37	33.15	8.09
TCDFs	12.22	14.84	12.45	77.06	3.34	3.44
PeCDF	10.27	11.0	9.95	100.22	2.86	2.76
HxCDF	9.52	8.38	6.61	39.36	2.78	1.81
HpCDD	11.36	11.73	4.64	20.86	3.94	1.29
OCDFs	10.69	15.86	3.57	10.14	2.92	0.88
Sum PCDDs/PCDFs	261.87	282.45	92.05	317.97	62.44	26.83
ΣPCDDs/ΣPCDFs	208.08/5	220.71/6	54.83/37	70.75/24	46.62/15	16.65/10
	3.79	1.75	0.22	7.85	0.82	0.18
	3.8	3.5	1.47	0.285	2.9	1.6
WHO-TE	1.756	1.75	1.402	11.294	0.462	0.387

Results showed some progressive increase in PCDDs concentrations relevant to the increase in chlorination in all stations, with octa-CDD showing the highest concentrations of all PCDDs in all stations. However, the concentrations of PCDFs detected in all stations, with the exception of station 4, decreased with increasing degree of chlorination. In the PCDD group of congeners, 1, 2, 3, 4, 6, 7, 8, 9 octa-CDD had the highest detected concentrations in all samples, unlike 2, 3, 7, 8 tetra-CDD, which showed the lowest concentrations.

Meanwhile, in the PCCF cluster, samples collected from three of the stations, namely 1, 3, and 6, had their tetra-CDFs congeners as the highest detected congeners, while octa-CDF, penta-CDF and hepta-CDF congeners had the highest concentrations in stations 2, 4 and 5, respectively.

In the present study, the total concentration is a measure of the contamination level of a sample. Meanwhile, the toxicity equivalent (TE) is a measurement of the toxicity derived from a number of toxic PCDDs/PCDFs congeners, in particular, 2, 3, 7, 8, TCDD, 1, 2, 3, 7, 8-PnCDD, 2, 3, 7, 8, TCDF, 1, 2, 3, 7, 8, PnCDF and 2, 3, 4, 7, 8, PnCDF.

The TEs were determined using the new TE factors recommended by the World Health Organization (WHO) for fish and mammals. In the present study, the WHO TEs ranged from 0.387 to 11.00 $\mathrm{ng\,kg^{-1}}$ lipid, with station 4 showing the highest value and station 6 showing the lowest recorded value. The high TE value recorded in sample 4 is probably related to the high level of relevant congeners, including tetra-CDD detected in this sample. Table I also lists the S PCDDs/S PCDFs values. D/F ratio values ranged between 0.289 in sample 4 that had the highest concentration of PCDD/PCDF, and the highest TE, and 3.8 in sample 1 which had the second highest concentration of PCDD/PCDF, with the second highest TE value. No particular pattern was inferred and no relationship was established between S PCDDs/S PCDFs and TE values.

For PCDD homologues, hexa-CDD was the most dominant homologue in all sediment samples analysed. Similarly, for PCDF homologues, hexa-CDD was also the most dominant congener. This result would add up to the previously mentioned view that the source of PCDD/PCDF contamination in the lake seems to be the same. This result may indicate that the source of contamination is not necessarily through atmospheric deposition that tends to enrich the Octa CDD congeners (Moon *et al.*, 2000).

The highest values for PCDDs and PCDFs were also found in station 4, hence indicating a high contamination load in this particular sampling site. There is currently no explanation for the elevated residues of PCDDs, PCDFs and dioxin-like PCBs detected at this site.

The PCDD and FCDD residues detected in the present study and their TEQ are much higher than the residues reported in the sediment samples collected from the Korean coast (Moon *et al.*, 2000) but lower than the residues detected in Detroit River, where the TEQ was as high as 306 $\mathrm{pg\,g^{-1}}$ in some sites (Marvin *et al.*, 2002), and the Panhandle Bay system in Florida, USA (Hemming, 2003).

The levels of dioxin-like PCB residues are listed in Table II. Results indicated an elevated level in sample collected at station 4, with a residue concentration of 99.111 $\mu\mathrm{g\,kg^{-1}}$, and an equivalent toxicity of 41.049 $\mu\mathrm{g\,kg^{-1}}$. Results also showed that IUPAC No. 118 congener, 2, 3, 4, 4, 5, pentachlorobiphenyl was the most concentrated of all detected congeners, with concentrations ranging between 0.039 and 43.201 $\mu\mathrm{g\,kg^{-1}}$ for stations 6 and 4, respectively.

Di-orthocongeners (Nos IUPAC 180 and 170) were also detected in all samples alike with concentrations ranging from 0.94 to 0.55 $\mu\mathrm{g\,kg^{-1}}$. Similar to PCCDs/PCCFs, the highest concentration of dioxin-like PCBs was detected in sampling site 4. The reasons for such high contamination in that particular site will be investigated in future studies. Table III lists the levels of PAH residues detected in the sampling stations. A variety of 15 PAH were detected in all sampling sites, with total concentrations ranging from 54.6 $\mu\mathrm{g\,kg^{-1}}$ to 27,784 $\mu\mathrm{g\,kg^{-1}}$.

TABLE II Residues of dioxin-like PCBs in Lake Temsah sediment samples ($\mu g\ kg^{-1}$ liquid).

PCBs	*1*	*2*	*3*	*4*	*5*	*6*
81[*]	<0.005	<0.005	<0.005	<0.01	<0.005	<0.005
77[*]	0.015	0.026	0.010	0.062	<0.005	<0.005
123[†]	0.007	0.015	0.006	2.115	<0.0005	<0.005
118[†]	0.416	0.4	0.215	43.201	0.095	0.039
114[†]	<0.005	<0.005	<0.0050	0.344	<0.005	<0.005
105[†]	0.107	0.102	<0.05	6.005	0.027	0.010
126[*]	<0.005	<0.005	<0.005	0.210	<0.005	<0.005
167[†]	0.030	0.026	0.013	12.932	<0.007	<0.005
156[†]	0.053	0.049	0.023	25.698	0.015	0.005
157[†]	0.010	0.009	0.005	2.266	<0.005	<0.005
169[*]	<0.005	<0.005	<0.005	<0.010	<0.005	<0.005
189[†]	0.010	0.015	0.005	6.279	<0.005	<0.005
Total	0.648	0.637	0.327	99.11	0.144	0.054
Total WHOTE	0.087	0.085	0.043	41.05	0.02	0.007
180[‡]	0.523	0.597	0.231	291.681	0.139	0.059
170[‡]	0.0277	0.023	0.131	179.118	0.071	0.035
Total di-ortho-PCBs	0.550	0.62	0.362	470.798	0.21	0.094

[*] Non-orthocongeners.
[†] Mono-orthocongeners.
[‡] Di-orthocongeners.

The levels of PCB residues detected in this study are far lower than those detected in the sediment of Porto Marghera's lagoon (Frignani *et al.,* 2000), sediment samples collected from south-eastern Korea (Choi *et al.,* 2000), and some parts of the USA (Thomas and Spiro, 1996).

The residue levels of PAHs recorded in this study are high, especially in station 5, where a total of 27,784 $\mu g\ kg^{-1}$ is alarmingly high. The proximity of station 5 to a dockyard and a ship-maintenance works where oil and other contaminants spills are likely could be the reason for such a high concentration of PAH. Benzo(a)pyrene has been detected in all sampling sites with varying concentrations. The highest concentration, 4452 $\mu g\ kg^{-1}$, was recorded in sampling site 5.

TABLE III Residues of polycyclic aromatic hydrocarbons detected in sediment samples collected from different stations ($\mu g\ kg^{-1}$ dry weight).

Compound	*Station 1*	*Station 2*	*Station 3*	*Station 4*	*Station 5*	*Station 6*
Naphthalene	22.8	104.6	40.5	50.4	21	6.1
Acenphthilene	6.3	17.9	5.5	4.1	1595	1.1
Acenaphthene	1.6	4.3	1.6	11.9	29	0.3
Fluorene	1.0	1.2	0.6	4.6	67	0.2
Fenanthrene	11.4	15.3	9.2	35.3	20	3.3
Anthracene	4.5	4.4	2.5	6.5	624	0.8
Fluoranthene	19.7	53.6	21.2	34.4	1468	5.4
Pyrene	26	52.9	27.3	29.6	9250	6.3
Benzo(a)anthracene	18.3	28.2	13.1	23.4	355	3.5
Crysene	18.1	30.9	16	28.1	611	4.5
Benzo(b,k,j)fluoranthene	59.3	72.8	40.5	67.5	5069	11.2
Benzo(a)pyrene	22.1	26.1	14.3	17.9	4452	4.2
Indeno(1,2,3,cd)pyrene	19.2	18.5	10.2	15.4	1940	3.6
Dibenzo(a,h)anthracene	3.1	3.3	1.6	5.8	395	0.6
Benzo(g,h,i)perilene	20.4	21.7	11.8	18.6	1888	3.5
Total	253.8	544.7	215.9	343.6	27,784	54.6

With the exception of station 5, the levels of PAH residues detected in the present study are lower than those reported in the sediment collected from Mill River, New Haven (White and Triplett, 2002).

Benzo(a)pyrene is a potent mutagen and carcinogen. Because it is formed when gasoline, garbage, or any animal or plant material burns, it is usually found in smoke and soot. The chemical combines with dust particles in the air and is carried into water and soil and onto crops. It is also found in the coal-tar pitch used in industry to join electrical parts together and is found in creosote, a chemical used to preserve wood.

Unlike PCCDs, PCCFs and dioxin-like PCBs, where their highest residues were detected in sampling site 4, the highest concentration of PAHs were detected in sampling station 5. This may indicate that the lake is exposed to different types of pollution stresses, hence impacting the lake in different spots.

Tawfic Ahmed *et al.* (2001) have reported the PAH level in the Lake Temsah ecosystem, including some marine organisms. The level of contamination in the present study would indicate a significant elevation in PAH residues. This is probably the result of the massive discharge of industrial effluent into the lake.

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