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PCDD/Fs into sediments of Aqaba Coastal City, Jordan: the relation to atmospheric pollution

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ABSTRACT

The present study investigated the source characterization of the polychlorinated dibenzo-*p*-dioxins and dibenzofurans (PCDD/Fs) into sediments of Aqaba Coastal City. Air samples from four location sites and sediment samples from five stations on the Jordanian coast of the Gulf of Aqaba, including 25 individual sediment samples merged by station to form five composite samples, were collected and analyzed for PCDD/Fs. Congener patterns of all air samples were characterized by high octa-chlorinated dibenzo-*p*-dioxin (OCDD) (78% of the ΣPCDDs), followed by octa-chlorinated dibenzofurans (OCDF) (38.5% of ΣPCDFs) and 1,2,3,4,6,7,8-HeptaCDD/Fs. The highest atmospheric concentration values of the total PCDD/Fs were measured at the middle of industrial complex. The results of sediment samples show that among the PCDD/Fs congeners, the OCDD, 1,2,3,4,6,7,8-HeptaCDF, 1,2,3,4,7,8,9-HeptaCDF and OCDF were the prevailing congeners (approximately 77.5%–90%), indicating air and sediment sharing the same main source of pollution. The most toxic congener of the dioxin group 2,3,7,8-TCDD was a very minor component with a contribution of less than 0.54% to the total PCDD/Fs. The toxic equivalents (TEQ) values ranged between 0.011 and 1.08 pg TEQ/g dry matter for all sediments. Concentrations in all sediment samples analyzed significantly below the safe limits. The substantial fraction PCDD/Fs emissions enter air and then carried into the waterway via urban storm water runoff could be the main source of the low concentrations recorded.

Keywords: PCDD/Fs; Dioxin; Furans; POPs; Sediments; Water; WHO-TEQ; Aqaba; Jordan

1. Introduction

Polychlorinated dibenzo-*p*-dioxins (PCDD) and polychlorinated dibenzofurans (PCDF) are the two main unintentionally produced persistent organic pollutants (POPs) included in the Stockholm Convention on POPs [1]. In the environment, PCDD/Fs have tendencies to persist, bioaccumulate in fatty tissues and undergo long range transport [1]. PCDD/Fs are released to the environment from a variety of industrial discharges, combustion processes and by-products in various chemical formulations [2]. Historically the production and combustion of chloro-organic chemicals have been major sources of PCDD/Fs in the environment [2,3].

PCDD/Fs are a class of planar tricyclic aromatic, non-polar, persistent, toxic compounds with similar chemical

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properties containing varying amounts of chlorine [4]. The 17 PCDD/F congeners, which are the most toxic and have a common mode of toxic action, have chlorine atoms attached in at least the 2, 3, 7 and 8 lateral positions.

PCDD/Fs are released into aquatic systems PCDD/Fs by atmospheric deposition due to direct and indirect discharges. Subsequently, they are transferred to sediment and water, taken up by aquatic organisms, ultimately depositing in sediments and eventually bioaccumulate and biomagnified in trophic levels. Hence, sediments may represent the background levels and the evolution of pollution inputs of PCDD/Fs over long period of time [5–7].

Jordan is a signatory to the Stockholm Convention on POPs in 15th August 2002. The first PCDD/Fs releases inventory conducted in Jordan was carried out by the Ministry of Environment through the Asian Project in 2003 based on the year 2000 as a reference year using UNEP Toolkit of 2001. The second inventory was conducted in 2005 using 2002 as a reference year using UNEP Toolkit of 2003. The concept of toxic equivalents (TEQ) is generally used as a single number to express the overall toxicity of PCDD/Fs and to compare the toxicity between sites having different compositions [8].

Hararah et al. [9] conducted PCDD/Fs emission inventory study in Aqaba using a standardized toolkit developed by UNEP Chemicals, and reported that the total emission rate in Aqaba was estimated as 0.875 g TEQ/year. The year 2012 is the reference date for this inventory. The category with the highest estimated emission is the transport sector (i.e., diesel engines), which was contributing to nearly 64.3% of total emissions to air. Heat and power generation are responsible for nearly 5.6% of the total emission to air. Disposal and land filling are estimated to be the only source of dioxin emissions to water and the highest estimated source of emission to residues with annual release of 0.007 g TEQ/y for water and 0.112 g TEQ/y for residues. Sewage/sewage treatment contributes to nearly 43% of the total emission to water and 98% to residue. Open water dumping contributes to nearly 57% of the total emission to water. Open burning processes are estimated to be the only significant source of dioxin emission to land through the 77 accidental fires in houses and factories, and 50 accidental fires in vehicles with annual release of 0.003 g TEQ/y [9].

There is no experimental data published in Jordan on PCDD/Fs levels in sediments, and the information available on PCDD/Fs contamination is scarce. Using experimental sediments and air samples, the present study constitutes the first scientific investigation that provides overview information on the influence of atmospheric input of PCDD/Fs on the delivery and accumulation of PCDD/Fs residues in sediments in the coastal city of Aqaba, as well as, on the entire Red Sea.

2. Materials and methods

2.1. Study area

Aqaba is a coastal city at the southern part of Jordan located at the northern tip of the Gulf of Aqaba (Fig. 1). The Gulf is positioned along the southeastern edge of the Sinai Peninsula. It is an inland extension of the Red Sea and has a length of 180 km, a width of 14–26 km and an average depth of 800 m [10]. The key coastline features are illustrated in Fig. 1.

In the tourism section, the infrastructure has been improved; mega residential and recreation projects have been constructed, more hotels, restaurants, recreation centers and roads have been built, causing more tourists to come to the city from inside the country and from abroad. In the trade sector, port activities have also been expanded; more free flow of goods to and from ports accompanied with an increase in road transportation of 30%. It is estimated that over 1,200 haulage trucks enter and leave Aqaba each day together with several hundred trucks to the industrial zone [11]. In parallel, the industrial activities that take place in the southern industrial area of the city have also been expanded causing more flow of raw materials to Aqaba and more flow of products to flow out. Major developments included expansion of the fertilizers complex by 40%, building new ports in the southern area, as well as increase of chemicals and fuel storage facilities. This development coupled with increasing population growth result in an increasing pressure on the environment, including marine life and air quality.

The topography of Aqaba is characterized by mountain ridges with elevations reaching up to 1,600 m perpendicular to the shoreline and interrupted by a series of intermittent

Fig. 1. The key coastline features (1, City of Aqaba; 2, tourism area; 3, the industrial zone).

valleys of various widths [11]. The network of valleys acts as wind channels that is eventually responsible for the erratic behavior of the wind direction in the vicinity of the Gulf shoreline. The coastal areas experience various changes in wind direction and temperature. The topography allows wind channeling from land through valleys to develop in the afternoon coupled with sea breeze [11].

The climate in Aqaba is arid with an average rainfall of about 30 mm. Daily average temperature ranges between 14°C in January to more than 35°C in August with relative humidity ranges between 30% and 55% [12]. The prevailing winds are primarily from the north (~81%), with around 7% from the south and the remaining calm wind. Periods of poor dispersion occur during winter and late autumn as a result of slow-moving anticyclones, typified by light winds and low mixing heights [12].

The Jordanian Gulf of Aqaba, the northeastern arm of the Red Sea, has suffered from air pollution since it was transformed into a free economic zone in 2001. The major sources of air pollution are emissions from vehicles, emissions from port activities and from the industrial area. One of the main industrial activities is phosphate industry which has been considered as a major source of polluting the environment.

2.2. Sediment samples collection

A total of 25 surface sediments from five locations (0–10 cm depth layer; five samples from each of sites S_1, S_2, S_3, S_4 and S_5) were collected using a solvent-rinsed stainless steel grab samples from the intertidal zone either by walking in the shallow water or by snorkeling. Table 1 describes each of these sample location sites.

A field blank, a blank and matrix spike sample (40–400 pg PCDD/Fs) were used in the analytical procedure for every nine samples for quality control. At each site, five sediment samples were collected from a rectangular area of about 100 m^2; one sample at every corner and a sample in the middle from each site. The five samples were grounded with a pestle and mortar, homogenized and combined into a single composite sample. Samples were freeze-dried, wrapped in prebaked aluminum foil, and then stored at −20°C until chemical analysis in laboratory. The sample locations of each site were geo-referenced using global positioning system technology, as shown in Fig. 2(a).

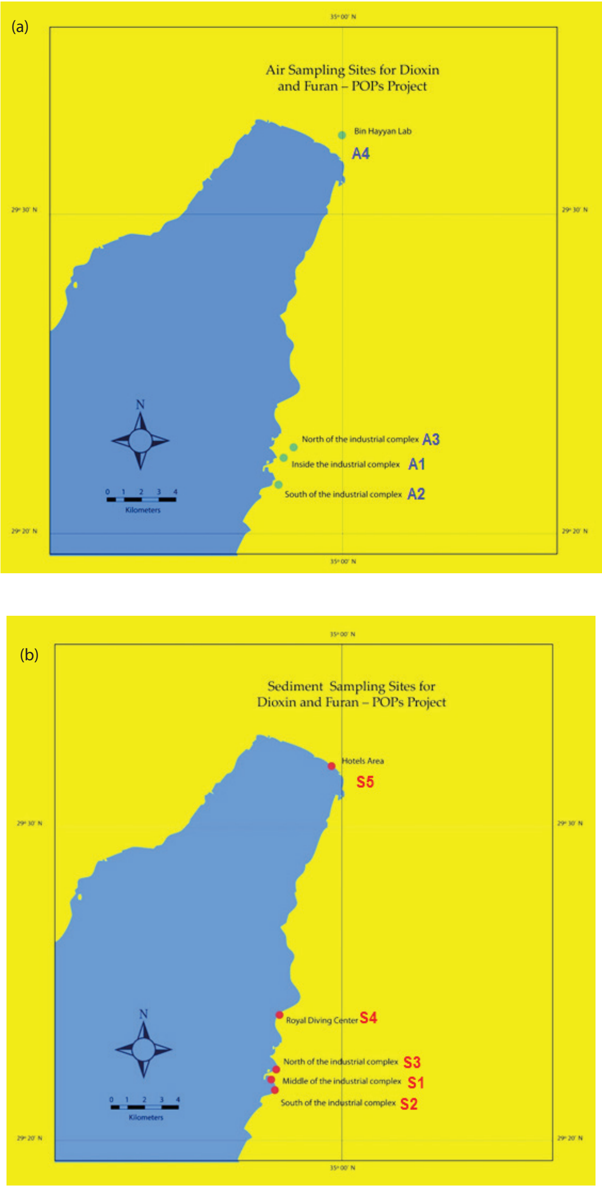

Fig. 2. (a) Geo-referenced air sampling location sites map. (b) Geo-referenced sediment sampling location sites map.

Table 1
Sediments and air sampling sites and descriptions

Sample type	Sample ID	Location and description		
		Sample location	Longitude	Latitude
Sediment	S_1	Middle of the industrial complex	34°57.756′ E	29°21.957′ N
	S_2	South of the industrial complex	34°57.882′ E	29°21.594′ N
	S_3	North of the industrial complex	34°57.906′ E	29°22.275′ N
	S_4	Diving activities area	34°58.019′ E	29°24.005′ N
	S_5	Tourism area and resort	34°59.673′ E	29°31.966′ N
Air	A_1	Inside the industrial complex	34°58.161′ E	29°22.370′ N
	A_2	South of the industrial complex	34°58.001′ E	29°21.525′ N
	A_3	North of the industrial complex	34°58.463′ E	29°22.685′ N
	A_4	Ben Hayyan Laboratories, Aqaba City	34°59.980′ E	29°32.461′ N

2.3. Air samples collection

Ambient air was sampled (20 samples) at four sites A_1, A_2, A_3 and A_4 (Fig. 2(b)). Table 1 describes each of these sample location sites. Long-term air collection was done by following US EPA Reference Method TO9A, with high-volume samplers at a height of 1.5 m, working for 40 h (1–2 d). The samplers consisted of a glass-fiber filter followed by a prewashed polyurethane foam (PUF) plug (10 cm diameter and 10 cm height) connected to a suction pump. The ambient air samples (field filter/PUF) were stored under ice (<4°C) at the analytical lab.

2.4. PCDD/Fs analytical procedure

The extraction, clean up and instrumental analysis were carried out at Ben Hayyan-Aqaba International Laboratories, Jordan. The PCDD/Fs in air and sediment samples were collected and measured by US EPA Method 1613 revision B [13]. Details of the methods used were described elsewhere [14]. Briefly, prior to extraction, 10 g of dried sediment was homogenized and spiked with known amounts of $^{13}C_{12}$-labeled internal standards (Wellington Laboratories, Canada). The samples were then extracted with hexane and dichloromethane using an accelerated solvent extractor. The extracts were concentrated and the residues were purified on an acidic silica gel column (treated with 44% sulfuric acid) and multilayer silica columns. The PCDD/Fs were fractionated on a basic alumina column. The final extracts were spiked with $^{13}C_{12}$-labeled injection standards of PCDD/Fs as appropriate for recovery quantification and subsequently injected into the injection port in splitless mode in the instrumental analysis using of Hewlett Packard 5890 gas chromatograph coupled to a VG 70-250S high resolution mass spectrometer (GC-HRMS).

Samples were injected at an injector temperature of 280°C (1 μL, splitless time: 2 min). Helium carrier gas was at 18 psi with a constant flow rate of 1.2 mL/min and the transfer line temperature and ion source temperature were both 250°C.

The following 17 PCDD/Fs were targeted for analysis: 2,3,7,8-tetra CDD; 1,2,3,7,8-penta CDD; 1,2,3,4,7,8-hexa CDD; 1,2,3,6,7,8-hexa CDD; 1,2,3,7,8,9-hexa CDD; 1,2,3,4,6,7,8-hepta CDD; octa CDD (OCDD); 2,3,7,8-tetra CDF; 1,2,3,7,8-penta CDF; 2,3,4,7,8-penta CDF; 1,2,3,4,7,8-hexa CDF; 1,2,3,6,7,8-hexa CDF; 1,2,3,7,8,9-hexa CDF; 2,3,4,6,7,8-hexa CDF; 1,2,3,4,6,7,8-hepta CDF; 1,2,3,4,7,8,9-hepta CDF and octa CDF (OCDF). The TEQ concentrations of PCDD/Fs were calculated using both of I-TEQs from TEFs proposed by NATO/CCMS [15], and the World Health Organization (WHO)-2005 toxic equivalency factors (TEF) [16]. Concentrations below detection limits were considered as zero (lower bound TEQ).

2.5. Quality control and quality assurance

The chemical analysis lab is accredited with ISO 17025. Routinely, a laboratory blank is performed and the values were within the acceptable limits for the adopted method. PCDD/Fs values were automatically corrected by taking into account the recovery rate of the ^{13}C-labeled internal standards. As stated before, the determination of the concentrations was performed according to the EPA Method 1613 revision B.

The results were statistically presented using ANOVA by showing the average value of the PCDD/Fs (with the least significant difference at the 0.1 level of probability) for each location samples.

3. Results and discussion

The results in Table 2 present the PCDD/F values for sediment samples. The concentration levels of total PCDD/Fs in sediments ranged from 23.1 to 54.25 ng/kg dry matter. The highest values were measured at location sites S_2, S_1 and S_5, respectively. Moreover, the results show that PCDF is by far the dominant congener (65.2% and 81.9% of the total amount of PCDD/Fs) in the sediment sample locations S_2, S_1 and S_5. This is very reasonable since PCDFs usually account for industrial impact [17–20]. Investigation shows that high PCDFs in samples S_2, S_1 and S_5 may be traced to the presence of factories and industrial-type activities. However, the PCDD and PCDF concentrations were found to be equal for

Table 2
Concentrations of PCDD/Fs and WHO-TEQ$_{PCDD/F}$ values (ng/kg dry matter) in coastal sediments (with least significant difference at the 0.1 level of probability)

Sample code	Concentration (ng/kg dry matter)				
	S_1	S_2	S_3	S_4	S_5
Dry residue (%)	89.72	85.92	83.84	72.51	78.14
ΣPCDD	11.53	11.6	11.31	11.62	5.66
ΣPCDF	21.63	42.65	11.79	11.62	25.62
ΣPCDD/F	33.16	54.25	23.1	23.24	31.28
WHO (1998) PCDD/F-TEQ (lower bound)	0.098	0.119	0.011	0.065	0.596
WHO (1998) PCDD/F-TEQ (upper bound)	1.05	1.08	0.992	1.03	0.89
WHO (2005) PCDD/F-TEQ (lower bound)	0.101	0.125	0.012	0.066	0.528
WHO (2005) PCDD/F-TEQ (upper bound)	0.962	0.993	0.901	0.936	0.823
I-TEQ (NATO/CCMS) excluding LOQ	0.111	0.149	0.018	0.071	0.608
I-TEQ (NATO/CCMS) including LOQ	0.956	1	0.89	0.924	0.848

sediment sample locations S_3 and S_4. According to Wagrowski and Hites [21] when PCDFs are predominant, the profile is classified as "source", while "sink" profiles are dominated by PCDDs. However, the similarities in the PCDD congener profiles in sample sites S_1, S_2, S_3 and S_4 suggest that the PCDD source or formation process is similar for these samples. However, with the generally very low concentrations recorded in the present study it is not possible to relate the concentrations to any pollution mainstream.

The atmospheric concentrations of the total PCDD/F values for the four sampling sites A_1, A_2, A_3 and A_4 are summarized in Table 3. The atmospheric concentration levels of total PCDD/Fs ranged from 0.653 to 0.867 pg/m^3. The highest atmospheric concentration values of the total PCDD/Fs were measured at middle of the industrial complex, sampling location site A_1 (0.8673 pg/m^3) and in the southern part of the industrial complex, sampling location site A_2 (0.7040 pg/m^3), in the direction of the prevailing wind (the predominant wind direction is from north (N) to northeast (NE)). Therefore, this indicates that industry had the most important influence on the dioxins and furans pollution, which was also reported in other areas [22,23].

However, lower atmospheric PCDD/F values were measured at both locations of A_3 (0.6794 pg/m^3) and A_4 (0.6531 pg/m^3) were approximately similar. This is because these two locations (A_3 and A_4) are both located in the opposite direction of the prevailing wind direction. Hence, the source of the atmospheric PCDD/F at A_3 and A_4 is most probably from the transportations (traffic emissions). Vehicle emissions could be one of the main sources of dioxin which have been confirmed by Lee et al. [24]. Other possible sources may include dioxins from long-distance transportation.

It is worth mentioning that in comparison with atmospheric PCDD/Fs concentration data in other cities in the world [22,23,25–27], the atmospheric TEQ concentrations of PCDD/Fs in Aqaba were higher than those in Europe and America, and similar to those of Japan, Taiwan, Guangzhou and Shanghai.

Congener patterns of all air samples are depicted in Fig. 3. OCDD was the most abundant congener for PCDD/Fs. Although the levels are different from site to site, the congener profiles of 2,3,7,8-sustituted PCDD/Fs for the air samples were generally similar for A_2, A_3 and A_4. They were characterized by high OCDD (78% of the ΣPCDDs), followed by OCDF (38.5% of ΣPCDFs) and 1,2,3,4,6,7,8-HeptaCDD/Fs.

At the location site A_1, the profiles of homologs in the air samples are predominated by octa-PCDD (75% of the ΣPCDDs) and octa-PCDF (24.7% of the ΣPCDFs), followed by 1,2,3,4,6,7,8-Hepta CDF (22.5% of the ΣPCDFs), and hexa-, penta- and tetra-PCDFs (which together contributed less than 47% of the ΣPCDFs). Moreover, the congener profiles of 2,3,7,8-sustituted PCDFs have higher levels (38.7% of the ΣPCDD/Fs) than congener profiles of 2,3,7,8-sustituted PCDDs (12% of the ΣPCDD/Fs) at the location site A_1.

Moreover, the atmospheric deposition cannot be claimed to be the main source of aquatic PCDD/Fs. If the fingerprints in air and sediment were the same, then this can be referred to the reason that the air and sediment share similar sources, and note that the air is the main source to the sediment. If the air is the main source to the sediment, then the mass of PCDD/Fs that enter the water column every year based on the atmospheric concentration can be estimated and multiplied by a reasonable dry particle deposition velocity (since PCDD/Fs are predominant in the particle phase). In most cases, by doing this calculation, one can find that atmospheric deposition just is not big enough to account for the mass of PCDD/Fs in the sediment. It is more likely that both the air and the sediment are affected by the same source (very likely motor vehicle emissions in most urban areas). These emissions enter the air, but a substantial fraction of them settle on the roadway without traveling very far or being measured in the atmosphere. From there, they are carried into the waterway via urban storm water runoff.

As shown in Fig. 4, the PCDD/F distribution in sediment was mainly characterized by OCDD, 1,2,3,4,6,7,8-hepta CDF, 1,2,3,4,7,8,9-hepta CDF and OCDF. The four compounds together account between 77.45% and 90% of the 17 toxic compounds. The most toxic congener of the dioxin group 2,3,7,8-TCDD, is a very minor component with a contribution of less than 0.54% to the total PCDD/Fs. Thus, the profiles of homologs in the sediment samples are predominated by octa- and hepta-polychlorinated PCDD/Fs, and this has been reported in several studies [28–30].

The results also show that among the PCDDs congeners, OCDD is clearly the dominant congener (79% of the total

Table 3
The atmospheric PCDD/Fs and WHO-TEQ$_{PCDD/F}$ values different sampling locations (with least significant difference at the 0.1 level of probability)

Sample code	Concentration (pg/m^3)			
	A_1	A_2	A_3	A_4
ΣPCDD	0.4216	0.4408	0.4089	0.4098
ΣPCDF	0.4456	0.2632	0.2705	0.2433
ΣPCDD/F	0.8673	0.7040	0.6794	0.6531
WHO (1998) PCDD/F-TEQ (lower bound)	0.0350	0.0002	0.0000	n.a.
WHO (1998) PCDD/F-TEQ (upper bound)	0.0560	0.0386	0.0360	0.0358
WHO (2005) PCDD/F-TEQ (lower bound)	0.0275	0.0002	0.0001	n.a.
WHO (2005) PCDD/F-TEQ (upper bound)	0.0486	0.0343	0.0323	0.0319
I-TEQ (NATO/CCMS) excluding LOQ	0.0350	0.0002	0.0001	n.a.
I-TEQ (NATO/CCMS) including LOQ	0.0524	0.0343	0.0323	0.0319

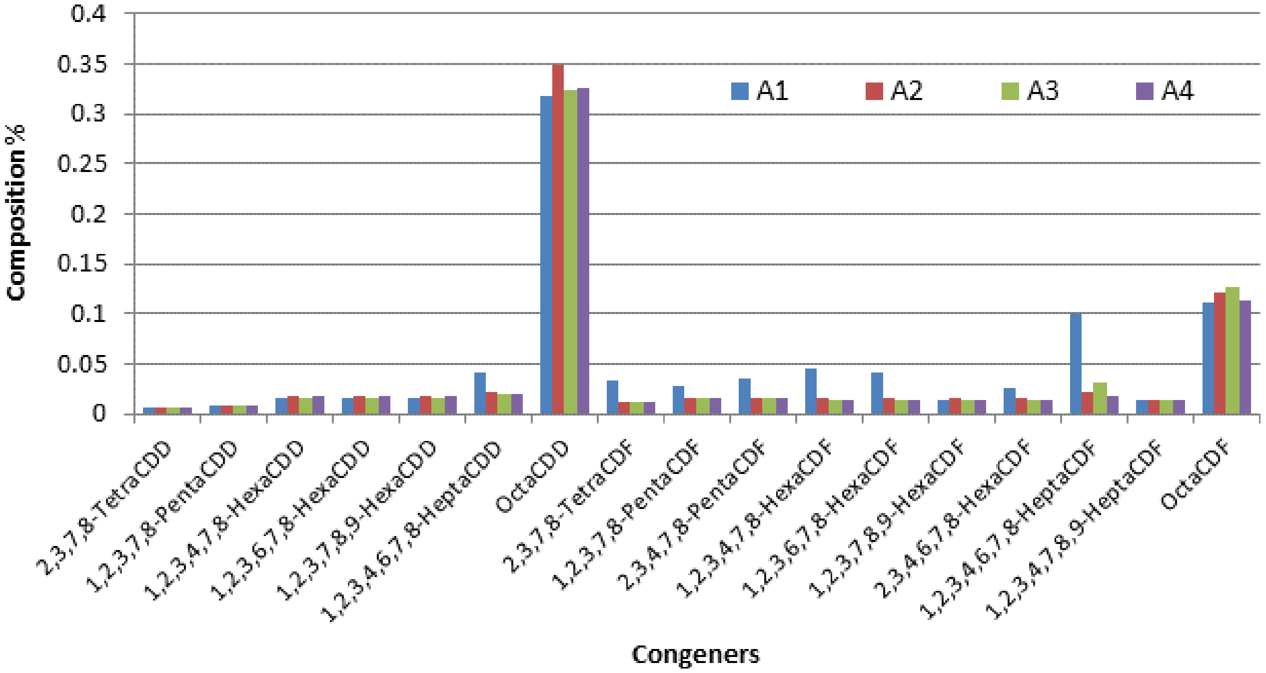

Fig. 3. Atmospheric PCDD/Fs congener profile in air samples at the four study sites.

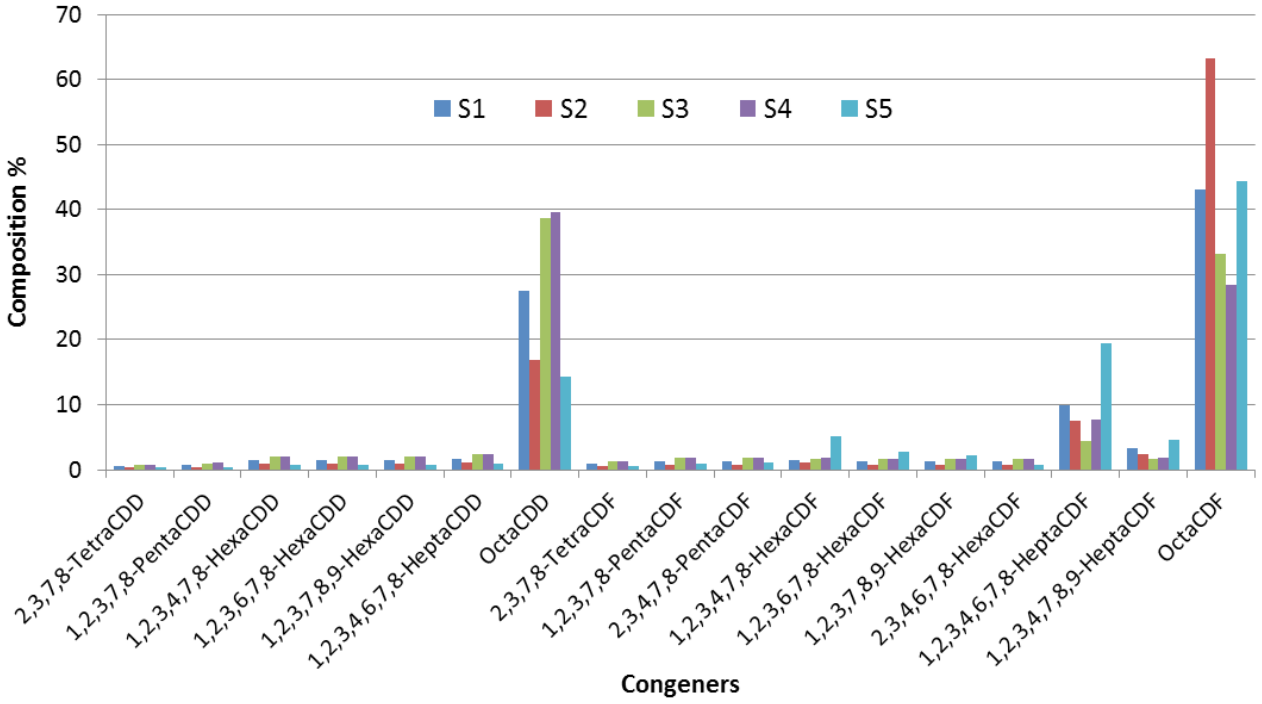

Fig. 4. PCDD/Fs congener profile in sediment samples at the five study sites.

amount of PCDDs), which is known to be associated with wastewater treatment [31,32]. In all samples (sites S_1, S_2, S_3, S_4 and S_5) the higher chlorinated CDDs (octa and hepta) predominated and this is in agreement with other studies that reported this PCDDs profile for regions of high atmospheric deposition [33]. The OCDF is the predominant congener among the PCDFs congeners (54% up to 81% of the total amounts of PCDFs).

As shown in Table 3, the WHO-TEQ lower bound (LB) and upper bound (UB) values calculated for five sediment samples using TEF proposed by WHO 1998 and 2005. The WHO (1998)-TEQ (LB) for PCDD/Fs ranged from 0.011 to

0.596 ng TEQ/kg dry matter, and the WHO (1998)-TEQ (UB) ranged between 0.89 and 1.08 ng TEQ/kg dry matter for the sediment samples.

The results are compatible with values of WHO (2005)-TEQ. This is also in agreement with the TEQ of PCDD/Fs values calculated using International TEFs according to NATO/CCMS (1988). Hence, the TEQ values of any of these sediment samples analyzed are far below the safe sediment value of 20 pg TEQ/g dry matter as proposed by Evers et al. [34].

4. Conclusions

The present study is the first investigation of the atmospheric input of PCDD/Fs into sediments of Aqaba Coastal City, and results published herein are most likely the first published results on the entire Red Sea. The TEQ values of PCDD/Fs recorded were significantly low as compared with the safe limits. However, PCDD/Fs were detected in all the five sediment samples of the study, showing their ubiquity in different areas on the Jordanian coast of the Gulf of Aqaba. The consistency of the PCDD/F congener patterns in atmospheric samples and sediments confirms the findings that air and sediment share similar sources of PCDD/Fs. While homogeneity on PCDDs pattern in all sampling sites may indicate natural baseline concentrations. Moreover, the congener profiles of dioxins and furans in sediments account for the predominance of OCDD, 1,2,3,4,6,7,8-hepta CDF, 1,2,3,4,7,8,9-hepta CDF and OCDF, which have relatively low toxicity equivalent factors.

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