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SOURCES AND DISTRIBUTION OF POLYCHLORINATED DIBENZO-*PARA*-DIOXINS AND DIBENZOFURANS IN SEDIMENTS FROM URBAN CASCADE RESERVOIRS, CENTRAL POLAND

The transport pattern of the organochlorine pollutants along the river-bed is an important issue due to their accumulation in reservoir bottom sediments and bioaccumulation in the food chain. In this study, seventeen most toxic 2,3,7,8-substituted dibenzo-*para*-dioxins (PCDDs) and dibenzofurans (PCDFs) were selected to determine sediment contamination in a cascade of five urban reservoirs.

The total concentrations of PCDD/Fs ranged from 22.98 to 254.56 ng kg⁻¹ d.w. with the minimum in the first reservoir, maximum in the middle reservoir, and decline at the end of the reservoirs' cascade. Concentrations of 2,3,7,8-tetrachlorinated dibenzo-*para*-dioxin (TeCDD) equivalents (TEQs) estimated based on WHO-TEFs decreased along the reservoir cascade (4.94; 3.02; 1.51 and 0.52 ng kg⁻¹ of d.w., respectively), excluding the first one (0.80 ng kg⁻¹ of d.w.). This situation can be related to the hydraulic transport and deposition of PCDD/Fs along the cascade system. The PCDD/PCDF ratio showed a different pattern with the pre-dominance of PCDFs in the first two cascade reservoirs (61.01% and 88.75%, respectively) and PCDDs in the last three reservoirs (86.16%, 87.83% and 91.41%, respectively).

1. INTRODUCTION

The City of Łódź is a city of 800 thousands inhabitants (agglomeration of 1 million inhabitants), located in central Poland. The city area is divided into 18 catchments drained by small urban streams (average flow < 1 m³ s⁻¹) with relatively high slope of stream channels (5–7‰). During the industrial revolution in the early 30 s

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of the 19th century, the streams were channelised and turned underground, becoming a part of storm water system (WAGNER et al. 2006; JOKIEL and MAKSYMIOUK 2002).

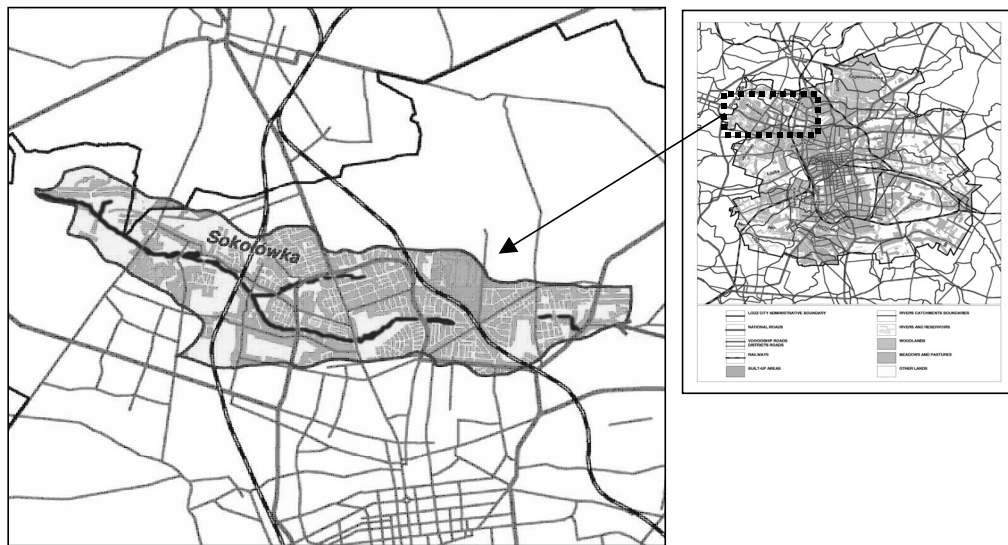


Fig. 1. Sokolowka River catchment (Wagner et al., 2006)

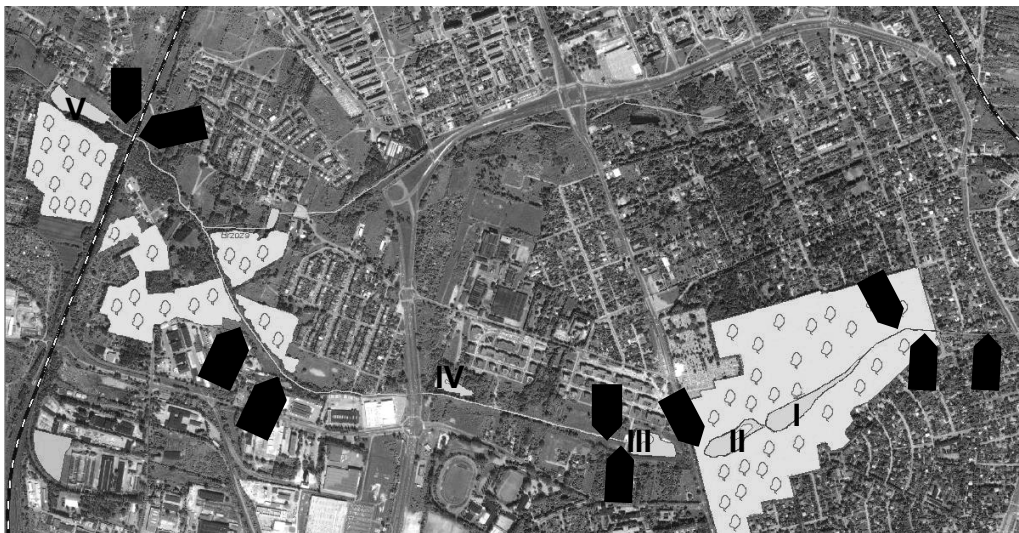


Fig. 2. Sediment samples collection sites and most important stormwater outlets (black arrows) situated along Sokolowka River (Jokiel and Maksymiuk, 2002)

The Sokolowka river (drainage area 45.4 km²), flowing across the northern part of the city (figure 1), appears as a typical urban storm water receiver supplied mostly by around 50 storm water outlets (figure 2). The main river bed was channelised by concrete slabs to straighten the course and deepen the bed for the purpose of detention of storm waters (WAGNER et al. 2006; BIEZANOWSKI 2003; KUJAWA and KUJAWA 2003; KONDRACKI 1978). The river length from the spring to the mouth is 13.3 km and 13.0 km – on the territory of the City of Łódź. More than 90% of the land used in the Sokolowka River basin is residential, commercial or industrial (table 1). Thus high concentrations of organic contaminants input, like Persistent Organic Pollutants (POPs), have impacted the quality of ecosystem services and in consequence citizens' health. Most of these substances accumulate in sediments which can receive inputs via various pathways including atmospheric deposition, industrial and domestic effluents, storm water, spills, and others. It is estimated that up to 97% of released POPs in a water column are retained in sediment (DIPINTO et al., 1993). Thus, concentration of POPs in sediments can provide a good indication of the level of environmental stress to ecosystems (SAPOZHNIKOWA et al. 2005).

Table 1

Characteristics of the Sokolowka River and its catchment
(JOKIEL and MAKSYMIUK 2002; BIEZANOWSKI 2003; KUJAWA and KUJAWA 2003)

Catchment	
Surface area (km ²)	45.40
Slop (‰)	6
Meadows and pastures (%)	0
Forests and coppices (%)	<10
Arable land and orchards (%)	0
Litology	Quaternary formation: clay, gravel, sand, silt
River	
Length (km)	13.4
Channel regulation (%)	100
Slop (‰)	0.55
Impoundment on the channel	5
Annual rainfall (mm)	535
Annual discharge (mm)	153
Q (m ³ s ⁻¹)	0.17
Q max (m ³ s ⁻¹)	2.61
Q min (m ³ s ⁻¹)	0.02

The objective of this study was to elucidate spatial differences in concentrations of PCDD/DF in sediments collected from reservoirs situated along a small urban river. Reservoirs were constructed for water retention, enhancement of the sedimentation rate and thus water purification as well as for recreational use (figs. 1–2, table 2).

Table 2

Characteristics of studying reservoirs (KUJAWA and KUJAWA 2003)

	I	II	III	IV	V
Surface area (m ²)	1600	1080	1200	400	1500
Capacity (m ³)	2250	1080	2200	410	2000
Retention time (days)	7.70	7.07	1.16	2.62	3.57
Age	old (19th century)	old (19th century)	new (2004)	new (2004)	old (1980)
Input flow:					
Min	0.006	0.007	0.007	0.008	0.013
Average	0.030	0.033	0.033	0.039	0.067
Max	4.5	1.7	1.5	1.2	10.5

2. MATERIAL AND METHODS

2.1. STUDY AREAS AND SAMPLING

Bottom sediment samples (0–25 cm) were collected by a Kayak sediment core sampler once in the autumn of 2007 from three stations (inlet part, medium part and dam part) in each reservoir situated on Sokolowka River (figure 2). Samples were filled into amber containers and transported in 4 °C to the laboratory. After that samples were frozen to –40 °C for 8 h and freeze-dried at –40 °C and 10⁻¹ mba for 72 h using Edwards freeze-dryer (Eppendorf, Germany), homogenized and mixed in proportion 1:1:1. They were analyzed to determine concentrations of 17 PCDD/Fs congeners.

2.2. CHEMICALS

The ¹³C-labelled internal standards were delivered by Cambridge Isotopes Laboratories (USA), all solvents and adsorbents used were purchased from LGC Promochem (Dziekanów Leśny, Poland). The silica gel was heated overnight at 550 °C to reduce background levels of contaminants.

2.3. EXTRACTION AND CLEAN-UP OF PCDD/DF

Extraction of PCDD/Fs was carried out using 2 g of dried sediment sample, applying an Accelerated Solvent Extractor (ASE 200) device (Dionex). Before extraction samples were spiked with isotopically labelled standard of known quantity to monitor sampling efficiency. Extraction of PCDD/Fs was performed with toluene at

150 atm (11 MPa) and the temperature was 175 °C. Two static cycles were applied for complete extraction.

To remove interferences from extracts multilayer columns packed with neutral silica and silica modified with 44% (w/w), 22% H₂SO₄, 3% KOH and 10% AgNO₃ were used. Elution was performed with 200 ml of hexane. After this step the sample solvent volume was reduced approximately to 5 ml by rotary evaporation and concentrated to 100 µl under a gentle stream of nitrogen. During concentration n-hexane was replaced by n-nonane and external standard was added (PN-EN 1948-3, 2002; EPA Method 1613, 1994).

2.4. ANALYSIS OF PCDD/DF

The PCDD/Fs analysis was performed using HRGC-HRMS: HP 6890N by Agilent Technologies equipped with a DB5-MS column (60 m × 0.25 mm i.d., film thickness 0.25 µm) in the splitless injection mode, coupled to high resolution mass spectrometer AutoSpec Ultima using perfluorokerosene (PFK) as a calibration reference. The GC temperature program was 150 °C for 2 min, 20 °C/min to 200 °C (0 min), 1 °C/min to 220 °C for 16 min and 3 °C/min to 320 °C for 3 min. The injector temperature was 270 °C. The MS was operated with a mass resolution of 10 000, and the electron impact ionization energy was 34.8 eV with an ion source temperature of 250 °C. Helium was used as carrier gas at a flow rate 1.6 ml/min. Samples were quantified with an isotope dilution method (ROBINSON et al. 2004; PN-EN 1948-3, 2002; EPA Method 1613, 1994).

3. QUALITY ASSURANCE/QUALITY CONTROL

The enforcing laboratory is operating a quality assurance system according to PN-EN ISO/IEC 17025:2005 and is accredited for the analysis of PCDD/DFs (certificate no. AB 215). The procedures used in laboratory were validated on the basis of internal and external reference materials. Additionally, each batch contained a method blank, a matrix spike, and duplicate samples. A reagent blank was used to assess artifacts and precision was verified by duplicate analyses (i.e. applied to two samples from each site). Samples spikes were used as an additional check of accuracy. Analyte recoveries were determined by analyzing samples spiked with PCDD/Fs standards. The recovery coefficient was taken into account for calculating the final concentrations of analytes.

The recoveries of ¹³C-labelled quantification standards were in a range of 85% to 105%. The limits of detection were in the range of 0.15 to 1.03 pg kg⁻¹ on dry weight basis.

4. STATISTICS

All data were subjected to statistical analyses using „Statistica” software for Windows. A Kruskal-Wallis ANOVA by Ranks was used to compare treatment levels. The statements of significance were based on a probability level of $P \leq 0.05$.

5. RESULTS AND DISCUSSION

The concentration levels of 17 selected PCDD/Fs (tetra to octa) in sediment samples are depicted in Table 3. The toxicity of chosen PCDD/Fs congeners (TEQ) are showed in Table 4.

Table 3

Concentration of 17 PCDD/DF congeners [ng kg^{-1} d.w.]
in sediment samples collected from Sokolowka river reservoirs during autumn period of 2007

	I	II	III	IV	V
2378-TCDD	n.d.	n.d.	0.30	n.d.	n.d.
12378-PeCDD	n.d.	1.81	n.d.	0.86	0.08
123478-HxCDD	n.d.	n.d.	n.d.	n.d.	n.d.
123678-HxCDD	n.d.	n.d.	1.35	1.24	0.06
123789-HxCDD	1.11	n.d.	3.13	n.d.	0.70
1234678-HpCDD	7.85	2.22	9.65	2.26	5.37
OCDD	n.d.	0.01	204.90	71.19	63.90
2378-TCDF	1.24	0.51	1.64	n.d.	n.d.
12378-PeCDF	1.44	1.71	2.64	0.51	n.d.
23478-PeCDF	n.d.	4.71	3.42	1.37	0.05
123478-HxCDF	n.d.	2.96	1.90	n.d.	0.21
123678-HxCDF	n.d.	4.69	1.47	n.d.	n.d.
234678-HxCDF	3.25	7.46	3.20	n.d.	2.26
123789-HxCDF	0.58	n.d.	0.59	n.d.	n.d.
1234678-HpCDF	5.84	7.45	12.19	5.25	2.26
1234789-HpCDF	0.55	n.d.	n.d.	n.d.	0.70
OCDF	1.12	2.35	8.19	3.35	1.11
Sum [ng kg^{-1} d.w.]	22.98	35.88	254.56	86.02	76.69

n.d. – not detected, measured concentration was below detection limit

The total concentrations of PCDD/Fs ranged from 22.98 in reservoir I to 254.56 ng kg^{-1} of dry weight in reservoir III. The toxicity measures as TEQ ranged from 0.52 in reservoir V to 4.94 ng TEQ kg^{-1} of dry weight in reservoir II. The high TEQ concentrations of sediment samples collected from reservoirs II and III were created by congener 2,3,4,7,8-PeCDF of high WHO-TEF value.

Table 4

TEQ concentration of 17 PCDD/DF congeners [ng TEQ kg⁻¹ d.w.]
in sediment samples collected from Sokolowka river reservoirs during autumn period of 2007

	TEF	I	II	III	IV	V
2378-TCDD	1	n.d.	n.d.	0.30	n.d.	n.d.
12378-PeCDD	1	n.d.	1.81	n.d.	0.86	0.08
123478-HxCDD	0.1	n.d.	n.d.	n.d.	n.d.	n.d.
123678-HxCDD	0.1	n.d.	n.d.	0.13	0.12	0.01
123789-HxCDD	0.1	0.11	n.d.	0.31	n.d.	0.07
1234678-HpCDD	0.01	0.08	0.02	0.10	0.02	0.05
OCDD	0.0003	n.d.	n.d.	0.06	0.02	0.02
2378-TCDF	0.1	0.12	0.05	0.16	n.d.	n.d.
12378-PeCDF	0.03	0.04	0.05	0.08	0.02	n.d.
23478-PeCDF	0.3	n.d.	1.41	1.02	0.41	0.01
123478-HxCDF	0.1	n.d.	0.30	0.19	n.d.	0.02
123678-HxCDF	0.1	n.d.	0.47	0.15	n.d.	n.d.
234678-HxCDF	0.1	0.33	0.75	0.32	n.d.	0.23
123789-HxCDF	0.1	0.06	n.d.	0.06	n.d.	n.d.
1234678-HpCDF	0.01	0.06	0.07	0.12	0.05	0.02
1234789-HpCDF	0.01	0.01	n.d.	n.d.	n.d.	0.01
OCDF	0.0003	n.d.	n.d.	n.d.	n.d.	n.d.
TEQ [ng kg ⁻¹ d.w.]		0.80	4.94	3.02	1.51	0.52

n.d. – not detected, measured concentration was below detection limit

The PCDDs/PCDFs ratio showed a pre-dominance of PCDDs in sediment samples collected from the last three reservoirs (86.16%; 87.83% and 91.41% in reservoir III, IV and V, respectively) mostly due to a high amount of OCDD congener. Whereas, in reservoir I and the II the PCDFs pre-dominance was observed (61.01% and 88.75%; respectively) (figures 3 and 4).

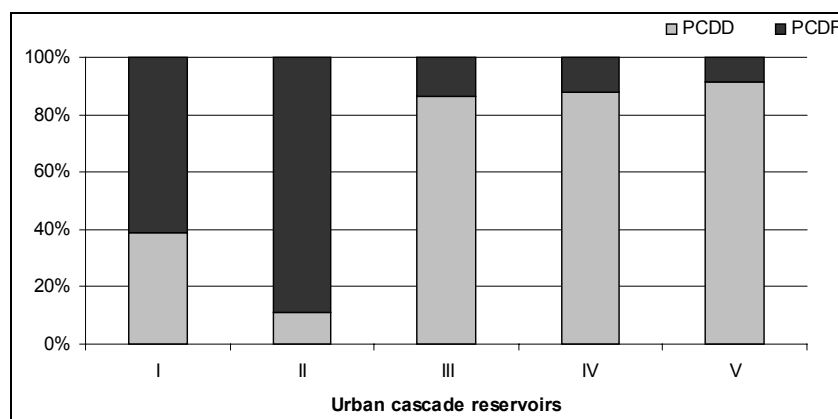


Fig. 3. Percentage content of PCDD/DF in sediment samples collected from 5 urban cascade reservoirs

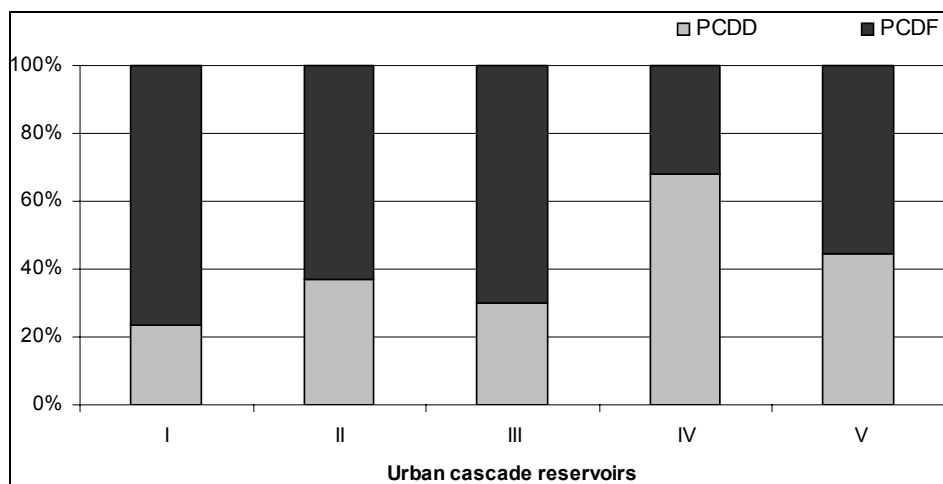


Fig. 4. Percentage content of PCDD/DF TEQ in sediment samples collected from 5 urban cascade reservoirs

The isomer pattern of PCDDs differed between locations. In the two first reservoirs the highest contribution was displayed by the 1,2,3,4,6,7,8-HpCDD congener (87.60% and 54.93% of the total amount of PCDDs in reservoir I and II, respectively). Whereas, the last three reservoirs were abundant in OCDD congener (93.42%, 94.24% and 91.14% of the total amount of PCDDs in reservoirs III, IV and V, respectively).

Congener profile of PCDFs was more diverse and showed pre-dominance of the 1,2,3,4,6,7,8-HpCDF in all reservoirs (41.65%, 23.39%, 34.58%, 50.12% and 34.27% of the total amount of PCDFs in reservoirs I–V, respectively). The second most abundant congener was 2,3,4,6,7,8-HxCDF, accounting for 23.18% in reservoir I, 23.43% in reservoir II and 34.27% in the last reservoir. Congener OCDF contributed 23.25% and 31.97% of the total PCDFs to reservoirs III and IV, respectively. Despite of above results, the Kruskal-Wallis ANOVA by Ranks test showed that there was no significant difference between PCDD/Fs concentrations among reservoirs.

To our knowledge no previous reports of PCDD or PCDF concentrations in sediments from the Łódź region are available for comparison. In general, it was found that sediments collected from the Sokolowka River reservoirs contained relatively low concentrations of the investigated PCDD/Fs with a mean level of 95.23 ng kg^{-1} and average TEQ concentrations of $2.16 \text{ ng TEQ kg}^{-1}$. In contrast, KANNAN et al. (2001) showed PCDD/Fs concentrations in sediments from the upper Detroit River and lower Rouge River (USA) in the range of 69 to $1415 \text{ ng kg}^{-1} \text{ d.w.}$ and TEQ concentrations between 3.99 to $62 \text{ ng TEQ kg}^{-1} \text{ d.w.}$ KOCH et al. (2004) reported the concentrations of PCDD/Fs in the sediment cores of North-East Germany that ranged from 19 to $20000 \text{ ng kg}^{-1} \text{ d.w.}$ Concentration of PCDDs in a reservoir situated in northern

Taiwan ranged between 0.95 to 14.40 ng TEQ kg⁻¹ (CHI et al. 2007). HILSCHEROVA et al. (2003) reported concentration from 59 to 120 for PCDDs and from 2400 to 53600 ng kg⁻¹ d.w. for PCDFs for upstream and downstream sections of the Tittabawse River, respectively.

Different sources of PCDD/Fs are characterized by different congener patterns. Major current sources to the Sokolowka River include sewage input and combined sanitary overflows. Although, re-suspension of contaminated bed sediments and direct urban runoff may also be other sources (IM et al. 2002) Concentrations of PCDD/Fs were highest in the sediments of reservoirs III, IV and V, but their patterns were different. The highest TEQ concentration (toxicity) was noted in reservoir II. These results suggest a considerable input of dioxins from untreated domestic sewage and polluted stormwater due to a number of large urban and residential agglomerations located within the described reservoir catchments (figures 2 and 3). Specially urban runoff has been reported to contain a detectable concentration of PCDD/Fs. It can be increased by some amount of PCDD/Fs in household wastewater (1.4 to 14 pg TEQ l⁻¹), washing machine effluent (17–25 pg TEQ l⁻¹) and shower water (12–210 pg), which have the same homolog profile like sewage (HORSTMANN and MCLACHLAN, 1995)

The results indicated that concentrations of 17 PCDD/Fs increased from reservoir I through III with the highest concentration in the middle reservoir, and decreasing in reservoirs IV and V. This situation may be related to the hydraulic transport of PCDD/Fs from reservoirs I and II during storm events and deposition of them in the middle reservoir. As an additional reason may be reported the strengthening of the reservoir bed by plastic material net, which may release some amounts of PCDD/Fs to the water and bottom sediments. The same material was used for construction of reservoirs III and IV and thus the highest concentrations of analyzed contaminants, especially OCDD, were observed in these ponds. Additionally, the fact that OCDD congener is more stable than other PCDD/Fs homologues in the environment may be a cause of its high amount in new reservoirs (XU et al. 2008; OGURA et al. 2001). For the same reason, the lack of OCDD congener in the two initial reservoirs may be related to their high age (more than 100 years). Moreover, textile industry, which was developed in Łódź from the 30s of the 19th to the end of the 80s of the 20th century, may have discharged some amounts of PCDD/Fs from pigments to the sewer system and subsequently to the river. Thus, the mixing of past (industrial) and new (residential) effluents with urban stormwater may be the main sources of PCDD/Fs, causing appreciable differences in the studied reservoirs.

Among other possible sources of PCDD/Fs, thermal processes, such as trash burning and diesel engine emission can result in the production of a range of analyzed congeners. Motor vehicles are suspected as an emission source which has been studied during the recent decades (KIM et al. 2003; RYAN and GULLET 2000; MARKLUND 1990). The total amount of PCDD/Fs concentration in diesel fuel engine exhaust was

reported to amount to about 1–218 pg TEQ N⁻¹ m³ (GEUEKE et al. 1999). Average PCDD/Fs concentrations per unit of exhaust gas with 25%, 50% and 75% load rates were 14.5, 6.9 and 6.5 pg TEQ /N m³, respectively, with a prevalence of high-chlorinated PCDD/Fs (1,2,3,4,6,7,8-HpCDD/F and OCDD/F) over low-chlorinated PCDD/Fs and greater contribution of PCDFs to TEQ (KIM et al. 2003). Thus relatively high percentages of PCDF in reservoir I and II (61.01% and 88.75%; respectively) may suggest the engine emission as another possible source.

In summary, a number of large urban and industrial agglomerations located within studied reservoir catchment as well as transformation and transfer along the reservoir cascade exert influence on the number and significance of possible sources of analyzed contaminants.

At least two main sources of PCDD/Fs have been identified: the first source is wastewater input by stormwater outlets and direct urban runoff. In particular, the lack of the sewage treatment facilities and un-permitted discharges could be responsible for the relatively great concentrations of PCDD/Fs in the reservoirs sediments. The second source may be presence of textile industry emissions in the past, despite lower concentrations of PCDD/DF in the sediments from the Sokolowka River reservoirs than those in sediments in other industrialized places.

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