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Seasonal Distribution of PCDDs/PCDFs in the Small urban Reservoirs

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ABSTRACT: Man-made reservoirs constructed on running waters, where a decrease in flow velocity and an increase in flocculent settling occurs, create perfect conditions for the deposition of allohtonic matter and adsorbed pollutants, such as toxic polychlorinated dibenzo-*p*-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs). The results obtained from the research on a cascade of five small reservoirs located along an urban river (Lodz, Poland) showed significantly lower concentrations of PCDDs/PCDFs in the spring season in the first three reservoirs; whereas the other two demonstrated an opposite tendency with the lower values observed during the autumn. Also, analysis of the seasonal variations of the TEQ concentrations showed lower values in the reservoirs located at the beginning of the cascade (I, II and III) in spring, while in the lower ponds (IV and V) lesser values were observed during autumn. The winter and spring snow and ice melting, and the accelerated run-off washing out organic and mineral matter with the associated micropollutants accumulated during this period, were recognized as the main reasons for such seasonal distribution of PCDDs/PCDFs. Intensive rains occurring during the autumn, especially after dry summers, may be in turn responsible for the raised total PCDDs/PCDFs and TEQ concentrations during the autumn sampling period. Furthermore, the discharges of polluted stormwater and illegal domestic sewage through the stormwater outlets located along the river boosted the reservoirs sediments in the PCDDs/PCDFs.

Key words: PCDDs/PCDFs, Urban river, Reservoirs, Seasonal distribution, Stormwater

INTRODUCTION

As a consequence of the rapid development of modern societies during 20th century, a significant amount of organic chemicals has been dispersed into the environment. Many have been used as pesticides, insecticides, defoliants, industrial chemicals or produced as undesirable by-products. From this group of substances the organochlorine compounds, including polychlorinated dibenzo-*p*-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs), have received the most attention due to their persistence in the environment, their bioaccumulation and the hazard they present to both humans and wildlife for several years (Makles *et al.*, 2001). Some of the most sensitive ecosystems with regard to this kind of chemicals are rivers. Their location in the lowest parts of the landscape results in their high exposure to the pollutants produced in their catchment, having the origin in various sources such as, atmospheric deposits, urban and agricultural run-off and others. Dam reservoirs located on rivers are even more sensitive to pollution, because of the relatively large catchment area and considerable accumulation of pollutants due to rapid decrease in river flow velocity and long water retention time (Tarczyńska *et al.*, 2002). High accumulation rate of PCDDs/PCDFs in aquatic ecosystems is related to their extremely low solubility in the water phase. This predisposes the substances to an association with the organic and mineral particulates present in the water and their further accumulation in the sediments. Therefore, man-made reservoirs constructed on running waters, with its hydrological characteristics, create perfect conditions for the deposition of allohtonic matter and associated PCDDs/PCDFs. It is estimated that majority of organochlorine compounds which reach the river are retained in reservoir sediments (DiPinto *et al.*, 1993). They serve as a storage compartment for the long-

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term release of the sediment-associated pollutants and pose a threat to aquatic organisms and, in consequence, human health e.g., due to the consumption of fish and the recreational use of reservoirs (Knezovich *et al*., 1987). Therefore, sediments are seen to be important in monitoring water ecosystem stress and health risk assessment (Thompson *et al*., 1996).

The overall aim of this research was to recognize the incidence, concentration and risk related to the presence of PCDDs/PCDFs in the Sokołówka River (Lodz, Poland) **-** a model storm-water receiving urban river. In particular, the seasonal distribution of PCDDs/PCDFs along the Sokołówka River cascade of reservoirs were tested.

MATERIALS & METHODS

The Sokołówka River (drainage area of 45.4 km2) is a small river of about 13 km length located in urban catchment with varying development and use (industrial, domestic in the upper part and single housing development and agricultural in the lower part). The river is contaminated with organic and inorganic compounds. The main channel of the river was regulated and collects stormwater sewage from 50 outlets. Consequently, Sokołówka River and a cascade of reservoirs situated along its continuum (Fig. 1) receive polluted stormwater, often together with illegally discharged wastewater. This results in water pollution leading to toxic algal blooms occurrence and accumulation of pollutants in the abiotic and biotic components of the reservoirs ecosystem (Wagner and Zalewski, 2009; Zerihun *et al*., 2012). Five reservoirs selected for the analysis located along the river differ from each other in their age, size, theoretical water residence time, light intensity and inflow (Table 1). All of them can be regarded as small, riverine reservoirs, with short water retention time.

Sediment samples (10-25 cm thickness) were collected from the inlet, middle and dam section of each reservoir during the spring and autumn periods of 2007 and 2008. The collected sediments were freeze-dried (-40 °C, 1mba, 72 h; Edwards Freeze Dryer), sieved through a 2 mm mesh sieve and the sediments from the inlet, middle and dam sections were mixed in proportion 1:1:1 in order to obtain one representative sample for each reservoir (Urbaniak *et al*., 2008; 2009ab; 2010; 2012).

PCDD/PCDF extractions, clean-up and analysis were performed according to the US EPA 1613 Method, and the PN/EN 1948: 3 2002 Norm (EPA 1613, PN/EN 1948: 3) using High Resolution Gas Chromatography/High Resolution Mass Spectrometry (HRGC/HRMS) and the samples were quantified with the isotope dilution method. The used methodology was widely described in our earlier publications by Urbaniak *et al*., (2008, 2009ab; 2010, 2012, 2013). Briefly, two grams of each sediment sample were spiked with isotopically-labelled standards (Cambridge Isotopes Laboratories, USA) and extracted by ASE (Accelerated Solvent Extraction) 200 Dionex at 150 atm (11 Mpa), and the oven was heated to 175^{o}C with toluene. The extracts

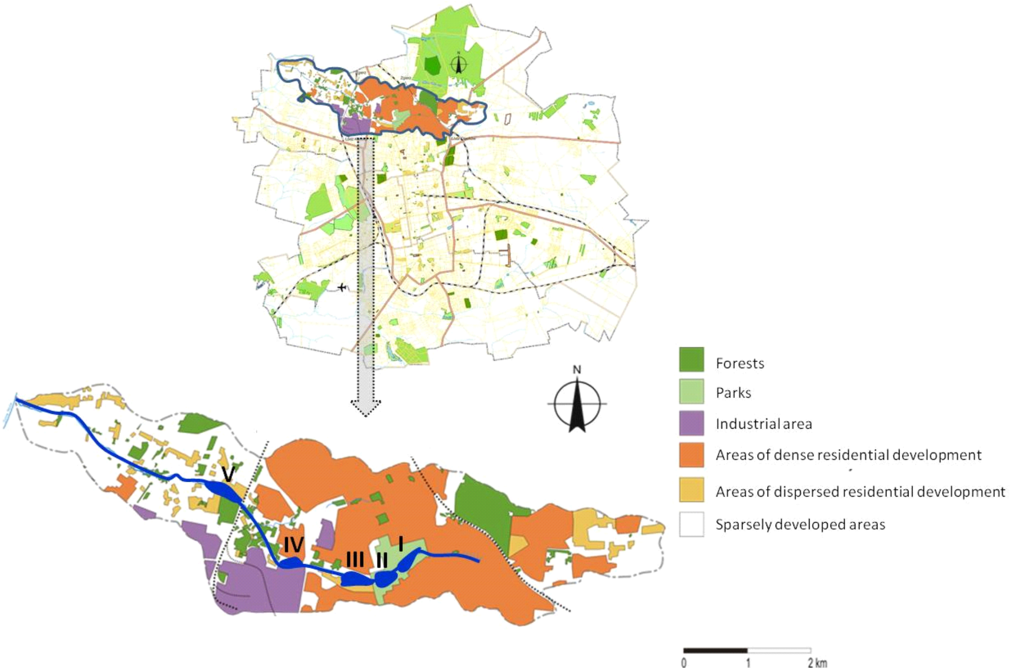

Fig. 1. Location of the studied Sokołówka reservoirs.

Table 1. Characteristics of the studied Sokołówka River and its reservoirs (Urbaniak *et al.*, 2012).

River	**I**	**II**	**III**	**IV**	**V**
Average Q (m^3 s^{-1})	0.03	0.03	0.04	0.04	0.07
Q max (m^3 s^{-1})	0.68	0.75	0.81	0.92	1.48
Q min (m^3 s^{-1})	0.006	0.006	0.007	0.008	0.014
River catchment above the reservoir					
Surface area (km^2)	6.25	6.87	7.37	7.87	13.97
Forests (%)	11	11	19	19	19
Agricultural lands (%)	13	13	23	23	23
Urban lands (%)	60	60	47	47	47
Others (%)	16	16	11	11	11
Reservoir					
Construction time	XIX	XIX	2004	2006	XIX
Max surface area (m^2)	16,400	11,000	18,600	4,000	15,000
Min surface area (m^2)	n.a.	n.a	14,000	n.a	15,000
Max depth (m)	1.5	1.5	2.0	1.0	2.0
Average depth (m)	1.0	1.0	1.3	n.a.	1.3
Capacity (m^3)	22,500	11,100	24,000	4,100	20,000
Average Q (m^3 s^{-1})	0.03	0.03	0.04	0.04	0.07
Average retention time (d)	9	4	8	n.a.	3
Macrophyte presence	-	-	+	-	-
Separators and sedimentation ponds presence	-	-	+	-	-

were purified with multilayer silica columns packed with neutral, acidic and basic silica gel and eluted with 200 mL of hexane. The obtained hexane extracts were concentrated to 5 mL by rotary evaporation and concentrated to 100 µL under a gentle stream of nitrogen, and the *n*-hexane was replaced by nonane. Identification and quantification of 7 congeners of PCDDs and 10 congeners of PCDFs were performed using HP 6890N Agilent Technologies GC coupled with a HRMS AutoSpec Ultima. The HRMS was operated in the splitless injection mode with perfluorokerosene as a calibration reference. Separation of PCDD/PCDF congeners was achieved using a DB5-MS column (60 m x 0.25 mm i.d., film thickness 0.25 µm). The oven temperature protocol was 150^{o}C for 2 min, 20^{o}C min^{-1} to 200^{o}C (0 min), 1^{o}C min^{-1} to 220^{o}C for 16 min and 3^{o}C min^{-1} to 320^{o}C for 3 min. The injector temperature was 270°C. The mass spectrometer was operated under positive electron ionization conditions: 34.8 eV electron energy at a resolving power of 10,000 with an ion source temperature of 250°C. Helium was used as a carrier gas at a flow-rate of 1.60 mL min^{-1}. Samples were quantified with the isotope dilution method. The analytical method used for PCDDs/PCDFs analysis was properly validated on the basis of internal reference materials, and the analytical laboratory was certified. All glassware and bottles used in the field and laboratory were cleaned with detergent, rinsed with ultra-pure water, followed by heating at 450°C overnight. Before using, the glassware was rinsed with acetone and hexane. Each analytical 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. Sample spikes were used as an additional check of accuracy. Analyte recoveries were determined by analysing the samples spiked with PCDDs/PCDFs standards. The recovery coefficient was taken into account for calculating the final concentrations of analytes. Additionally, in order to assess the method correctness, Standard Reference Materials - Fly Ash (PCDDs, PCDFs) BCR - were used. All data were statistically analysed using “Statistica” software for Windows. The Wilcoxon tests were used to compare the PCDD/PCDF concentration levels in sediments samples at the spring and autumn seasons. The statements of significance were based on a probability level of $P \leq 0.05$.

RESULTS & DISCUSSION

Urban water systems are very sensitive and dependent upon weather conditions, mostly precipitation which is the major vehicle in causing wet deposition and pollutant run-off. Therefore, rainfall frequency and intensity determine the removal of deposits originating in the atmosphere from the catchment surface. The total annual precipitation in large industrialized cities is generally 5-10% higher than in the surrounding areas, and for individual storms, the increase in precipitation can be as high as 30% (Marsalek *et al*., 2006). Therefore, varying meteorological changes occurring during the research, including periods of snow melting (February/March), dry weather (April, May, September) and intensive storms (e.g. up to 38 mm/day in August 2008), influenced the PCDDs/PCDFs inflow and transport down the cascade. This happened as a result of three processes: intensive run-off and the transportation of PCDDs/PCDFs from the catchment, their deposition in sediments during low flow periods, and their resultant re-suspension from the riverbed and reservoirs during high flows. In the collected sediments we found significantly higher concentrations of PCDDs and PCDFs in the autumn in the first three reservoirs of the cascade: I, II and III, with mean values of, respectively, 9.30, 109.57 and 183.67 ng/kg d.m. for PCDDs and 22.24, 39.38 and 22.27 ng/kg for PCDFs. In the same reservoirs the concentrations in the spring were as follows: 7.94, 4.41 and 69.72 ng/kg for PCDD and 4.33, 10.80 and 7.64 ng/kg for PCDF. The average concentrations of PCDDs and PCDFs in the last two reservoirs: IV and V, were higher in the spring season and amounted to 539.57 and 15,399.39 ng/kg for PCDD and 129.12 and 136.84 ng/kg for PCDF, while in the samples collected during the autumn the concentrations recorded to 67.07 and 5457.07 ng/kg for PCDDs and 8.90 and 192.17 ng/kg for PCDFs. Nevertheless, statistical analysis using the

Table 2. Seasonal variation (spring and autumn) of the individual PCDD/PCDF congener concentrations in the cascade of small urban reservoirs

Reservoir	*I*		*II*		*III*		*IV*		*V*	
Season	**spring**	**autumn**	**spring**	**autumn**	**spring**	**autumn**	**spring**	**autumn**	**spring**	**autumn**
2378-TCDD	-	-	-	-	-	0.15	-	-	-	-
12378-PeCDD	-	0.96	0.03	1.65	0.13	-	-	0.43	-	0.04
123478-HxCDD	-	-	0.06	0.79	-	-	-	-	-	2.01
123678-HxCDD	0.10	2.17	-	-	0.40	1.16	-	0.62	-	13.73
123789-HxCDD	4.27	3.21	0.85	-	-	1.57	-	-	-	9.79
1234678-HpCDD	0.06	3.92	0.17	13.48	3.47	10.40	54.74	4.69	1114.34	357.02
OCDD	3.51	-	3.30	96.09	65.85	170.54	484.83	61.76	14285.05	5074.53
Sum of PCDD [ng/kg]	**7.94**	**9.30**	**4.41**	**109.57**	**69.72**	**183.67**	**539.57**	**67.07**	**15399.39**	**5455.07**
2378-TCDF	3.07	0.62	0.13	0.26	-	0.82	1.66	-	-	-
12378-PeCDF	0.12	3.43	0.06	0.85	0.61	1.32	1.43	0.26	-	-
23478-PeCDF	-	2.84	0.05	3.83	0.58	1.71	3.08	0.68	-	1.75
123478-HxCDF	0.01	3.69	0.03	3.46	0.73	1.57	2.39	0.31		14.39
123678-HxCDF	-	-	0.06	4.14	0.53	1.16	1.31	0.47	-	
234678-HxCDF	-	7.62	5.88	7.32	0.94	2.28	5.85	0.20	52.37	47.69
123789-HxCDF	-	0.29	1.34	1.30	0.69	0.29	1.50	0.15	-	-
1234678-HpCDF	-	2.92	2.13	10.22	1.07	7.33	18.74	3.93	84.47	33.38
1234789-HpCDF	-	0.27	-	-	0.75	-	3.04	-	-	0.35
OCDF	1.13	0.56	0.40	8.00	1.74	5.79	90.12	2.90	-	94.61
Sum of PCDF [ng/kg]	**4.33**	**22.24**	**10.80**	**39.38**	**7.64**	**22.27**	**129.12**	**8.90**	**136.84**	**192.17**
Sum of PCDD/PCDF [ng/kg]	**12.27**	**31.54**	**15.21**	**148.95**	**77.36**	**205.94**	**668.69**	**75.97**	**15536.23**	**5647.24**
TEQ concentration [ng TEQ/kg]	**0.75**	**3.75**	**0.91**	**4.82**	**0.72**	**1.82**	**3.18**	**0.92**	**21.51**	**14.78**

Wilcoxon test showed no statistically significant differences between the mean concentrations of PCDDs in the samples collected in the spring and autumn season in four of the five reservoirs, I, II, IV and V (I: T = 6, P = 0.68 , II: T = 2, p = 0.13, IV: T = 3, p = 0.46, V: T = 10, p = 0.91). Only the values obtained for samples collected from the middle – III – reservoir, showed statistically significant differences between the seasons of spring and autumn (T = 1, p = 0.046). In the case of PCDFs no statistically significant differences between the mean concentrations of congeners in the spring and autumn season were shown for the first (I) (T = 7, P = 0.06) and the last (V) reservoir (T = 8, p = 0.60); whereas in other ponds, the significant seasonal variation was confirmed (II: T = 1, p = 0.01; III: T = 5, p = 0.02; IV: T = 0, p = 0.005) (Table 2). Also, analysis of the seasonal variations of the TEQ concentrations showed that in the reservoirs located at the upper part of the river (I, II and III) higher values were detected in autumn samples, while in the lower ponds (IV and V) higher values were observed in spring (Table 2).

The elevated values of PCDDs/PCDFs in the spring samples may be related to the accelerated run-off of organic and mineral matter due to winter/spring snow/ice melt (Cailleaud *et al.* 2007; Herbert *et al.*, 2006; Kawamura and Kaplan, 1986; Gregor *et al.*, 1995; Lohman and Jones, 1998). Moreover, the PCDDs/PCDFs emission into the atmosphere in winter and spring is higher due to domestic heating (especially in the area of dense residential development, see fig. 1) and the pollutants are longer deposited at the catchment surface comparing to the summer season. It is estimated that the emission from domestic burning contributes to about 60% of the total PCDDs/PCDFs emission to the atmosphere (US EPA, 1994). This, together with the larger catchment area for each reservoir downstream the catchment (eg. 7.87 km^2 for reservoir IV and 13.97 km^2 for reservoir V, see table 1), increases the volume of stormwater flushed into the river and thus sediments accumulated in the reservoirs (Urbaniak *et al.*, 2013).

The very high total and TEQ PCDDs/PCDFs concentration was observed in the lowest, oldest (ca 100 years) reservoir, which also receives water from the Sokołówka tributary – the Brzoza River, which drains a 30,000 inhabitants residential estate (Urbaniak *et al.*, 2013). Therefore, the long time of sediment accumulation together with the contribution of run-off water from the surrounding housing estates and

Table 3. Seasonal variation (spring and autumn) of the average percentage of PCDD/PCDF in the cascade of small urban reservoirs

Reservoir	***I***		***II***		***III***		***IV***		***V***	
Season	**spring**	**autumn**	**spring**	**autumn**	**spring**	**autumn**	**spring**	**autumn**	**spring**	**autumn**
2378-TCDD	0.00	0.00	0.00	0.00	0.00	0.08	0.00	0.00	0.00	0.00
12378-PeCDD	0.00	9.32	0.68	1.47	0.19	0.00	0.00	0.64	0.00	0.00
123478-HxCDD	0.00	0.00	1.36	0.70	0.00	0.00	0.00	0.00	0.00	0.04
123678-HxCDD	1.20	21.11	0.00	0.00	0.57	0.63	0.00	0.92	0.00	0.25
123789-HxCDD	53.83	31.32	19.18	0.00	0.00	0.85	0.00	0.00	0.00	0.18
1234678-HpCDD	0.70	38.25	3.92	12.03	4.97	5.66	10.14	6.95	7.24	6.54
OCDD	44.27	0.01	74.86	85.79	94.28	92.78	89.86	91.50	92.76	92.99
2378-TCDF	70.87	2.79	1.27	0.65	0.00	3.68	1.29	0.00	0.00	0.00
12378-PeCDF	2.83	15.43	0.62	2.17	7.94	5.93	1.11	2.88	0.00	0.00
23478-PeCDF	0.00	12.76	0.49	9.73	7.58	7.67	2.38	7.67	0.00	0.91
123478-HxCDF	0.30	16.58	0.26	8.80	9.59	7.05	1.85	3.52	0.00	7.49
123678-HxCDF	0.00	0.00	0.61	10.52	6.97	5.21	1.01	5.32	0.00	0.00
234678-HxCDF	0.00	34.26	58.37	18.59	12.35	10.24	4.53	2.19	38.27	24.82
123789-HxCDF	0.00	1.31	13.29	3.29	8.97	1.32	1.16	1.71	0.00	0.00
1234678-HpCDF	0.00	13.13	21.14	25.95	14.06	32.90	14.52	44.10	61.73	17.37
1234789-HpCDF	0.00	1.23	0.00	0.00	9.80	0.00	2.35	0.00	0.00	0.18
OCDF	26.00	2.52	3.95	20.31	22.75	25.99	69.80	32.61	0.00	49.24

industrial areas led to exceed not only the Sediment Quality Limit of 0.85 ng TEQ/kg d.w. but also the Probable Effect Level of 21.5 ng TEQ/kg d.w., with reference to the Sediment Quality Guidelines (www.ec.gc.ca/ceqgrcqe/English/Html/GAAG_PCDDs/PCDFsFuransSediment_e.cfm).

At the last, the microorganisms inability to biodegrade the PCDDs/PCDFs as well as decreased light intensity and reduced photodegradation during the winter and spring may also contribute to the final higher concentrations of analyzed compounds at the spring season (Urbaniak, 2007).

During the autumn season, in turn, the process which may contribute to the increase in total and TEQ PCDDs/PCDFs concentrations in the water of reservoirs I and II may be the deposition of the lower chlorinated, and thus more toxic, congeners on the leaves of the surrounding trees during the summer dry weather (Nizetto *et al.*, 2006) and their washout during intensive rains (Kirchner *et al.*, 2006). Such a changing weather events may also cause flushing of PCDDs/PCDFs from reservoirs I and II and their deposition in reservoir III, a phenomenon possibly exemplified by the July-August rains in both 2007 and 2008.

The biotic interactions within the water ecosystems may also have an effect on the concentration and seasonal distribution of PCDDs/PCDFs. As reported by Berglund *et al.* (2001) and Roessink *et al.* (2008) the concentrations of organochlorine compounds in aquatic organisms decreases with the trophic status of lakes. PCDDs/PCDFs associated with phytoplankton undergo sedimentation, thus purging the water column during the summer/autumn season in favor of the reservoirs' sediments (Larsson *et al.*, 1998; Roessink *et al.*, 2008; Berglund *et al.*, 2001). One further reason for the raised autumn concentrations in the first reservoirs (I and II) may be light limitation, due to the dense tree cover in the park and thus lower photodegradation rate as compared with the other sunlit reservoirs (Urbaniak, 2007). The research shows also the variation of the percentage of individual analyzed PCDDs and PCDFs. High predominance of PCDDs in the total PCDD/PCDF concentration was observed in all the sediment samples (accounted for up to 98.45%). This was in majority generated by the raised concentration of the OCDD congener which reached up to 94.28% in the case of the III reservoir (Table 3). This demonstrates the serious effects caused by the illegal disposal of untreated sewage, which was demonstrated in our earlier research (Urbaniak *et al.*, 2010, 2013; Zerihun *et al.*, 2012) and supported by other Polish studies (Lulek, 2001; Dudzińska 2002A,B; 2004; 2005; Dudzińska & Czerwiński, 2002, 2003; Dudzińska *et al.*, 2004, 2008).

CONCLUSIONS

In the shallow, turbid, hypertrophic Sokołówka River urban reservoirs the distribution of PCDDs/PCDFs is mostly dependent on the weather-related pattern of allohtonic matter transport down the cascade, especially during the winter/spring snow melt and the intensive storm events. The periods of dry weather interrupted by intensive storm events, intensify the flushing of PCDDs/PCDFs down the cascade, increasing their concentration in the downstream reservoirs. Moreover, stormwater outlets supplying the river with stormwater and illegal domestic disposals further contributes to increase of the PCDDs/PCDFs concentrations in sediments.

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