SEEKING POTENTIALLY ANOMALOUS HUMAN EXPOSURES TO PCDD/Fs AND PCBs BY SEWAGE SLUDGE CHARACTERIZATION

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SUMMARY: An approach to detect anomalies in the exposure to Persistent Organic Pollutants (POPs) throughout the food chain is presented. A steel making plant and its surrounding area were selected as a case-study. To investigate the possible effects of the plant on the settled population, sewage sludge samples from four wastewater treatment plants (WWTPs) were taken: one of these was chosen as reference for the population exposed to the emissions of the mill; the remaining three plants were chosen to provide background information about the POP content of sludge. No clear anomalies in dioxins (PCDD/Fs) were detected for the potentially exposed population. In terms of Polychlorinated Biphenyls (PCBs), the reference WWTP showed a total concentration between 2.7 and 4.8 times higher than the other plants; in terms of equivalent toxicity, only slightly higher concentrations were found for the reference WWTP. Therefore, considering acceptable the daily intake from the diet of the unexposed population, the absence of a dioxin and dioxin-like emergency in the area of the mill is demonstrated. This method well assesses situations of permanent exposure to POP levels that are higher than the background, even if it can lead to misleading results in presence of acute episodes.

1. INTRODUCTION

Within the large family of Persistent Organic Pollutants (POPs), organochlorine substances like polychlorinated dibenzo-p-dioxins (PCDDs), polychlorinated dibenzofurans (PCDFs) and polychlorinated biphenyls (PCBs) are considered the most toxic and diffuse compounds (Rada et al., 2011). PCDD/Fs are commonly named dioxin and are composed of a total number of 210 congeners, whose the most toxic for humans and animals could be grouped in 17 congeners, 7 for PCDDs and 10 for PCDFs (Rada et al., 2006). PCBs are known to be made up a total 209 congeners, but the most toxic ones are the co-planar PCBs, which are 12 and present a toxic behavior similar to PCDD/Fs; for this reason, these congeners are named dioxin-like PCBs. The exposure to such compounds has been object of important concern in the last decades (Rada and Ragazzi, 2008), especially in the light of their health effects. Acute toxicity does not represent the most important hazard; the primary risk is related to chronic exposures at lower concentrations: indeed, 2,3,7,8-TCDD, the most toxic PCDD/F congener, was classified in Group 1 as carcinogenic to humans, being the most known cancer promoter, since increased risks for lung cancer, soft-tissue sarcoma, non-Hodgkin lymphoma and other malignant neoplasms in the most informative cohort studies were reported (IARC, 1997; Silberg and

Gasiewicz, 1989). There is also clear evidence that PCBs cause cancer in animals, especially liver and thyroid neoplasms (USEPA, 2012; EFSA, 2005). In addition, the results of a number of epidemiological studies raise concerns for the potential carcinogenicity of PCBs on humans (USEPA, 2012). For these reasons, IARC classified PCBs in the Group 2A, as potential carcinogenic to humans (IARC, 1987).

Since POPs are capable to enter the food chain, due to the lipophilic properties of these compounds, bioaccumulation represents the most important way of contamination. Contamination of food is primarily caused by deposition of emissions coming from various sources on farmland and the subsequent accumulation in the food chain (Liem et al., 2000; Van Ham et al., 2011). On European scale, it was estimated that steel making plants would constitute the most important source of PCDD/Fs (Quaß et al., 2004). The metal sector represents also an important PCB source as demonstrated by a study about the wastewater of scrap metal recycling plants (Van Ham et al., 2011). Some studies focused on the mass balance of POPs throughout the human body (Juan et al., 1999; Moser and McLachlan, 2001) showing the important role of bioaccumulation. An alternative approach to assess the existence of unexpected high POP levels into the food chain could be represented by the analysis of sewage sludge at wastewater treatment plants (WWTPs).

Aim of this study is then to propose a methodology to detect anomalies throughout the food chain, as a consequence of the release of POPs in air from industrial sources. This study focuses on a steel making plant located in a West-East oriented Alpine valley in the North of Italy. Three domestic WWTPs, located outside the area of influence of the plant, were chosen as background reference for assessing the content of POPs in their sewage sludge. Another domestic WWTP, which receives the wastewater from the villages located in the vicinity of the plant, was chosen as representative of the population exposed to the intake of POPs released by the plant. Sewage sludge samples were analyzed and the results discussed in order to assess the presence of possible anomalies in the population exposure to POPs.

2. MATERIALS AND METHODS

To highlight the effect of PCDD/F and PCB emissions from the steel making plant on the food chain and to reconstruct the fate of these compounds into the environment, an analysis of four sewage sludge samples from four different WWTPs was carried out. One of them was chosen as reference to assess the direct impact of the emissions from the steel making plant on the food chain of the population that lives inside the area of influence of the mill. The choice of this WWTP started from previous dispersion simulations of PM emitted by the plant, whose results allowed detecting the population potentially exposed to contamination, through direct inhalation and assumption via food intake after deposition (Rada et al., 2012). The region surrounding the plant hosts several cultivated lands, cattle and dairy farms. Due to the abundance of meat, dairy products, fruit and vegetables produced in the study region, the percentage of consumption of locally produced food can be assumed significant. This is important in the light of the fact that more than the 90% of the average dioxin daily intake is estimated deriving from food consumption, primarily dairy products, followed by cereals and vegetables, meat and fish (Fürst and Wilmers, 1997; Eduljee and Gair, 1996). In fact, POPs have lipophilic properties and bioaccumulation represents the prevailing way of contamination of the food chain: atmospheric deposition of POPs coming from various sources (e.g. waste treatment plants, production of chemicals, metal industry) firstly contaminates soil, hay, vegetables and fruit (Liem et al., 2000); contaminated soil and hay may transfer the POPs accumulated to the adipose tissues of cattle and, subsequently to the consumption of meat and dairy products, to the humans, whilst contaminated vegetables and fruit may transfer their POP content to cattle but also directly to humans. The absorption of PCDD/Fs and PCBs from the diet can result higher than 80% in situations of elevated POP intake (Moser and McLachlan, 2001). The absorption is followed by excretion from the body, which does not depend on fluctuation of the concentration in the diet, but only on the concentration in the body: in fact, the fecal elimination occurs continuously at a rate that depends on the blood concentration (Moser and McLachlan, 2001). The body, thus, seems to be capable to equalize the effects of peak intakes of POPs.

The reference WWTP, located inside the area of influence of the steel making plant, was finally chosen from those plants whose catchment area includes the villages directly exposed to the emissions, according to the dispersion simulations. The input for this WWTP was assumed composed for 60% by the wastewater from the potentially exposed population; the remaining 40% was assumed composed by wastewater from other residential unexposed population; moreover, the WWTP line receives liquid streams from the thermal drying of provincial sewage sludge, but they can be supposed poor of dioxin as dominated by the liquid phase: indeed, a positive association between dioxin levels and suspended solids was found in a previous study, which supports the theory that dioxin-like compounds will partition almost exclusively onto the organic matter in sludge in preference to water (Telliard et al., 1990).

As a term of comparison, three more WWTPs were chosen as representative of the population living outside the study area, hence providing background information on the PCDD/F and PCB levels in the food chain. Similarly, these three plants were chosen so that their catchment areas include villages located outside the area of influence of the plant. Consultation of the local emission inventory excluded the presence of other relevant sources of POPs. Locations of the WWTPs and their distances from the steel making plant are presented in Figure 1.

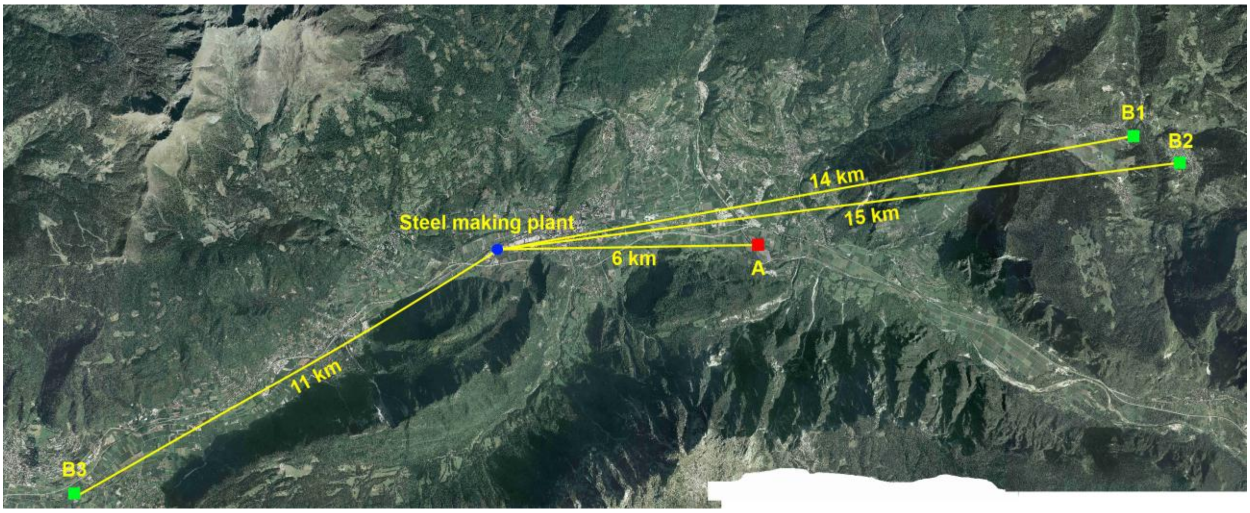

Figure 1. Location of the steel making plant (blue circle), the reference WWTP (red square), the three background WWTPs (green squares) and respective distances from the steel making plant.

The choice of taking sludge samples instead of wastewater samples is related to the fact that sludge is more concentrated in POPs than water. As a matter of fact, the activated sludge treatment that is applied to WWTPs removes hydrophobic compounds like PCDD/Fs and PCBs from wastewater by sorption to sludge (Ju et al., 2007). The four sewage sludge samples (sample volume of 2 l each), one for the reference WWTP and three for the background WWTPs, were taken on the 12th December 2011, between 9 a.m. and 11 a.m.. The choice of this period of the year is crucial and is due to the need of avoiding contributions from tourist peaks in the region, which usually occur during summer or during Christmas holidays. In fact, by taking the sludge samples during the low season, mainly the contribution of the resident population is counted. On the other hand, if the sampling had been carried out during a tourist period, the results would have shown the diluting effect of considering the contribution of a foreign unexposed population. The sludge samples were taken before the chemical conditioning with polyelectrolytes, adopted in every treatment plant considered. Since polyelectrolytes are synthesis products, the possibility exists that such substances may contain dioxin compounds and, thus, they may increase the dioxin content of the unconditioned sludge. Choosing to sample sewage sludge before conditioning gives the certainty that no additional sources of dioxin (other than anthropic input and, possibly, negligible deposition contributions at the liquid surface of the tanks in the WWTPs) influenced the sludge samplings.

Moisture of the samples was detected by calculation of the dry residual mass after evaporation at 105°C, according to the method EN 14346:2007 (UNI, 2007). The content of PCDD/Fs was measured by isotope dilution, high resolution capillary column gas chromatography (HRGC)/high resolution mass spectrometry (HRMS), in accordance with the EPA 1613B method (USEPA, 1994). The content of dioxin-like PCBs in the sludge samples was then determined by isotope dilution and internal standard HRGC/HRMS, following the EPA 1668B methodology (USEPA, 2008).

3. RESULTS AND DISCUSSION

The results of the analysis on the four sludge samples from the WWTP taken as reference for the population potentially exposed to contamination (A) and for the three background WWTPs (B1, B2 and B3) are presented in Table 1 and Table 2, for PCDD/Fs and PCBs respectively.

For some PCDD/F congeners (e.g., 2,3,7,8-TCDD and 1,2,3,7,8,9-HxCDF), the concentration measured in sewage sludge is lower than the instrumental detection limit (DL) for all the WWTPs considered. Conventionally, concentration below the DL are assumed as half the DL itself.

As the results show, the total PCDD/F concentrations measured at the reference WWTP (A) are slightly higher than those measured elsewhere, being the congeners 1,2,3,4,6,7,8-HpCDD and OCDD predominant. On the other hand, in terms of total WHO-TEQ concentrations, the A plant shows the lowest dioxin content with respect to the others. Considering that WHO-TEQ concentrations are related to the toxicity for humans, no clear anomalies in PCDD/Fs could be detected in the food chain for the potentially exposed population.

These results are in agreement with the fact that PCDD/F deposition in the surroundings of the steel making plant is substantially low and can be considered similar to that normally found in rural areas, as demonstrated by a monitoring campaign carried out between 2010 and 2011 (Ragazzi et al., 2011). The use of wood as source for domestic heating, which is typical of mountainous regions where this material is abundant, may have had a certain influence in making the results of the four samples comparable.

Table 1. PCDD/F concentrations and respective WHO-TEQ concentrations in sludge samples for the WWTP taken as reference for the population exposed to POP contamination (A) and for the background WWTPs (B1, B2 and B3); concentrations below the DL are assumed equal to half the DL itself. DM = dry matter

Congener	**WHO-TEF**	**Concentration** [ng kg^{-1}_{DM}]				**WHO-TEQ Concentration** [ng WHO-TEQ kg^{-1}_{DM}]			
		A	B1	B2	B3	A	B1	B2	B3
2,3,7,8-TCDD	1	0.05[a]	0.05[a]	0.05[a]	0.05[a]	0.050	0.050	0.050	0.050
1,2,3,7,8-PCDD	1	0.25[a]	0.60	0.25[a]	0.25[a]	0.250	0.600	0.250	0.250
1,2,3,4,7,8-HxCDD	0.1	0.25[a]	0.25[a]	0.60	0.25[a]	0.025	0.025	0.060	0.025
1,2,3,6,7,8-HxCDD	0.1	2.60	1.10	4.00	0.25[a]	0.260	0.110	0.400	0.025
1,2,3,7,8,9-HxCDD	0.1	0.25[a]	0.25[a]	1.20	1.70	0.025	0.025	0.120	0.170
1,2,3,4,6,7,8-HpCDD	0.01	57.00	31.00	46.00	48.00	0.570	0.310	0.460	0.480
OCDD	0.0001	466.00	323.00	355.00	412.00	0.047	0.032	0.036	0.041
2,3,7,8-TCDF	0.1	4.10	1.60	2.40	3.30	0.410	0.160	0.240	0.330
1,2,3,7,8-PCDF	0.05	0.80	0.60	0.60	0.90	0.040	0.030	0.030	0.045
2,3,4,7,8-PCDF	0.5	0.25[a]	0.50	1.70	1.70	0.125	0.250	0.850	0.850
1,2,3,4,7,8-HxCDF	0.1	0.25[a]	0.70	1.40	1.60	0.025	0.070	0.140	0.160
1,2,3,6,7,8-HxCDF	0.1	0.80	1.70	0.90	1.00	0.080	0.170	0.090	0.100
2,3,4,6,7,8-HxCDF	0.1	0.60	1.60	2.00	0.25	0.060	0.160	0.200	0.025
1,2,3,7,8,9-HxCDF	0.1	0.25[a]	0.25[a]	0.25[a]	0.25[a]	0.025	0.025	0.025	0.025
1,2,3,4,6,7,8-HpCDF	0.01	17.90	16.70	6.20	16.40	0.179	0.167	0.062	0.164
1,2,3,4,7,8,9-HpCDF	0.01	0.25[a]	0.50	0.60	0.25[a]	0.003	0.005	0.006	0.003
OCDF	0.0001	44.00	49.00	59.00	52.00	0.004	0.005	0.006	0.005
Total	-	595.60	429.40	482.15	540.15	2.178	2.194	3.024	2.748

[a] **Below the DL**

Table 2. PCB concentrations and respective WHO-TEQ concentrations in sludge samples for the WWTP taken as reference for the population exposed to POP contamination (A) and for the background WWTPs (B1, B2 and B3); concentrations below the DL are assumed equal to half the DL itself.

Congener	WHO-TEF	Concentration [ng $kg^{-1}{}_{DM}$]				WHO-TEQ Concentration [ng WHO-TEQ $kg^{-1}{}_{DM}$]			
		A	B1	B2	B3	A	B1	B2	B3
PCB 77	0.0001	492.0	71.0	172.0	191.0	0.0492	0.0071	0.0172	0.0191
PCB 81	0.0003	19.6	0.5[a]	1.4	1.9	0.0059	0.0002	0.0004	0.0006
PCB 105	0.00003	1890.0	233.0	650.0	700.0	0.0567	0.0070	0.0195	0.0210
PCB 114	0.00003	162.0	0.5[a]	48.0	59.0	0.0049	0	0.0014	0.0018
PCB 118	0.00003	5210.0	1290.0	1800.0	1800.0	0.1563	0.0387	0.0540	0.0540
PCB 123	0.00003	403.0	35.0	136.0	126.0	0.0121	0.0011	0.0041	0.0038
PCB 126	0.1	5.2	0.5[a]	1.4	7.1	0.5200	0.0500	0.1400	0.7100
PCB 156	0.00003	600.0	151.0	343.0	343.0	0.0180	0.0045	0.0103	0.0103
PCB 157	0.00003	99.0	32.0	67.0	52.0	0.0030	0.0010	0.0020	0.0016
PCB 167	0.00003	141.0	55.0	97.0	114.0	0.0042	0.0017	0.0029	0.0034
PCB 169	0.03	7.0	2.1	2.8	6.4	0.2100	0.0630	0.0840	0.1920
PCB 189	0.00003	53.0	14.0	20.0	18.8	0.0016	0.0004	0.0006	0.0006
Total	-	9081.8	1884.6	3338.6	3419.2	1.0418	0.1746	0.3365	1.0181

[a] **Below the DL**

With regard to PCB concentrations, some considerations that differ slightly from the latter can be made: the A plant shows a total concentration between 2.7 and 4.8 times higher than the other plants; the same sludge sample shows a WHO-TEQ concentration which is slightly higher than the other samples, even if the concentration of the most toxic congener (PCB 126) is higher in one of the background WWTPs (B3). The different urbanization, compared to cases B1 and B2, can explain this last difference, since case B3 is characterized by a moderate presence of small industrial activities.

Some considerations about the sensitivity of the methodology can be also expressed: assuming that the consumption of local food is about 10% of the total consumption, considering that the exposure to POPs may be accounted for 90% to food ingestion and assuming that, in conditions of permanent exposure to a certain POP level, POP excretions can be assumed equal to intakes, a concentration about 100 times higher in the locally produced food should be necessary in order to observe an about 10 times higher concentration in feces. Considering that only 60% of the stream is composed by wastewater from the exposed population, a 6 times higher concentration in sewage sludge should be observed at the A plant with respect to the background. Moreover, some authors reported that, in conditions of permanent exposure to normal levels following a higher exposure in the past, the PCDD/F excretion can result twice higher than the intake (Schrey et al., 1998). In such conditions, if the concentration in locally produced food had been 100 times higher than the background and more recently normal, the concentration in sewage sludge at the A plant would be 12 times higher with respect to samples taken from the other WWTPs. On the other hand, the method is not able to detect acute episodes of exposure, since, as previously stated, in such situations even only 20% of the assumed POPs can be excreted: this condition would imply the same concentration in sludge that could be achieved if the POP concentrations in locally produced food, in permanent conditions, were 12 times higher than the background, thus getting to misleading results. A replication of the sludge characterization is then compulsory when anomalous data are found. Finally, because of the lower excretion that occurs when POP intakes increase, some difficulties in assessing the results could be encountered also when the exposure of the local population is slowly increasing.

Assuming acceptable the dietary daily intake of people living in the areas B1, B2 and B3 (for the absence of relevant sources of POPs), the fact that people in area A generate sewage sludge with similar concentrations demonstrates the absence of a dioxin and dioxin-like emergency in the area of the steel making plant.

4. CONCLUSIONS

A methodology to detect anomalies in the accumulation of POPs throughout the food chain, as a consequence of their release into the atmosphere from industrial sources, was presented.

A steel making plant and its surrounding area, located in an Alpine valley in Northern Italy, were selected as a case-study. To investigate the possible effects of the plant on the population living nearby, exposed to POPs both via inhalation and via consumption of locally produced food, sewage sludge samples from four WWTPs were taken: on the basis of available dispersion maps of PCDD/Fs emitted from the mill, one WWTP was chosen as reference for the population settled near the steel making plant and exposed to its emissions; the remaining three plants, located outside the area of influence of the mill, were chosen to provide background information about the POP content of sludge. No clear anomalies in PCDD/Fs were detected for the potentially exposed population, confirming the results obtained during a previous deposition monitoring campaign, that measured low deposition as that normally found in rural areas. In terms of PCBs, the A plant shows a total concentration between 2.7 and 4.8 times higher than the other plants; in terms of WHO-TEQ concentrations, only slightly higher PCB levels were found for the A plant, even though the most toxic congener (PCB 126) presented a higher concentration in the B3 plant. However, if we consider acceptable the daily intake from the diet of the population living in the areas not affected by the steel making plant, the absence of a dioxin and dioxin-like emergency in the area of the steel making plant is demonstrated by the similar concentrations of PCDD/F and PCB in the samples sewage sludge.

The method well assesses situations of permanent exposure of the population to POP levels that are higher than the background, even if difficulties in the interpretation of the results can occur in presence of acute episodes. A replication of the sludge characterization, in case of anomalous comparative data, can help for a correct management of the method.

AKNOWLEDGEMENTS

The authors would like to thank Dr Paola Foladori for her important support in this activity, Ms Roberta Villa and Mr Alessandro Chistè for their assistance in the sampling campaign. The authors are also grateful to Dr Werner Tirler and his research group for the analysis of the samples. The authors are also grateful to the Environmental Protection Agency of the Province of Trento (APPA) for the financial support and, in particular, to Mr Maurizio Tava for his precious contribution.

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