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Levels and Congener Profiles of Particle-bound Polybrominated Dibenzo-*p*-dioxins/furans (PBDD/Fs) in Ambient Air around Guangzhou, China

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Abstract The mean particulate polybrominated dibenzo-*p*-dioxins/furans (PBDD/Fs) levels for Guangzhou were in the range of 225–2244 fg/m^3 and showed the spatial trend of background < suburb areas < urban area < industrial area, suggesting the industrial and urban sources of PBDD/Fs. The most abundant congeners were 2,3,7,8-TBDF, 1,2,3,7,8-PeBDF and 2,3,4,7,8-PeBDF. The mean toxicity equivalency quantities (TEQs) of PBDD/Fs were in the range of 89.3–456 fg I-TEQ/m^3 for Guangzhou, which were several times higher than that of the background area (49.1 fg I-TEQ/m^3). The rough inhalation risk evaluation showed that the residents in Guangzhou were in relatively higher exposure levels of PBDD/Fs.

Keywords Dioxins · Furans · Brominated flame retardants · Polybrominated diphenyl ethers

Polybrominated dibenzo-*p*-dioxins and dibenzofurans (PBDD/Fs) have similar physical and chemical properties with their chlorinated analogues and are expected to bioaccumulate in similar way to polychlorinated dibenzo-*p*-dioxins/furans (PCDD/Fs) (Brinbaum et al. 2003; WHO 1998). But being compared with PCDD/Fs, they have received less concern and data about their environmental level are very scarce. A certain number of reports and conferences have showed the occurrence of PBDD/Fs in ambient air (Hayakawa et al. 2004; Li et al. 2007, 2008; Wang et al. 2008), consumer products like plastics and TV sets (WHO 1998), sediments (Choi et al. 2003a; Ren et al. 2009), biota samples (Fernandes et al. 2008) and even human samples (Choi et al. 2003b), which proved that PBDD/Fs were ubiquitous in the environment. Toxicity equivalency quantity (TEQ) contributed by PBDD/Fs counted up to 15% of the total dioxin TEQ of five analyzed persistent organic pollutants (POPs) (Jogsten et al. 2010). Moreover, PBDD/Fs seem to be more persistent towards mammalian metabolism for the longer half life of *tetra*-PBDF than that of *tetra*- PCDF in mice and rat (Birnbaum et al. 2003; Hagberg 2009). Consequently, it is necessary to pay more attention to the environmental behavior and risk assessment of PBDD/Fs.

PBDD/Fs are known to be produced unintentionally as by-products during the processes of manufacturing, using and recycling of brominated organic chemicals (WHO 1998), especially brominated flame retardants (BFRs), e.g., polybrominated diphenyl ethers (PBDEs) (Sakai et al. 2001; WHO 1998). Therefore, the large-scale usage of BFRs and the products containing BFRs is a potential source for increased PBDD/Fs' exposure to humans as well as wildlife (Birnbaum et al. 2003), which is reflected by the detectable level and increasing trend of PBDD/Fs in various environment matrices (Hagberg 2009). The incineration, combustion and some illegal disposal of BFRs-containing wastes have also been proved to cause the pollution of PBDD/Fs (Li et al. 2007; Wang and Chang-Chien 2007; WHO 1998). Besides, natural production of organobrominated compounds by marine organism was also suspected as an important source of PBDD/Fs (Haglund et al. 2007).

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Generally, PBDD/Fs are expected mostly being particle-bound at ambient temperatures for their high melting points and very low vapor pressures (WHO 1998). Guangzhou is the central city of the well-known industrial area Pearl River Delta (PRD) in South China, where have been heavy-polluted for its rapid economic growth and being one of the largest productions sites for computers and electronic products in the world. The atmospheric levels of PBDEs, the most important precursors of PBDD/Fs, in Guangzhou were reported higher than those in North America and Europe, especially BDE-209 (Chen et al. 2006). With this background, we conduct this preliminary study to investigate the levels, congener profiles and possible emission sources of PBDD/Fs in ambient air around Guangzhou.

Materials and Methods

In this study, four districts representing industrial area (Huangpu, HP), prosperous urban area (Tianhe, TH), and suburb areas (Panyu, PY and Huadu, HD), respectively were selected to evaluate the atmospheric particulate contamination of PBDD/Fs in Guangzhou (Li et al. 2007; Yu et al. 2006). For comparison, air particles were also collected from a Chinese National Key Scenery Area named Dinghu Mountain (DH) with good environment protection and no pollution source around. Fig. 1 shows the locations of the sampling sites and the relevant landmarks. Short sampling term of about 1 week without rain or snow was schemed at each of the five sampling spots with intelligent high-volume air samplers (Tianhong Intelligent Instrument Plant in Wuhan, China). All samplers were calibrated with an air sampler calibrator each time before and after air sampling interval. The total suspended particulate was collected with a glass fiber filter (20.3 cm × 25.4 cm, Whatman) which was heated at 450°C for 4 h in air to remove possible organic background contaminants and sealed in aluminum foil prior to use. After sampling, the filters were packaged with aluminum foil and stored <4°C until analyzed.

The analysis of PBDD/Fs followed the U.S. EPA Method 1613 and Compendium Method TO-9A for PCDD/Fs. Briefly to say, samples were added $^{13}C_{12}$-PBDD/Fs standards and extracted with toluene for 48 h. The extracts were cleaned orderly through acid silica gel bed (H_2SO_4: silica gel = 40:60, w/w, 50 g/sample, in n-Hexane), multi-layer silica gel column and Florisil column. The final eluent was concentrated to 20 μL under gentle nitrogen stream and spiked with the internal standards of PBDD/Fs for instrumental analysis. HRGC-HRMS (Thermo Finnigan MAT95 XP, Germany) running in positive electron impact (EI^+) mode with selected ion monitoring (SIM) at the resolution >10,000 was used for PBDD/Fs analysis. DB-5 MS fused silica capillary column (J&W Scientific, CA, 30 m × 0.25 mm i.d., 0.1 μm). Oven temperature: 150°C (2.0 min) to 220°C at 40°C/min, 220°C to 300°C (5.0 min) at 7.4°C/min. Detailed analysis method for PBDD/Fs, PCDD/Fs and elemental carbon (EC) can be referred to our previous work (Li et al. 2007, 2008; Yu et al. 2006).

The solvents and reagents used in the analytical procedure were all pesticide grade or higher. All $^{13}C_{12}$-labeled PBDD/F standards were obtained from Cambridge Isotope Laboratories Inc., USA. The silica gel (70−230 mesh, Aldrich, USA) and the Florisil (60−100 mesh, Merk, Germany) were Soxhlet extracted with DCM for 24 h, vacuum-dried and activated at 180°C for 5 h and 135°C for 24 h, respectively before use. Each sample was spiked with $^{13}C_{12}$-labeled PBDD/Fs standards with the recoveries range of 66%–122%. Field GFF blank, laboratory blank, ongoing precision and recovery sample (OPR, EPA 1613), and standard reference material (SRM, EDF-2513, CIL, U.S.) were analyzed for each batch of twelve samples for quality control. The detection limits were quantified as three times the standard deviations for the mean concentration in the

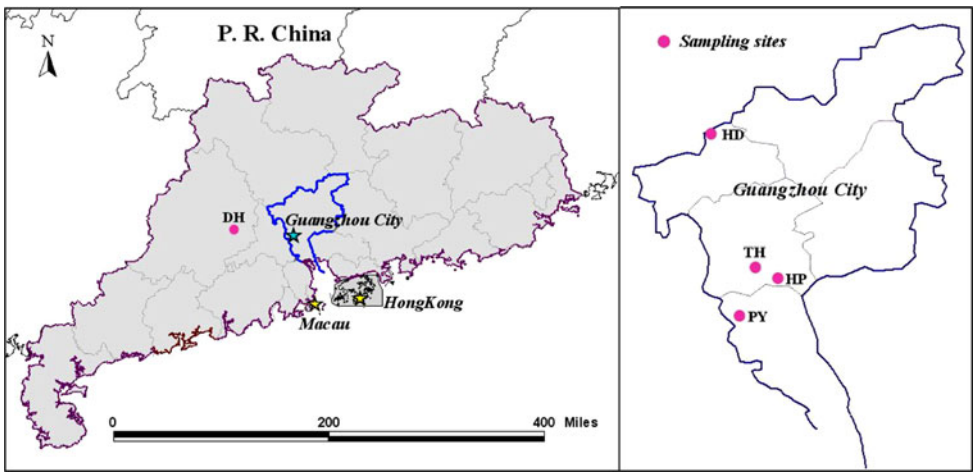

Fig. 1 The detailed locations of the sampling sites of Dinghu Mountain (DH) and different districts of Guangzhou (HD-Huadu, HP-Huangpu, PY-Panyu, TH-Tianhe)

blanks. In this study, the detect limits of the method for PBDD/Fs were 0.25 pg/sample for TBDF and 0.3 pg/sample for TBDD, 0.5 pg/sample for PeBDF and 1.0 pg/sample for PeBDD, 1.5 pg/sample for HxBDD/Fs. During data processing, values below the detection limit were considered equal to zero.

Results and Discussion

The analysis of PBDD/Fs is often disturbed by the presence of PBDEs because the fragment ions forming by PBDEs losing HBr or Br_2 in the ionization chamber of the MS cannot be distinguished from those formed by PBDF (Ebert et al. 1999). Furthermore, PBDD/Fs can also be formed from BFRs via the pyrolysis of some highly brominated and thermally unstable BFRs at high temperatures (Hagberg 2009). The highly brominated PBDD/F homologues could be an indication for such an artifact formation. Therefore, effective clean-up measure to remove PBDEs in samples is one of the most critical sides of QA/QC for PBDD/Fs analysis (Hagberg 2009). Both Florisil column (Ebert et al. 1999; Li et al. 2008) and active carbon column (Choi et al. 2003a; Wang and Chang-Chien 2007) were reported being effective to separate PBDEs from PBDD/Fs. In this study, Florisil column was adopted to avoid the usage of toluene. To verify the separation results, some parallel samples were treated with the clean-up procedures for PBDEs analysis (Chen et al. 2006), and were performed GC–MS (Agilent 7890A GC-Agilent 5975C MS in negative electron ionization mode) analysis with the method for PBDEs. Standards of PBDEs and the corresponding samples prepared for PBDD/Fs analysis were also injected and analyzed with the same method for comparison. The results showed that almost all (>99%) of the PBDEs could be removed from the samples, which suggested the inferences and side reactions caused by PBDEs could be effectively controlled in present study.

Owing to the lack of reference standard compounds, only eight 2,3,7,8-substituted PBDD/Fs congeners were analyzed in air particle samples from Guangzhou and Dinghu Mountain, China. Comparatively, the highest mean concentration (TEQ) of PBDD/Fs was found in the industrial area HP with the value of 2243 fg/m^3 (455.7 fg I-TEQ/m^3), followed by urban area TH (999 fg/m^3, 266.5 fg I-TEQ/m^3), and then the suburban areas PY (701 fg/m^3, 106 fg I-TEQ/m^3) and HD (506 fg/m^3, 89.3 fg I-TEQ/m^3). No significant differences, however, were found between the concentrations of the two suburban areas ($p > 0.05$). The background area DH held the lowest mean PBDD/Fs level of 225 fg/m^3 (49.1 fg I-TEQ/m^3), which is about 10 times lower than that of HP. This changing trend indicated that industries had the most important influence on PBDD/Fs' environmental levels, which was also reported in other areas (Li et al. 2008; Wang et al. 2008). HP is the most important industrial base of Guangzhou with the main industries of petrifaction, automobile, shipbuilding and electronics, some of which are PBDEs-involved industries and may contribute to direct PBDD/Fs emission into the environment. Data of our previous work have showed that the atmospheric concentrations of 11 PBDEs (BDE-209 was included) were 5202–11629 pg/m^3 for HP industrial area, which were much higher than the urban area and background area in Guangzhou, and most areas in Europe and America, especially BDE-209 (Chen et al. 2006). Furthermore, formation of PBDD/Fs from the photolysis of PBDEs was found occurring at outdoor sunlight exposure, and the major photoproducts of BDE-209 were reported as mainly lower brominated PBDFs (WHO 1998).

Urban activities e.g., usage and combustion of BFRs-containing products (Sakai et al. 2001), automobiles traffic, fuels incineration have also been proved as potential emission sources of PBDEs (WHO 1998). There is a thermal power plant locating in HP with the highest electricity capacity of 26.4 million kilowatt-hours per day. Traffic problem in urban areas of Guangzhou is serious and some parking places possess relatively high atmospheric PBDD/Fs levels (unpublished data). EC has been reported as a good indicator of pollutants from combustion processes e.g., PCDD/Fs, polycyclic aromatic hydrocarbons (Yu et al. 2006). In this study, particulate concentrations of PBDD/Fs of HP and TH were found positively correlated with those of EC (Pearson analysis coefficients: 0.782 and 0.844, linear relation coefficients R^2: 0.612 and 0.712, for HP and TH respectively), which suggested that combustion processes also act as one of the emission sources of PBDD/Fs in these two districts.

Studies about PBDD/Fs occurrence in ambient air were very limited and only a few data were available, which were summarized in Table 1. In comparison, ambient air around some special sources, e.g., motorway tunnel in Germany (WHO 1998), recycling center in Taiwan (WHO 1998), e-waste recycling area in China (Li et al. 2007), industrial areas of Shanghai and Taiwan (Li et al. 2008; Wang et al. 2008), held higher PBDD/Fs levels than general urban areas and remote/background areas. The ever-reported highest atmospheric concentrations of ΣPBDD/Fs from source area were of an e-waste recycling area in China (Li et al. 2007) with the values being 23.2–461 pg/m^3 in winter and 8.12–38.6 pg/m^3 in summer, respectively. The highest atmospheric ΣPBDD/Fs levels of urban areas were found in Osaka (1.0–12.5 pg/m^3, WHO 1998) and Kyoto (1.76–12.1 pg/m^3, Hayakawa et al. 2004), Japan. Comparatively, our results (0.057–4.17 pg/m^3) were higher than those of Research Triangle Park of USA, urban areas

Table 1 The summary of PBDD/Fs concentrations (pg/m^3) in ambient air around different areas in the world

Country	Sampling year	Description	Site	ΣPBDD/Fs Conc. Mean (range)	References
Germany	1989	PBDD/Fs (*mono-* to *tetra-*)	Motorway tunnel	(n.d.−0.85)	WHO (1998)
Germany	1990	PBDD/Fs (*tri-* to—*hexa-*)	Motorway tunnel	(0.7–22.3)	WHO (1998)
			Urban area	(0.08–1.97)	
			Suburban area	(n.d.−3.4)	
North California, USA	1990–1991	PBDD/Fs (*tetra-* to—*hexa-*)	Research triangle park	(0.13–0.72)	WHO (1998)
Taiwan	Not available	PBDD/Fs (*tri-* to—*hexa-*)	Recycling center	(3.5–6.7)	WHO (1998)
Osaka, Japan	Not available	PBDD/Fs (*tri-* to—*hexa-*)	Urban area	(1.0–12.5)	WHO (1998)
Osaka, Japan	2001	Eleven 2,3,7,8-PBDD/Fs	Urban areas	0.036	Ohta et al. 2002
Kyoto, Japan	2000–2001	PBDD/Fs (*mono-* to *octa-*)	Urban areas	4.48 (1.76–12.1)	Hayakawa et al. 2004
Guiyu, China	2005	Eight 2,3,7,8-PBDD/Fs	E-waste recycling area	21.9 (8.12–38.6)[a]	Li et al. 2007
				118 (23.2–461)[b]	
Chendian, China	2005	Eight 2,3,7,8-PBDD/Fs	Vincity area of Guiyu	0.912 (0.327–1.41)[a]	Li et al. 2007
				6.31 (0.364–19.1)[b]	
Shanghai, China	2006	Eight 2,3,7,8-PBDD/Fs	New developing area	1.234 (0.066–4.16)	Li et al. 2008
			Commercial area	0.699 (0.032–1.96)	
			Residential area	0.709 (0.161–2.60)	
			Industrial area	1.36 (0.264–2.67)	
Taiwan	2004–2005	Eight 2,3,7,8-PBDD/Fs	Rural area	0.011 (0.003–0.020)	Wang et al. 2008
			Urban area	0.024 (0.015–0.030)	
			Industrial area	0.046 (0.028–0.058)	
			Science park	0.095 (0.058–0.130)	
Guangzhou, China	2004–2005	Eight 2,3,7,8-PBDD/Fs	Background area	0.225 (0.057–0.393)	This study[c]
			Suburban area 1	0.506 (0.143–1.47)	
			Suburban area 2	0.701 (0.270–1.61)	
			Urban area	0.874 (0.227–1.72)	
			Industrial area	2.24 (0.412–4.17)	

[a] Results of summer samples

[b] Results of winter samples

[c] Only particle concentrations were calculated

of Germany (WHO 1998) and Taiwan (Wang et al. 2008), and were similar to Shanghai, China (Li et al. 2008).

Concentrations of seventeen 2,3,7,8-substituted PCDD/Fs in the air particle samples from Guangzhou were also calculated and the mean values ranged from 3648 ± 468 pg/m^3 (130 ± 10 pg I-TEQ/m^3) to 9867 ± 3515 pg/m^3 (653 ± 249 pg I-TEQ/m^3). Yu et al. (2006) also analyzed the particle-bound PCDD/Fs of HD, TH and HP of Guangzhou with the mean TEQ values of 105 ± 16, 164 ± 45 and 769 ± 172 pg I-TEQ/m^3, which showed no significant different from our corresponding data ($p > 0.05$). By contrast, PBDD/Fs' concentrations for the five sampling sites were several times lower than those of PCDD/Fs, while the discrepancies between their TEQ concentrations were small and no significant differences were found between them ($p > 0.05$), indicating the TEQs of air particle samples from Guangzhou contributed by eight 2,3,7,8-substituted PBDD/Fs congeners were comparable to those of seventeen 2,3,7,8-PCDD/Fs congeners. In this situation, it is strongly suggested that more attention should be paid to PBDD/Fs' contamination for the great usage of BFRs in the whole world.

For their very low vapor pressures, PBDD/Fs are predicted mostly being bound to particles at ambient temperatures (WHO 1998). We have found that most PBDD/Fs congeners (>70%) were absorbed in atmospheric particle phase during summer time (29–34°C) and almost all congeners (>90%) were partitioned to particulate phase in winter (10–23°C) in our previous work (Li et al. 2007, 2008). In present study, the average sampling temperatures were in the range of 24–35°C and the gas samples were not available for some objective reasons. Only particulate concentrations will underestimate the total PBDD/Fs level. Based on our previous work, we reckoned the mean total atmospheric PBDD/Fs as 321, 723, 1,001, 1,249 and 3,206 pg/m^3 for DH, HD, PY, TH and HP, respectively

Table 2 Rough inhalation risk evaluation of PBDD/Fs and PCDD/Fs for Dinghu Mountain (DH) and four districts in Guangzhou, China (HD-Huadu, PY-Panyu, TH-Tianhe, HP-Huangpu)

Sampling sites	Particulate TEQ (pg TEQ/m^3)		Atmospheric TEQ[a] (pg TEQ/m^3)		Inhalation exposure dose (pg I-TEQ kg^{-1} day^{-1})					
					PBDD/Fs		PCDD/Fs		Total	
	PBDD/Fs	PCDD/Fs	PBDD/Fs	PCDD/Fs	Adult	Children	Adult	Children	Adult	Children
DH	0.049	0.047	0.070	0.067	0.0150	0.0266	0.0144	0.0255	0.0294	0.0521
HD	0.089	0.130	0.127	0.186	0.0272	0.0483	0.0398	0.0706	0.0670	0.119
PY	0.106	0.289	0.151	0.413	0.0324	0.0575	0.0885	0.157	0.121	0.214
TH	0.195	0.272	0.279	0.389	0.0597	0.106	0.0833	0.148	0.143	0.254
HP	0.456	0.653	0.651	0.933	0.140	0.248	0.200	0.354	0.339	0.602

[a] Calculated from the particle-bound concentrations with the assumption that particulate fraction of 70%

with the assumption that the particulate percentage was 70% at these temperatures.

When the homologue patterns of PBDD/Fs were investigated, PBDFs were found to a greater extent than PBDDs, which is in accordance with the published reports (Hayakawa et al. 2004; Wang et al. 2008; WHO 1998), and three PBDFs isomers contributed 52.8%–95.8% to the total PBDD/Fs. The lower brominated PBDFs homologues dominated in all the samples, especially *penta*-PBDFs. *Hepta*- and *octa*- PBDD/Fs were not included in this study but they were reported being absent in many environmental samples (Hayakawa et al. 2004; WHO 1998). As for the congener profile, the most abundant congener was 2,3,7,8-TBDF, which meanly accounted for 9.6–49.6% of the total eight 2,3,7,8-sunstituted PBDD/Fs concentrations, followed by 1,2,3,7,8-PeBDF and 2,3,4,7,8-PeBDF with the mean relative abundances of 14.6%–40.6% and 14.2%–31.6%, respectively. However, 2,3,4,7,8-PeBDF was the major TEQ contributor with the mean relative percentage changing from 40.9% to 65.9%, then were 2,3,7,8-TBDF (4.5%–21.2%) and 2,3,7,8-TBDD (3.4%–30.3%).

People's direct inhalation exposure to the atmospheric dioxins could be calculated as the average daily respiration intake of I-TEQ equivalents per unit body weight with the assumption that individuals were exposed to polluted air 24 h/day and the indoor air pollution was equal to the outdoor exposure. Respiratory bioavailability is conservatively assumed as 100%. The daily PCDD/Fs exposure doses for adults and children were computed by the following equation (Yu et al. 2006):

$$\boldsymbol{Inh} = \frac{\boldsymbol{V_r C_{air} f_r t_f}}{\mathrm{BW}}$$

Where $\boldsymbol{Inh}$ is the inhalation exposure in pg I-TEQ kg^{-1} day^{-1}, $\boldsymbol{V_r}$ means the ventilation rate and the value is 20 m^3 day^{-1} for adults and 7.6 m^3 day^{-1} for children, $\boldsymbol{C_{air}}$ is the average air concentration of dioxin in pg I-TEQ m^{-3}. $\boldsymbol{f_r}$ is the alveolar fraction retained in the lungs and 0.75 is taken for both adults and children, $\boldsymbol{t_f}$ is the exposed time fraction and were assumed conservatively as 1, BW is the body weight (70 kg for adults and 15 kg for children) (Yu et al. 2006).

In this study, the daily inhalation exposure doses of PBDD/Fs were also calculated with the above-mentioned formula and the results were tabulated in Table 2. As can be seen, residents in the four districts were in high exposure to PBDD/Fs with the daily inhalation doses being 0.0272–0.140 pg I-TEQ kg^{-1} day^{-1} for adult and 0.0483–0.289 pg TEQ kg^{-1} day^{-1} for children, which were a little higher than that of Shanghai (Li et al. 2008), but were much lower than those of the e-waste recycling area and its vicinity area (Li et al. 2007). Counting PCDD/Fs in would double the values to 0.0670–0.339 pg TEQ kg^{-1} day^{-1} for adult and 0.119–0.602 pg TEQ kg^{-1} day^{-1} for children. The tolerable daily intake of PCDD/Fs established by WHO (1998) is 1–4 pg I-TEQ kg^{-1} day^{-1} and the inhalation intake doses of PCDD/Fs have been reported contributing approximately 2.61% to the total daily intake (Yu et al. 2006). Some our previous researches have roughly estimated the total daily intake doses of PCDD/Fs according to this value (Li et al. 2007, 2008; Yu et al. 2006). But there are still no reference values for the percentage of inhalation intake dose in the total daily intake of PBDD/Fs up to now. The total daily intake PBDD/Fs doses can't be calculated out for the scarcity of data about other exposure pathways and more further research are needed in the future.

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