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Flame retardants and organochlorines in indoor dust from several e-waste recycling sites in South China: Composition variations and implications for human exposure

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ABSTRACT

Several classes of flame retardants, such as polybrominated diphenyl ethers (PBDEs), novel brominated flame retardants (NBFRs), dechlorane plus (DPs), and organophosphate flame retardants (PFRs), together with polychlorinated biphenyls (PCBs) were measured in indoor dust from five villages located in three e-waste recycling regions in Guangdong Province, South China. The medians of PBDEs, NBFRs, and PFRs in dust in five sites ranged from 685–67,500, 1460–50,010, and 2180–29,000 ng/g, respectively. These concentrations were much higher than the medians of PCBs (52–2900 ng/g). BDE 209 and decabromodiphenyl ethane (DBDPE) were the two major halogen flame retardants in dust, while tris-(1-chloro-2-propyl) phosphate (TCIPP) and triphenyl phosphate (TPHP) were the major PFRs. Principle component analysis revealed the different pollutant patterns among different sites. The estimated median human exposures of PBDEs, NBFRs, PFRs, and PCBs via dust ingestion were 1.1–24.1, 0.73–20.3, 1.36–23.5, and 0.04–0.93 ng/kg bw/day for adults, and 16.2–352, 10.7–296, 19.9–343, 0.05–0.61, 0.65–13.6 ng/kg bw/day for toddlers, respectively. Residents from Site 5 had the highest exposure (95 percentile levels and high dust ingestion for toddlers) of PBDEs (3920 ng/kg bw/day), NBFRs (3200 ng/kg bw/day), and PFRs (5280 ng/kg bw/day). More attention should be paid to the contamination with NBFRs and PFRs, instead of PCBs, in these e-waste recycling regions, and local public health threat from PBDE alternatives should remain of concern. To the best of our knowledge, this is the first study on human exposure assessment of PFRs at e-waste sites.

1. Introduction

Improper electronic-waste (e-waste) recycling is an increasingly important issue for global environment and public health. Approximately 70% of global e-waste is processed in China every year (Robinson, 2009). At most recycling sites, the obsolete electronic items, including computers, cables, television sets, and mobile phones, are simply recycled by primitive techniques, such as acid bath and outdoor burning. Toxic metals, as well as organic pollutants, such as flame retardants (FRs) and polychlorinated biphenyls (PCBs), are released into the environment during the improper dismantling of e-waste, which poses huge threats to human health (Ni et al., 2010, 2013; Yang et al., 2008).

Several studies have reported the occurrence of organic contaminants, such as PCBs and polybrominated diphenyl ethers (PBDEs), at e-waste recycling sites (Bi et al., 2007; Deng et al., 2007; Lee et al., 2004; Leung et al., 2007; Venier and Hites, 2008). Atmospheric PBDE concentrations in e-waste recycling areas, such as Qingyuan (3260 ng/m^3) (Tian et al., 2011) and Guiyu (8760 ng/m^3) (Chen et al., 2009; Deng et al., 2007), were higher than those from other parts of the world (Lee et al., 2004; Venier and Hites, 2008). As a result of e-waste treatment, elevated PBDE levels were found in soil from an acid leaching (2720–4250 ng/g) site and an e-waste dumping site (893–2890 ng/g) in Guiyu (Leung et al., 2007). The median PBDE concentration (580 ng/g lw, lipid weight) detected in the serum of Guiyu residents was three times higher than that in individuals from a rural area away from the e-waste site (Bi et al., 2007).

With the phase-out of PBDEs (European Court of Justice, 2008; UNEP, 2009; US EPA, 2009), the market demand for alternative FRs, such as dechlorane plus (DPs), novel brominated flame retardants (NBFRs), and organophosphate flame retardants, (PFRs) has been increasing in recent years (Covaci et al., 2011; Sverko et al., 2011; Van der Veen and de Boer, 2012). Considering the short life cycle of electronic products, it is expected that the alternative FRs will increasingly be found in e-waste. However, the contamination status of e-waste recycling sites with NBFRs, PFRs, and DPs has been only seldom

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reported. DBDPE and 1,2-*bis*(2,4,6-tribromophenoxy) ethane (BTBPE) have been found in air, dust, soil, and also biota samples from e-waste recycling sites (She et al., 2013; Tian et al., 2011; Wang et al., 2010; Zheng et al., 2012). Recently, 2-ethylhexyl-2,3,4,5-tetrabromobenzoate (EH-TBB, 3.09–62.2 ng/g lw) and *bis*-(2-ethylhexyl)-3,4,5,6-tetrabromophthalate (BEH-TEBP, 0.81–16.3 ng/g lw) were detected in food samples from e-waste sites located in Eastern China (Labunska et al., 2015). The occurrence of PFRs in e-waste sites is still unknown, although they have been ubiquitously found worldwide in air, house dust, soil, and biota (Cequier et al., 2014; Van der Veen and de Boer, 2012).

At e-waste recycling sites, human health is seriously threatened via several direct exposure routes such as inhalation, dust ingestion, and dietary ingestion (Deng et al., 2007; Labunska et al., 2013, 2014; Wang et al., 2010). Dust ingestion is considered to be an important pathway of human exposure to organic contaminants (Alves et al., 2014). The potential health risks are particularly higher for infants and toddlers due to their more frequent hand-to-mouth activities and close-to floor behavior (Lioy et al., 2002; Mercier et al., 2011). In the e-waste recycling sites, a large amount of e-waste is domestically dismantled in workshops or even in living rooms, which makes indoor dust ingestion a significantly important exposure pathway for local workers and residents. Poor hygiene awareness and habit of workers would even multiply their exposure risk.

In the present study, a broad range of organic contaminants, including FRs and PCBs, were reported in indoor dust collected from workshops and houses in five e-waste recycling sites in Longtang, Dali, and Guiyu Counties located in South China. The aims of the present study were (1) to provide data about the major organic contaminants pollution, especially NBFRs and PFRs in dust from various e-waste sites; (2) to investigate the site-specific pollution status in contaminants profiles, so as to provide valuable information on the emerging pollution status in the e-waste sites from China; and (3) to estimate the human exposure via dust ingestion from e-waste site area for both adults and toddlers.

2. Materials and methods

2.1. Sampling

Indoor dust samples (n = 56) were collected from five villages located in South China from September to November, 2013. E-waste recycling activities have been conducted in these five villages for many years. Villages 1 (23°32′N, 113°03′E; n = 9), 2 (23°36′N, 113°04′E; n = 7) and 3 (23°34′N, 113°02′E; n = 13) are located in Longtang, Qingyuan City, while villages 4 (23°06′N, 113°07′E; n = 13) and 5 (23°19′N, 116°21′E; n = 14) are located in Dali town in Foshan City and Guiyu town in Shantou City (Fig. 1). Longtang, Dali and Guiyu are among the largest e-waste dismantling and recycling sites in China. Dali is about 50 km away from Longtang and about 300 km from Guiyu. The indoor dust samples from workshops and nearby residence houses were obtained from the surface of furniture, windowsills and floors using woolen brushes which were pre-cleaned with ethyl alcohol. Individual samples were wrapped in aluminum foil and sealed in polyethylene zip bags, and after being transported to the laboratory all samples were kept at −20 °C until chemical analysis.

2.2. Sample preparation and analysis

The sample preparation and analysis method was described in details elsewhere (Van den Eede et al., 2012). Briefly, about 50 mg of sample was spiked with internal standards (BDE 77, BDE 128, ^{13}C-BDE 209, CB 143, ^{13}C-BEH-TEBP, ^{13}C-*anti*-DP, ^{13}C-*syn*-DP, triamyl phosphate (TAP), triphenyl phosphate (TPHP-d_{15}), tris-(2,3-dichloropropyl)-phosphate (TDCPP-d_{15}), tris-(2-butoxyethyl)-phosphate (TBOEP-d_6), tris-(chloroethyl)-phosphate (TCEP-d_{12})), then extracted by ultrasonication and vortexed with hexane:acetone (3:1, v/v). Supelclean™ ENVI™-Florisil cartridges (500 mg, 3 mL, Supleco, Bellefonte, PA, USA) were used for clean-up and fractionations: the first fraction was eluted with 8 mL hexane and the second fraction was eluted with 10 mL ethyl acetate. All PCBs, PBDEs, DPs and DBDPE were present in the 1st fraction, while EH-TBB, BEH-TEBP, BTBPE and PFRs were present in the 2nd fraction. After concentration under nitrogen, the 1st fraction was further cleaned on acid silica (44%, precleaned with 10 mL hexane) and analytes were eluted with 10 mL hexane:dichloromethane (1:1, v/v). After evaporation to nearly dryness, both fractions were resolubilized in 100 μL of iso-octane prior to GC–MS analysis.

The details of the analyzed compounds were shown in Table S1, Supporting Information. The analysis of PCBs, NBFRs and PBDEs was performed by 6890 Agilent (Palo Alto, CA, USA) gas chromatography (GC) coupled to a 5973 mass spectrometer (MS) operated in electron capture negative ionization (ECNI). A DB-5 column

Fig. 1. The location of five e-waste recycling sites in China: Longtang (Sites 1, 2, and 3), Dali (Site 4), and Guiyu (Site 5).

(15 m × 0.25 mm × 0.10 μm) was used for separation and the MS was deployed in selected ion monitoring (SIM) mode. The analysis of PFRs was performed by GC–MS in electron ionization (EI) mode. A HT-8 column (25 m × 0.22 mm × 0.25 μm) was used and the MS was operated in SIM mode with two characteristic ions acquired for each compound. Detailed information about analytical parameters was provided by Van den Eede et al. (2012).

2.3. Quantification and quality assurance

As a part of the quality assurance protocol, procedural blanks (n = 9) and standard reference material — SRM 2585 (n = 3, purchased from NIST) were analyzed in the same batches with the dust samples to account for eventual external contamination during sampling, sample preparation and instrumental analysis, and to evaluate method accuracy, respectively. Levels of target analytes were blank-corrected in all samples. The values of PCBs, PBDEs, NBFRs and PFRs in SRM 2585 were in agreement (RSD < 15%) with reference values or published indicative values (Ali et al., 2013; Van den Eede et al., 2012). The limits of quantification (LOQs) were set as the mean values of target compounds detected in procedural blanks plus three times the standard deviation. BDE 153, CBs 118, 170, 180, and 187, *anti*-DP, and BEH-TEBP were found less than 100 pg in blanks, and BDE 209, DBDPE, TEHP, TNBP, and TDCPP were detected less than 0.70 ng in blanks. These values in blanks were less than 1% of values in most samples. For the undetectable compounds in blanks, the LOQs were estimated as a signal to noise ratio of 10. The LOQs ranged from 0.2 to 1 ng/g (dry weight) for PCBs, DPs, EH-TBB, BTBPE, and PBDEs (except for BDE 209), 5 to 10 ng/g for BDE 209, BEH-TEBP, and DBDPE, 10 to 20 ng/g for PFRs (except for TBOEP), and 100 ng/g for TBOEP (Table 1).

2.4. Statistical analysis

Statistical analysis was performed with SPSS 16 for Windows (SPSS, Inc., Chicago, IL). After the data were log-transformed to display a normal distribution, one-way analysis of variance (ANOVA) was conducted to investigate the differences in contaminant levels between different sites, and Pearson's correlation analysis between levels of EH-TBB and BEH-TEBP was performed. Sample concentrations below LOQs were replaced with 1/2 ∗ LOQ in statistical analysis. Principal component analysis (PCA) was conducted to compare the profiles of contaminants. The first two principle components (PCs) were considered to account for a significant contribution to the total variance according to the latent root criterion. Factor loadings and factor scores were determined and used in interpreting PC patterns.

3. Results and discussion

3.1. Concentrations and congener profiles of pollutants

3.1.1. Organochlorines

PCBs exhibited median levels of 52–2900 ng/g in indoor dust from five sampling areas. CBs 118, 138, and 153 were the predominant congeners. The PCB congener patterns in Site 2 were consistent with that in indoor dust from the same area collected several years ago (Zhu et al., 2014) (Fig. S1, Supporting information). Only limited studies reported the occurrence of PCBs in indoor dust from e-waste sites in recent years (Tue et al., 2013; Zhu et al., 2014). The PCB levels in indoor dust in the present study were higher than those from e-waste sites in Vietnam (4.8–320 ng/g) (Tue et al., 2013). The current PCB concentrations in Site 2 were several times lower than those (9269 ng/g) in dust sample collected five years ago in the same area (Zhu et al.,

Table 1

Concentrations (median (min–max), ng/g) of PCBs, PBDEs, NBFRs, and PFRs in indoor dust from five e-waste recycling sites.

	LOQ	Longtang			Dali	Guiyu
		Site 1 (n = 9)	Site 2 (n = 7)	Site 3 (n = 13)	Site 4 (n = 13)	Site 5 (n = 14)
CB 101	1.00	16 (8.2–156)	104 (29–510)	634 (224–2100)	131 (nd–546)	15 (nd–94)
CB 118	1.00	21 (5.4–252)	208 (28–910)	810 (366–2600)	126 (8.8–850)	18 (5.2–150)
CB 153	1.00	14 (4.5–200)	139 (13–363)	415 (226–1400)	89 (4.7–426)	13 (5.5–79)
CB 138	1.00	15 (4.3–225)	194 (16–364)	462 (268–2050)	86 (5.8–695)	17 (5.3–86)
CB 187	1.00	5.6 (nd–44)	16 (1.6–169)	63 (32–161)	31 (1.1–178)	2.4 (nd–21)
CB 180	1.00	4.8 (1.4–106)	51 (2.9–288)	120 (59–405)	47 (2–279)	3.9 (nd–46)
CB 170	1.00	2.5 (0.8–58)	38 (1.6–126)	65 (36–307)	19 (1.2–82)	2.6 (nd–26)
∑ PCBs		74 (26–1040)	750 (92–2300)	2900 (1280–9030)	544 (36–2720)	52 (20–474)
BDE 28	0.50	(nd–7.8)	9.1 (1–168)	8.1 (6.2–24)	2 (0.5–7.9)	48 (9.1–176)
BDE 47	0.50	12 (nd–186)	146 (6.6–1405)	143 (82–362)	55 (5–92)	984 (26–3440)
BDE 100	0.50	2.5 (nd–51)	24 (nd–170)	39 (19–84)	14 (2.3–31)	135 (10–1340)
BDE 99	0.50	13 (2.1–275)	227 (7.5–1630)	232 (129–670)	100 (13–201)	1370 (48–7270)
BDE 154	1.00	2.3 (0.8–136)	40 (1.9–252)	107 (82–729)	18 (2.3–329)	140 (15–730)
BDE 153	1.00	3.6 (0.7–103)	135 (3.7–884)	148 (42–1430)	23 (2.7–67)	636 (45–4790)
BDE 183	1.00	9.2 (3–422)	323 (7.8–2710)	214 (117–617)	57 (3.6–155)	2030 (60–24,500)
BDE 209	5.00	644 (146–19,100)	11,100 (385–73,100)	22,500 (10,200–195,000)	6470 (391–51,900)	55,100 (1770–232,000)
∑ PBDEs		685 (173–20,010)	12,600 (414–78,700)	23,600 (11,000–196,000)	6980 (420–52,300)	63,300 (3520–237,000)
Syn-DP	0.50	23 (8.7–279)	401 (12–1180)	1000 (531–14,500)	146 (6.3–418)	1050 (49–4530)
Anti-DP	0.50	80 (20–572)	1150 (39–3410)	2100 (832–9820)	250 (17–730)	2378 (196–9940)
∑ DPs		103 (32–811)	1480 (51–4600)	2990 (1410–23,600)	450 (23–1150)	3460 (244–14,500)
BEH-TEBP	5.00	88 (10–268)	160 (8.2–652)	7120 (nd–17,600)	193 (nd–928)	49 (nd–779)
EH-TBB	1.00	7.5 (nd–192)	29 (nd–75)	311 (4.9–862)	36 (7.1–116)	60 (nd–178)
BTBPE	0.50	28 (5.8–374)	148 (7.2–12,700)	101 (2.8–432)	40 (3.8–227)	3870 (20–27,600)
DBDPE	10.0	1160 (nd–6540)	10,900 (nd–96,800)	26,300 (11,300–181,000)	6400 (1920–23,400)	45,400 (197–201,000)
∑ NBFRs		1460 (39–6880)	11,700 (31–109,600)	39,500 (13,400–199,000)	6580 (2000–23,600)	50,010 (365–228,000)
TEHP	10.0	41 (nd–936)	49 (nd–131)	170 (57–1500)	189 (68–566)	75 (13–2550)
TNBP	10.0	(nd–92)	(nd–29)	16 (nd–53)	61 (19–2100)	78 (24–789)
TCEP	20.0	177 (nd–671)	322 (nd–528)	269 (131–977)	394 (76–1740)	633 (149–6920)
TBOEP	100	nd	nd	301	220 (nd–1060)	(nd–1860)
TPHP	20.0	1110 (86–15,800)	2500 (122–16,500)	3320 (119–6030)	1740 (31–6660)	9810 (371–332,000)
EHDPP	20.0	88 (28–731)	201 (34–1380)	221 (111–630)	406 (28–4550)	2180 (222–6900)
TDCPP	10.0	97 (nd–636)	252 (nd–1500)	288 (128–1610)	317 (nd–1730)	1500 (228–14,100)
∑ TCPP	10.0	386 (48–4940)	1300 (527–2840)	2310 (878–4800)	3110 (640–8790)	3760 (854–10,000)
∑ PFRs		2180 (200–22,000)	5560 (1190–20,400)	6750 (2160–12,800)	7600 (934–21,500)	33,100 (4660–350,000)

2014). This is probably caused by the fact that different e-wastes were dismantled instead of transformers in recent years, which bring less PCBs into the environment than before.

DPs have been used over 40 years and have been recognized as alternative FRs for PBDEs (Sverko et al., 2011). The median values of DPs in dust samples from Sites 1–5 varied from 103 to 3260 ng/g. Zheng et al. (2010) reported DP levels (mean of 1515 ng/g) in dust from e-waste recycling workshops located in Longtang town. These concentrations were comparable with those in dust samples from Sites 2 and 3 in the present study. The DP levels in the present study were also higher than those in kindergarten dust (231 ng/g) from Beijing, China (Cao et al., 2014b). The f_{anti} values ($f_{\text{anti}} = \textit{anti}\text{-DP} / (\textit{anti}\text{-DP} + \textit{syn}\text{-DP})$) in dust from all sites ranged from 0.61 to 0.74 (mean values) (Fig. S2, Supporting information), which were similar to the reported values for commercial mixtures (Sverko et al., 2011).

3.1.2. Brominated flame retardants

The median PBDE levels in dust from Sites 1 to 5 were 685, 12,600, 23,600, 6980, and 67,500 ng/g (Table 1), respectively, which were much higher than those of PCBs. BDE 209 was the major PBDE congener in all dust samples and contributed up to 90% of PBDEs at Sites 1–4 and 84% in Site 5 (Fig. 2). This indicates that e-waste recycled in the workshops contain mainly Deca-BDE. Concentrations of PBDEs in dust from Site 1 in the present study were lower than those in dust (1441 ng/g) collected in the same sampling area five years ago (Wang et al., 2010). However, the PBDE levels in Sites 2 and 3 were more than ten times higher than those collected five years ago (Wang et al., 2010). The median PBDE levels in indoor dust from Sites 2–5 were at least one order of magnitude higher than those in house dust from Canada (2200 ng/g) (Shoeib et al., 2012), the UK (2900 ng/g) (Harrad et al., 2008), and the USA (3500 ng/g) (Harrad et al., 2008), and they were also higher than the median concentrations in office dust (500–7500 ng/g) from Beijing, China (Cao et al., 2014a) (Table S2, Supporting Information). The PBDE concentrations in the present study were also 1–2 orders of magnitude higher than those in house dust from Kuwait (356 ng/g), Pakistan (145 ng/g), Romania (375 ng/g), Belgium (613 ng/g), and Australia (527 ng/g) (Ali et al., 2013; D'Hollander et al., 2010; Dirtu et al., 2012; Toms et al., 2009). Although PBDEs have been gradually phased-out worldwide, our results indicate that PBDEs are still released into the environment, especially in e-waste sites, due to the hysteresis of obsolete electronic and electrical items.

DBDPE was detected at median concentrations of 1160, 10,900, 26,300, 6400, and 44,000 ng/g in Sites 1–5, respectively (Table 1). Levels of EH-TBB (median: 7.5–311 ng/g), BEH-TEBP (median: 49–7120 ng/g), and BTBPE (median: 28–3870 ng/g) were at least four times lower than those of DBDPE. Compared with concentrations of BTBPE and DBDPE in dust collected from Sites 1–3 five years ago (Wang et al., 2010), the concentrations of BTBPE and DBDPE in the present study increased by 1–8 and 40–400 times, respectively, which might be due to the increasing amount of BTBPE and DBDPE used in electronic products (Covaci et al., 2011) and the corresponding recycling activities of e-wastes containing BTBPE and DBDPE in recent years. It could be hypothesized that BTBPE and DBDPE contamination at e-waste recycling sites will further increase in the future with ongoing recycling activities, because "new" waste should contain more BTBPE and DBDPE instead of PBDEs. This should be of concern in e-waste recycling sites. The concentrations of BTBPE and DBDPE in the present study were up to three orders of magnitude higher than those in dust from other countries (Ali et al., 2013; Dirtu et al., 2012; García et al., 2007; Shoeib et al., 2012; Stapleton et al., 2008; Van den Eede et al., 2011) (Table S2, Supporting Information). EH-TBB and BEH-TEBP levels in the present study were comparable with those in house dusts from the UK (25 and 96 ng/g for EH-TBB and BEH-TEBP), Canada (120 and 99 ng/g), and the USA (133 and 142 ng/g) (Ali et al., 2011; Shoeib et al., 2012; Stapleton et al., 2008).

3.1.3. Organophosphate flame retardants

PFRs had comparable levels with DBDPE, with medians of 2180, 5560, 6750, 7600, and 29,000 ng/g in Sites 1–5, respectively (Table 1). The levels of PFRs in dust samples from Sites 1–4 were at the high-end of reported PFR levels (Ali et al., 2013; Dirtu et al., 2012), while PFR levels (median of 29,000 ng/g) at Site 5 were the highest that have been reported in the dust sample by far, much higher than those in Kuwait (6555 ng/g) and Pakistan (575 ng/g), Belgium (16,500 ng/g), Romania (7000 ng/g), and Spain (21,500 ng/g) (Ali et al., 2013; Dirtu et al., 2012; García et al., 2007; Van den Eede et al., 2011) (Table S2, Supporting Information). TPHP and $\sum$TCPP (tris(chloropropyl)phosphate) were the most abundant PFRs and collectively accounted for over 70% of the total PFR concentrations (Fig. 3). TCIPP (tris-(1-chloro-2-propyl) phosphate), a TCPP isomer, accounted for 70 to 85% of the $\sum$TCPP in 55 out of 56 samples. To facilitate the comparison, the $\sum$TCPP is discussed throughout this study rather than the individual isomers. $\sum$TCPP and tri-(2-butoxyethyl)-phosphate (TBOEP) were reported as the most abundant PFRs in dust samples from Kuwait, Pakistan, Belgium, Romania, and Spain (Ali et al., 2013; Dirtu et al., 2012; García et al., 2007; Van den Eede et al., 2011). Higher levels of TPHP (7360 ng/g) than $\sum$TCPP (572 ng/g) were only found in house dust from the USA (Stapleton et al., 2009). TPHP is widely used as a plasticizer in unsaturated polyester resins and in PVC, and also in commercial mixtures, such as AC073 from Supresta and FM 550 from Chemtura (Van der Veen and de Boer, 2012). The high TPHP levels in dust from e-waste sites indicate the different pollution profile introduced by e-waste recycling compared with previous studies.

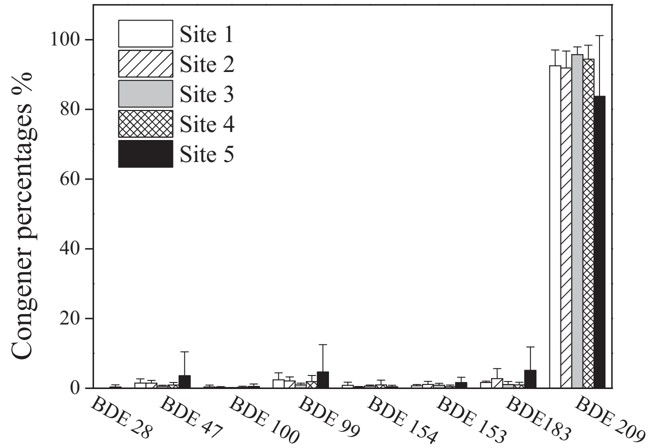

Fig. 2. Congener profiles of PBDEs in indoor dust from e-waste recycling sites. Error bars represent standard deviation.

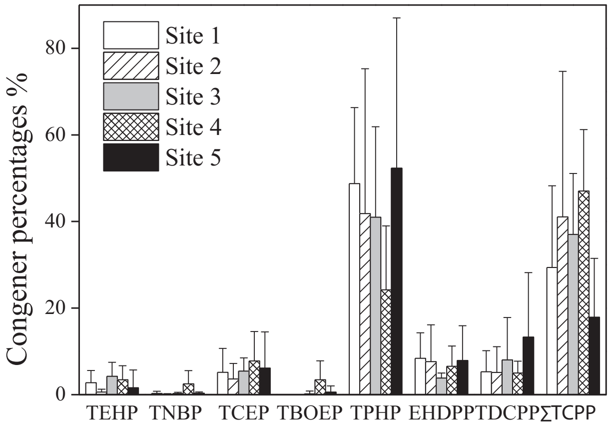

Fig. 3. Congener profiles of PFRs in indoor dust from e-waste recycling sites. Error bars represent standard deviation.

3.2. Spatial variations on levels and sources of pollutants

Sites 3 and 5 had significantly higher levels of PBDEs, NBFRs, and PFRs than Sites 1 and 4 (one-way ANOVA, $p < 0.05$). Significantly lower levels of PFRs at Site 2 than Site 5 were also observed ($p < 0.05$). The higher concentrations of FRs in Sites 3 and 5 could possibly be attributed to the more intensive recycling activities, which result in the permanent introduction of contaminants.

BFRs, including PBDEs and NBFRs, were the main pollutants in all sites with the exception of Site 1, where PFRs were the most abundant pollutants accounting for 48% of the total contaminants (Fig. 4). Almost equal abundance of PBDEs and NBFRs was observed in Sites 2 and 4. The contributions of NBFRs overwhelmed those of PBDEs in Sites 1 and 3 while PBDEs outweighed NBFRs in Site 5. PCBs and DPs contributed less than 8% to total contaminants in all studied areas. This contaminant pattern reflected the usage of FRs in electrical and electronic products which have been discarded. The variations in contaminant compositions also indicate the different types of e-waste dismantled during recycling activities in the five studied areas.

To gain more insight into the pollution pattern in different sites, a principal component analysis (PCA) based on log10 transformed data (data for TBOEP was excluded since it was only detected in Site 4) was conducted. The first two factors identified in PCA accounted for 50% and 22% of the total variance (Fig. 5a). PC1 was heavily influenced by PBDE congeners, DPs, DBDPE, and BTBPE, reflecting a source of halogenated FRs in e-waste. PC2 was heavily influenced by PCB congeners, indicating a source of PCBs in e-waste, such as transformers and capacitors. Most PFRs had high loadings in PC1, implying that these chemicals may come from similar sources to halogenated flame retardants. EH-TBB, BEH-TEBP, and TEHP had low loadings in both factors; this suggests that these chemicals may have different sources from other chemicals. Factor score plot (Fig. 5b) showed that Site 5 was separated from the other sites and had a high score in factor 1, indicating different contaminant patterns from other sites. According to Yang et al. (2008), in Longtang and Dali (Sites 1–4 in the present study), e-wastes sometimes come from the same overseas importation. That could be a possible reason for the different factor score of site 5 in Fig. 5b. Samples in Site 3 had high scores in factor 2, indicating more PCB pollution in this area, which could be the result of the more intensive transformer recycling than in other sites in the past several years. The factor scores of Sites 1 and 2 scattered in a wide range indicated the variation of the e-waste types recycling in different workshops. Based on our previous filed observations, electrical wires, household electrical appliances (e.g., televisions, fridges and computers), and electrical components were historically recycled in Sites 1, 2, and 3, respectively (Zheng et al., 2012). However, it is still difficult to find a clear relationship between the e-waste types and pollution status. More research is necessary to trace the e-waste recycling activities and the release of pollutants into the environment during recycling processes.

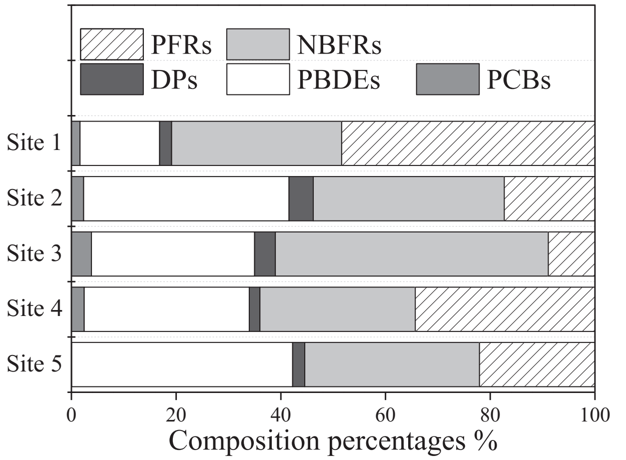

Fig. 4. Composition profiles of different groups of contaminants (PCBs, PBDEs, DPs, NBFRs, and PFRs).

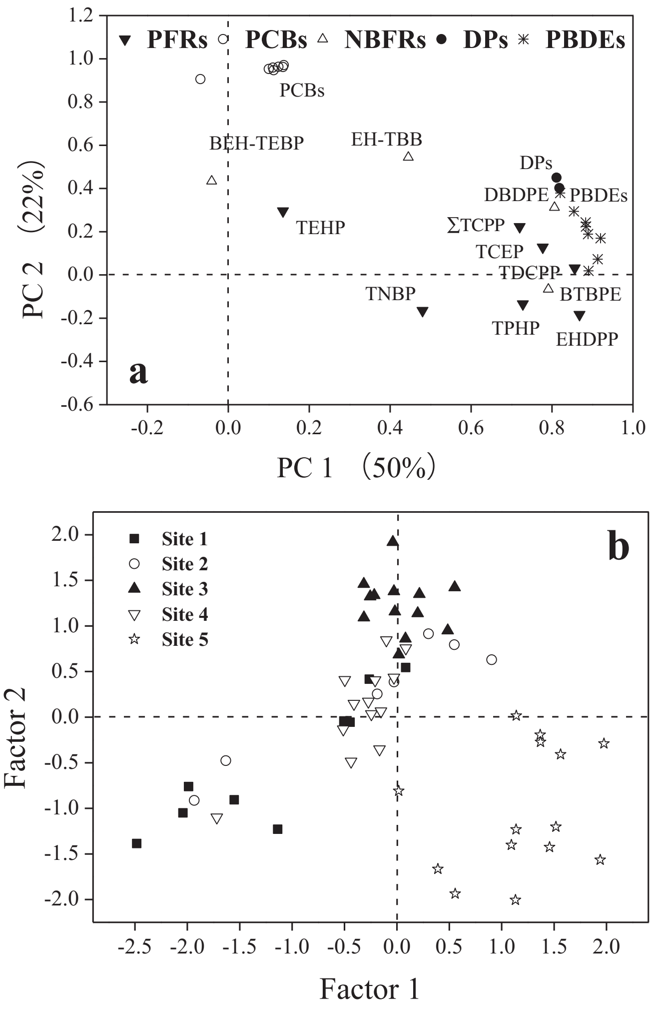

Fig. 5. Principal component analysis results based on the log 10 transformed concentrations of PCB, PBDE, NBFR, and PFR congeners (PC1, 50% variance; PC2, 22% variance). The figure legends represent the factor loadings (a) and factor scores (b).

BEH-TEBP could have the same source as EH-TBB in commercial mixtures. For example, Firemaster 550 consists of about 35% of EH-TBB and 15% of BEH-TEBP (Bearr et al., 2010; Chemtura, 2008) and Firemaster BZ-54 consists of about 70% of EH-TBB and 30% of BEH-TEBP (Bearr et al., 2010). On the other hand, BEH-TEBP also has other sources, such as the commercial mixture DP-45 which contains only BEH-TEBP (Great Lakes, 2004). In order to reveal the sources of EH-TBB and BEH-TEBP in different e-waste recycling sites, we performed a correlation (Pearson's correlation) analysis between EH-TBB and BEH-TEBP in dust samples. A strong correlation was found in dust from Sites 3 and 5 ($R = 0.74$; $p < 0.001$), while no significant correlations were observed at Sites 1, 2, and 4 ($p > 0.05$) (Fig. S3). Therefore, at Sites 1, 2, and 4, DP-45 was likely to be the source of BEH-TEBP, while EH-TBB and BEH-TEBP were more likely derived from the same commercial mixtures, such as Firemaster 550 or BZ-54, in Sites 3 and 5.

3.3. Human exposure assessment

Ingestion of indoor dust is a significant pathway of human exposure to environmental pollutants (Alves et al., 2014). However, limited data are available for the exposure of residents via dust ingestion in e-waste

Table 2
Human exposure assessment of contaminants (ng/kg bw/day) via indoor dust for adults and toddlers from e-waste sites.

		Adults								Toddlers							
		Average dust ingestion				High dust ingestion				Average dust ingestion				High dust ingestion			
		Median	Mean	5th	95th	Median	Mean	5th	95th	Median	Mean	5th	95th	Median	Mean	5th	95th
Site 1	PCBs	0.02	0.08	<0.01	0.26	0.05	0.20	0.02	0.64	0.31	1.16	0.11	3.73	1.23	4.66	0.46	14.9
	PBDEs	0.20	1.11	0.05	3.98	0.49	2.78	0.14	9.95	2.85	16.2	0.79	58.0	11.4	64.8	3.15	232
	DPs	0.03	0.08	0.01	0.22	0.07	0.20	0.02	0.54	0.43	1.18	0.15	3.15	1.72	4.72	0.60	12.6
	NBFRs	0.42	0.73	0.01	1.76	1.04	1.83	0.03	4.39	6.09	10.7	0.20	25.6	24.4	42.7	0.79	102
	PFRs	0.62	1.36	0.06	4.56	1.56	3.41	0.16	11.4	9.07	19.9	0.95	66.6	36.3	79.6	3.80	266
Site 2	PCBs	0.21	0.29	0.04	0.62	0.54	0.74	0.10	1.54	3.13	4.29	0.57	8.98	12.5	17.2	2.28	35.9
	PBDEs	3.60	6.25	0.46	18.3	9.00	15.6	1.15	45.7	52.5	91.1	6.71	267	210	364	26.8	1070
	DPs	0.42	0.61	0.02	1.26	1.06	1.53	0.04	3.16	6.17	8.92	0.26	18.4	24.7	35.7	1.03	73.7
	NBFRs	3.35	7.33	0.01	24.7	8.37	18.3	0.04	61.7	48.8	107	0.23	360	195	428	0.91	1440
	PFRs	1.59	2.11	0.37	4.88	3.97	5.26	0.92	12.2	23.2	30.7	5.34	71.2	92.7	123	21.4	285
Site 3	PCBs	0.83	0.93	0.40	1.79	2.07	2.33	0.99	4.48	12.1	13.6	5.78	26.1	48.3	54.3	23.1	104
	PBDEs	6.75	12.6	3.39	39.7	16.9	31.6	8.49	99.2	98.4	184	49.5	578	394	736	198	2310
	DPs	0.85	1.73	0.44	5.11	2.14	4.33	1.10	12.8	12.5	25.3	6.42	74.6	49.9	101	25.7	298
	NBFRs	11.3	16.1	5.27	41.5	28.2	40.3	13.2	104	165	235	76.9	606	659	939	308	2420
	PFRs	1.93	1.90	0.71	3.28	4.82	4.76	1.78	8.20	28.1	27.8	10.4	47.8	112	111	41.5	191
Site 4	PCBs	0.16	0.20	0.02	0.62	0.39	0.51	0.06	1.54	2.27	2.97	0.35	8.97	9.06	11.9	1.40	35. 9
	PBDEs	2.00	3.22	0.54	10.2	4.99	8.05	1.34	25.5	29.1	47.0	7.82	149	116	188	31.3	594
	DPs	0.13	0.14	0.04	0.27	0.32	0.34	0.10	0.67	1.88	1.98	0.60	3.93	7.51	7.94	2.40	15.7
	NBFRs	1.88	2.93	0.91	5.81	4.70	7.32	2.28	14.5	27.4	42.7	13.3	84.7	110	171	53.2	339
	PFRs	2.17	2.33	0.69	4.80	5.43	5.83	1.73	12.0	31.7	34.0	10.1	70.0	127	136	40.4	280
Site 5	PCBs	0.01	0.04	<0.01	0.12	0.04	0.11	0.01	0.30	0.22	0.65	0.08	1.77	0.87	2.59	0.34	7.09
	PBDEs	18.1	24.1	1.41	67.1	45.2	60.3	3.53	168	264	352	20.6	979	1060	1410	82.5	3920
	DPs	0.99	1.25	0.10	3.00	2.47	3.12	0.24	7.49	14.4	18.2	1.39	43.7	57.7	72.7	5.55	175
	NBFRs	14.3	20.3	2.13	54.8	35.7	50.7	5.33	137	208	296	31.1	799	834	1180	124	3200
	PFRs	9.45	23.5	1.45	90.5	23.6	58.7	3.62	226	138	343	21.1	1320	551	1370	84.5	5280

recycling sites (Wang et al., 2010; Zheng et al., 2010; Zhu et al., 2014). If applying an average body weight (bw) of 70 kg for adults and 12 kg for toddlers, 100% absorption of contaminants, with average dust ingestion of 20 and 50 mg/day and high dust ingestion of 50 and 200 mg/day for adults and toddlers (Jones-Otazo et al., 2005), the daily intakes of contaminants for adults and toddlers via dust ingestion could be calculated, on the basis of the 5th percentile, median, arithmetic mean, and 95th percentile of concentrations of dust samples from five e-waste recycling sites (Table 2).

The intakes of PBDEs, NBFRs, and PFRs were 1–3 orders of magnitude higher than those of PCBs at all sites. The estimated daily intake (EDI) of PBDEs, NBFRs, and PFRs (median levels) ranged from 0.20–18.1, 0.42–14.3, and 0.62–9.45 ng/kg bw/day for adults, and 2.85–264, 6.09–208, and 9.07–138 ng/kg bw/day for toddlers, respectively. The highest EDI values (95% levels) for PBDEs, NBFRs, and PFRs were estimated as 168, 165, and 226 ng/kg bw/day for adults, and 3915, 3844, and 5282 ng/kg bw/day for toddlers, respectively. BDE 209 (maximum 3770 ng/kg bw/day), DBDPE (maximum 2640 ng/kg bw/day), and TPHP (maximum 5060 ng/kg bw/day) were the major contaminants to which the residents of e-waste sites were exposed via dust ingestion. For residents in all e-waste sites, the estimated exposure levels of NBFRs, PBDEs and PFRs (high intake and 95% levels) were higher or at the high-end of the intakes reported in the literature (Ali et al., 2013; Van den Eede et al., 2011; Wang et al., 2010). Additionally, the calculated intake values of BDEs 47, 99, and 209 for toddlers at Site 5 were, respectively, 44, 79, and 54% of corresponding RfDs recommended by the US EPA (US EPA, 2008).

The EDI values of PBDEs and NBFRs (95% levels) from Sites 3 and 5 were up to 30 times higher than those from Sites 1, 2, and 4. The results indicate the heavier exposure for residents from Sites 3 and 5, which could be due to the on-going recycling activities in these two sites. Meanwhile, PFR exposure via dust ingestion (95% levels) in Site 5 was >20 times higher than at all other four sites. The estimated dust ingestion of FRs seemed to vary largely among different e-waste sites. In the present study, the high estimated exposure of organic contaminants via indoor dust, especially for NBFRs and PFRs, suggests a great health threat to the local people. Guiyu town (Site 5) has been long known as the most important e-waste recycling area in China. Our results also showed the highest human exposure risks for PBDEs, NBFRs, and PFRs in Site 5 among all e-waste sites investigated in this study. More attention should be paid on the indoor dust exposure and the health risks for the local residents in e-waste sites, especially for Site 5.

4. Conclusions

The present study reported the occurrence and human exposure assessment of several organic contaminants in indoor dust samples from five villages located in three e-waste recycling regions in South China. BDE 209, DBDPE, TPHP and TCIPP were the dominant chemicals for PBDEs, NBFRs, and PFRs, respectively. The concentrations of PBDEs, NBFRs, and PFRs were several orders of magnitude higher than in other studies, emphasizing the greater pollution introduced by e-waste recycling. The contaminant patterns depended on the types of e-waste recycled in different sites. The concentrations of BTBPE and DBDPE, both being PBDE alternatives, were found to sharply increase as compared to our previous studies of the same sites, which suggest the importance of investigation of alternative FRs at e-waste sites. The human exposure of contaminants via dust ingestion was also higher than most available data. The highest contamination burden of PBDEs, NBFRs, and PFRs was observed for local residents in Site 5 (Guiyu town). More attention should be paid on the contamination of NBFRs and PFRs at e-waste recycling sites, while the increasing levels of PBDEs at some sites should also be of concern.

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Appendix A. Supplementary data

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