

Organophosphate Flame Retardants in House Dust from South China and Related Human Exposure Risks

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Abstract House dust associated with organic pollutants is not only a potential source of pollutants to the outdoor environment, but also a source to human exposure. The present study investigated the occurrence and concentrations of organophosphate flame retardants (OPFRs) in house dust collected from South China dwellings (n=20). The results revealed a universal presence of most target OPFRs in house dust, with concentrations of Σ OPFRs ranging from 2.06 to 19.95 $\mu\text{g/g}$. The median concentration of Σ OPFR (9.20 $\mu\text{g/g}$) was one order of magnitude greater than that of polybrominated diphenyl ethers (0.80 $\mu\text{g/g}$). The composition of OPFR chemicals in house dust was dominated by chlorinated OPFRs, such as tris(1,3-dichloro-2-propyl) phosphate (TDCPP) and tris(1-chloro-2-propyl) phosphate (TCPP). This compositional pattern was different from what has been reported in indoor dust from many other countries, where tris(2-butoxyethyl) phosphate (TBEP) was generally dominant. The daily intake of OPFRs by adults

was estimated to be 1.6 and 4.2 ng/kg body weight/day under average and high exposure scenarios, respectively, and 31.7 and 127 ng/kg body weight/day for toddlers.

Keywords Organophosphate flame retardants · Polybrominated diphenyl ethers · House dust · Daily intake · South China

Flame retardants (FRs) represent a group of anthropogenic chemicals that are added to consumer products in order to meet fire retardancy needs. Among a large variety of FRs, polybrominated diphenyl ethers (PBDEs) have attracted the most attention, due to their global distribution and persistent, bioaccumulative and toxic nature (Law et al. 2014; Darnerud 2003). Along with the discontinuation of PBDE production in North America and Europe, many other FR substances have been produced or subject to increased use in order to replace PBDEs while meeting flame retardancy needs. Among them, organophosphate flame retardants (OPFRs) are of particular concern, due to their high production volumes and potential toxicity (van der Veen and de Boer 2012). OPFRs represent a group of halogenated or non-halogenated compounds with tri-ester structures, including tris(2-chloroethyl) phosphate (TCEP), tris(1-chloro-2-propyl) phosphate (TCPP), tris(1,3-dichloro-2-propyl) phosphate (TDCPP), tributyl phosphate (TBP), triphenyl phosphate (TPhP), tris(2-butoxyethyl) phosphate (TBEP), tris(2-ethylhexyl) phosphate (TEHP), and others (van der Veen and de Boer 2012). They are used as FRs as well as plasticizers in many commercial products, including plastics, foam, textiles, furniture and many others (Reemtsma et al. 2008). These OPFRs are different from PBDEs in physicochemical properties, environmental

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behavior, and fate, thus likely representing different risks to the environment and human health.

As most FR chemicals are not chemically bound to consumer products, a portion of them may migrate from host products, partitioning between indoor air and dust or primarily associated with settled dust particles (Harrad et al. 2010). Chemicals associated with dust can enter the body via inhalation of reentrained dust particles, inadvertent ingestion after hand-to-mouth contact, or direct absorption through the skin (Whitehead et al. 2011). For some individuals, especially children, dust may contribute a substantial proportion of the overall FR intake (Whitehead et al. 2011). Therefore, indoor dust likely represents a significant vector of FRs to humans. Additionally, house dust may also represent a potential source of pollutants, such as FRs, to the outdoor environment (Butt et al. 2004).

Previous studies have demonstrated the universal presence of PBDEs in indoor dust (Harrad et al. 2010). Correlations between dust PBDE concentrations and human health effects were also reported (Vorkamp et al. 2011; Johnson et al. 2013). However, compared to numerous PBDE studies, investigations on OPFRs in indoor environments are fewer in number. In the present study, we conducted a preliminary investigation on OPFRs in house dust from the city of Guangzhou, South China. Specific objectives were to: (1) determine OPFR concentrations and compositions in house dust; (2) compare our finding with the data from international studies; and, (3) estimate human exposure risks via dust ingestion.

Materials and Methods

Reference standards of 20 PBDE congeners (BDE-28, -47, -49, -66, -85, -99, -100, -138, -153, -154, -183, -196, -197, -201, -202, -203, -206, -207, -208, -209) were purchased from AccuStandard (New Haven, CT). A total of 14 OPFRs, including 2-ethylhexyl-diphenyl phosphate (EHDPP), TBEP, TBP, TCEP, TCPP, tricresyl phosphate (TCrP), tris(2,3-dibromopropyl) phosphate (TDBPP), TDCPP, tris(2-ethylhexyl) phosphate (TEHP), triethyl phosphate (TEP), TPhP, tris(3,5-dimethylphenyl) phosphate (T35DMPP), tris(2-isopropylphenyl) phosphate (T2IPPP) and tripropyl phosphate (TPrP), were purchased from AccuStandard or Wellington Laboratories (Guelph, Canada). Surrogate standards, 4'-fluoro-2,3',4,6-tetrabromodiphenyl ether (F-BDE69), 4'-fluoro-2,3,3',4,5,6-hexabromodiphenyl ether (F-BDE160), d_{15} -TPhP, d_{12} -TCEP, d_{15} -TDCPP, d_{27} -TBP, and tris(2-butoxy-[13C2]-ethyl) phosphate (M_6 -TBEP), as well as internal standards, 3-fluoro-2,3',4,4',5',6-hexabromodiphenyl ether (F-BDE154) and coumaphos- d_{10} , were purchased from AccuStandard, Wellington, or

Cambridge Isotope Laboratories (Andover, MA). High performance liquid chromatography (HPLC) grade solvents were purchased from Fisher Scientific (Hanover Park, IL).

A total of 20 different families from the city of Guangzhou voluntarily participated in this study. These families were randomly chosen and their dwellings were scattered across the city. None of the dwellings was from the same building, which avoided pseudo-replication. A customized and pre-cleaned nylon bag with a pore size of $\sim 25 \mu\text{m}$ was attached to the floor attachment of a commercial vacuum cleaner (Electrolux, ZMO1511, 1400W). After room floors from each dwelling were vacuumed, the nylon bag was detached, wrapped with clean aluminum foil, and transported back to the analytical laboratory on ice. Dust on the nylon bag was sieved through a $500 \mu\text{m}$ stainless cloth sieve (Hogentogler & Co., Inc., Columbia, MD) and the sieved dust (approximately 50 mg) was spiked with surrogate standards and extracted with 5 mL of a mixture of hexane and dichloromethane (1:1, v/v) under sonication. Sonication was conducted in three cycles each of which lasted for 5 min. After each cycle, the tube was centrifuged and the supernatant extract was collected. Combined extract from three cycles was divided into two halves. The first half was concentrated and spiked with internal standard F-BDE154 for PBDE determination. The second half was concentrated and cleaned through an ammonium silica (Biotage, Charlotte, NC) solid phase extraction cartridge (Chen et al. 2012). The resulting extract was concentrated and spiked with internal standard, coumaphos- d_{10} , for OPFR analysis.

PBDEs were analyzed on an Agilent 7890B gas chromatogram (GC) coupled to an Agilent 5977A mass spectrometer (MS; Agilent Tech., Palo Alto, CA). The GC was equipped with a 15 m DB-5HT column (0.35 mm i.d., 0.1 μm film thickness, J&W Scientific, Folsom, CA). The GC oven program and the MS parameters were introduced in Boyles et al. (2017). OPFRs were determined on an Agilent 1260 HPLC interfaced with a 3200 Q Trap triple quadrupole/linear ion trap MS (AB Sciex; Toronto, Canada). The HPLC system was equipped with a Waters Xterra[®] phenyl column (2.1 \times 100 mm, 3.5 μm particle size; Waters Corporation, Milford, Massachusetts, USA). The LC-MS/MS protocols were introduced in Chen et al. (2012).

Quality assurance and control procedures included the evaluation of analyte recoveries in spiking experiments, procedural blanks and recoveries of surrogate standards in authentic samples. PBDE and OPFR analytes (50 ng each) were spiked into a dust composite and analyzed using the above methods. The mean (\pm standard deviation) of recoveries of spiked individual analytes ranged from $83.6\% \pm 5.6\%$ to $123\% \pm 3.5\%$ for PBDEs and $66.6\% \pm 12.8\%$ to $115\% \pm 13\%$ for OPFRs in five replicates. Procedural blanks contained no detectable analytes except TBP, TPhP and TBEP, which were present in

blanks at concentrations of up to 8–16 ng/g dw. Blank contamination was subtracted from dust concentrations. Average recoveries of surrogate standards ranged from 70% to 95% for PBDE analysis and 58%–83% for OPFR analysis. Reported concentrations of analytes were corrected based on the recoveries of relevant surrogate standards. The limit of quantification (LOQ), defined as an analyte response ten times the standard deviation of the noise, ranged from 2 to 10 ng/g for PBDEs (tri- and tetra-BDEs: 2 ng/g; penta- and hexa-BDEs: 4 ng/g; hepta- and octa-BDEs: 6 ng/g; non-BDEs: 8 ng/g; deca-BDE: 10 ng/g) and 2–12 ng/g for OPFRs (Table 1). The summed concentrations of all PBDE congeners and OPFR substances were referred to as Σ PBDEs and Σ OPFRs, respectively.

Results and Discussion

Among the target OPFR substances, 12 out of 14 were detected at a frequency of 50%–100% in dwellings from South China, demonstrating a broad occurrence in the indoor environment (Table 1). Concentrations of Σ OPFRs ranged from 2.06 to 19.95 $\mu\text{g/g}$, with the median concentration (9.20 $\mu\text{g/g}$) one order of magnitude greater than that of Σ PBDEs (median: 0.80 $\mu\text{g/g}$; range: 0.20–28.00 $\mu\text{g/g}$) in the same dust (Fig. 1). The greater OPFR concentrations in house dust may be due to broader applications and higher use amounts of OPFRs compared with PBDEs. In addition to their wide applications in various fiber types in textiles, OPFRs are also compatible with other processing chemicals and are easy to use (van der Veen and de Boer 2012). While the market demand data are lacking in China, the European data estimated that OPFRs surpassed brominated

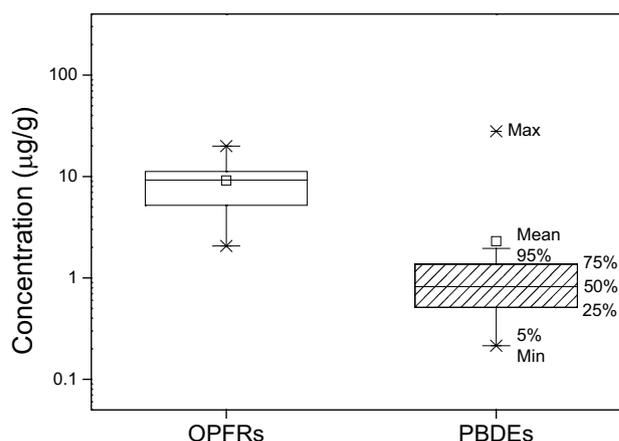


Fig. 1 Concentrations ($\mu\text{g/g}$) of organophosphate flame retardants (OPFRs) and polybrominated diphenyl ethers (PBDEs) in house dust from 20 dwellings in South China

FRs in market consumption (Reemtsma et al. 2008). OPFRs also have potential to replace PBDEs in many applications, such as in textile back-coatings (van der Veen and de Boer 2012). Thus the discontinuation of PBDEs may further increase the use of OPFRs in household products and subsequent releases to the indoor environment. An increasing input of OPFRs from household products to the outdoor environment is also anticipated, given that house dust has been suggested as a potential source of indoor pollutants to the outdoor environment (Butt et al. 2004).

The compositions of OPFRs in house dust were dominated by TDCPP (0.42–10.19 $\mu\text{g/g}$) and TCPP (0.11–4.59 $\mu\text{g/g}$) (Table 1). This should be given some attention, as most chlorinated OPFRs are believed to be

Table 1 Concentrations ($\mu\text{g/g}$) of individual OPFRs detected in South China house dust

OPFR	Full name	Median	Mean	Range	DF ^a (%)	LOQ ^b
EHDPP	2-Ethylhexyl-diphenyl phosphate	1.02	1.24	0.25–6.53	100	0.01
TBEP	Tris(2-butoxyethyl) phosphate	0.40	0.55	0.08–2.07	100	0.006
TBP	Tributyl phosphate	0.11	0.18	0.02–1.17	100	0.002
TCEP	Tris(2-chloroethyl) phosphate	0.41	0.53	0.05–3.13	100	0.010
TCPP	Tris(2-chloroisopropyl) phosphate	0.62	1.24	0.11–4.59	100	0.008
TCrP	Tricresyl phosphate	0.21	0.27	0.03–1.40	100	0.008
TDBPP	Tris(2,3-dibromopropyl) phosphate	nd ^c	nd	nd	0	0.012
TDCPP	Tris(1,3-dichloro-2-propyl) phosphate	3.23	3.51	0.42–10.19	100	0.008
TEHP	Tris(2-ethylhexyl) phosphate	0.77	0.72	0.14–1.22	100	0.008
TEP	Triethyl phosphate	0.05	0.08	<0.006–0.26	90	0.006
TPhP	Triphenyl phosphate	0.61	0.75	0.1–3.55	100	0.01
T35DMPP	Tris(3,5-dimethylphenyl) phosphate	0.03	0.05	<0.01–0.11	50	0.01
T2IPPP	Tris(2-isopropylphenyl) phosphate	0.06	0.08	<0.01–0.22	70	0.01
TPrP	Tripropyl phosphate	nd	nd	nd	0	0.01
Σ OPFRs		9.20	9.12	2.06–19.95	100	

^aDetection frequency; ^blimit of quantification; ^cnon-detectable

carcinogenic. Oral TDCPP exposure to rats induced tumors in the liver, kidneys and testes (van der Veen and de Boer 2012). TDCPP and TCPP also exhibited concentration-dependent neurotoxicity, inhibited DNA synthesis, and decreased cell number and altered neurodifferentiation (Dishaw et al. 2011). Another chlorinated OPFR, TCEP, which was detected in our house dust at concentrations of 0.05–3.13 $\mu\text{g/g}$, is carcinogenic for animals (WHO 1998), toxic to aquatic organisms, and can cause neurological and reproductive effects in rodents (Tilson et al. 1990; Umezu et al. 1998; Chapin et al. 1997). Human studies have reported that OPFRs (e.g., TDCPP and TPP) in house dust may be associated with altered hormone levels and decreased semen quality in men (Meeker and Stapleton 2010). Therefore, the dominance of chlorinated OPFRs in house dust may elevate the overall toxic risks via dust ingestion.

The OPFR composition profiles differed among studies from different countries or regions. Studies from northern China and an electronic-waste recycling site in South China revealed similar patterns in house dust as we observed, i.e. the dominance by chlorinated OPFRs (Cao et al. 2014; He et al. 2015; Zheng et al. 2015). Similar compositional profiles were also reported in indoor dust from Kuwait, Pakistan, Saudi Arabia, and Spain (Abdallah et al. 2014; Ali et al. 2013, 2016; Cristale et al. 2016). This is in contrast with the patterns observed in many other countries, such as Belgium, Canada, Japan, Norway, Sweden, the United Kingdom (UK) and the U.S., where TBEP was usually dominant in indoor dust (Bergh et al. 2011; Brommer and Harrad 2015; Dodson et al. 2012; Fan et al. 2014; Mizouchi et al. 2015; Van den Eede et al. 2011; Xu et al. 2016). The discrepancy may reflect country-specific applications of different OPFR substances.

The concentrations of ΣOPFRs in house dust from South China were in the middle of the concentration ranges reported worldwide (Fig. 2). The greatest concentrations were reported in Japan where median ΣOPFR concentrations were 97 and 300 $\mu\text{g/g}$ in indoor dust from domestic houses and elementary schools, respectively (Mizouchi et al. 2015). A survey of 134 Canadian dwellings revealed a median ΣOPFR concentration of 41.9 $\mu\text{g/g}$ in dust (Fan et al. 2014). Lowest concentrations were reported in Egypt and Philippines dwellings, where median ΣOPFR concentrations were generally less than 1 $\mu\text{g/g}$ (Kim et al. 2013; Abdallah et al. 2014). Market demands on OPFRs as well as the applications may differ largely among countries or regions, resulting in large variances in indoor OPFR concentrations. Nevertheless, ΣOPFR concentrations surpassed those of ΣPBDEs in most international dust studies where data of these two groups of FRs were available.

Given that ingestion of indoor dust represents an important exposure pathway to flame retardants, we estimated the

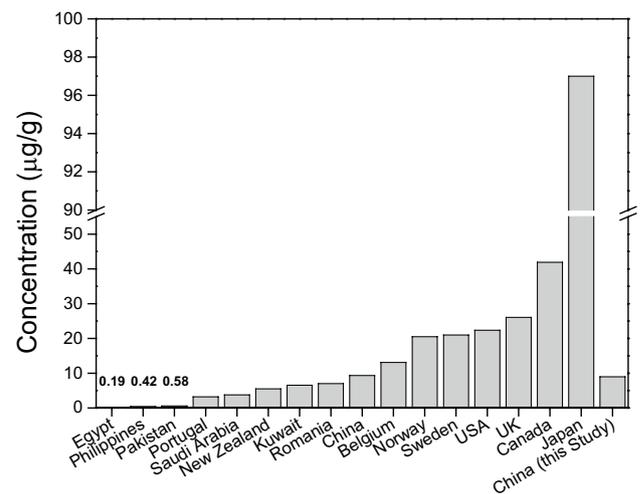


Fig. 2 Concentrations of ΣOPFRs in indoor dust reported in other studies. Literature data were from Ali et al. (2012, 2013, 2016); Abdallah et al. (2014); Bergh et al. (2011); Brommer and Harrad (2015); Coelho et al. (2016); Dirtu et al. (2012); Dodson et al. (2012); Fan et al. (2014); He et al. (2015); Kim et al. (2013); Mizouchi et al. (2015); Van den Eede et al. (2011); and Xu et al. (2016)

daily intake of OPFRs and PBDEs via indoor dust ingestion using the following equation (Liao et al. 2012):

$$EDI = \frac{C \times DIR \times IEF}{BW} \quad (1)$$

where EDI is the estimated daily intake (ng/kg body weight/day), C is the concentration of a chemical in house dust, DIR is the dust ingestion rate (g/day), IEF is the indoor exposure fraction (hours spent over a day in an indoor environment), and BW represents body weight. In this study, we assumed 100% absorption of contamination from ingested dust. The average and high DIR were assumed to be 20 and 50 mg/day for adults and 50 and 200 mg/day for toddlers, respectively (Ali et al. 2013; Abdallah et al. 2014). We also assumed that the average body weights of adults and toddlers are 70 and 20 kg, respectively, and that adults and toddlers spend an average of 63.8% and 86.1% of their time at home, respectively (Abdallah et al. 2014; Klepeis et al. 2001). Therefore, the median daily intake of OPFRs for adults via dust ingestion in South China was estimated to be 1.6 and 4.2 ng/kg bw/day under the average and high exposure scenarios, respectively (Table 2). Elevated intake was estimated for toddlers to be 31.7 and 127 ng/kg bw/day under the average and high exposure scenarios, respectively (Table 2). These daily intake estimates were more than ten times greater than those for PBDEs (Table 2). Van den Eede et al. (2011) reported a reference dose of 1300–8000 ng/kg bw/day for a variety of OPFRs, including TBEP, TBP, TCEP, TCPP, TDCPP, TCrP, and TPhP. The estimated human intake of individual OPFRs in the present study was

Table 2 Summary of estimated daily intake (EDI) of OPFRs and PBDEs by toddlers and adults under different exposure scenarios

	Toddler				Adult			
	Mean	Median	5th	95th	Mean	Median	5th	95th
Average dust ingestion ^a								
ΣOPFRs	31.7	32.1	8.4	62.5	1.6	1.7	0.4	3.2
ΣPBDEs	8.0	2.9	0.9	14.3	0.4	0.2	0.1	0.7
High dust ingestion ^b								
ΣOPFRs	128.0	127.0	33.8	250.1	4.1	4.1	1.1	8.0
ΣPBDEs	11.4	32.0	3.7	57.3	0.4	1.0	0.1	1.8

^aAssuming 20 mg/day for adults and 50 mg/day for toddlers

^bAssuming 50 mg/day for adults and 200 mg/day for toddlers

generally 2–4 orders of magnitude lower than the recommended reference doses. However, potential health risks should not be overlooked given possible additive or synergistic effects among OPFRs or between OPFRs and other persistent organic pollutants present in house dust. Long-term exposure to even low dose of OPFRs may also represent a potential risk, but the relevant studies were very limited to date.

In summary, we investigated the concentrations and compositions of a suite of OPFRs in house dust from South China. The results revealed that ΣOPFR concentrations were generally one order of magnitude greater than those of ΣPBDEs. Chlorinated OPFRs dominated the composition profiles in South China house dust, different from what has been reported in many other countries. Human intake estimates suggested that toddlers have much greater intake of OPFRs than adults, given that toddlers usually have higher dust ingestion rates. The findings from this preliminary study developed a baseline for further research addressing the sources, transport and fate of OPFRs and related human exposure risks in South China.

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