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## Sources of halogenated brominated retardants in house dust in an industrial city in southern China and associated human exposure

She-Jun Chen<sup>a</sup>, Nan Ding<sup>a,b</sup>, Zhi-Cheng Zhu<sup>a,b</sup>, Mi Tian<sup>c</sup>, Xiao-Jun Luo<sup>a</sup>, Bi-Xian Mai<sup>a,\*</sup><sup>a</sup> State Key Laboratory of Organic Geochemistry, Guangzhou Institute of Geochemistry, Chinese Academy of Sciences, Guangzhou 510640, China<sup>b</sup> University of Chinese Academy of Sciences, Beijing 100049, China<sup>c</sup> Chongqing Institute of Green and Intelligent Technology, Chinese Academy of Sciences, Chongqing 400714, China

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## ABSTRACT

Halogenated flame retardants (HFRs) are a class of ubiquitous pollutants in the environment and attract increasing attention. In the present study, HFR concentrations were measured in indoor and outdoor dust in an important industrial city (Dongguan) in southern China, in which their presence and associated human exposure are unknown. The HFRs were dominated by polybrominated diphenyl ethers (PBDEs) and decabromodiphenyl ethane (DBDPE), with mean concentrations of 2365 and 2441 ng/g in the indoor dust, respectively, which were 2–3 order of magnitude higher the concentrations of other HFRs. However elevated tri- to hepta-BDE concentrations (869 ng/g) were found in Houjie Town, a furniture manufacturing center. The mean indoor/outdoor (I/O) ratios of HFR concentrations in the dust were all larger than one (1.55–16.4), suggesting the importance of indoors sources for HFRs in indoor dust in this industrial city. Principal component analysis (PCA) showed that the correlations among the HFRs in the indoor dust probably revealed differences in their commercial applications, while most HFRs in the outdoor dust have similar sources except for phased-out BDE47 and 99. The compositions of lower brominated PBDEs varied among the towns, probably due to their different sources or influence of photo-degradation. Nevertheless, the similar composition of highly brominated congeners indicated little photo-degradation encountered in the ambient environment. The non-cancer risk associated with indoor dust ingestion is low for the general population in Dongguan, but some children in the furniture manufacturing center have significantly high risk of exposure to banned PBDEs.

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## 1. Introduction

Halogenated flame retardants (HFRs) are an important class of chemicals that have been widely used in textiles, furniture, electronics, construction materials, and automotive interiors since the 1970s to improve the resistance to fire (Batterman et al., 2009). HFRs are ubiquitous and persistent in the environment and pose potential health hazards to humans and wildlife (Talsness, 2008; Covaci et al., 2011). Some of these chemicals, such as tetra-through heptabrominated diphenyl ethers (PBDEs) and hexabromocyclododecane (HBCD) have been included in the Stockholm Convention on Persistent Organic Pollutants (POPs) for elimination. However, application products containing these flame retardants are still in use and remain an emission source of HFRs, because most of them are physically mixed into product applications and readily to release into the ambient environment.

\* Corresponding author.

E-mail address: [nancymai@gig.ac.cn](mailto:nancymai@gig.ac.cn) (B.-X. Mai).

Growing evidence has indicated that house dust play a significant role in human exposure to HFRs, due to people's unintentional dust ingestion every day and high HFR concentrations in this medium that have been detected globally (Wu et al., 2007; Lorber, 2008; Johnson et al., 2010). This exposure route is particularly important for young children because of their tendency to play on the floors and frequent hand-to-mouth contact, in agreement with the much greater PBDE exposure of toddlers than adults in previous studies (Jones-Otazo et al., 2005; Fischer et al., 2006). Furthermore, adverse effects such as developmental neurotoxicity and cardiovascular function by prenatal and early-life exposure to PBDEs have been demonstrated in recent studies (Costa and Giordano, 2007; Gascon et al., 2012; Gump et al., 2014), underlining the importance of human exposure to HFRs associated with house dust and other sources (Domingo, 2012).

Release from household products such as textiles, furniture, and electronics in homes is primarily responsible for the elevated HFR concentrations in house dust. Suzuki et al. (2009) found that these additives are transferred from products through miniaturization

and migration into dust as plastic and textile fragments or through vaporization and airborne transfer of microparticulates. Zhang et al. (2011) suggest emission of PBDEs from various indoor products and subsequent partitioning between air and dust. In addition, indoor source is also an important source of PBDEs to the outdoors as evidenced by higher PBDE concentrations in the indoor environment than outdoor levels (Harrad et al., 2008b). However, most of these results were observed in North America and Europe where significant outdoor emissions of HFRs are usually lacking. Little is known about the sources of HFRs in indoor dust in regions where significant outdoor emissions are present and their influence on indoor dust chemicals. Li and co-workers measured PBDEs in the organic films on indoor and outdoor window surfaces in southern China. They found higher concentrations in the exterior films than interior films and suggested that PBDEs were derived mainly from ambient environment (Li et al., 2010).

High HFR levels in the ambient air have been reported in both urban and rural regions in China because of the fast economic development (Chen et al., 2006; Zhang et al., 2009; Qiu et al., 2010; Yu et al., 2011). The study area, Dongguan City, is located in the Pearl River Delta in southern China, which is an important world manufacturing center. Electronic information manufacturing is the priority pillar industry, and electrical machinery and equipment, textile and garments, furniture, and toy manufacturing are also the important industries of this city. High concentrations of PBDEs have been reported in the riverine sediment in Dongguan (Zhang et al., 2009); however, no information is available about the occurrence of HFRs in house dust and associated human exposure to these chemicals in this city. It is not known if the outdoor sources have a significant influence on the HFR contamination in the indoor dust in this industrial region.

In the present study, HFR concentrations were measured in the indoor and outdoor dust in different towns of Dongguan. We aim to understand the HFR levels, compositions, sources, and environmental processes in the house dust, to examine the possible relationships between the industrial development and HFRs contamination in dust in different towns, and to evaluate the risk of human exposure to HFRs through indoor dust ingestion.

## 2. Materials and methods

### 2.1. Sample collection

Dongguan consists of 28 towns and one downtown area and has an area of 2562 km<sup>2</sup> and a population of 1.9 million in 2013 (Fig. S1). Dongguan is an important international production center of computer products and accessories. Electronic and electric equipment manufacturing accounts for nearly half of the city's industrial output. There are over 3300 electronic information manufacturing enterprises that are widely distributed over all the towns. Other industries are mainly in specific towns such as furniture manufacturing in Houjie Town, textile and garments in Humen and Dalingshan Towns, and toy manufacturing in the downtown.

Dust samples (indoor:  $n=102$ ; outdoor:  $n=20$ ) were collected from 11 boroughs of Dongguan (Table S1). The indoor dust samples were obtained from the surface of furniture, tables, and floors of the bedroom and living room where people spend most of their time, using a vacuum cleaner. Likewise, the outdoor dust samples were obtained from the surface of objects (such as floor, tables, and stools) in/around yards or buildings using vacuum cleaner or solvent-cleaned brushes, which are mainly a mixture of deposited atmospheric particles. The samples were wrapped in aluminum foil and sealed in small polyethylene zip bags.

### 2.2. Extraction and cleanup

The dust samples were sieved through a stainless steel 80-mesh sieve. Dust (0.5–1.5 g) was spiked with BDE77, BDE181, and BDE205 and Soxhlet extracted with an acetone: hexane mixture (1:1, v:v) for 48 h. The extract was concentrated to 1–2 mL using a rotary evaporator and then solvent exchanged to hexane. The extracts were purified through a silica column packed with neutral silica (8 cm, 3% deactivated), 44% sulfuric acid silica (8 cm) and anhydrous sodium sulfate (1 cm) from the bottom to top. The effluents were concentrated to 200  $\mu$ L under a gentle nitrogen stream. A known amount of internal standards (4-F-BDE67, 3-F-BDE153, BDE118, and BDE128) were added to the final extracts prior to instrumental analysis.

### 2.3. Instrumental analysis

Di- through hepta-BDEs (BDE28, 47, 66, 85, 99, 100, 138, 153, 154, and 183), pentabromotoluene (PBT), hexabromobenzene (HBB), pentabromoethylbenzene (PBEB), and Dechlorane Plus were analyzed by an Agilent 6980 N gas chromatograph coupled with an Agilent 5975B mass spectrometer (GC–MS) using electron capture negative ionization (ECNI) in the selected ion monitoring mode. A DB-XLB (30 m  $\times$  0.25 mm i.d., 0.25  $\mu$ m film thickness; J&W Scientific, CA) capillary column was used for separation. For octa- through deca-BDEs (BDE196, 197, 201, 202, 203, 206, 207, 208, and 209), decabromodiphenyl ethane (DBDPE), BTBPT, and polybrominated biphenyl (PBB209), the analysis was performed with a Shimadzu 2010 GC–ECNI–MS system equipped with a DB-5HT (15 m  $\times$  0.25 mm i.d., 0.10  $\mu$ m film thickness; J&W Scientific, CA) column. The method detection limits (MDL), defined as the mean blank mass plus three standard deviations, were between 0.2 and 1.0 ng/g based on average dust mass.

### 2.4. Quality control

A procedural blank was run with each batch of samples. Only trace amounts of BDE47, 99, 206, 207, 208, and 209 were detected in the procedural blanks, and they were subtracted from the amounts in the sample extracts. The surrogate recoveries were  $86.3 \pm 11.2\%$  for BDE77,  $107 \pm 8.6\%$  for BDE181, and  $97.6 \pm 19.7\%$  for BDE205 in all the samples. The recoveries of target compounds were 85.7–120% (standard deviations < 13.7%) in the spiked blanks and 79.5–132% (< 16.8%) in matrix spiked samples. The relative standard deviations were within 1.56–16.7% for all compounds in the triplicate samples. Reported concentrations were not surrogate-recovery corrected.

### 2.5. Data analysis

The Pearson product moment correlation and *t*-test were performed on Sigmaplot 12.0, and principal component analysis (PCA) was carried out using SPSS 16.0 for Windows. Concentration data that did not follow a normal distribution were log-normalized. In the PCA, concentrations that were below the LOQ, LOQ values were assigned. A confidence level of 95% was used for the statistical tests and correlation analysis.

## 3. Results and discussion

### 3.1. Concentrations

The total concentrations of HFRs in the indoor dust ranged from 514 to 20,600 ng/g (with a mean of 4986 ng/g), which were significantly higher than those in the outdoor dust ranging from

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