



Distribution pattern of legacy and “novel” brominated flame retardants in different particle size fractions of indoor dust in Birmingham, United Kingdom



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HIGHLIGHTS

- PBDEs and NBFrs measured in floor dust (FD) and elevated surface dust (ESD).
- Particle size distribution of PBDEs and NBFrs studied.
- Concentrations of most target BFRs in ESD exceed significantly those in FD.
- Concentrations of more volatile BFRs higher in finer particle size fractions.
- Concentrations of less volatile BFRs do not vary significantly with particle size.

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ABSTRACT

This study investigates the particle size distribution of eight polybrominated diphenyl ethers (PBDEs) and five “novel” brominated flame retardants (NBFrs) in settled house dust. Elevated surface dust (ESD) and floor dust (FD) were collected from 5 homes in Birmingham, UK, yielding a total of 10 samples. Each sample was fractionated into three different particle sizes: 125–250 μm (P1), 63–125 μm (P2) and 25–63 μm (P3). Non-fractionated bulk dust samples (BD) were also analysed. BDE-209 predominated, comprising an average 74.3%, 77.3%, 69.2%, and 62.7% ΣBFRs of BD, P1, P2 and P3 respectively. $\Sigma_5\text{NBFrs}$ contributed 24.2%, 21.5%, 29.0% and 35.3% ΣBFRs , while $\Sigma_7\text{tri-hepta-BDEs}$ represented 1.5%, 1.2%, 1.7%, and 2.0% ΣBFRs . BEH-TEBP was the predominant NBFr contributing 76.9%, 75.1%, 83.1%, and 83.9% ΣNBFrs in BD, P1, P2 and P3 respectively; followed by DBDPE which contributed 20.1%, 21.9%, 14.1% and 13.9% ΣNBFrs . EH-TBB, BTBPE and PBEB were the least abundant NBFrs. Concentrations of $\Sigma_7\text{tri-hepta-BDEs}$ and BEH-TEBP in P3 exceeded significantly ($P < 0.05$) those in P2, with those in P2 exceeding significantly those in P1. In contrast, no significant differences were found between concentrations of BDE-209, EH-TBB, BTBPE, and DBDPE in different particle size fractions. Concentrations of $\Sigma_7\text{tri-hepta-BDEs}$, BDE-209, and BEH-TEBP in ESD exceeded significantly those in FD ($P < 0.05$). Normalising BFR concentrations to organic carbon content, did not alter these findings. This suggests that differences in BFR concentrations between different particle size fractions are caused by variations in particle surface area to volume ratio, rather than by variations in organic carbon content.

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1. Introduction

Polybrominated diphenyl ethers (PBDEs) are a group of brominated flame retardants (BFRs) added to several polymers used in

various consumer products such as electronics, textiles, and polyurethane foams. They are blended physically with – rather than chemically bound to – polymeric materials, which likely lead them to migrate into the environment (WHO, 1997; USEPA, 2010 Alae, 2003). PBDEs are persistent and bioaccumulative toxicants, with available information suggesting that they affect negatively the nervous system, the liver and the thyroid system (NICNAS, 2007; USEPA, 2008; Shaw et al., 2009; Noyes et al., 2010; Yu et al., 2011; Johnson et al., 2013; Yang et al., 2014; Zhang et al., 2014; Fang

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and Stapleton, 2014). Hence, their manufacture and new use is now banned or severely restricted in many jurisdictions, and they are listed – or in the case of the Deca-BDE product nominated for listing – under the Stockholm Convention on Persistent Organic Pollutants (POPs) (UNEP, 2008, 2013a; b). Such bans and restriction on the use of established BFRs like PBDEs has resulted in increased production of several other BFRs collectively referred to here as novel brominated flame retardants (NBFRs). These NBFRs display physicochemical properties similar to those of established BFRs (Covaci et al., 2011; EFSA, 2012), and there is growing concern about increasing environmental contamination with NBFRs (Shi et al., 2009; Shoeib et al., 2012; Ali et al., 2013; Fromme et al., 2014; Poma et al., 2014; Hassan and Shoeib, 2015; Al-Omran and Harrad, in press; Newton et al., 2015) and their potential negative health effects (Johnson et al., 2013).

Among the various human exposure pathways, ingestion of indoor settled dust appears to represent a major pathway for human exposure to BFRs in North America and Europe, particularly for young children as a result of normal hand-to-mouth activity (Besis and Samara, 2012). Indoor settled dust refers to “particles in building interiors that have settled onto objects, surfaces, floors, and carpeting. These particles may include soil particles that have been tracked or blown into the indoor environment from outdoors as well as organic matter” (USEPA, 2011).

In general, indoor dust falls into a wide range of particle size fractions ranging from <2.5 μm to over 2 mm and possessing organic contents between 5% and 95% (Morawska and Salthammer, 2003). However, for human exposure assessment, dust particles <250 μm are considered of particular concern (USEPA, 2000, 2003), as these have been proposed as those most likely to stick to hands and be ingested (Duggan and Inskip, 1985; Hee et al., 1985; Edwards and Lioy, 1999; Yamamoto et al., 2006).

A large and growing body of literature has investigated human exposure to BFRs via indoor dust using a disparate range of particle sizes, for example, < 75 μm (Xu et al., 2015), <100 (Kang et al., 2011), <125 μm (Wu et al., 2007), <150 μm (Wilford et al., 2005; Shoeib et al., 2012; Whitehead et al., 2012), 25–250 μm (Al-Omran and Harrad, in press), <500 μm (Allen et al., 2008; Wang et al., 2010), 25–500 μm (Harrad et al., 2008a, 2008b; Muenhor et al., 2010; Brommer et al., 2012), <1000 μm (Suzuki et al., 2006), <2000 μm (Gevao et al., 2006) and all fractions (Takigami et al., 2008). Early studies on polycyclic aromatic hydrocarbons (PAHs) and pesticides in indoor dust and airborne particles, indicate that, due to the inverse relationship between particle size and specific surface area, levels of these pollutants increase gradually with decreasing particle size (Lewis et al., 1999; Sugiyama et al., 2000).

The first study to investigate PBDE concentrations as a function of dust particle size was ten years later (Wei et al., 2009). By analysing four different dust fractions (250–420 μm , 150–250 μm , 75–150 μm , and <75 μm) in indoor dust (1 car and 2 homes), Wei et al. (2009) found that PBDE levels in car dust were inversely related to particle size, while they were comparable in homes. Limited studies since, suggest BFR concentrations are influenced significantly by dust particle size. Kefeni and Okonkwo, (2014) reported that of the PBDEs detected in dust particles < 250 μm from 2 homes and two offices; 93.4% were associated with particles < 150 μm . In a similar study, Chao et al. (2014) found no significant difference in concentrations of Σ_{28} PBDE in different particle sizes of house dust and electronic dust. Based on analysis of office dust particle size fractions, Cao et al. (2013) reported some variation in concentrations of PBDEs with particle size. Concentrations of tri-hexa PBDEs were highest in the 74–100 μm and 100–200 μm particle size fractions, those of hepta-PBDEs were greatest in 200–300 μm and 300–400 μm fractions, octa- and deca-PBDE concentrations peaked in particles <50 μm , while 2-bis

(2,4,6-tribromophenoxy) ethane (BTBPE) was highest in the 50–74 μm and 75–100 μm size range. In a subsequent study by the same authors, Cao et al. (2014) found that in several non-domestic microenvironments, BDE-209 showed higher levels in coarser particles in kindergartens (500–900 μm) and dormitories (900–2000 μm). Moreover, BFR concentrations did not increase constantly with decreasing particle size. Instead, the variation of concentrations with particle size was multi-modal, with the highest levels associated with particle sizes around 900, 100, and 10 μm (Cao et al., 2014). A later study revealed no significant variation in concentrations of HBCDs between different particle size fractions (Cao et al. (2015)). Other studies have used forensic microscopy to show that the highest levels of BFRs are present in particles with different morphology, containing more fibre-like material (Wei et al., 2009; Cao et al., 2013).

From the above, it is apparent that while concentrations of many BFRs vary according to the dust particle size fraction, the exact nature of this variation remains unclear. Moreover, the reasons for such variation have yet to be conclusively elucidated. A related issue is whether BFR concentrations differ significantly between floor and elevated surface dust. We hypothesised previously in a study of house dust from Basrah, Iraq, that the existence of such differences would suggest a need to sample both dust sample types, in order to fully reflect exposure of the room occupants (Al-Omran and Harrad, in press). In that same study, we found that differences in the organic carbon content of dust samples, could not explain the higher concentrations of PBDEs and NBFRs detected in elevated surface dust compared to floor dust in the same rooms. It instead seems more plausible that BFR concentrations will be greater in dust particles with a greater surface area to mass ratio, a hypothesis consistent with the higher proportion of finer particles found in elevated surface compared to floor dust in Basrah (Al-Omran and Harrad, in press).

This study therefore reports concentrations of eight PBDEs (BDE-28, BDE-47, BDE-99, BDE-100, BDE-153, BDE-154, BDE-183 and BDE-209) and five NBFRs: pentabromoethylbenzene (PBEB), 2-ethylhexyl-2,3,4,5-tetrabromobenzoate (EH-TBB), bis (2-ethylhexyl) 3,4,5,6-tetrabromophthalate (BEH-TEBP), 2-bis (2,4,6-tribromophenoxy) ethane (BTBPE), and decabromodiphenylethane (DBDPE) in both elevated surface dust and floor dust from Birmingham, UK. Each dust sample is separated into three different particle size fractions (125–250 μm , 63–125 μm , and 25–63 μm). BFR concentrations are reported both as dry weight and normalised to dust organic carbon content. These data are used to test our hypotheses that: (a) BFR concentrations in elevated surface dust will exceed those in floor dust; (b) BFR concentrations will increase with decreasing particle size; and (c) variations in dust organic carbon content cannot account for any variations in BFR concentrations between different particle size fractions.

2. Materials and methods

2.1. Chemicals and standards

Native and labelled PBDEs (BDE-28, BDE-47, BDE-77, BDE-99, BDE-100, BDE-128, BDE-153, BDE-154, BDE-183, BDE-209 and ^{13}C -BDE-209) and NBFRs (PBEB, EH-TBB, BTBPE, ^{13}C -BTBPE, BEH-TEBP, ^{13}C -BEH-TEBP and DBDPE) standards were purchased from Wellington Laboratories Inc. Guelph, Canada as stock solutions in iso-octane, except BDE-209 and ^{13}C -BDE-209 which were purchased as stock solutions in nonane. The recovery determination standard PCB-129 in hexane was purchased from Qmx Laboratories, UK. The purity of all standards is >98%. Ethyl acetate (EA), Acetone (Ac), *n*-Hexane, dichloromethane (DCM), iso-octane, phosphoric acid and sulfuric acid were obtained from Fisher

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