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Identifying transfer mechanisms and sources of decabromodiphenyl ether (BDE 209) in indoor environments using environmental forensic microscopy

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Abstract

Although the presence of polybrominated diphenyl ethers (PBDEs) in house dust has been linked to consumer products, the mechanism of transfer remains poorly understood. We conjecture that volatilized PBDEs will be associated with dust particles containing organic matter and will be homogeneously distributed in house dust. In contrast, PBDEs arising from weathering or abrasion of polymers should remain bound to particles of the original polymer matrix and will be heterogeneously distributed within the dust. We used scanning electron microscopy and other tools of environmental forensic microscopy to investigate PBDEs in dust, examining U.S.A. and U.K. dust samples with extremely high levels of BDE 209 (260–2600 µg/g), a non-volatile compound at room temperature. We found that the bromine in these samples was concentrated in widely scattered, highly contaminated particles. In the house dust samples from Boston (U.S.), bromine was associated with a polymer/organic matrix. These results suggest that the BDE 209 was transferred to dust via physical processes such as abrasion or weathering. In conjunction with more traditional tools of environmental chemistry, such as gas chromatography-mass spectrometry (GC/MS), environmental forensic microscopy provides novel insights into the origins of BDE 209 in dust and their mechanisms of transfer from products.

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Disclaimer

Certain commercial equipment, instruments, or materials are identified in this paper to specify adequately the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology (Gaithersburg, Maryland), nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

Brief: Scanning electron microscopy and other environmental forensic microscopy techniques suggest that BDE 209 can be transferred from products to dust via abrasion or weathering.

Introduction

Polybrominated diphenyl ethers (PBDEs) are examples of “indoor POPs,” persistent organic pollutants that are typically found in much higher concentrations indoors than outdoors. PBDEs are a class of brominated flame retardant (BFR) synthesized and sold in three different commercial formulations referred to as PentaBDE, OctaBDE and DecaBDE (1). While consumer products (e.g., furniture and electronic devices) are thought to be the indoor sources of these compounds, the links between products and PBDEs in house dust and indoor air remains an emergent field of investigation. Allen et al. recently reported associations between PentaBDE in house dust and bromine-containing foam furniture as measured by X-ray fluorescence, as well as between DecaBDE in dust and bromine-containing electronics, primarily televisions (2). Longitudinal analysis of air collected from an office using passive air samplers suggested that a computer was the main source of PentaBDEs in the air (3). Although such field studies do not tell us how PBDEs are transferred from product to dust or air, at least three mechanisms have been proposed: volatilization from products (presumably increased during heating), direct partitioning between PBDE in polymers and dust, and physical weathering or abrasion (2,4–9). While chamber experiments have documented volatilization of PentaBDE congeners from both foam and electronics (6,10), this work does not explain the very high levels of DecaBDE found in some indoor dust samples, concentrations of the order of 1 mg/g (11,12). We hypothesize that volatilized/partitioned PBDEs will be associated with dust particles containing organic matter (8) and will be homogeneously distributed in house dust. In contrast, PBDEs arising from weathering or abrasion of polymers should remain bound to particles of the original polymer matrix and will be heterogeneously distributed within the dust.

Environmental forensic microscopy (13,14) provides one potential method for identifying and characterizing the materials in dust that contain PBDEs and may aid in distinguishing between different transfer mechanisms. Among the most important and versatile tools is scanning electron microscopy (SEM). Secondary electrons produced by inelastic scattering of the electron beam provide high resolution images of materials. Backscatter electrons, produced by elastic collisions, show the location of atoms with higher atomic numbers (which scatter these electrons more strongly and thus appear brighter in backscatter images). Characteristic X-rays, produced when outer shell electrons fill vacancies produced by collisions of the electron beam with inner shell electrons, provide elemental spectra (15).

The goal of this study is to determine whether environmental forensic microscopy can help characterize BFRs in dust and investigate mechanisms of transfer of these compounds from products to dust. To do so, we analyzed several samples of dust with extremely high levels of BDE 209, the main congener present in DecaBDE (>97%), a flame retardant primarily used as an additive in high impact polystyrene housing for electronic goods and furniture textile fabrics (1). BDE 209 is the main PBDE congener occurring in many dust samples (11,12). As BDE 209 has very low volatility (16), we hypothesized that abrasion is a more plausible transfer mechanism than volatilization.

Materials and Methods

Dust Samples

In this exploratory analysis we used two sources of indoor dust, targeting samples with very high concentrations of BDE 209 and low levels of other BFRs:

1) Dust was collected from Boston homes as described previously (2,11). Briefly, indoor dust samples (n=108) were collected from urban residences in the Boston (U.S.A.) area during two sampling rounds in the winter of 2005–2006 and the fall of 2006. Researchers collected dust

separately from the bedrooms and living rooms of each home using a Eureka Mighty-Mite vacuum cleaner (Model 3670) and crevice tool attachment fitted with a cellulose extraction thimble; the residents' vacuum cleaner bag was also obtained. Large debris was removed using metal sieves certified to ASTM-International standards to collect dust particles <500 μm in size. We analyzed samples for a suite of 38 PBDE congeners using a gas chromatograph (Agilent 6890N) coupled to a mass spectrometer (Agilent 5975) operated in electron capture negative ionization mode (GC/ECNI-MS) (17). We used a questionnaire to collect basic information about the homes including cleaning habits. We used a portable X-ray fluorescence device (Innov-X Systems) to measure bromine concentrations in foam-containing furniture, electrical devices, rugs and other consumer products (2). The concentrations of PentaBDE, OctaBDE and DecaBDE in dust were independently and log-normally distributed (11). The two highest samples, containing 270 and 540 μg PBDEs/g dust, were obtained from the vacuum cleaner bags of the same home, sampled eight months apart. Both consisted overwhelmingly of BDE 209, with concentrations of 260 and 530 $\mu\text{g/g}$ respectively. These extreme points had BDE 209 concentrations two orders of magnitude higher than the geometric mean of all dust samples, 2.5 $\mu\text{g/g}$. Compared to BDE 209, the concentrations of other BFRs identified in these samples were orders of magnitude lower: other PBDEs, hexabromocyclododecanes (HBCD), 1,2-bis(2,4,6-tribromophenoxy)ethane (BTBPE, TBE), decabromodiphenyl ethane (DBDPE), and the brominated components of Firemaster 550, 2-ethylhexyl 2,3,4,5-tetrabromobenzoate and (2-ethylhexyl) tetrabromophthalate (11,18). Thus, BDE 209 (83% bromine by weight) appears to be the major BFR in the two dust samples.

2) Dust was collected in 2006–2007 in various microenvironments—homes ($n=30$), offices ($n=18$) and cars ($n=20$)—in the West Midlands and Hampshire areas of the U.K. as described previously (19). Researchers collected dust using a Nilfisk Sprint Plus 1600 W vacuum cleaner using nylon sample socks (25 μm pore size) that were mounted in the furniture attachment tube of the vacuum cleaner. In cars, we vacuumed the surface of the seats with which occupants would have direct contact. Dust was sieved to <500 μm and analyzed for 13 PBDE congeners via GC/MS as described elsewhere (19,20). The concentrations of BDE 209 in these dust samples were log-normally distributed with a median of 8.1 $\mu\text{g/g}$. The highest concentration, 2600 $\mu\text{g/g}$ of BDE 209, was in dust from a 9 year old automobile; when re-sampled months later the concentration was 2200 $\mu\text{g/g}$. This sample contained much lower concentrations of other BFRs: other PBDEs (1.1 $\mu\text{g/g}$ Σ tri-hexa-BDEs), HBCD (2.1 $\mu\text{g/g}$ Σ HBCDs), DBDPE (0.15 $\mu\text{g/g}$), and BTBPE (0.029 $\mu\text{g/g}$) (19,21).

We report here on the microscopic analysis of the two Boston dust samples containing 260 and 530 $\mu\text{g/g}$ and the UK dust sample that contained 2600 $\mu\text{g/g}$ BDE 209.

Microscopic Analysis

The two Boston dust samples were examined by MVA Scientific Consultants in several ways: stereomicroscopy utilizing a Zeiss Stemi 2000 stereomicroscope (magnification 6.5X to 47X) and polarized light microscopy including microchemical tests utilizing an Olympus BH-2 polarized light microscope (40X to 1000X). The composition of the dust was analyzed as described previously (22,23). Representative portions of the samples were examined by SEM using a JEOL 6500F field emission scanning electron microscope coupled with a Noran X-ray energy dispersive spectrometry (EDS) system. One type of dust particle was analyzed by Fourier transform infrared microspectrophotometry (micro-FTIR) utilizing a SensIR FTIR equipped with a diamond ATR objective; the results were compared with relevant reference materials (14). As expected, preliminary analysis of a sample of commercial DecaBDE showed bright backscatter electron images with high bromine concentrations confirmed by EDS (data not shown).

The Boston dust samples were also examined at the Surface and Microanalysis Science Division of the National Institute of Standards and Technology (NIST). A multi-instrumental approach was favored due to the heterogeneous distribution of the bromine-containing dust particles. Aluminum stubs (2.54 cm) were covered with double-sided carbon tape and aliquots of well-mixed dust samples were spread on the carbon tape. Excess dust was removed by tapping the stub on side of the sample container. An Eagle III micro-x-ray fluorescence (micro-XRF) elemental analyzer (EDAX, Inc., Mahwah, NJ) was used to map the entire sample stub (40 kV, dwell time of 1 second per pixel), thereby pinpointing pixels ($100\text{ }\mu\text{m} \times 100\text{ }\mu\text{m}$) of measurable bromine content. Detection limits for bromine depend on details of particle morphology, size and dwell time, but are on the order of $200\text{ }\mu\text{g/g}$. Composite elemental maps were created by combining maps of individual elements using NIST Lispix (24). Next, sample stubs were placed in an environmental scanning electron microscope (Quanta 200F, FEI, Hillsboro, OR), operating at high vacuum, for both imaging and compositional analysis via X-ray EDS (EDAX, Inc.). EDS can be used to examine specific areas of particles and/or to create detailed elemental maps of particles. The beam energy used for imaging varied between 1 kV and 20 kV and for EDS was 30 kV. Similar results were found in samples surface coated with carbon and those without.

The Birmingham automobile dust sample was adhered to SEM stubs using double-sided carbon sticky tabs (Agar Scientific) prior to coating with evaporated carbon in an Emscope SC500 evaporation unit. Microscopic examination was conducted at high vacuum in an FEI XL-30 FEG environmental scanning electron microscope (ESEM). Chemical analysis for the determination of bromine content was carried out by energy dispersive X-ray microanalysis using an Oxford Instruments INCA instrument.

Results and Discussion

Boston house dust

Light microscopic analysis showed that the two Boston dust samples were similar, containing primarily plant fragments, soil minerals and construction debris (Table 1). The types of materials were similar to those found in other residential dusts (22,23) with the exception of some flakes of clear, elastic material found in both samples. Figure 1 presents a large-scale composite elemental map obtained via microscopic XRF analysis. It illustrates the typical and extremely uneven distribution of bromine in the dust samples relative to common elements detectable with this approach, iron and calcium. The micro-XRF analysis suggests that bromine is detectable in approximately 0.1% of the sampled pixels. However, the detection limit is generally higher for larger particles than for smaller ones ($<50\text{ }\mu\text{m}$) for a given bromine content. Consequently, smaller bromine-containing particles may not be detected using this technique unless operating conditions (e.g., dwell time) are adjusted. These results are most consistent with the abrasion hypothesis, but they do not rule out highly localized absorption of BDE 209 or non-detectable concentrations of bromine present on a wider distribution of particles.

Using scanning electron microscopy in the backscattered electron mode, followed by EDS, it was possible to find bromine-containing particles in both Boston samples (Figure 2–Figure 5 show representative micrographs). Figure 3–Figure 5 show that the composition of dust is also heterogeneous on the scale of individual particles: bromine typically occurs in micron-size patches, often in conjunction with patches containing calcium or other elements. More generally, bromine-rich particles were associated with a polymeric/organic matrix and were not observed free in the dust; they appear to be embedded in the organic material and associated with calcium. FTIR analysis showed that the flakes of clear, elastic material are consistent with a plastic, such as an acrylic (Figure 6). Calcium carbonate is a very common additive in plastics; clays such as kaolin are also used as fillers (25). Taken together, the evidence suggests that the

BDE 209 is primarily present in the dust samples as inclusions in plastic flakes, consistent with the abrasion hypothesis.

The two Boston dust samples were collected from the same home eight months apart. Both samples were obtained from the residents' home vacuum cleaner bag, which was changed between samples. Interestingly, the BDE 209 concentrations in the researcher-collected dust from the living room and bedroom of this home were one to two orders of magnitude lower. Questionnaire data indicated that the residents used their vacuum cleaner almost exclusively in one room, a carpeted, sparsely furnished, enclosed porch. XRF analysis of the two chairs and small table on this porch found non-detectable levels of bromine ($<5 \mu\text{g/g}$). The carpet contained moderate levels of bromine ($5,000 \mu\text{g/g}$) that seem insufficient to explain the high concentration of BDE 209 in the dust (260 and $530 \mu\text{g/g}$) given the apparent rarity of bromine-containing particles, about 0.1% of pixels (Figure 1): a material containing $5,000 \mu\text{g/g}$ of bromine present in only 0.1% of particles would give an overall bromine concentration in dust of only $5 \mu\text{g/g}$. While it is possible that the vacuum cleaner may be a source of DecaBDE, the external components were screened using XRF and the bromine levels were below the limit of detection. It is also possible that the use of vacuum cleaners may itself cause abrasion distinct from other indoor processes. Other methods for collecting dust (9) could be explored to rule out this possibility. Additional research on the source of the BDE 209 in these dust samples is continuing.

British dust sample from a car

In the British sample of car dust containing $2600 \mu\text{g/g}$ of BDE 209, analysis via backscattered electrons found a range of bromine-rich particles generally ranging in size from <1 to $20 \mu\text{m}$. Figure 7 shows a particle (on the order of $30 \mu\text{m}$ across) with unevenly distributed patches on the surface containing high levels of bromine as confirmed by EDS. We hypothesize that these particles arise as a result of abrasion of fabric treated with DecaBDE, given the large amount of fabric in the car, the relative ease with which fabric may be abraded, and the method of dust sampling (vacuuming of seats).

In each of the high BDE 209 samples studied, bromine was concentrated in widely scattered, highly contaminated particles. In the Boston samples, bromine was associated with a polymer/organic matrix. These data suggest that BDE 209 was transferred to dust via physical processes such as abrasion or weathering, rather than volatilization which should produce a more uniform distribution of bromine in association with the organic matter in dust. However, we only examined a small number of dust samples with high concentrations of BDE 209, a relatively non-volatile compound at room temperature. Caution is therefore needed in extrapolating these results to other dust samples or to other BFRs. It is possible, for example, that the more volatile PentaBDE may transfer to dust via both volatilization and physical degradation of foam. Additional studies of other dust samples are needed to better understand the sources and distribution of brominated flame retardants in dust.

The usefulness of environmental forensic microscopy in this exploratory investigation—a first attempt to use these techniques to investigate the origins of PBDEs in dust—indicates that these tools can provide novel insights into the distribution of BFRs in dust and their mechanisms of transfer from products. Nevertheless, our methods have certain limitations. For analysis of heterogeneous samples such as dust, micro-XRF and EDS are best thought of as showing relative abundances of elements, not precise quantitative measurements. Because we could only examine the distribution of elements, we examined dust samples with very large concentrations of one BFR. We are currently investigating microscopic methods for determining the chemical identity of the bromine-containing compounds in dust particles.

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Figure 1.

A large-scale, microscopic XRF image of Boston house dust showing the uneven distribution of bromine, about 0.1% of pixels. Red=bromine, blue=calcium, green=iron. The scanned area is 9.4 mm by 7.8 mm; the pixel size is 0.1 mm by 0.1 mm.

Figure 2.
A moderate-scale, composite image created using secondary electrons (shown in blue-green) and backscatter electronics (shown in red). Red areas may contain bromine; this can be confirmed at any given location using EDS.

Figure 3. Close-up, backscattered electron image of a dust particle. EDS shows that the two bright areas, marked 1 and 2, are bromine rich. Areas 3–5 are enriched in calcium or aluminum.

Figure 4.

A close-up composite image of a dust particle created using secondary electrons (shown in red) and backscatter electrons (shown in green). EDS showed that the green patches contain bromine, calcium and/or iron.

Figure 5.
a). Close-up secondary electron image of a bromine-containing dust particle. b) Elemental map constructed using EDS of the boxed area in 5a (white=carbon, blue=calcium, red=bromine).

Figure 6.
Comparison of infrared spectra from clear, elastic material in the dust sample (top) and a reference acrylic material (bottom)

Figure 7.

Environmental scanning electron microscopic images using backscatter electrons: a) a British car dust sample containing 2600 $\mu\text{g/g}$ BDE 209, b) close-up of a patch in the center of 7a (circled) that is enriched in bromine as confirmed by EDS. Shiny white areas indicate the presence of bromine.

Table 1

Composition of dust samples from the same Boston home, as ascertained by light microscopy (% values are volume/volume estimates). Both samples, collected from the same home eight months apart, had extremely high concentrations of BDE 209: 260 µg/g and 530 µg/g.

Constituent	Sample 1 (Winter)	Sample 2 (Fall)
Soil minerals ¹	10–20%	15–25%
Synthetic fibers	1–5%	1–5%
Plant fragments ²	40–60%	50–70%
Debris ³	20–40%	15–25%
Other ⁴	5–15%	1–5%

¹ Includes quartz, feldspar, mica and amphibole mineral grains

² Includes cellulose, decomposed plant material and wood fragments

³ Includes carbonate, gypsum and flakes of clear elastic material

⁴ Includes skin cells, salt, pollen grains, rubber, soot, paint