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**Characterizing the sorption of polybrominated diphenyl ethers (PBDEs) to cotton and polyester fabrics under controlled conditions.**

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## 1. Introduction

Textiles constitute the largest surface area of all materials indoors (Molander et al., 2012) and hence are expected to play an important role as a phase into which chemicals emitted indoors will partition. Textiles, or fibres, can be divided into the categories of natural (e.g., cotton, wool, linen, silk), synthetic (e.g., many polyesters, nylon, acrylic), and semi-synthetic (e.g., rayon). Natural fibres tend to be relatively polar and have reactive functional groups (Fig. S1). Cotton and linen, being derived from plants, consist of 88–96% cellulose (in raw fibres) with hydroxyl functional groups on the glucose monomer (Mather and Wardman, 2011). The cellulose polymer chains participate in hydrogen bonding that can confer a highly crystalline structure (Mather and Wardman, 2011). Animal-derived wool and silk consist of proteins with the amide group and polar side chain with -OH groups available for hydrogen bonding. In contrast, the most popular form of polyester consists of the terephthalic acid monomer that contains a benzene ring, carbonyl group and an aliphatic chain which together have relatively low polarity and fewer functional groups available for bonding than natural polymers (Mather and Wardman, 2011) (Fig. S1).

Fibres also differ in their physical morphology. Cellulose fibres, from the cotton seed, have an irregular and convoluted surface. In contrast, the artificial spinning of a polymer, including cellulose in the case of rayon, gives it a more uniform surface morphology (Mather and Wardman, 2011). Thus, natural fibres that have not been artificially spun, have a larger micro-surface area than synthetic fibres and natural fibres that have been artificially spun. The greater surface area of natural, non-artificially spun fibres may offer more binding sites for the sorption of organic compounds. Apart from surface morphology, fibres also differ in degree of polymerization and crystallinity of structure which can also affect the availability of binding sites.

Differences in the properties of fabrics and other indoor surfaces have been investigated for their propensity to sorb chemicals. For instance, Piadé et al. (1999), Noble (2000), Petrick et al. (2010) and Chien et al. (2011) reported greater affinity of natural fabrics, such as cotton and wool, to polar nicotine and chemicals in environmental tobacco smoke (ETS) in contrast to polyester. Similarly, Morrison et al. (2015b) reported the greatest sorption of methamphetamine

(a relatively polar compound) to cotton and a cotton-polyester blend upholstery fabric than polyester. They speculated that the highest sorption of methamphetamine to the upholstery fabric was due to the addition of sizing additives, many of which are water-soluble, and are intended to control the surface properties of textiles. They did not find a difference in sorption between cotton that was clean versus soiled (presumably with oily compounds). Won et al. (2000, 2001) reported the affinity of non-polar volatile organic compounds (VOCs) for less polar substrates such as synthetic fibre carpet and carpet/pad combination than polar gypsum board. Consistent with these observations, McQueen et al. (2008) commented that the hydrophobic and oleophilic nature of polyester readily attracted secreted body oils (i.e., compounds responsible for body odour) and provided a favorable environment for biotransformation and release of the resultant VOCs in comparison to cotton that sorbed and tended not to release these compounds.

Numerous studies have used chambers to determine the sorption of air-borne chemicals to materials. Examples of such studies include the sorption of VOCs to indoor materials such as carpet and gypsum board (Won et al., 2000, 2001), ETS to clothing (Noble, 2000; Chien et al., 2011), and nicotine to indoor surfaces including fabrics, glass and wood material (Piadé et al., 1999; Petrick et al., 2010). Conditions such as temperature, air exchange rate and relative humidity can be controlled and thus investigated in chamber experiments. Recently, Morrison et al. (2015a) reported fabric-air partition coefficients of phthalates (Diethyl phthalate, DEP and Di-n-butyl phthalate, DnBP) obtained by exposing the fabrics to phthalate-equilibrated air in a closed chamber experiment over 10 days. Rauert et al. (2014, 2015) designed a chamber to test the mechanisms responsible for transferring to dust the flame retardants (FRs) polybrominated diphenyl ethers (PBDEs) from an electronic casing and hexabromocyclododecane (HBCDD) from an impregnated curtain. In both studies, they found that abrasion of FR-enriched particles and fibres from the casing and curtain was likely a more significant pathway with subsequent deposition to dust.

The goal of this study was to investigate the sorption of gas-phase PBDEs to cotton and polyester fabrics. PBDEs, available as three commercial mixtures differing in bromination, were used as additive flame retardants in a wide variety of products, including textiles such as curtains and upholstery fabric (e.g., Abbasi et al., 2015). Although new production of commercial penta- and octa-BDE mixtures came under national and international control starting in 2003 and deca-

BDE has also been slated for control (UNEP, 2010, 2013; Environment Canada, 2013), PBDEs remain amongst one of the most prevalent classes of flame retardant found indoors (Bradman et al., 2014; Abbasi et al., 2016 *inter alia*). Based on the body of evidence presented above, we hypothesized that polyester has a higher affinity for non-polar semi-volatile organic compounds (SVOCs) compared to polar cotton. We designed a chamber study to test this hypothesis using PBDEs as test chemicals. Cotton and polyester that differed in physical and chemical characteristics were tested for PBDE sorption as a function of exposure duration, air flow and temperature. The study used the chamber designed by Rauert et al. (2014). Solid-state  $^{13}\text{C}$  nuclear magnetic resonance (NMR) was also performed to characterize aliphatic versus aromatic structural moieties in the test fabrics. These structural differences between cotton and polyester could provide insight into the sorptive behaviour of non-polar PBDEs.

## **2. Experimental method**

### *2.1. Test Material*

Cotton and polyester fabrics (purchased from a local store) were pre-cleaned by pressurized liquid extraction using Dionex ASE 350 (Thermo Scientific, USA) with hexane (HPLC grade, Fisher scientific). Pre-cleaned fabrics were wrapped in cleaned aluminium foil and stored at  $-4^{\circ}\text{C}$  until use. For each experiment, fabrics were cut into  $5\times 5\text{ cm}^2$  squares and weighed before placement in the test chambers.

### *2.2. Test chambers*

A detailed explanation of the test chambers is given by Rauert et al. (2015). Briefly, stainless steel cylindrical chambers of 10 cm diameter and 20 cm height were used at the University of Birmingham, UK. The total volume of the chamber was  $1570\text{ cm}^3$ . The lid of the chamber allowed for the inflow and outflow of air using a low volume pump. Two types of experiments were conducted: without (closed) and with (open) airflow. For the air flow experiments, a constant air flow of 10 L/min through the chamber (air exchange rate of 6.4 exchanges per minute) was achieved using a Capex L2 Diaphragm Pump (Charles Austin Pumps Ltd, Surrey, UK). Inflowing air was purified by a polyurethane foam (PUF) disk (140 mm diameter, 12 mm thickness,  $360.6\text{ cm}^2$  surface area, PACS, Leicester, UK) held in a glass assembly with

attachment to the inlet using polypropylene tubing. A similar assembly of two PUF disks was attached to the outlet to collect PBDEs in outflow air. Only the 'chamber-side' outlet PUF was treated as a sample as the air-side PUF did not show any breakthrough loss during experimental design development (Rauert et al., 2015). The length of polypropylene tubing attached to the outlet was kept at 2 cm to minimize the loss of PBDEs due to sorption to the tubing surface (Rauert et al., 2015). Two platforms inside the chamber were made of wire mesh placed on stainless steel O-rings attached within the chambers. The entire chamber assembly was rinsed once each with hexane, dichloromethane (DCM) and methanol (HPLC grade, Fisher scientific) before use. The chambers were heated to the desired temperature by placing them in a hot water bath. A filter paper (47 mm PTFE membrane filter, 1.0  $\mu\text{m}$  pore size, Whatman, UK) spiked with a known amount of native PBDE standards was placed on the upper platform in the chamber to act as an emission source (Fig. S2). One  $5\times 5\text{ cm}^2$  square of pre-cleaned cotton and a similarly dimensioned square of pre-cleaned polyester fabric were placed side-by-side at the bottom of the chamber (Fig. S2).

After completion of each experiment, PUF disks at the outlet, spiked filter paper, and each of the fabrics squares were collected and stored at  $-18^\circ\text{C}$  for further laboratory analysis. Internal walls of the chamber were rinsed three times with hexane and DCM (1:1, v/v) to collect analytes absorbed onto the walls. The solvent rinse was collected in a glass bottle for further analysis.

Chamber experiments were run without air flow for 24 hours at  $40^\circ\text{C}$  and  $60^\circ\text{C}$ , whereas chamber experiments with air flow were conducted for one week at room temperature ( $\sim 25^\circ\text{C}$ ), and for 72 hours at  $40^\circ\text{C}$  and  $60^\circ\text{C}$ . Each chamber experiment without air flow was repeated 4 times, whereas 2 replicates were conducted for the experiments with air flow.

### 2.3. *Extraction and analysis*

Full details of extraction and analytical procedures are given in the supplementary information (SI). Briefly, each sample was extracted using ASE. The crude extract was then reduced under gentle stream of nitrogen to 0.5 mL in a Zymark Turbovap (TurboVap II concentration workstation, Caliper Life Science, Massachusetts, USA) followed by clean-up by loading onto SPE cartridges filled with 2 g of pre-cleaned alumina and 5 g of pre-cleaned sodium sulphate (SPE cartridge were self-packed in the laboratory). The analytes were eluted with 30

mL of hexane: DCM (1:1, v/v). The eluate was then reduced to incipient dryness in the Zymark Turbovap and the dried extract was reconstituted to 100  $\mu$ L using  $^{13}\text{C}$ -BDE-100 (Wellington laboratories, Guelph, Canada) in methanol as internal standards. The final solution was analysed for PBDEs on LC-MS/MS using the method described by Rauert et al. (2015) and briefly given in SI.

#### 2.4. *QA/QC*

Samples were analysed according to established QA/QC methods. Laboratory blanks were extracted and analysed with samples from each set of experiment. Samples were spiked with mass-labeled surrogate standards  $^{13}\text{C}$ -BDE-47, -99, -153 and -153 to determine recoveries. A set of 5 calibration standards with concentrations ranging from 20 to 900 ng/mL were also run before and after each batch of samples to monitor the sensitivity of the instrument. Average recoveries of surrogate standards ranged between 77-81%. Blank correction was done using the criteria explained by Saini et al. (2015). Ninety % of samples had blanks <5% of the sample concentration and thus did not require correction. Statistical analyses were performed using Microsoft Office Excel 2007 and STATISTICA software version 8 (StatSoft Inc., Oklahoma, US), respectively. Non-parametric Mann-Whitney U test (MWU) was performed and used a significance level of 5%.

#### 2.5. *Nuclear Magnetic Resonance (NMR) analysis*

Solid-state  $^{13}\text{C}$  NMR analysis was performed using a Bruker BioSpin Avance III 500MHz NMR spectrometer fitted with a H-X solid-state NMR probe. Prior to NMR analysis, fabrics were finely ground into a powder using a Wig-L-Bug mechanical grinder. Powdered fabric samples were packed into a 4mm Zirconium rotor and sealed with a Kel-F cap. Ramped amplitude cross polarization magic angle spinning (CP-MAS) NMR spectra were acquired with the following parameters: CP contact time (1ms), MAS spinning speed of 11kHz, and a recycle delay of 1s. NMR spectra were processed using a zero filling factor of 2 and line broadening of 50Hz. NMR spectra were phased and baseline corrected using Advanced Chemistry Development (version 15) software.

## 2.6. *Scanning Electron microscopic (SEM) images*

Scanning electron microscope images of cotton and polyester fabrics were taken at the University of Toronto using a tungsten filament JEOL JSM6610LV microscope operated in secondary electron imaging mode (JOEL USA, Peabody, MA). Fabric pieces and single fibre strands were fixed to aluminum stubs using double side carbon (conductive) tape and were coated with 30 nm thick gold layer using a gold sputter coater (Polaron Range, SC7620, Thermo VG Microtech UK). Images were captured with 30× and 2,000× magnification at a working distance of 22 mm using an electron beam high voltage of 15 kV. The fabric weave and surface structure of single strands of cotton and polyester were captured in images to see the differences in surface morphology and area.

## 2.7. *Density and thickness measurements*

Density measurements (mass per unit area) of the fabrics were made using the standard method CAN/CGSB-4.2 No. 5.1-M90 Unit Mass of Fabrics (CGSB, 2004). The fabrics were conditioned for minimum of 24 hours at 20°C±1°C and 65%±2% RH (ISO 139: 2005 Textiles - Standard atmospheres for conditioning and testing). The fabrics were cut into circular pieces of 5 cm diameter of area 19.635 cm<sup>2</sup>. Fabric thickness was measured following CAN/CGSB-4.2 No. 37-2002 Fabric Thickness Method (CGSB, 2002) using 28.66 mm diameter presser foot under an air pressure of 1.0 kPa. Ten different pieces of each fabric were used as replicates for each measurement and their averages are reported here.

## 2.8. *Specific surface area (SSA) measurements*

SSA of cotton and polyester fabrics were measured using Brenauer-Emmett-Teller (BET) adsorption method (Rouquerol et al., 2014). Briefly, fabrics were cut into fine pieces and samples were kept under vacuum at 60°C for 16 hours to outgas any pre-sorbed chemicals. Adsorption isotherms were obtained using an Autosorb-iQ gas sorption analyzer (Quantachrome, Boynton Beach, FL, USA). Adsorbate gas, Krypton (Kr, 99.999%), and purge gas, Helium (He, 99.999%), were purchased from MEGS. Kr sorption isotherms were obtained at 77 K using a liquid nitrogen (N) bath. Kr was used instead of N to obtain adsorption isotherms as the fabrics' SSA were < 5 m<sup>2</sup>/g.

### 3. Results

#### 3.1. NMR spectra

The solid-state  $^{13}\text{C}$  NMR spectra for polyester and cotton fabrics are shown in Fig. 1. The NMR spectrum for polyester showed resonances for reported components within polyester (Colletti and Mathias, 1988; Gan et al., 2004). Resonances at 36-44 ppm (labelled as 'a') and 62-72 ppm (labelled as 'b') are consistent with mid-chain  $\text{CH}_2$  and  $\text{CH}_2$  next to carboxylic groups respectively. The aromatic (labelled as 'c') and carboxylic (labelled as 'd') carbon were also observed in the spectrum for polyester. Overall, the solid-state NMR spectrum revealed the aromatic-rich nature of the polyester fabric used in this study, which is consistent with a terephthalate monomer. The NMR spectrum of the cotton reflected its cellulose-rich nature (Horii et al., 1987; Castelvetro et al., 2007). Hexose ring carbons were visible at 62–70 ppm and 72–90 ppm (labelled as 'e' and 'f') and the anomeric carbon was observed at 104–110 ppm. Sorbent characteristics such as polarity and aromaticity have been correlated to different sorption behaviour (Gustafsson et al., 1997; Bucheli and Gustafsson, 2000; Accardi-Dey and Gschwend, 2002; Salloum et al., 2002; Chen et al., 2005). It is inferred based on these spectra that polyester would sorb more PBDEs under equilibrium conditions due to the high aromaticity compared to cotton, if the physical characteristics such as densities of fabrics are similar.

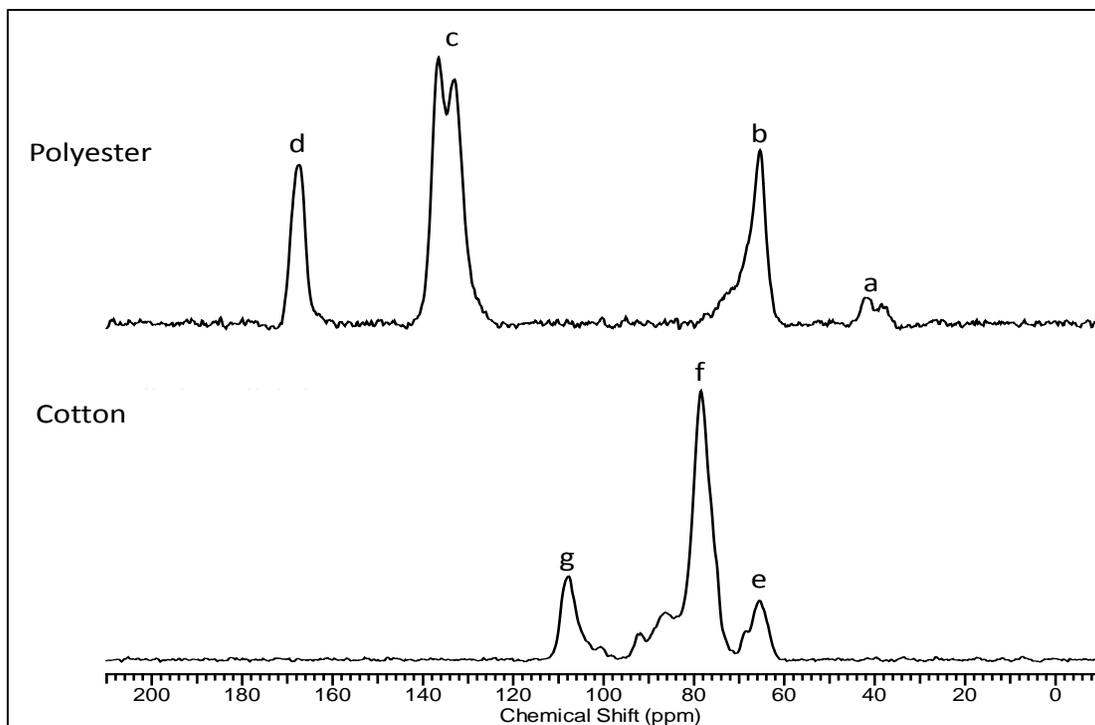


Fig. 1. Solid-state <sup>13</sup>C NMR spectra of polyester and cotton. Chemical shift assignments correspond to: a) mid-chain CH<sub>2</sub> groups, b) CH<sub>2</sub> groups adjacent to COOH groups, c) aromatic carbon, d) carboxylic carbon, e) hexose ring carbons in cellulose, f) hexose ring carbons in cellulose closer to O, and g) anomeric carbon in cellulose.

### 3.2. SEM images, density and specific surface area

Cotton and polyester fabric samples differed in weaving pattern and surface morphology under 30× and 2000× magnification (Fig. 2). Cotton had a dense weave whereas polyester was less dense. Single strands of cotton under 2000× magnification showed a convoluted structure with grooves and folds on its surface consistent with its natural origin (Mather and Wardman, 2011). In comparison, polyester had a smooth surface consistent with its synthetic origin and spinning (Mather and Wardman, 2011). The average areal densities of cotton and polyester samples measured at 20°C±1°C and 65%±2% RH were 164±1.3 and 45±0.6 g/m<sup>2</sup>, respectively. Average thickness of the samples of cotton and polyester were 0.05±0.002 and 0.02 cm, respectively. Thus, volumetric densities were 310,510±12720 and 253,390±3460 g/m<sup>3</sup> for cotton and polyester, respectively. BET-SSAs of cotton and polyester were 0.72 (0.07 < P/P<sub>0</sub> < 0.18, C = 7.2) and 0.07 (0.13 < P/P<sub>0</sub> < 0.21, C = 2.0) m<sup>2</sup>/g, respectively. Ten times higher BET-SSA of cotton than polyester is consistent with differences seen in the SEM images (Fig. 2).

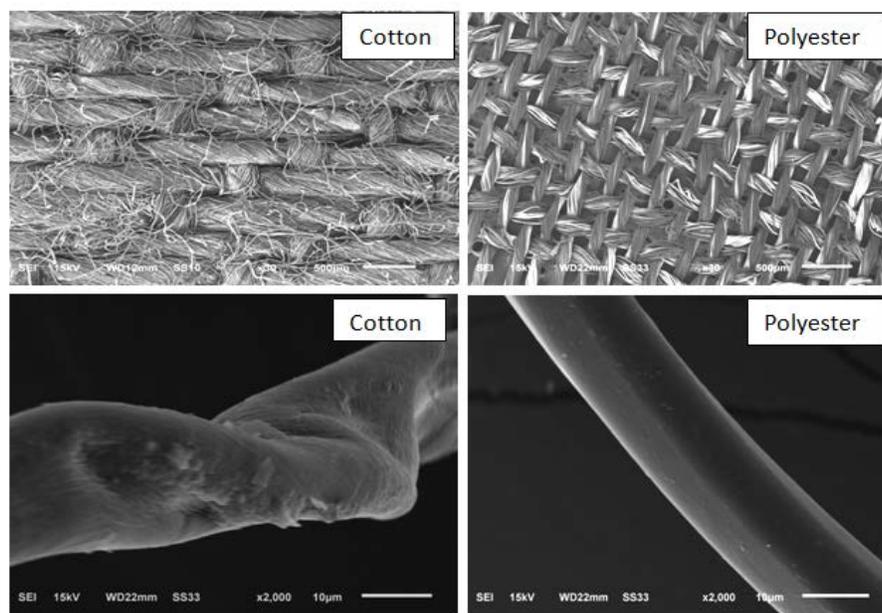


Fig. 2. SEM images of cotton and polyester fabrics (top) under 30× magnification and single strand structure (bottom) under 2000× magnification.

### 3.3. Recoveries of PBDEs from chambers

Total recoveries of  $\Sigma_7$ PBDEs from all chamber compartments (e.g., filter paper, fabrics, chamber wall rinses and outflow PUF) ranged between 64–91% and 60–100% for chambers without and with air flow, respectively (Fig. S3). Recoveries tended to be lowest at 60°C, particularly with air flow (although not statistically significant), which is consistent with greater chemical loss from the filter paper and significantly higher outflow air concentrations ( $\Sigma_7$ PBDEs: 174 ng/PUF) compared to that at room temperature and 40°C ( $\Sigma_7$ PBDEs: 38–40 ng/PUF) (MWU,  $p < 0.05$ ; Fig. S5). Less than 100% recoveries were found for most analytes which could be due to (i) analytical uncertainties, (ii) sink effects of the chamber that were not captured by the chamber wall rinses, and (iii) unaccounted loss of analytes to air while opening the chamber for sample collection.

*Chambers without air flow:* The mass of spiked PBDEs that remained on the filter paper (FP) was statistically indistinguishable at 40°C and 60°C. Total remaining mass on the filter paper ranged between 173–179 ng/FP and increased with decreasing congener vapour pressure from 4–20% remaining of BDE-47,-85, -99 and -100 compared to 38% of BDE-153, 25% of -154, and 71-74% of -183 (Fig. S4). The percentage distribution and total mass sorbed to chamber walls

was also statistically indistinguishable at 40°C ( $\Sigma_7$ PBDEs 243 ng) and 60°C ( $\Sigma_7$ PBDEs 325 ng). Similarly, mass sorbed to both fabrics was statistically indistinguishable at 40° versus 60°C with  $\Sigma_7$ PBDEs ranging between 25–33 ng/fabric square.

*Chambers with air flow:* Total mass remaining on the filter paper at 60°C ( $\Sigma_7$ PBDEs 72 ng/FP) was significantly less (MWU,  $p < 0.05$ ) than that at 40°C ( $\Sigma_7$ PBDEs 332 and 335 ng/FP, respectively), indicating more volatilization at 60°C (Fig. S5). In terms of percentages, 4–84% remained on the filter paper at room temperature and 40°C compared to 1–48% at 60°C. Mass sorbed to chamber walls was statistically indistinguishable at room temperature, 40°C and 60°C with  $\Sigma_7$ PBDEs ranging between 227–272 ng. Mass sorbed to cotton was statistically indistinguishable at room temperature, 40°C and 60°C with  $\Sigma_7$ PBDEs ranging between 15–19 ng/fabric square, whereas mass sorbed to polyester at 40°C ( $\Sigma_7$ PBDEs 9 ng/square) was statistically less than at room temperature and 60°C ( $\Sigma_7$ PBDEs 15 and 20 ng/square, respectively), recalling that the experiment at room temperature was of 1 week duration versus 72 hours for the 40°C and 60°C experiments (MWU,  $p < 0.05$ ).

On an area basis, the chamber walls sorbed more PBDEs than fabric due to the large internal surface area of 785 cm<sup>2</sup> compared to 25 cm<sup>2</sup> fabric squares (Fig. S6). However, fabrics had comparable or up to 5 times greater sorption than the chamber walls when normalized per planar surface area (Fig. S6). The wall sink effect has been discussed in numerous chamber studies as an unavoidable feature of such studies (e.g., Uhde and Salthammer, 2006; Katsumata et al., 2008; Rauert et al., 2014, 2015).

#### 3.4. Sorption of PBDEs to cotton and polyester

*Chambers without air flow:* At 40°C and 60°C after 24 hours, cotton and polyester had statistically similar concentrations of  $\Sigma_7$ PBDEs ranging between 1–1.3 ng/cm<sup>2</sup> (planar surface area) with maximum sorption of BDE-47 ranging between 0.5–0.6 ng/cm<sup>2</sup> (MWU,  $p > 0.05$ ) (Fig. 3). However at 40°C and 60°C,  $\Sigma_7$ PBDEs were 0.35 and 0.36 ng/cm<sup>2</sup>-BET sorbed to polyester compared 0.007 and 0.009 ng/cm<sup>2</sup>-BET sorbed to cotton, respectively (MWU,  $p < 0.01$ ) (Fig. 4). The difference between cotton and polyester of 40–50 times of greater sorption normalized to

BET-SSA versus planar surface area is far greater than the 10 times higher BET-SSA of cotton than polyester.

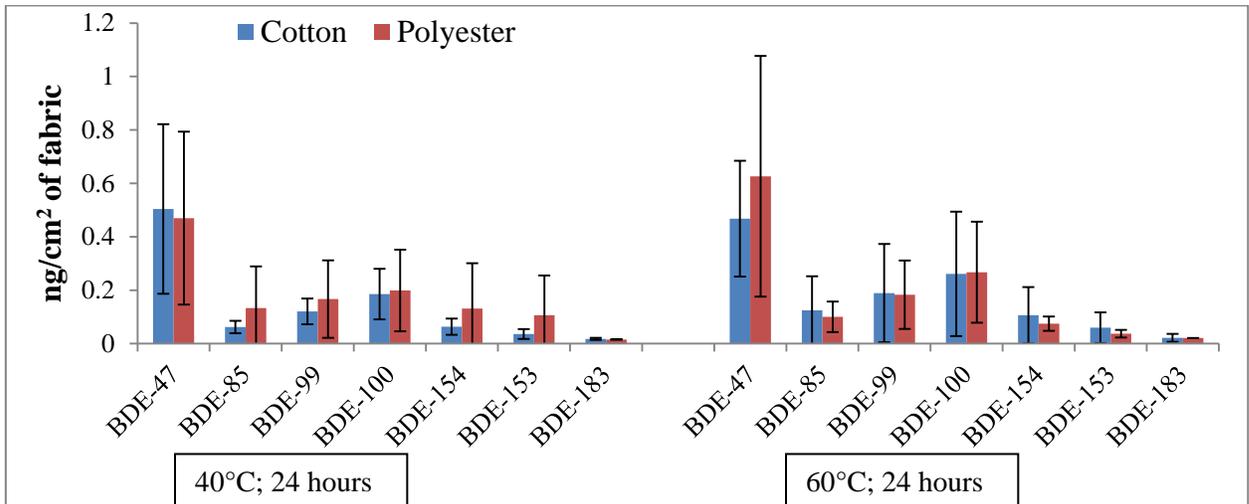


Fig. 3. PBDEs sorbed to cotton and polyester expressed per  $\text{cm}^2$  planar surface area in chambers without air flow at  $40^\circ\text{C}$  and  $60^\circ\text{C}$  after 24 hours (error bars show maximum and minimum concentration).

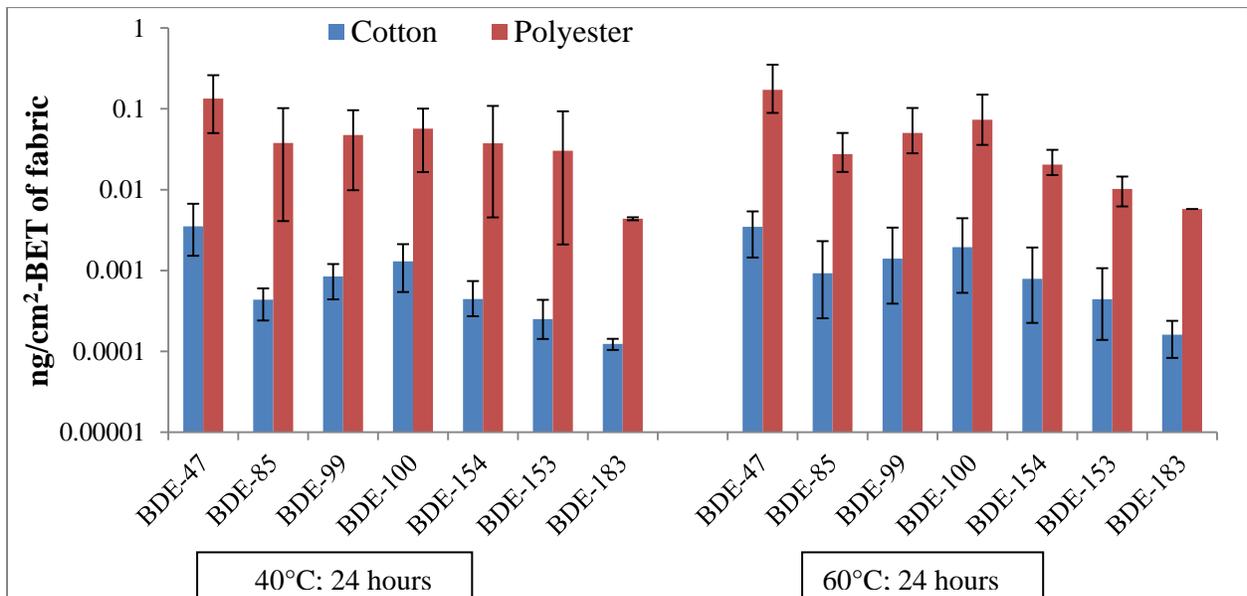


Fig. 4. PBDEs sorbed to cotton and polyester expressed per  $\text{cm}^2$ -BET in chambers without air flow at  $40^\circ\text{C}$  and  $60^\circ\text{C}$  after 24 hours (error bars show maximum and minimum concentration). Note: Y-axis is a log scale.

With 3.6 times lower density of polyester than cotton, polyester had 3–10 times higher concentrations of PBDEs when expressed per unit mass of fabric, i.e.,  $\Sigma_7$ PBDEs concentrations of 247 and 255 ng/g for polyester compared to 50 and 66 ng/g for cotton at 40°C and 60°C, respectively (MWU,  $p < 0.05$ ; Fig. 5). Thus, at both temperatures we observed no differences among PBDE concentrations between polyester and cotton when normalized to per unit planar surface area, but 40–50 times greater sorption when normalized to BET-SSA, and 3–10 times for when normalized to mass (MWU,  $p < 0.05$ ). The higher sorption of PBDEs to polyester than cotton when considering BET-SSA and fabric mass suggest either a higher affinity of PBDEs for polyester than cotton, presumably due to both being non-polar and the high aromaticity of polyester, or that PBDEs were ‘diluted’ by the large surface area of cotton relative to polyester. We note that the difference in sorption per unit BET-SSA (40-50 times) was greater than the surface area difference (10 times) between the fabrics, which is the basis of the dilution hypothesis.

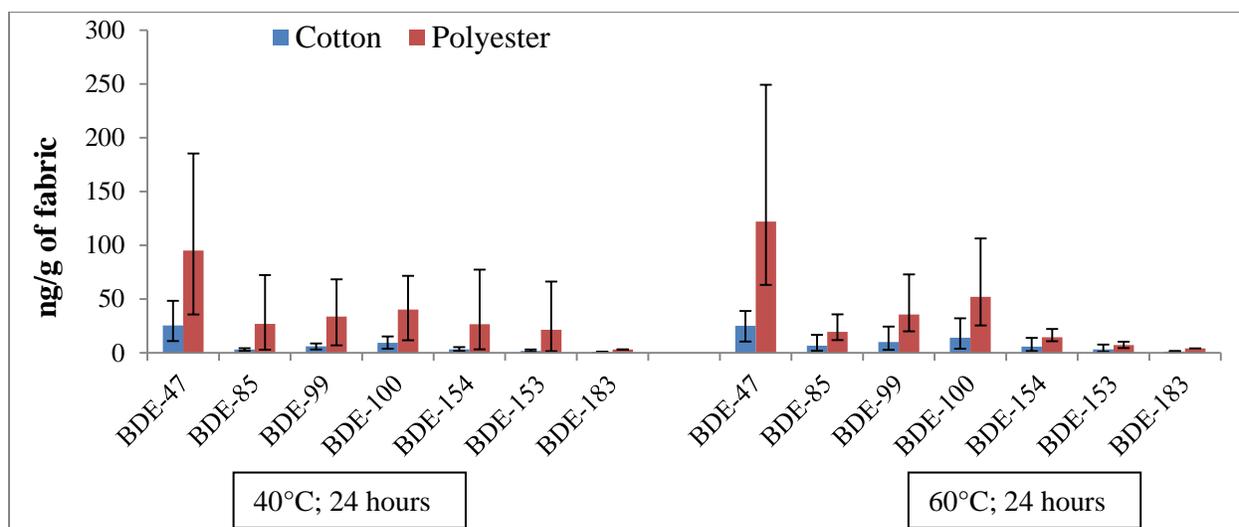


Fig. 5. PBDEs sorbed to cotton and polyester expressed per gram of fabric in chambers without air flow at 40°C and 60°C after 24 hours (error bars show maximum and minimum concentration).

*Chambers with air flow:* Similarly to experiments without air flow, cotton and polyester had statistically similar PBDE mass sorbed per unit planar surface area at room temperature, 40°C and 60°C (MWU,  $p > 0.05$ ) (Fig. 6).  $\Sigma_7$ PBDEs concentrations ranged from 0.6–0.8 and 0.4–0.8 ng/cm<sup>2</sup> for cotton and polyester, respectively. Again, significantly more PBDEs were sorbed per

unit BET-SSA of polyester ( $\Sigma_7$ PBDEs 0.1–2.4 ng/cm<sup>2</sup>-BET) than cotton ( $\Sigma_7$ PBDEs ~0.005 ng/cm<sup>2</sup>-BET) with the difference of 20–50 times being comparable to that under closed conditions (MWU,  $p < 0.01$ ) and again, much greater than the 10-fold difference in BET-SSA (Fig. 7). Similarly, PBDEs sorbed per unit mass of polyester ( $\Sigma_7$ PBDEs 70–170 ng/g) was significantly greater than cotton ( $\Sigma_7$ PBDEs 36–40 ng/g; MWU,  $p < 0.05$ ) (Fig. 8). In summary, PBDE concentrations did not differ between polyester and cotton when normalized to planar surface area, but polyester concentrations were 20–50 times greater when normalized to BET-SSA, and 2–4 times greater when normalized to mass (MWU  $p < 0.05$ ).

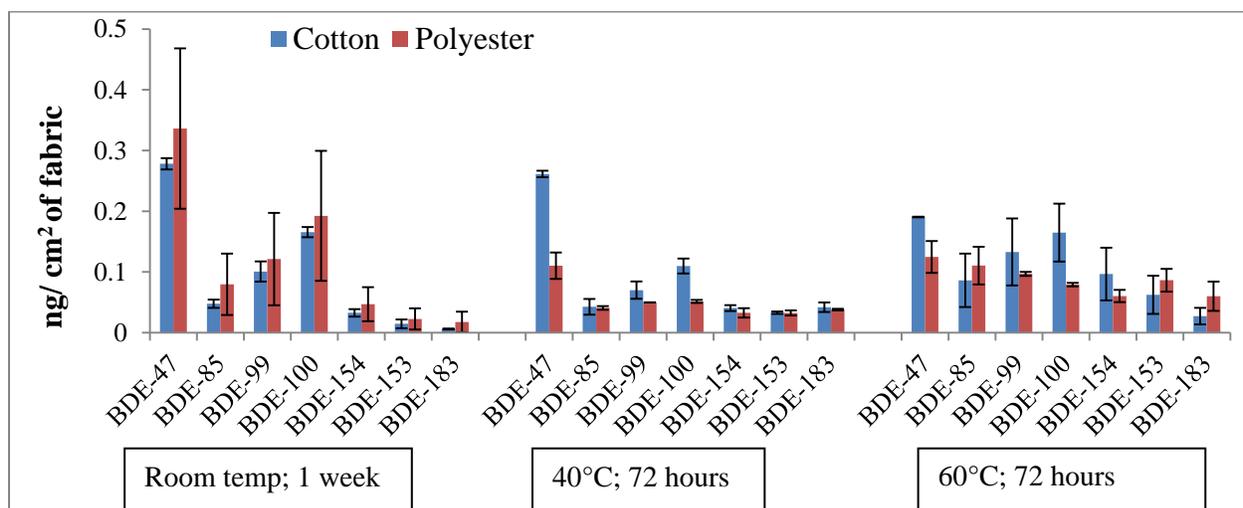


Fig. 6. PBDEs sorbed to cotton and polyester per cm<sup>2</sup> planar surface area of fabric in experiments with air flow (error bars show maximum and minimum concentration).

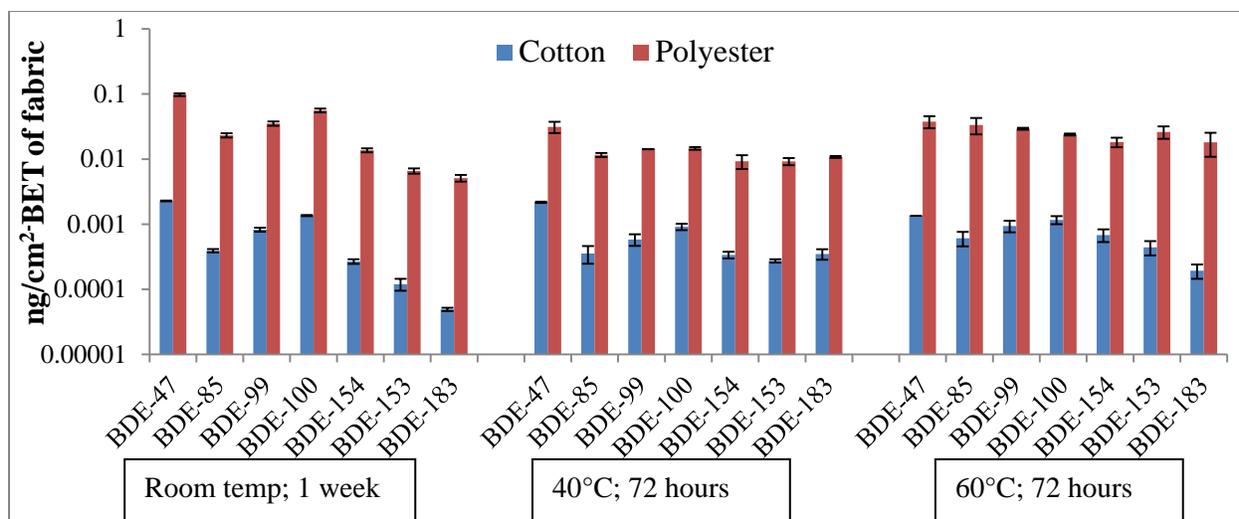


Fig. 7. PBDEs sorbed to cotton and polyester expressed per cm<sup>2</sup>-BET of fabric in chambers with air flow (error bars show maximum and minimum concentration). Note: Y-axis is a log scale.

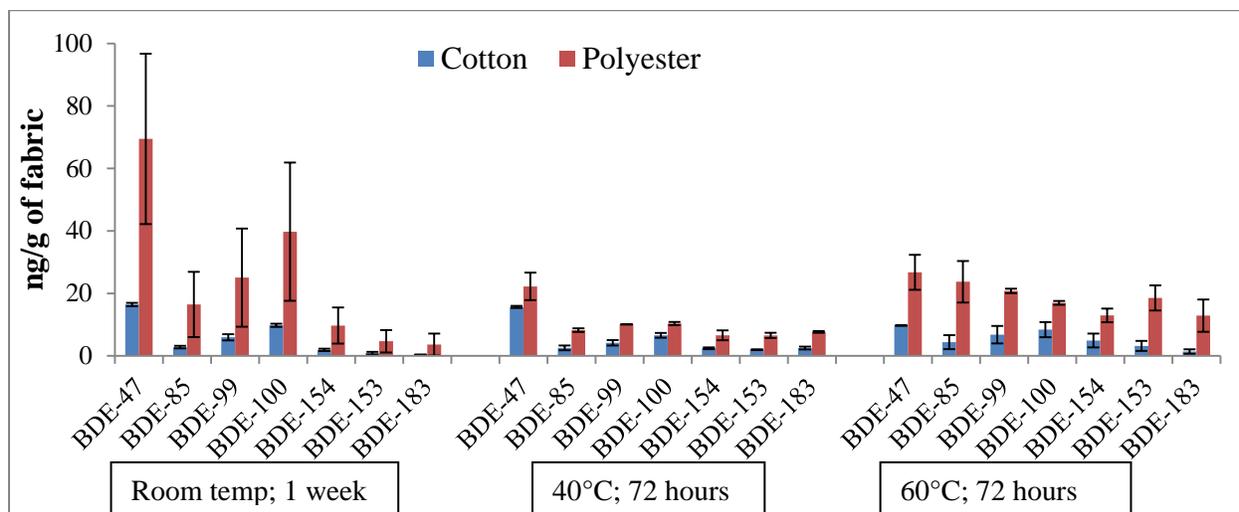


Fig. 8. PBDEs sorbed to cotton and polyester per gram of fabric in experiments with air flow (error bars show maximum and minimum concentration).

### 3.5. Distribution coefficient, $K'_D$ ( $C_{fabric}$ (or steel)/ $C_{chamber air}$ )

In the experiments with air flow, chamber air concentrations were calculated from the PBDE mass sorbed to PUF at the chamber exit and air flow rate (10 L/min) (Table 3.1). Distribution coefficients,  $K'_D$  (area normalized, unit of m) were calculated as ratios of mass sorbed to fabrics or steel of the chamber walls (pg/m<sup>2</sup>, planar surface area of fabrics) and corresponding air concentrations (pg/m<sup>3</sup>). Since time to reach equilibrium for fabrics was estimated as >10 years

based on cotton-air equilibrium partition coefficients estimated with COSMO-RS solvation theory under typical indoor conditions (Saini et al., unpublished results), it is highly unlikely that PBDEs had attained equilibrium in the chambers with exposure times of 72 hours to one week. Thus, we use the term distribution coefficient rather than partition coefficient to denote that the system was not at equilibrium.

Table 3.1. Average measured chamber air concentrations and planar area-normalized distribution coefficients ( $\text{pg}/\text{m}^2$  fabric or chamber to  $\text{pg}/\text{m}^3$  air concentration;  $K'_{\text{cotton-air}}$ ,  $K'_{\text{polyester-air}}$ , and  $K'_{\text{steel-air}}$  m) at room temperature (one week), and 40°C and 60°C (72 hours).

	BDE-47	BDE-85	BDE-99	BDE-100	BDE-153	BDE-154	BDE-183
	Chamber air concentrations ( $\text{pg}/\text{m}^3$ )						
Room temp. (~25°C)	210	16	59	84	9.9	19	2.7
40°C	422	57	126	199	24	51	12
60°C	1107	438	699	896	297	546	57
	$K'_{\text{cotton-air}}$ (m)						
Room temp. (~25°C)	$1.3 \times 10^4$	$2.9 \times 10^4$	$1.7 \times 10^4$	$2.0 \times 10^4$	$1.5 \times 10^4$	$1.7 \times 10^4$	$2.2 \times 10^4$
40°C	$6.2 \times 10^3$	$7.5 \times 10^3$	$5.6 \times 10^3$	$5.5 \times 10^3$	$1.4 \times 10^4$	$8.0 \times 10^3$	$3.4 \times 10^4$
60°C	$1.7 \times 10^3$	$2.0 \times 10^3$	$1.9 \times 10^3$	$1.8 \times 10^3$	$2.1 \times 10^3$	$1.8 \times 10^3$	$4.7 \times 10^3$
	$K'_{\text{polyester-air}}$ (m)						
Room temp. (~25°C)	$1.6 \times 10^4$	$4.8 \times 10^4$	$2.1 \times 10^4$	$2.3 \times 10^4$	$2.3 \times 10^4$	$2.5 \times 10^4$	$6.4 \times 10^4$
40°C	$2.6 \times 10^3$	$7.2 \times 10^3$	$4.0 \times 10^3$	$2.6 \times 10^3$	$1.3 \times 10^4$	$6.5 \times 10^3$	$3.1 \times 10^4$
60°C	$1.1 \times 10^3$	$2.5 \times 10^3$	$1.4 \times 10^3$	$8.8 \times 10^2$	$2.9 \times 10^3$	$1.1 \times 10^3$	$1.0 \times 10^4$
	$K'_{\text{steel}}$ (m)						
Room temp. (~25°C)	$2.5 \times 10^3$	$2.5 \times 10^4$	$9.8 \times 10^3$	$8.7 \times 10^3$	$2.2 \times 10^4$	$1.8 \times 10^4$	$2.8 \times 10^4$
40°C	$1.3 \times 10^3$	$8.9 \times 10^3$	$5.3 \times 10^3$	$3.7 \times 10^3$	$1.5 \times 10^4$	$1.0 \times 10^4$	$8.1 \times 10^3$
60°C	$3.0 \times 10^2$	$1.3 \times 10^3$	$8.8 \times 10^2$	$6.2 \times 10^2$	$1.7 \times 10^3$	$1.1 \times 10^3$	$4.7 \times 10^3$

PBDE air concentrations were 5–30 times higher at 60°C than at room temperature, consistent with a tendency to partition more into gas phase than the condensed phases at higher temperatures. Similar findings were reported by Clausen et al. (2012), with up to 211-fold increase in gas-phase concentration of di-(2-ethylhexyl) phthalate (DEHP) emitted from a vinyl flooring test piece kept in a chamber with a 38°C increase in chamber temperature at steady state. PBDE concentrations in chambers at room temperature were also in the range of those reported in literature for indoor air, which emphasize the real world relevance of these results (Abdallah and Harrad, 2010; Zhang et al., 2011; Saini et al., 2015 *inter alia*). As expected, BDE-47, -99 and -100 had consistently higher air concentrations than other PBDEs at every

temperature. This explains the inverse relationship between PBDE sorbed to cotton, polyester and steel versus octanol-air partition coefficient,  $K_{OA}$ , and the positive relationship with vapour pressure (Fig. S7).

$K'_D$  values normalized to planar surface area for cotton, polyester and steel were comparable for each congener (Table 3.1).  $K'_D$  values for polyester were 14–104 times higher than those for cotton (MWU,  $p < 0.01$ ), when normalized to BET-SSA (BET-SSA was not available for steel, Table S3).

Log  $K'_D$  values for all materials increased with log  $K_{OA}$  and decreased with vapour pressure (Fig. S8).  $K'_{\text{steel-air}}$  showed the strongest relationship, whereas  $K'_{\text{cotton-air}}$  showed the weakest relationship that was not significant ( $p > 0.05$ ). We attribute the latter to less uniformity in physical structure amongst samples of cotton than polyester, which in turn is less uniform than steel chamber walls. As expected,  $K'_D$  values for fabric was consistently greatest at the lowest temperature and decreased with increasing temperature.

#### 4. Discussion

These results confirm that cotton and polyester fabrics sorb gas-phase PBDEs from surrounding air, with an area normalized distribution coefficients,  $K'_D$ , of  $\sim 10^3$  to  $10^4$  m after one week at room temperature. These  $K'_D$  values imply that 1 m<sup>2</sup> of these fabrics would sorb PBDEs present in  $10^3$  to  $10^4$  m<sup>3</sup> of equivalent air volume under the given conditions. Similar  $K'_D$  for cotton and polyester fabrics indicates air side controlled uptake of PBDEs under kinetic or non-equilibrium conditions. Kinetic phase of uptake is relevant for “real life” scenarios where it is highly unlikely that equilibrium will be reached between fabrics and air, given the expected high sorptive capacity of fabrics.

On a planar surface area basis, cotton and polyester had statistically similar sorption of PBDEs. However, polyester showed 3–10 times greater sorption when expressed per gram of fabric relative to planar surface area and 20–50 times greater than cotton when the BET-SSA was considered, which could have two explanations. First, the large BET-SSA of cotton could have ‘diluted’ sorbed PBDEs. If this is the case, then further testing is necessary to determine if cotton ultimately achieves similar sorption as polyester, given sufficient time for the chemical to

penetrate the interstices of cotton. Alternatively or in addition, polyester could have sorbed more than cotton because of chemical compatibility between the non-polar PBDEs and the polyester sorbent (e.g., Won et al., 2000, 2001). This explanation is consistent with the sorption difference being greater than the difference in BET-SSA between the two fabrics. The importance of considering BET-SSA is that it indicates the potential of cotton to remain in kinetic uptake stage for longer compared to polyester due to more binding sites. Due to difference in physical morphology and chemical structure, McQueen et al. (2008) also suggested the greater availability of binding sites in cotton for body oils than polyester, which resulted in less bioavailability of those compounds for microbial degradation into odour-producing compounds. Analogously, we hypothesize that greater sorption of SVOCs by cotton than polyester could lead to less availability for dermal uptake.

Solid-state  $^{13}\text{C}$  NMR analysis confirmed that the polyester fabric exhibited high aromaticity, whereas cotton was dominated by cellulose. Abundance of non-polar moieties is expected to favour sorption of non-polar organic compounds. Cellulose has been shown to be a poor sorbent for a range of non-polar compounds due to the lack of aromaticity and polar nature (Xing et al., 1994a, 1994b, 1994c; Salloum et al., 2002). Wang and Xing (2007) also showed that charring resulted in enhanced aromaticity of cellulose along with increased surface area and porosity, hence increased the sorption of phenanthrene and naphthalene. Therefore, sorption can also be driven by physical characteristics such as surface area and porosity of sorbent apart from chemical characteristics (Wang and Xing, 2007).

If we assume the chamber as an indoor mesocosm, with the chamber walls mimicking indoor surfaces, these results show that fabrics (e.g., clothing, upholstery) with their large surface area, will act as a substantial sink for these chemicals under ambient conditions. The chamber experiments were conducted for a short duration. In reality, the time available for chemical sorption is much greater, particularly if the chemical is not lost during laundering (Schreder and La Guardia, 2014, Saini et al., unpublished results).

These results are also significant for human exposure, since dermal uptake of flame retardants has been shown to occur (Abdallah et al., 2015, 2016). Sorption and distribution coefficients estimated at room temperature are relevant for chemical uptake from air to the air-side of fabrics

whereas the data for 40°C could be relevant for the skin-side of fabrics worn as clothing. These results suggest greater sorption from air due to cooler ambient temperatures; whether the sorbed chemicals are released to the fabric-skin air space at a higher skin temperature (as the distribution coefficient decreases) remains to be tested.

## 5. Conclusions

Chamber studies conducted to test the sorption of a range of gas-phase PBDE congeners to cotton and polyester showed that 1 m<sup>2</sup> of these fabrics can sorb PBDEs present in 10<sup>3</sup> to 10<sup>4</sup> m<sup>3</sup> of equivalent air volume after one week at room temperature under conditions with air flow. As expected, the distribution coefficients were proportional to  $K_{OA}$  and inversely related to vapour pressure. The hypothesis that polyester sorbed more PBDEs than cotton was not supported when considering planar surface area. In contrast, polyester sorbed 3–10 times more than cotton per gram of fabric and 20–50 times more when considering BET surface area. Greater sorption of PBDEs by polyester than cotton could be explained by ‘dilution’ due to the large surface area of cotton however the ‘dilution’ factor of 20–50 times is greater than the 10 times difference in BET surface area. Alternatively, the difference in sorption could be due to the greater affinity of polyester for non-polar PBDEs which is consistent with the NMR analysis and reports from the literature. We hypothesize that lower PBDE concentrations in cotton than polyester on a BET surface area basis could reduce the potential for dermal transfer. The results also raise the question of whether fabrics that sorb relatively more chemical from air at cooler ambient temperatures could subsequently release them to the fabric-skin space at relatively higher skin temperatures. Finally, the results point to the importance of fabrics (e.g., clothing, draperies, and upholstery) as a sink for PBDEs and other non-polar compounds emitted to the indoor environment.

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