



Levels and profiles of organochlorines and flame retardants in car and house dust from Kuwait and Pakistan: Implication for human exposure via dust ingestion

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ARTICLE INFO

Article history:

Received 12 October 2012

Accepted 12 February 2013

Available online 21 March 2013

Keywords:

Organochlorines
Flame retardants
Pakistan
Kuwait
Car and house dust
Human exposure

ABSTRACT

There are only few studies documenting indoor pollution in the Middle East and the Indian subcontinent. In present study, we have evaluated the occurrence of various organochlorines (OCs) and flame retardants (FRs) in dust from cars and houses of Pakistan and Kuwait. Polybrominated diphenyl ethers (PBDEs), novel brominated flame retardants (NBFRs), organophosphate FRs (PFRs), polychlorinated biphenyls (PCBs), and organochlorine pesticides (OCPs) were investigated in indoor dust from urban houses (N = 15 per country) and cars (N = 15 per country). PFRs were the major analytes in all four microenvironments, followed by PBDEs > NBFRs > OCPs > PCBs. For all classes of analytes, relatively lower levels were observed in car and house dust from Pakistan than Kuwait. Levels of \sum PBDEs, \sum NBFRs and \sum PFRs were higher in car dust, while \sum OCPs and \sum PCBs were higher in house dust from both countries. \sum PFRs occurred at average concentrations of 16,900, 87,900, 475, and 2500 ng/g in Kuwaiti house and car, and Pakistani house and car dust, respectively. For both countries, the profiles of analytes in car dust were different from those in the house dust. Different exposure scenarios using 5th percentile, median, mean, and 95th percentile levels were estimated for adult, taxi drivers and toddlers. For Kuwaiti toddlers, assuming high dust intake and mean and 95th percentile concentrations, the values computed for \sum OCPs (1500 ng/kg bw/day) were higher than RfD values, while for \sum PCBs (14.5 ng/kg bw/day) it was only two-fold lower than the corresponding RfDs.

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1. Introduction

Different environmental contaminants, such organochlorine pesticides (OCPs), polychlorinated biphenyls (PCBs), and flame retardants (FRs) have been reported worldwide in various environmental media, several are suspected to be associated with various effects on human health (ATSDR, 2000, 2002, 2004, 2005; Darnierud, 2003). As a consequence, many countries have restricted or banned their marketing and ratified legislation that limits their continued use. Several OCPs and PCBs were listed under Stockholm Convention years ago, while more recently new chemicals, such as the penta- and octa-brominated diphenyl ethers (BDEs), were also included (Stockholm Convention, 2009).

Some industrialized countries already have started replacing restricted FRs with new chemical formulations, such as novel BFRs (NBFRs) or organophosphate FRs (PFRs). This has already been evidenced in matrices which are relevant for human exposure (Covaci et al., 2011; Stapleton et al., 2008, 2009; Van den Eede et al., 2011). Indoor dust has been associated with human exposure to various organic contaminants,

although most of the studies reported in the literature have focused on BFRs (Harrad et al., 2008; Roosens et al., 2010; Vorkamp et al., 2011). Recent reports have suggested that indoor dust might play an important role in the human exposure to OCPs, e.g., DDTs and HCHs (Bräuner et al., 2011) particularly in countries with a lower degree of industrialization, where FRs contamination is expected to be less important (Dirtu and Covaci, 2010; Dirtu et al., 2012).

Most of these organic contaminants are recognized as environmental contaminants, but there are still various regions, such as the Middle East and the Indian subcontinent, where limited information is known about their occurrence, as well as about their dynamics. Therefore, reporting these priority chemicals in indoor media from these regions is a high necessity. It is also important to investigate whether a link exist between the extent of indoor contamination and socio-economic status.

Recently, there has been a considerable focus on the risk posed to human health by indoor contaminants particularly to the most vulnerable groups, such as toddlers and infants etc. (Mitchell et al., 2007; Wu et al., 2007). Regarding indoor contaminants, settled dust has been considered and important exposure medium, in particular to infants and toddlers due to their more frequent hand-to-mouth activities (Lioy et al., 2002; Mercier et al., 2011). By employing similar

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dust collection protocols and identical analytical procedures, several classes of organic chemicals listed in Table 1, were investigated in car and house dust samples. House dust was the main matrix considered in this study, as people spend approximately 95% of their time indoors due to extreme weather conditions especially in summer particularly in the Middle East (Gevao et al., 2006). Car dust were also collected from these countries since many engineered materials, such as acrylonitrile butadiene styrene, polypropylene polymers, electronics, textiles, polyurethane foam, are used in the automotive industry and may contain several of the organic contaminants targeted in this study.

The aims of the present study were: (i) to evaluate the levels and profiles of several classes of organic contaminants in indoor dust from two microenvironments of Kuwait and Pakistan, (ii) to establish potential sources of targeted organic contaminants in the studied areas, (iii) to assess the magnitude of contamination of the Kuwaiti and Pakistani indoor environment by comparing the obtained results against data from other locations worldwide, (iv) to evaluate the evidence that alternative FRs are replacing PBDEs, (v) to estimate exposure to these chemicals of toddlers and adults from Pakistan and Kuwait via dust ingestion.

2. Experiment methodology

2.1. Materials

Standards for OCPs, PCBs and PBDEs were acquired from Dr. Ehrenstorfer Laboratories (Augsburg, Germany). Standards of NBFs were purchased from Wellington Laboratories (Canada). Standards of TEP, TnPP, TiBP, TnBP, TPhP, TCEP, TCP (mixture of 4 isomers), TBEP and TDCPP (mixture of 2 isomers) were purchased from Chiron AS (Trondheim, Norway), while TCPP (mixture of 3 isomers) were obtained from Pfaltz & Bauer (Waterbury, CT, USA). Purity of analytical standards for PFRs was >98%, except for TBEP (>94%). Internal standards (ISs) BDE 77 and BDE 128 (AccuStandard

Inc., USA), $^{13}\text{C}_{12}$ -BDE 209 (Wellington Laboratories), TAP (TCI Europe, Zwijndrecht, Belgium) and labeled TPP-d₁₅ (Sigma) were used to quantify these chemicals.

All solvents used during the analysis were of pesticide-grade. Acetone (Ace), dichloromethane (DCM), ethyl acetate, *iso*-octane and toluene were purchased from Merck (Darmstadt, Germany), while *n*-Hexane (Hex) was purchased from Acros Organics (Geel, Belgium). Concentrated sulfuric acid (98%) (H₂SO₄) and silica gel were purchased from Merck. Empty polypropylene filtration tubes (3 mL) SPE cartridges and 500 mg/3 mL Supelclean™ ENVI™ – Florisil® cartridges were purchased from Supelco (Bellefonte, PA, USA). Silica gel was washed with Hex and activated overnight at 160 °C. Prior to each experiment, silica was heated for 2 h at 160 °C for activation. Acid impregnated silica (44%, w/w) was prepared by adding dropwise 22 mL concentrated sulfuric acid (98%) to 50 g silica under continuous stirring. All glassware was soaked for 12 h in an alkali solution (diluted RBS 35, pH 11–12) to degrade any remaining chemicals. After washing, the glassware was rinsed with water and dried at 400 °C for 5 h. Prior to use the all glassware was rinsed with Hex.

2.2. Sampling and sample preparation

Indoor dust samples (N = 60) were collected from Kuwait city (houses N = 15; cars N = 15) and Faisalabad, Pakistan (houses N = 15; cars N = 15). For dust collection, active sampling was done using a commercial vacuum cleaner. Details about dust (Table S-1) samples and their collection are given in Supplementary information (SI).

The sample extraction and purification method was described in detail elsewhere (Van den Eede et al., 2012). Briefly, an accurately weighed aliquot of dust (typically 75 mg) was spiked with internal standards and extracted by ultrasonication and vortexed with Hex: Ace (3:1, v/v). Florisil was used to obtain two fractions; a 1st fraction was eluted with 8 mL Hex and a 2nd fraction with 10 mL ethyl acetate. All PCBs, OCPs, PBDEs and NBFs, except TBPH, were present in the 1st fraction, while TBPH and PFRs were present in the 2nd fraction. After concentration under nitrogen, the 1st fraction was further cleaned on acid silica and analytes were eluted with 10 mL Hex:DCM (1:1, v/v). After evaporation to dryness, each fraction was resolubilized in 100 µL of *iso*-octane prior to GC–MS analysis.

2.3. Analysis

Details about the instrumental analysis are found elsewhere (Ali et al., 2012b; Van den Eede et al., 2012). Briefly, the analysis of PCBs, OCPs, NBFs and PBDEs was performed by 6890 Agilent (Palo Alto, CA, USA) gas chromatography (GC) coupled to a 5973 mass spectrometer (MS) operated in electron capture negative ionization (ECNI). A DB-5 column (15 m × 0.25 mm × 0.10 µm) was used for separation and the MS was deployed in selected ion monitoring (SIM) mode. The ion source, quadrupole and interface temperatures were set at 200, 150 and 300 °C, respectively. The analysis of PFRs was performed by GC–MS in electron ionization (EI) mode. A HT-8 column (25 m × 0.22 mm × 0.25 µm) was used and the MS was operated in SIM mode with two characteristic ions acquired for each compound. The ion source, quadrupole and interface temperatures were set at 230, 150 and 300 °C, respectively.

2.4. Quantification and quality assurance

As a part of the quality assurance protocol, field blanks (N = 3 for each set of samples), laboratory blanks (N = 10) and indoor dust standard reference materials (SRMs) from National Institute of Standards & Technology SRM 2585 (N = 4) and 2584 (N = 4) were analyzed in parallel with the dust samples to account for eventual external contamination during sampling, sample preparation and instrumental analysis, and to evaluate method accuracy. Levels of targeted analytes were blank-corrected in all samples. To avoid photodegradation of analytes,

Table 1
Organic contaminants studied in the indoor dust during present study.

Novel brominated flame retardants (NBFs)
1,2-Bis(2,4,6-tribromophenoxy)ethane (BTBPE)
2-Ethylhexyl-2,3,4,5-tetrabromobenzoate (TBB)
Bis(2-ethylhexyl)-3,4,5,6-tetrabromophthalate (TBPH)
Decabromodiphenylethane (DBDPE)
Hexabromobenzene (HBB)
Hexachlorocyclopentadienyl-dibromocyclooctane (HCDBCO)
Organochlorine pesticides (OCPs)
Hexachlorobenzene (HCB)
<i>cis</i> -Chlordane (CC)
<i>trans</i> -Chlordane (TC)
Oxychlordane (OxC)
<i>trans</i> -Nonachlor (TN)
Dichlorodiphenyldichloroethane (<i>p,p'</i> -DDD)
Dichlorodiphenyldichloroethylene (<i>p,p'</i> -DDE)
Dichlorodiphenyltrichloroethane (<i>p,p'</i> -DDT)
Hexachlorohexane (α -HCH, β -HCH, γ -HCH)
Organophosphate flame retardants (PFRs)
Tri-ethyl-phosphate (TEP)
Tris-(2-chloroethyl)-phosphate (TCEP)
Tris-(1,3-dichloro-isopropyl)-phosphate (TDCPP) (mixture of 2 isomers)
Tri- <i>n</i> -propyl-phosphate (TnPP)
Tri- <i>iso</i> -butyl-phosphate (TiBP)
Tris-(1-chloro-2-propyl)-phosphate (TCPP) (mixture of 3 isomers)
Tri-(2-butoxyethyl)-phosphate
Tri-cresyl-phosphate (TCP) (mixture of 4 isomers)
Tri-phenyl-phosphate (TPhP)
Tri- <i>n</i> -butyl-phosphate (TnBP)
Polychlorinated biphenyls (PCBs)
99, 101, 105, 118, 138, 153, 156, 170, 180, 183, 187, 194, 199, 196/203, 206, 209
Polybrominated diphenyl ethers (PBDEs)
28, 47, 99, 100, 153, 154, 183, 196, 197, 203, 209

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