

Advances in the study of current-use non-PBDE brominated flame retardants and dechlorane plus in the environment and humans

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The fate of the high production volume, currently in use, and not regulated non-polybrominated diphenyl ether (PBDE) flame retardants, such as tetrabromobisphenol A (TBBPA), hexabromocyclododecane (HBCD) and dechlorane plus (DP), and the alternative flame retardants of PBDE, such as BTBPE and DBDPE, in the environment has attracted increasing attention and aroused concern due to the increasing regulation and phasing-out of PBDEs. This paper reviews the distribution, bioaccumulation, human exposure and environmental behavior of those non-PBDE flame retardants in various environmental compartments. The data gaps and needs for future research are discussed.

TBBPA, HBCD, BTBPE, DBDPE, DP

1 Introduction

Halogenated organic flame retardants are chemicals that are added to polymeric materials and other materials to prevent fires. Brominated and chlorinated compounds are the sole halogen compounds possessing commercial significance as flame-retardant chemicals. Polybrominated diphenyl ethers (PBDEs), tetrabromobisphenol A (TBBPA), and hexabromocyclododecane (HBCD) are the three classes of most widely used brominated flame retardants (BFRs). The world production volumes of PBDEs, TBBPA, and HBCD in 2001 (the most recent figures available from the industry) were 67000, 119700, and 16700 t, respectively [1]. The production capacities of TBBPA and HBCD in China in 2007 were 18000 and 7500 t, respectively [2]. Since PBDEs were found in environmental samples remote from sources [3], the contamination of BFRs, especially PBDEs, has become one of the most extensively studied subject. PBDEs, TBBPA, and HBCD have been identified to be ubiquitous

environmental contaminants and bioaccumulated in the tissues of wildlife and humans [4–6]. Penta and octa-mix PBDE formulations have been officially listed as persistent organic pollutants due to their persistence, potential for bioaccumulation, and toxicity to wildlife and humans [7]. The deca-mix product has been banned in Europe in July 2008. There are no restrictions on the production and use of TBBPA and HBCD. However, HBCD has been considered as a candidate for POPs, and both TBBPA and HBCD are included in the OSPAR (the Convention for the Protection of the Marine Environment of the North-East Atlantic, OSPAR) list of chemicals for priority action under their Hazardous Substances Strategy [8].

In response to the increasing regulation and phasing-out of the commercial PBDE products, the BFR industry has begun to market alternative bromine based compounds to replace the BDE formulations. 1,2-bis(2,4,6-dibromophenoxy) ethane (BTBPE) and decabromodiphenylethane (DBDPE) are two potential replacements, with the former for octa-BDE and the latter for deca-BDE. BTBPE was first produced in the 1970s and used as an additive BFR in ABS resin and in products such as computers, televisions, and

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mobile phones. Annual production volumes were about 4500–22500 t during the period from 1986 to 1994 and decreased to 450–4500 t after 1998 [9]. As it has been announced as a replacement of penta-BDE, a high production of BTBPE is expected. DBDPE was first introduced in the early 1990s under the trade name SAYTEX 8010 by Albemarle Corporation [10]. Its application was the same with that of BDE209. As a replacement of deca-BDE, it is expected that the production and use of DBDPE will mount in the coming future. It was estimated that the production of DBDPE grows with a rate of 80% per year in China [11]. BTBPE has been detected in a variety of biotic and abiotic matrices [9, 12–15] and DBDPE has been found in air, indoor dust, sediments, fish, and birds around the world [10–15].

A chlorinated flamer retardant, bis(hexachlorocyclopentadieno)cyclooctane, commonly named as Dechlorane Plus (DP), was identified in ambient air, fish, and sediment samples from the Great Lake areas and in air and fish samples from China [16–19], although it has been used for over 40 years as a flame retardant in electrical wires and cables, computer connectors, and plastic roofing materials. It is categorized as a high production volume (HPV) chemical by US EPA and is listed on Canada's Domestic Substances list [18].

The comprehensive reviews on PBDEs are available in recent publications [4, 20] and HBCD in the environment and humans until the end of 2005 has been overviewed by Covaci *et al.* [5]. In this paper, we review the reports published in recent years on non-PBDE flame retardants, including TBBPA, HBCD, BTBPE, DBDPE, and DP. The aim was to summarize the distribution, bioaccumulation and environmental behavior of those non-PBDE flame retardants in various environmental compartments. The knowledge gaps and needs for further research are discussed.

2 Tetrabromobisphenol A (TBBPA)

In 2009, Covaci *et al.* [21] summarized the publications until February 2008 concerning the analytical and environmental aspects of TBBPA. TBBPA analytical methods including sample preparation, chromatographic separation, detection techniques, and quality control were discussed in their review. The dataset available on TBBPA and its derivatives in abiotic matrices, such as air, indoor dust, water, soil, sewage sludge and sediments, in biological matrices, food, and human was also reviewed. Generally, fewer data on TBBPA in both abiotic and biotic matrices are available compared with other BFRs, such as PBDEs and HBCD. Levels of TBBPA in abiotic and biotic matrices were generally low except samples from locations near industrial emissions and electronic product recycling plants [22, 23]. TBBPA was usually used as a reactive flame retardant, which could be used to explain the low levels of TBBPA

found in the environment matrix. As a consequence, the expected human exposure may be low.

A few reports on TBBPA in indoor dust, sediments, human tissues, and human exposure have been published since the review of Covaci *et al.* [21]. Geens *et al.* [24] determined TBBPA in dust samples from 18 houses and 2 offices throughout Flanders (Belgium). TBBPA concentrations in two offices (45 ng/g and 100 ng/g) were 5 to 10 times higher than those in the domestic dust samples (mean 10 ng/g). The human exposure by dust intake (0.2 ng/day) was found minor compared to diet exposure (2.8 ng/day). Mäkinen *et al.* [25] detected TBBPA in air samples, including personal samples and stationary samples, collected from a furniture workshop, a circuit board factory, two electronics dismantling facilities, a computer classroom, and offices and social premises. TBBPA was mainly found in two electronics dismantling facilities with concentrations less than the detection limit up to 14600 ng/m³. This result further confirmed that the high TBBPA level was mainly found in occupational environments. Takigami *et al.* [26] reported TBBPA in indoor and outdoor air, dust, and exhaust from two houses in Japan. No significant difference in TBBPA concentration was found between indoor and outdoor air. In dust, TBBPA concentrations were 520 ng/g and 490 ng/g.

Harrad *et al.* [27] reported TBBPA in water ($n = 27$), sediment ($n = 9$), and fish samples ($n = 30$) from English Lakes and found concentrations of TBBPA ranged from 140 to 3200 pg/L (water), 330 to 3800 pg/g dry weight (sediment), and <0.29 to 1.7 ng/g lipid weight (fish). The sediment TBBPA levels in this report are at the low end of those previously reported concentrations in river sediments in England (9800 ng/g dw in sediments from the River Skene near a BFR manufacturing site and an averaged 25 ng/g in its downstream). This is because the samples in this study were collected from lakes located far away from the emission sources of TBBP-A while the previous sediments were collected from sites impacted to varying degrees by a BFR production facility. Sánchez-Brunete *et al.* [28] determined TBBPA in industrial and agricultural soils from Spain. TBBPA was found in various soil samples ($n = 3$) at levels ranging from 3.4 to 32.2 ng/g in industrial soils and at 0.3 ng/g in one agricultural soil.

TBBPA was detected at concentrations ranging from 3.8 to 225 ng/g dry wt in river sediments ($n = 17$) collected from an industrial area (Dongguan) in South China, which houses a vast number of electronics manufacturing and assembly plants [29]. These values were higher than the concentrations in sediments from Europe and the United States in most recent reports, but lower than the values in sediments from the River Skene. In addition, a significantly increased trend for TBBPA level was found in the upper layers in two sediment cores collected from the Chinese Industrial site. This result suggested a significant increase of the use of TBBPA in the study area [29].

Hayama *et al.* [30] determined TBBPA in five human

serum samples from healthy volunteers from Japan. The concentrations of TBBPA in serum samples are in between 6.2 and 8.7 pg/g serum with mean of 7.4 pg/g serum. These levels of TBBPA are almost the same as European levels (< 1 to 3.4 pmol/g for computer technicians in England and 0.71 ng/g lipid in Norway) [31, 32] though Asia is the largest consumer of brominated flame-retardants containing TBBPA in the world [21]. Shi *et al.* [33] investigated the TBBPA levels in 24 pooled human milk samples and 48 food samples in China and the dietary exposure of Chinese adults and nursing infants to TBBPA. The mean TBBPA concentrations in meat, eggs, and aquatic food were 263, 194, and 738 pg/g lipid, respectively. TBBPA in human milk ranged from < LOD to 5124 pg/g lipid. The average estimated daily intake (EDI) of TBBPA via human milk for nursing infants was 5094 pg/kg bodyweight (bw)/day with a range 320–37240 pg/kg bw/day. The medium bound (< LOD = 1/2LOD) EDI_{TBBPA} for a “reference” man via animal origin foods was 256 pg/kg bw/day. Meat and meat products were the main sources in the total dietary intake of TBBPA. EDI values of TBBPA by Chinese population were lower than those observed in U.K. (5980 pg/kg bw/day) but higher than those in the Dutch population (40 pg/kg bw/day).

The research concerning the environmental fate of TBBPA revealed that TBBPA was not as persistent as PBDEs. Ronen *et al.* [34] found a decrease by 80% in TBBPA concentration in an anaerobic incubation of sediments with TBBPA and a metabolite, bisphenol A, was identified. Gerecke *et al.* [35] investigated the degradation of TBBPA under anaerobic conditions in sewage sludge in the laboratory. The half-life of TBBPA was 0.59 days, indicating the degradation of TBBPA occurred in full-scale anaerobic digesters as well. In a study of microbial O-methylation of TBBPA, George *et al.* [36] observed that more than 50% of the original TBBPA was transformed to TBBPA mono- and dimethyl ether in the Kearny Marsh sediment under aerobic conditions after 80 days' incubating and approximately 10% of the total aerobic heterotrophs were capable of O-methylation. Recently, studies on the fate of TBBPA in freshwater sediments were conducted by Luo *et al.* [37, 38]. Bound TBBPA, defined as the fraction which cannot be extracted by organic solvents, has been detected in river surface sediments from Dongjiang River at concentrations ranging from 1.2 to 20 ng/g dry wt, accounting for 6.5% to 48% of total TBBPA. In the sediment cores, the percentages of bound TBBPA were increased from 22% to 92% with increasing depth, indicating that TBBPA was easily to be bound in sediments. The formation mechanism of bound TBBPA was suggested mainly via covalent bonding to organic matter through phenolic ether linkages. This result can also be used, at least partly, to explain the low concentrations of TBBPA found in sediments.

Watanabe *et al.* [39] detected TBBPA and its derivatives in mussel collected in Osaka, Japan. TBBPA was not de-

tected in mussel but its derivatives, dimethyl ether derivative of TBBPA, was present in mussel with a concentration about 5 ng/g wet wt. The derivatives of TBBPA were supposed to be formed by microbial transformation but the metabolism of TBBPA in mussel cannot be ruled out. A same observation was also reported by Vorkamp *et al.* [40]. They collected thirty-three peregrine falcon eggs from South Greenland and analyzed TBBPA and its derivatives. TBBPA could not be detected in any of the eggs whereas dime-TBBPA was quantifiable in 29 out of 33 eggs. Concentrations of dime-TBBPA ranged between < 0.1 and 940 ng/g lw, with a mean concentration of 280 ng/g lw.

3 Hexabromocyclododecane (HBCD)

A review of HBCD in the environment until the end of 2005 has been conducted by Covaci *et al.* [5]. Since then, more data on the HBCD in abiotic and biotic samples has become available.

Takigami *et al.* [41] measured HBCD in both indoor air and dust from two Japanese homes in late 2006. The total HBCD (no diastereoisomer-specific) concentrations ranged from 6.7 to 280 pg/m³ in the air samples and from 240 to 13000 ng/g in the dust samples. Abdallah *et al.* [42] determined HBCD in 31 UK houses' dust and 6 UK offices' dust using LC-MS/MS. The mean HBCD concentrations (sum of the α -, β -, and γ -diastereoisomers) were 6000 ng/g and 1400 ng/g, respectively. These concentrations were in line with those detected in North American domestic dust. Yu *et al.* [43] reported the diastereoisomer- and enantiomer-specific profiles of HBCD in the atmosphere of an urban city (Guangzhou) in South China. The concentrations of HBCD ranged from 0.69 to 3.09 pg/m³ ($n=4$), which were similar to those reported in the US but were significantly lower than those in Europe and Japan. α -HBCD was the dominant isomer and β -HBCD was a minor isomer in all air samples. The result of enantiomeric fractions indicated that β - and γ -HBCD were racemic mixtures and that (–)- α -HBCD was slightly enriched in the typical urban site, implying that the HBCD in this site might be a mixture of emission from both local sources and soil-air exchange.

Most of the HBCD data in sediments was reported in Europe in recent years. Klamer *et al.* [44] reported HBCD in the size fraction less than 63 μ m of North Sea surface sediments at concentrations from less than 0.2 to 6.9 ng/g dry wt. Verslycke *et al.* [45] determined HBCD in sediments ($n=3$) from the Scheldt estuary in the Netherlands. HBCD concentrations were between 14 and 71 ng/g dry wt. Evenset *et al.* [46] determined HBCD in four sediment cores from the deepest part of Lake Ellasjøen. HBCD was exclusively found in the section of 1 to 2 cm with α -HBCD concentration of 0.43 ng/g dry wt and γ -HBCD concentration of 3.9 ng/g dry wt. Sediments from nine English lakes

were analyzed for HBCD by using LC-MS/MS. Concentrations of HBCD ranged from 0.88 to 4.8 pg/g dry wt and HBCD chiral signatures are racemic in sediments [25]. Haukås *et al.* [47] reported concentrations and spatial distribution of α -, β -, and γ -HBCD in a contaminated Norwegian fjord. The results indicated α -HBCD gradient with decreasing concentrations at increasing distance from the point source in sediments. Mean concentration of total HBCD at the locations close to the point source was 9000 ng/g TOC. There was one study reporting HBCD in sediments from locations other than Europe. Minh *et al.* [48] measured HBCD in six surface sediments and three sediment cores from Tokyo Bay at concentrations ranging from 0.06 to 2.3 ng/g dry wt. An increasing trend of HBCD concentration was observed in sediment cores since it first appeared in the mid-1970s.

More information on diastereoisomer- and enantiomer-specific profiles of HBCD in biota samples is available. The commercial HBCD products are composed of three non-aromatic diastereoisomers: α -(6%), β -(8%) and γ -HBCD (80%). The HBCD diastereoisomeric compositions in biota are variable and significantly different from that of the technical mixtures with an increase of α -HBCD and a concurrent decrease of γ -HBCD.

Verslycke *et al.* [45] reported HBCD with concentrations of 562 to 727 ng/g lipid in mysid shrimp (*Neomysis integer*, $n=3$) collected from the Scheldt estuary. Fish samples from Irish water were analyzed for HBCD on a diastereoisomer-specific basis. HBCD was detected in farmed salmon ($n=4$) with the concentration between 1.1 to 2.7 ng/g wet wt, and in one sample of tuna at 0.3 ng/g wet wt [49]. Skipjack tuna (*Katsuwonus pelamis*) was used as a bioindicator to investigate the distribution of HBCD in the Asia-Pacific region with concentrations between < 0.1 to 45 ng/g lipid. The concentrations of HBCD were found higher in the northern hemisphere than the southern one [50]. HBCD concentration in fish ($n=30$) collected from nine English lakes ranged from 14 to 270 ng/g lipid. An enrichment of (-) α -HBCD and (+) γ -HBCD was found in fish samples [27]. In north-west Atlantic, HBCD was detected in 87% of the fish samples at concentrations ranging from 2.4 to 38.1 ng/g lipid [51].

Elevated levels of HBCD have been reported in marine mammals. Law *et al.* [52] studied HBCD in the blubber of harbor porpoises from the UK during the period from 1994 to 2003. α -HBCD was detected in all samples ($n=85$) and was the dominant isomer at concentrations ranging from 10 to 19200 ng/g lipid. Three isomers of HBCD were determined in blubber samples from male California sea lions (*Zalophus californianus*) stranded between 1993 and 2003 [53]. Total HBCD ranged from <0.4 ng/g to 96 ng/g lipid with α -HBCD contribution of 62%–100%. The concentrations of HBCD increased from 0.7 ng/g to 12.0 ng/g ww in sea lion blubber between 1993 and 2003. Johnson-

Restrepo *et al.* [54] determined HBCD in three marine top-level predators-bottlenose dolphin, bull shark, and Atlantic sharpnose shark-collected from coastal waters of Florida, USA. Total HBCD concentrations in bottlenose dolphin blubber, bull shark muscle, and Atlantic sharpnose shark muscle were 7.4, 77.7, and 54.5 ng/g lipid, respectively.

HBCDs were also found in the liver of fulmars ($n=15$) breeding in Bjørnøya at concentrations ranging from 3.8 to 62 ng/g lipid [55]. In blood plasma of glaucous gulls (*Larus hyperboreus*) collected in 2004 from Svalbard, HBCD (no diastereoisomer-specific) was found at concentrations ranging from 0.07 to 1.2 ng/g lipid [56]. Lundstedt-Enkel *et al.* [57] reported HBCD at the concentration 65 ng/g lipid in birds from Karso islands collected in 2000. The geometric mean of HBCD in eggs collected from the island of Stora Karso during the period from 2000 to 2002 was 77 ng/g lipid. Sagerup *et al.* [58] determined HBCD in glaucous gulls (*Larus hyperboreus*) at Bjørnøya. α -HBCD was detected at concentrations ranging from 200 to 15000 ng/g lipid in the liver and 5 to 500 ng/g lipid in the brain.

The data of HBCD in human tissues remains limited. HBCD was detected in 24 pooled human milk samples (1237 individual samples) collected in China in 2007 [33]. α -HBCD was generally the most abundant isomer with concentrations from < LOD to 2776 pg/g lipid. α -HBCD was also detected in 11 pooled human milk samples in Japan between 1988 and 2006 with the levels ranging from 430 to 1900 pg/g lipid, which were similar to those in China [59]. HBCD was detected in human milk from A Coruna (NW of Spain) at concentrations between 3 to 188 ng/g lipid, which were one to two orders of magnitudes higher than those in Japan and China [60]. Different from other studies, γ -HBCD other than α -HBCD was found to be the dominated isomer in Spanish study. An enrichment of (-) α -HBCD was observed in human milk but there was no clear preference for γ -HBCD enantiomer. HBCD concentrations in the serum of nonoccupationally exposed populations in Belgium (< 0.5 to 11.3 ng/g lipid) [61] and Netherlands (< 0.2 to 7.0) [62] were slightly lower than those in Norway (< 1.0–52) [63]. Occupationally exposed populations in an industry plant in Norway had HBCD concentrations ranging from 6 to 856 ng/g lipid [64]. The overall mean HBCD concentration was 0.33 ng/g lipid wt in human adipose tissue obtained in New York City, USA [54]. Concentrations of HBCD in human samples from the United States were 1–5 fold lower than those reported from several European countries.

Dietary exposure, inhalation of indoor air, and ingestion of indoor dust are the major routes for nonoccupationally exposed persons to intake HBCD. A study concerning dietary exposure of HBCD in China revealed that the estimated daily intake of HBCD was 5.8 ng/kg bw/day [33]. This value was similar to those in UK (5.9–7.9 ng/kg bw/day) [65], in Dutch (8.3 ng/kg bw/day) [66], and in Sweden (2.15

ng/kg bw/day) [67], but higher than that in Norway (0.3 ng/kg bw/day) [68]. Abdallah *et al.* [69] used the data obtained in indoor house dust and office dust in UK to quantify the personal exposure to HBCD via inhalation of indoor air and ingestion of indoor dust. The result showed that personal exposure ranged from 4.5 to 1851 ng/day under an average dust ingestion scenario and ranged from 11 to 4630 ng/day under a high dust ingestion scenario. Recently, Roosens *et al.* [61] investigated the correlation between concentrations of HBCD in human serum and dust ingestion or food intake. The averaged dietary intake of HBCD was 7.2 ng/day, whereas those estimated under average and high dust ingestion scenarios were 3.2 ng/day and 8.0 ng/day, respectively. However, it is the dust ingestion not the food intake correlates with HBCD levels in human serum, suggesting dust ingestion may play a more significant role than dietary intakes in determination of the intake of HBCD.

A few studies have investigated the transfer of HBCD through the food web. Tomy *et al.* [70] studied the enantioselective bioaccumulation of HBCD in an eastern Canadian arctic marine food web consisting of zooplankton, shrimp, clama, red fish, arctic cod, walrus, beluga, and narwhal. α -HBCD and γ -HBCD were detected in all species and total HBCD concentrations ranged from 0.6 ng/g lipid in arctic cod to 3.9 ng/g lipid in narwhal. Trophic magnification was observed for α -HBCD, whereas trophic dilution was found for γ -HBCD in the food web. Only the ($-$)- α -HBCD showed a strong positive relationship between the concentration and trophic level (TL) with a trophic magnification factor (TMF) value of 2.2. A significant increase in the enantiomeric fraction value of the α -enantiomers with TL was observed, implying that there is an overall preferential enrichment of the ($-$)- α -enantiomer due to the great bioaccumulation potential of the ($-$)- α -enantiomer and/or due to the great susceptibility of the (+)- α -enantiomer. In an arctic marine food chain, no biomagnification from ringed seals to polar bears was found, which may be due to the biodegradability of HBCD in the polar bears. However, the substantial biomagnification of HBCD from polar cod to ringed seals indicates its high bioaccumulation potential in other species [71].

Tomy *et al.* [72] studied HBCD in a western Canadian arctic marine food web using LC-MS/MS. Different HBCD isomer profiles were found in this food web. In the top TL marine mammal (beluga), α -HBCD accounts for about 95% of the total HBCD. Whereas in its primary prey species (arctic cod), HBCD profile is dominated by γ -HBCD. This result further confirms that the selective isomer metabolism and isomer conversion are possible to occur through the food web. Haukås *et al.* [73] performed experiments of oral exposure of rainbow trout to HBCD to assess the role of selective uptake on diastereoisomer-specific accumulation and disposition of HBCD to the liver, brain and muscle. Their results indicated that a diastereoisomer-selective uptake

of α - and β -HBCD in the rainbow trout occurred after exposure. The differences in HBCD pattern between organs at the end of the experiment supported a proposal of an organ-specific diastereoisomer accumulation.

Several studies reported the time trend of HBCD in the biota samples. α -HBCD concentrations were reported to have increased in eggs from three species of birds, herring gulls (*Larus argentatus*), Atlantic puffins (*Fratercula arctica*), and black-legged kittiwakes (*Rissa tridactyla*) in North Norway throughout the 20-year period (1983–2003) [74]. Law *et al.* [52] reported an increase in HBCD concentration in blubber in the period from 1993 to 2003. Recently, they conducted a further study using samples collected from 2003 to 2006 [75]. The results indicated a significant downturn in levels of HBCD in the blubber of harbor porpoises in UK since 2003. This decrease was attributed to the closure of an HBCD manufacturing plant in NE England in 2003 and two voluntary schemes intended to reduce emissions of HBCD to the environment from the industry. Peck *et al.* [76] reported HBCD in white-sided dolphins from 1993 to 2004. α -HBCD was exclusively detected in all samples with concentrations ranging from 19 ng/g lipid to 380 ng/g lipid. No significant temporal trend was found for HBCD in white-sided dolphin blubber. Kakimoto *et al.* [59] investigated the time trend of HBCD in the breast milk of Japanese women from 1973 to 2006. HBCD appeared in the samples after 1983 and increased in those collected since 1988. The time trend of HBCD was found to be related to that of the industrial HBCD consumption volume in Japan.

One remarkable advance on HBCD study is in those reports concerning the degradation and metabolism of HBCD in the environment since the review of Covci *et al.* Gerecke *et al.* [35] investigated the degradation of HBCD under anaerobic conditions in sewage sludge in a laboratory system. The half-life of HBCD (as the technical mixture) was 0.66 day, and the half-life of α -HBCD was about two times of β - and γ -HBCD. Harrad *et al.* [77] studied the cause of variability in concentrations and diastereoisomer patterns of HBCD in indoor dust by analysis of HBCD and pentabromocyclododecenes (PBCDs—degradation products of HBCDs) in dust in the presence and absence of light. A significant shift from γ -HBCD to α -HBCD occurred within 1 week in the presence of light. There is no significant change in enantiomer fractions. However, there is degradative loss of HBCD, as evidenced by the decrease in HBCD levels and the increase in PBCDs with time. These data suggests a rapid photolytically-mediated shift in the diastereoisomer profile, a slower loss of HBCD via delimitation of HBr, and that neither of these processes are enantioselective.

Barandsma *et al.* [78] reported the presence of hydroxylated metabolites of HBCD in three wildlife species (tern eggs, seals, and flounders) and 28-day exposed Wistar rats for the first time. Four different groups of hydroxylated HBCD were identified in rat adipose, liver, lung, and muscle

tissues by LC-MS and GC-MS. Debromination of HBCD to PBCD was found to be another metabolic pathway. In tern eggs from the Western Scheldt, the monohydroxy-HBCD was found and the monohydroxy metabolites of HBCD and PBDE were found in the blubber of harbor seals (Wadden Sea). No hydroxylated metabolites were detected in the tissues of flounders. Covaci *et al.* [79] also found the OH-HBCD in the liver of harbor seals from the Northwest Atlantic by using LC-MS/MS in the MRM mode. Concentrations of OH-HBCD (2.1 to 15.0 ng/g lipid) were lower than that of HBCD.

4 1,2-bis (2,4,6-dibromophenoxy)ethane (BTBPE) and decabromodiphenylethane (DBDPE)

The data for BTBPE and DBDPE in abiotic and biotic matrices are shown in Table 1. A few studies reported the occurrence of BTBPE in indoor dust and air samples in Europe, the United States, and China. Sjödin *et al.* [22] detected BTBPE for the first time in air from an electronics recycling plant and other work environments in Sweden and found the concentration of 41 pg/m³ in a factory assembling printed circuit boards, 3 to 11 pg/m³ in a computer repair facility, 5.8 pg/m³ in an office with computers, and 3 to 4.8 pg/m³ in a computer teaching hall. Pettersson-Julander *et al.* [80] measured BTBPE in personal air samples collected by glass fiber filters combined with XAD-2 adsorbent in a closed face cassette from an e-waste recycling facility in Sweden. The concentration of BTBPE ranged from 1.1 to 39 ng/m³. In the same recycling facility, Julander *et al.* [81] determined BTBPE in air samples at concentrations ranging from 7.4 to 11.9 ng/m³ and the highest BTBPE concentrations (8.2 to 12.2 ng/m³) were found in the inhalable dust fraction. Harrad *et al.* [82] reported BTBPE in dust from the United Kingdom cars, homes, and offices with mean concentrations of 120, 7.2, and 7.7 ng/g, respectively, which were significantly lower than those of PBDEs. In house dust collected in 19 homes in the greater Boston, MA area during 2006, the mean concentration of BTBPE was found to be 30 ng/g, which is slightly higher than that in Europe [13]. In five sites near Great Lake areas, BTBPE was detected at concentrations from 0.5 to 1.2 pg/m³ [12]. Hoh *et al.* [9] reported the presence of BTBPE in air samples from various sites in the United State. The concentrations of BTBPE in air samples ranged from 2.8 to 70 pg/m³, which were comparable to those of tetra- through hexa-PBDEs but higher than those of BDE209. Zhu and Hites [83] detected BTBPE in tree bark samples collected near two manufacturing facilities in southern Arkansas. The two manufacturing facilities were found to be the sources of this compound. Shi *et al.* [84] determined the BTBPE in air collected in Guangzhou City in China. The levels of BTBPE were in the range of 3.38 to 37 pg/m³, comparable with those in the United State.

In the study of Shi *et al.* [84], BTBPE was found in sediments collected in Dongjiang River in 2006 and 2002 at respective concentrations ranging from 0.05 to 2.07 ng/g ($n=3$) and from 0.27 to 21.9 ng/g dry wt ($n=3$). Hoh *et al.* also reported the presence of BTBPE in sediments [9]. BTBPE has been detected in the sediment core from Lake Michigan and the concentration was found to increase with time. The concentrations of BTBPE were lower than that of BDE209 but were 10 times higher than the sums of BDE47, 99, and 100.

Limited studies reported the occurrence of BTBPE in fish. Law *et al.* [14] measured some brominated flame retardants in a Lake Winnipeg (Canada) food web. BTBPE was detected in all species samples at average concentrations ranging from 0.13 to 1.96 ng/g lipid. Biomagnification of BTBPE within certain predator/prey relationships was also observed in this food web, suggesting a potential biomagnification in aquatic food webs. In fish samples collected from an e-waste region in South China, BTBPE was detected in fish muscle and liver tissues with the concentration range from <0.012 to 0.15 ng/g lipid, three orders of magnitude lower than those of PBDEs [84]. Ismail *et al.* [85] reported the time trend of BTBPE in lake trout (*Salvelinus namaycush*) collected from Lake Ontario between 1979 and 2004. Concentrations of BTBPE in the lake trout increased exponentially between 1979 (0.6 ng/g lipid) and 1993 (2.6 ng/g) with a doubling-time of six years. After 1993, it decreased by 40% between 1993 and 2004. This is the first study that reported the time trend of BTBPE in the environment.

BTBPE has been detected in several water birds including glaucous gulls, herring gulls, and Northern fulmars. In northern fulmar eggs from the Faroe Islands, BTBPE was detected in eight of nine samples with a mean concentration of 0.11 ng/g lipid, 150 times lower than the average total PBDE concentration, which may be due to its low volumes in use [86]. Verreault *et al.* [87] determined BTBPE in glaucous gull plasma and egg samples from the Norwegian Arctic. BTBPE was confirmed in one plasma sample alone (0.26 ng/g wet weight) and less than 29% of the egg yolk samples (<MLOQ to 0.96 ng/g wet weight). The levels of BTBPE in the egg yolk exceed those reported in northern fulmars from the Faroe Island. Recently, Gauthier *et al.* [15] reported the temporal trends and spatial distribution of non-PBDE flame retardants in the eggs of colonial populations of Great Lakes herring gulls. BTBPE was found mainly in sampling years from mid-1990s to 2006 and in eggs from all seven herring gull colonies at averaged concentration ranging from <0.06 to 0.20 ng/g wet weight. Shi *et al.* [84] detected BTBPE in water birds collected in an e-waste region from South China at concentrations ranging from 0.07 to 2.41 ng/g lipid.

Few studies were conducted to investigate the environmental behavior of BTBPE. Hakk *et al.* [88] investigated

Table 1 Concentrations of BTBPE and DBDPE in abiotic and biotic matrices

Samples	Location	BTBPE	DBDPE	Ref.
abiotic samples				
air	Sweden	3–41 pg/m ³		[22]
personal air samples	Sweden	1.1–39 ng/m ³		[80]
dust	Sweden	7.4–11.9 ng/m ³	<0.02–0.08 ng/m ³	[81]
air	Sweden		0.6 ng/ m ³	[10]
air	the United States	2.8–70 pg/m ³		[9]
air	Near the Great Lake	0.5–1.2 pg/m ³	1.0–22 pg/m ³	[12]
dust in cars	United Kingdom	120 ng/g	270 ng/g	[82]
dust in homes		7.2 ng/g	170 ng/g	
dust in offices		7.7 ng/g	400 ng/g	
household dust	Sweden		nd–121 ng/g dw	[90]
dust in homes	The United States	1.6–789 ng/g	<10.0–11070 ng/g	[13]
air	Guangzhou, South China	3.38–37 pg/m ³ ,	402–3578 pg/m ³	[84]
tree bark	North America	nd–24 ng/g lipid	nd–100 ng/g lipid	[83]
sewage sludge	12 countries		nd–220 nd/g dw	[91]
sewage sludge	Sweden		100 ng/g dw	[10]
sediment	Western Scheldt		24 ng/g dw	[10]
sediment	Dongjiang River, South China		23–431 ng/g dw	[29]
sediment core	Lake Michigan	0.1–<10 ng/g		
biotic samples				
fish	Lake Winnipeg	0.13–1.96 ng/g lipid	nd–3.3 ng/d lipid	[14]
archived fish	Lake Ontario	0.6–2.6 ng/g lipid		[85]
fish	Qingyuan, South China	nd–0.15 ng/g lipid		[84]
water bird	Qingyuan, South China	0.07–2.41 ng/g lipid	9.6–124 ng/g lipid	
glaucous gull egg	Faroe Islands	<0.02–0.17 ng/g ww		[90]
glaucous gull egg	Norwegian Arctic	<MLOQ–0.96 ng/g ww		[87]
herring gull egg	Great Lakes	<0.06–0.20 ng/g w w	nd–288 ng/g ww	[15]
water bird	Yellow River Estuary		nd–2.4 ng/g lp	[93]
water bird	Qingyuan, South China		nd–800 ng/g	[92]
giant panda, red panda	China		nd–863 ng/g lp	[94]

the metabolism, tissue disposition, and excretion of BTBPE in male Sprague-Dawley rats. A limited gastrointestinal absorption and high biotransformation were found, and as a consequence, leading to low residual levels in tissues. Metabolites, characterized as monohydroxylated, monohydroxylated with debromination, were also detected but in low levels. A dietary exposure study on BTBPE in juvenile rainbow trout (*Oncorhynchus mykiss*) was performed by Tomy *et al.* [89], suggesting that this chemical has a high potential for biomagnification in aquatic food webs with a biomagnification factor of 2.3. No debrominated and hydroxylated metabolites were detected in liver extracts, suggesting the biotransformation or storage of BTBPE in the hepatic system of fish is minor.

DBDPE was first detected by Kierkegaard *et al.* [10] in Swedish sewage sludge (about 100 ng/g dry wt), in sediments from Western Scheldt (24 ng/g dry wt), and in air samples (0.6 ng/m³) from a Swedish electronics dismantling facility. Since then, DBDPE has been detected in air, indoor dust, sediments, fish, and birds around the world.

DBDPE was detected in air samples collected in five sites from the Great Lake region with a minimum average concentration of 1.0 pg/m³ and a maximum average concentration of 22 pg/m³ [11]. In air samples from an electronics recycling facility in Sweden, DBDPE was detected at concentrations ranging from 0.06 to 0.8 ng/m³. The highest concentration was found in the inhalable fraction of dust [81]. Stapleton *et al.* [13] determined DBDPE in 19 dust

samples collected in the greater Boston, MA area during 2006. DBDPE levels ranged from less than the detection (< 10 ng/g) to 10240 ng/g. The DBDPE levels in living rooms (138 ng/g) and bedrooms (153 ng/g) are one order of magnitude higher than those detected in household dust (47 ng/g) in Sweden [90]. Harrad *et al.* [82] detected DBDPE in dust from the United Kingdom cars, homes, and offices. The mean concentrations of DBDPE in homes, offices, and cars were 270, 170, and 400 ng/g, respectively.

Zhang *et al.* [29] studied the DBDPE in sediments and sediment cores from Dongjiang River which runs through Dongguan city, a largest manufacturing base of electronic products in South China. DBDPE concentrations were in the range of 23 to 431 ng/g dry wt ($n=17$) and a significant correlation was observed between DBDPE and PBDEs, indicating that the emission sources of DBDPE were similar to those of PBDEs. A rapid increase of DBDPE was found in the upper layer of sediment cores and the segments where the sharp increase of DBDPE concentration matched well with the depth where the concentration of deca-BDE declined. This result indicated that deca-BDE mixture was gradually replaced by DBDPE in this industrialized region. Ricklund *et al.* [91] conducted an international survey of DBDPE in sewage sludge samples collected from 42 wastewater treatment plants in 12 different countries, revealing that DBDPE was present in sludge from all countries with a highest concentration of 216 ng/g dry wt. This result indicated that the contaminant of DBDPE may be a worldwide concern.

Law *et al.* [14] investigated BFRs in a food web from the Lake Winnipeg (Canada). DBDPE was not detected in zooplankton, mussels, and whitefish but was found in other five species of fish. The highest concentration was found in walleye (mean of 1.0 ng/g lipid). In their study, DBDPE was found biomagnified with the highest biomagnification factor up to 9.2, indicating a potential bioaccumulation in fish species. Recently, DBDPE has been detected in water birds from an e-waste recycling region in South China [92], water birds from Yellow River Estuary, North China [93], and captive giant pandas (*Ailuropoda melanoleuca*) and red pandas (*Ailurus fulgens*) from China [94], indicating a wide contamination of DBDPE in biota. Gauthier *et al.* [15] studied non-PBDE flame retardants in the eggs of colonial populations of Great Lakes' herring gulls. DBDPE was not detected in any eggs collected prior to 1996 but showed increasing occurrence and concentrations in 2004, 2005, and /or 2006. The highest concentration of DBDPE in eggs was up to 288 ng/g wet wt and the level of DBDPE surpassed the levels of the ubiquitous BDE-209 between 1982 and 2006 at selected sites.

5 Dechlorane plus (DP)

All available DP data in abiotic and biotic matrices is shown

in Table 2. DP was first detected in environmental matrices in the Great Lake areas. Hoh *et al.* [16] reported for the first time the occurrence of DP in sediment cores from Lake Eric and Lake Michigan. The peak DP concentration in the sediment core from Lake Eric (40 ng/g dry wt) was comparable to that of BDE209 (48 ng/g dry wt), but it was 30 times lower than BDE209 concentrations (122 ng/g dry wt) in cores from Lake Michigan. A study concerning DP in sediment cores from Lake Ontario conducted by Qiu *et al.* [17] revealed that the level of DP in sediments increased rapidly starting in the mid-1970s and reached its peak concentration (310 ng/g dw) in the mid-1990s. The concentration of DP in Lake Ontario sediments exceeds that of the BFRs. Sverko *et al.* [95] reported that the DP levels in Lake Ontario (586 ng/g dw) were over 60-fold higher than those in Lake Eric (8.62 ng/g dw). A decreasing trend for DP from 1980 to 2002 was found in sediment samples from the Niagara River, suggesting a possible decrease in production or the reduction of free DP released into the environment. The highest DP concentration (averaged 7590 ng/g dw) was reported in sediments from a reservoir contaminated by e-waste in South China [96]. This concentration was more than one order of magnitude higher than the highest concentration reported in sediments (586 ng/g dw) from Lake Ontario, which implies that e-waste was one of the major sources of DP in the environment. With regard to the stereoisomer profile of DP in sediments, the average syn-DP fractional abundance in sediments of the lower Great Lakes was less than that in the commercial DP products [95] and the anti-DP fractional abundance in sediment cores from the Ontario Lake increased with the depth [17]. These results suggested that the anti-isomer is more persistent than the syn-isomer in sediments.

DP was first detected in air samples in the Great Lake region by Hoh *et al.* [16]. The atmospheric DP concentrations are higher in the eastern Great Lake sites (Sturgeon Point, New York, and Cleveland, Ohio) than the western Great Lake sites (Eagle Harbor, Michigan, Chicago, Illinois, and Sleeping Bear Dunes, Michigan) and the highest concentration was up to 490 pg/m³. In another study DP was detected in the atmosphere near the Great Lakes, and DP concentrations in the five sites were in the range of 0.8 to 20 pg/m³ [12]. Ren *et al.* [19] measured DP in air samples across 97 Chinese urban and rural sites. DP was detected in 51 of these sites with the mean concentration of 15.6 pg/m³ in urban centers and 3.5 pg/m³ in rural areas. Elevated urban levels were measured along the southeastern coast and in south-central China. No significant differences in stereoisomer profile of DP were found between samples in air and commercial mixture, no matter in China or the Great Lake regions.

Qiu *et al.* [97] used tree bark as a passive sampler to monitor the DP distribution in the northeastern United States. The DP concentrations ranged from 0.02 to 115 ng/g bark and most of the DP concentrations in tree bark were

Table 2 Concentrations of DP in abiotic and biotic matrices

Samples	Location	Concentration	Ref.
air	Great Lakes	<490 pg/m ³	[16]
atmosphere	Great Lakes	0.8–20 pg/m ³	[12]
air	China	<0.8–66 pg/m ³	[19]
residential indoor dust	Ottawa, Canada	2.3–182 ng/g	[98]
tree bark	Northeastern United States	0.025–115 ng/g bark	[97]
tree bark	Halle, Germany	0.032 ng/g bark	
tree bark	Trieste, Italy	0.067 ng/g bark	
tree bark	Hanam, South Korea	1.4 ng/g bark	
tree bark	Shenzhen, China	0.30 ng/g bark	
tree bark	Hangzhou, China	0.23 ng/g bark	
tree bark	Tianjin, China	0.18 ng/g bark	
surface sediment	Lake Eric	0.061–8.62 ng/g	[95]
surface sediment	Lake Ontario	2.23–586 ng/g	
sediment	A pool in South China	7590 ng/g dw	[96]
archived fish	Lake Eric	0.14–0.91	[16]
fish	Lake Winnipeg	0.037–0.816 ng/g lipid	[102]
aquatic species	Lake Ontario	0.015–4.41 ng/g lipid	
lake trout	Lake Ontario	2.3–7.2 ng/g lipid	[95]
aquatic species	A pool in South China	19.1–9630 ng/g lipid	[96]
herring gull eggs	Laurentian Great Lakes	<15 ng/g ww	[100]
human serum	Guiyu, South China	7.8 to 465 ng/g lipid	[101]
human serum	Haojiang, South China	0.98 to 50.5 ng/g lipid	

much higher than those of BFR, such as PBDEs, TBE, and DBDPE. The highest concentration of DP was found in the city of Niagara Fall, decreasing rapidly with distance from the potential sources. Qiu *et al.* also used tree bark to investigate DP pollution in Canada, Europe and Asia. In Germany and Italy, DP concentrations were similar to the lowest concentrations measured in bark from the northeastern United States, but DP concentrations in China and Korea were nearly one order of magnitude higher than the lowest level in the northeastern United States. This implied that there may be Asia-specific sources of DP in the environment. In addition, no stereoselective DP enrichment was found in tree bark samples from the northeastern United States. Combining the observation in air samples from the Great Lake region and China, we concluded that the two isomers have similar atmospheric persistence.

DP was detected in the residential indoor dust ($n = 69$) collected in the city of Ottawa at concentrations ranging from 2.3 to 182 ng/g [98]. The presence of DP in indoor dust indicated that dust may play a role in human exposure to this compound. The large variation of the ratios of syn- to anti- DP isomers in the dust samples suggests a complex degradation process of DP in the environment.

Hoh *et al.* [16] reported DP in archived fish from Lake Erie. The DP levels ranged from 0.14 to 0.91 ng/g lipid. Tomy *et al.* [18] studied DP in archived food web samples from Lake Winnipeg and Lake Ontario. The mean DP concentrations in Lake Winnipeg and Lake Ontario were in the range of 0.035 to 0.82 ng/g lipid and 0.015 to 4.41 ng/g lipid, respectively. Ismail *et al.* [85] reported DP at concentrations ranging from 2.3 to 7.2 ng/g lipid in lake trout in Lake Ontario. DP concentrations were found to decrease significantly during the period from 1979 to 2004, which is in line with the study of Sverko *et al.* [95] Recently, Wu *et al.* [96] investigated DP in aquatic species including six fish species and one reptile, water snake, in a highly contaminated reservoir with e-waste in South China. High DP concentrations (19.1–9630) were measured in these samples, which were one to four orders of magnitude higher than those in the Great Lake region. In the archived fish samples from Lake Erie [16] and aquatic species in the contaminated site in South China [96], an enrichment of syn-DP was observed, and the fraction of syn-DP was found to further increase upon moving up the trophic levels in the aquatic food web. However, in Lake Winnipeg, syn-DP was exclusively found in lower trophic level organisms while the anti-DP

was dominant in higher trophic level organisms [18]. All these results suggested a stereoselective enrichment of syn-DP or anti-DP isomer in fish.

DP has also been detected in birds in the Great Lake regions. Gauthier *et al.* [99, 100] investigated DP in the eggs of herring gulls (*Larus argentatus*) taken from the Laurentian Great Lakes over the period from 1982 to 2006. The DP concentrations in eggs were generally less than 15 ng/g wet wt and they were generally high post mid-1990s. The mean fraction of anti-DP (0.69) in the herring gull egg was very similar to that in commercial products (0.75–0.77), indicating no stereoselective enrichment of syn-DP or anti-DP isomer in herring gull eggs.

Ren *et al.* [101] determined DP in the serum samples collected from electronics dismantling workers in an e-waste site (Guiyu) in South China and a nearby region of the e-waste site (Haojiang). DP was detected in all serum samples with concentration ranging from 7.8 to 465 ng/g lipid in Guiyu ($n=20$) and 0.98 to 50.5 ng/g lipid in Haojiang ($n=20$). The average anti-DP fractional abundance was 0.58 and 0.64 for Guiyu and Haojiang, respectively. Tentatively identified dechlorination products were found in 19/40 serum samples. These results suggested DP could bioaccumulate in a stereoselective manner, and undergo dechlorination metabolism.

Studies on the bioaccumulation and trophic transfer of DP in organisms are limited. A recent diet-exposure study using juvenile rainbow trout demonstrated that the syn-DP has a higher assimilation efficiency and a lower depuration rate relative to anti-DP [15]. This could be used to explain why syn-DP was enriched in most fish samples. Two studies reported the trophic transfer of DP isomers in food web. Tomy *et al.* [102] reported the bioaccumulation behavior of DP in Lake Winnipeg and Lake Ontario food web. The anti-DP was observed to biomagnify with a TMF of 2.5 while the syn-DP was diluted through the aquatic food web from Lake Winnipeg. In the food web from Lake Ontario, no statistically significant TMFs were found for either isomer. Wu *et al.* [96] found that both syn-DP and anti-DP were significantly biomagnified with a TMF of 11.3 and 6.6, respectively, in the food web from a highly contaminated reservoir with e-waste in South China. The trophic magnification potentials of the DP isomers were generally comparable to or lower than those of the highly recalcitrant PCB congeners, but were 2–3 times greater than those of PBDE congeners.

6 Concluding remarks

As the highest production volume BFRs, TBBPA has been less studied compared with PBDEs and HBCD and fewer data are available for environmental assessments. The low level found in the environment and humans is mainly be-

cause of its use as a reactive flame retardant, which makes it problematic to release from products. Most of the data regarding TBBPA in the environment came from Europe in which the usage volume of TBBPA was much lower than that in America and Asia. Fewer data of TBBPA are reported in Asia, the major TBBPA user. The high levels and rapid increasing trend of TBBPA in sediments from electronics manufacturing and assembly centres in South China suggested the worst case scenarios for possible environmental impacts of TBBP-A would be found in regions where TBBP-A is manufactured, transported and used. Therefore, more work is required to focus on the environmental fate and the human exposure to TBBPA around production or usage sites, especially in Asia, in the future. There are two chemical reactive hydroxyl groups in TBBPA, which readily lead to chemical or biological interactions. The high sequestration of TBBPA in sediments and the fact that the derivatives of TBBPA rather than the parent compound were detected in mussel and bird eggs support this hypothesis. Thus, research of the possibility of degradation of TBBPA in sediments and soil, biotransformation and the possible pathways of TBBPA as well as the factors affecting these processes is urgently needed.

In the recent three to four years, data concerning HBCD in the environment and humans has increased rapidly. These data cover a wide range of geographic areas including Europe, America, and Asia and more information on diastereoisomer- and enantiomer-specific profiles of HBCD was provided in abiotic and biotic samples. Although a few studies reported HBCD in the environment and humans in Asia and America, the data in these two regions remains limited and no data is available for South America, Africa, Australia and New Zealand. More studies on these regions are encouraged. The time trend studies on HBCD in biotic and human tissue samples indicated an increased trend. This trend may be confirmed by abiotic samples (sediment cores). Compared with aquatic environment, the data of HBCD in terrestrial environment is scarce. A big breakthrough concerning HBCD is the detection of metabolites of HBCD in biota samples.

As for “novel” flame retardants, our review shows that there are considerable data gaps in previous reports. Most of the studies concerning DP, BTBPE, and DBDPE concentrated in the Great Lake regions and a few were conducted in South China. No data from other regions is available. The geographic distribution, time trend, bioavailability, human exposure, and possible degradation in abiotic and biotic matrices are not clear. More studies concerning these items are required. Analytical instruments with higher sensitivity need to be employed for analysis of higher brominated flame retardants such as DBDPE, and extraction methods need to be improved to allow greater efficiency of DBDPE recovery from environmental media, which could significantly improve the environmental fate studies. Research

may be oriented towards these chemicals' potential bioaccumulation and toxicity.

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