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A data fusion algorithm based study on the presence of polybrominated diphenyl ethers (PBDEs) and novel brominated flame retardants (NBFRs) in copper laminate sorting residues

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ABSTRACT

The sorting residue of copper clad laminate stacked on site contains polybrominated diphenyl ethers (PBDEs) and novel brominated flame retardants (NBFRs). If not properly treated, they will be discarded into the surroundings and cause secondary pollution. The PBDEs and several NBFRs were detected in the sorting residue of copper clad laminate (SRCCL) of the storage yard. The ∑9PBDEs and ∑5NBFRs concentrations ranged from 2.71 to 122.83mg/kg. Different storage yards displayed three composition patterns of PBDEs, indicating that their sources were different, with domestic and imported ones. All results indicate that untreated SRCCL dumping sites are an important source of PBDEs and their emissions.

Keywords: Bromodiphenyl Ether, Non-brominated Flame Retardants, SRCCL, Non-methane Total Hydrocarbons, Polychlorinated Biphenyls

1. Introduction

In recent years, with the rapid development of synthetic materials, synthetic plastics, rubbers, fibers are more and more used in various sectors of the national economy and people's production and life, but at the same time, the fires caused by the ignition of polymers are also greatly increased, so the importance of brominated flame retardants and flame retardant materials is no longer in doubt [1-4]. From the current production of brominated flame retardants in China, both in terms of variety and quantity, there is a greater development than in previous years, especially polybrominated diphenyl ethers (PBDEs) and new brominated flame retardants (NBFRs), on the one hand, after China's

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accession to the WTO, there are strict requirements for flame retardant performance in exported products, especially plastic products for electrical and electronic products, which are required to meet the internationally recognized flame retardant standards [5-8]. On the other hand, the Fire Protection Law has been implemented in China, especially with the formulation of the standard of the Ministry of Public Security, "Requirements for Combustion Performance and Labeling of Flame-Retardant Products in Public Places", which will further promote the production of brominated flame retardants to develop rapidly [9-11].

PBDEs are a new type of persistent organic pollutants (POPs) due to their high flame retardant efficiency, good stability and low cost. Therefore, they are often used as flame retardants to reduce the frequency of fire and the degree of harm, and are widely used in petroleum, textiles, plastic products, building materials, transportation equipment and electronic products [12-15]. Brominated flame retardants (BFRs) are a class of brominated organic compounds with good stability, low price, and good flame retardant efficiency, which are often added to furniture, carpets, building materials, textiles, and electronic products to reduce the occurrence of fire accidents, and are closely related to daily life [16-19]. Currently, it is believed that humans are exposed to brominated flame retardants in three main ways, i.e., dust ingestion, food ingestion, and dermal absorption [20-21]. Many studies have confirmed that traditional brominated flame retardants (BFRs) have adverse effects on human health at low exposure levels, so they have been banned and phased out globally, and a variety of new brominated flame retardants (BFRs) have been developed and are now widely used [22-25].

This study first detects PBDEs and several novel non-BFRs' concentrations in SRCCL from the yard. Secondly, the pollution gas emission results during the disassembly of WCCLs residue are verified through PUF adsorption measurement. This result will help investigate the sources of SRCCL pollution and subsequent pollution prevention and control.

2. Materials and methods

These sampling sites are in a small village in Qingyuan City, Guangdong Province, which is a copper recovery area in southern China (Figure 1). The study collects SRCCL from five different stacking points. All stacking points are stacked with tons of SRCCL. Improper handling can cause secondary pollution. They are named Banchong Landfill (BCLF), Banchong Prison (BCJL), Shaxi Station (SXST), Yunlu Village (YLVL), and Sihe Village (SHVL). All samples are packed in polyethylene zipper bags, delivered to the lab, and reserved at 20oC.

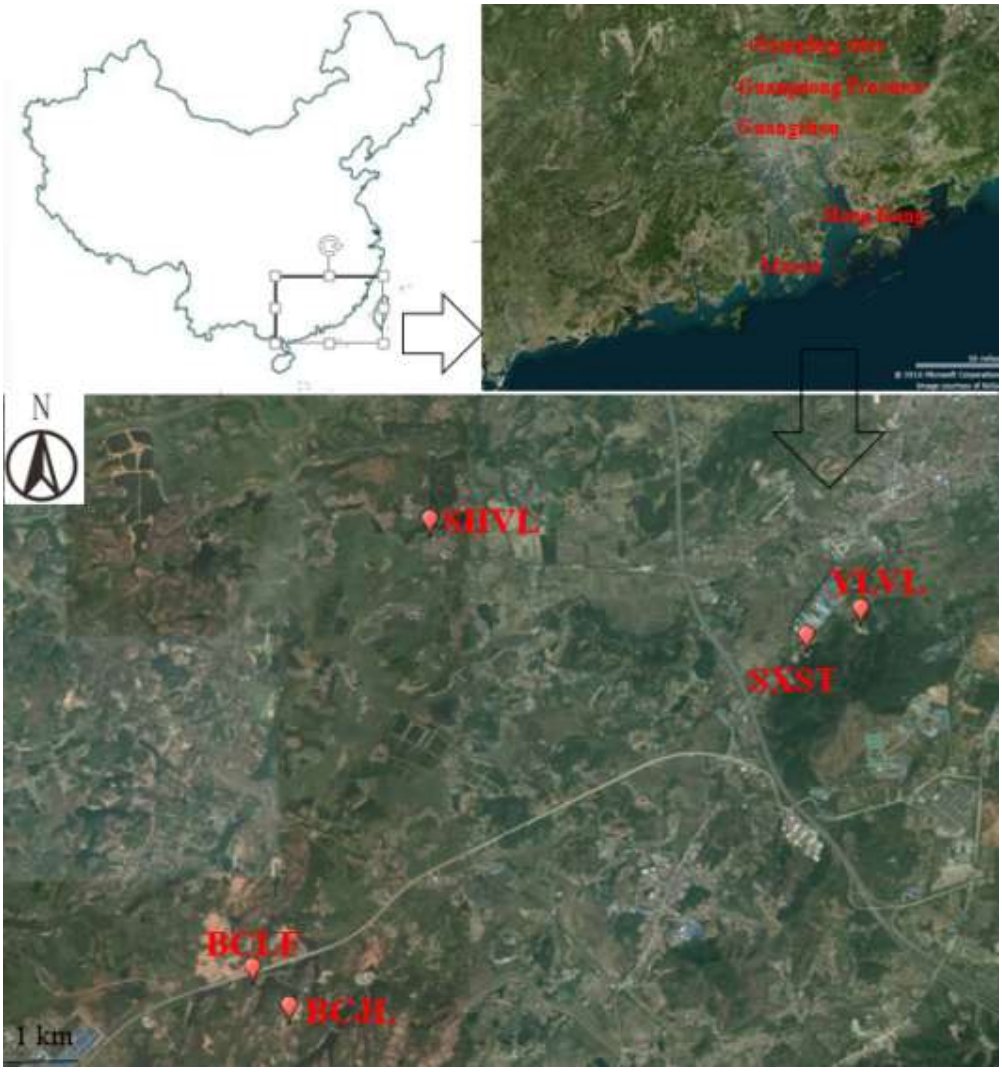

Fig. 1. Sampling site.

2.1. Chemicals and materials

PBDEs and five NBFR standard substances are sourced from Sigma Aldrich in the United States. All solvents utilized in extraction and purification are HPLC grade. Neutral silicone (MERCK, Germany), aluminum oxide (MERCK, Germany), and anhydrous sodium sulfate (ENOX, China) are baked at 450°C for 12 hours and sealed for storage. All glassware utilized is pre soaked overnight in RBS detergent, rinsed 3 cycles using tap water, then rinsed with distillation water and heated in the furnace (450°C for 6 hours) to remove impurities and target compounds.

2.2. Sample extraction and analysis

This sampling adopts a three-cycle method of manual assembly line disassembly, circuit board and CCL baking board, and component processing. The circuit board and CCL baking board are the most crucial components. During sampling, each sampling personnel should be familiar with the sampling process and work independently. Each workbench is equipped with a dust collector to separate gases and prevent pollutants from damaging human health. Figure 2 shows the workflow of the entire CCL heating disassembly.

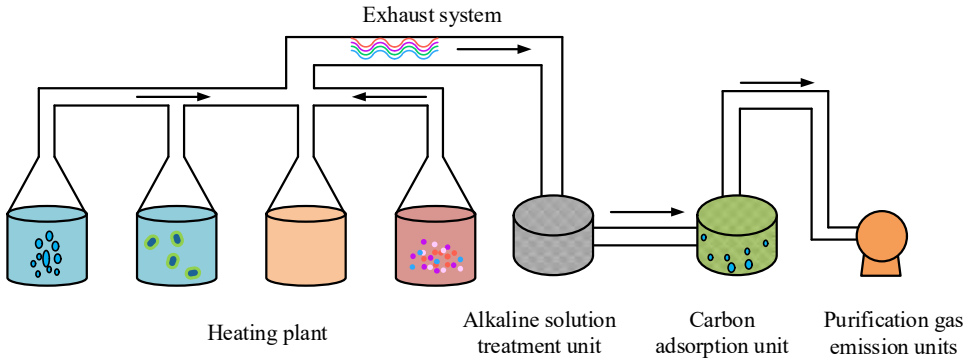

Fig. 2. Schematic diagram of the workflow for heating and disassembling copper clad plates.

Polyurethane foam (PUF) is utilized to collect PBDEs and NBFRs in the residue in real-time. This material has the characteristic of efficient adsorption of organic pollutants. Therefore, PUF is chosen as the most suitable sampling medium. The sampling device includes a high flow air sampler connected to a PUF collector. The sampling collector is widely distributed in every corner of the heating laboratory. The sampling time is set to 72 hours. After sampling, take out the foam block in the PUF collector and freeze it immediately to prevent the volatilization of pollutants. After thawing the frozen PUF sample, soak and extract it with a high-efficiency solvent, such as dichloromethane or ethyl acetate. The extraction process lasts for 24 hours to ensure that the pollutants are completely transferred to the solvent. The extracted solution is concentrated to 1mL using a rotary evaporator and then transferred to a glass vial. Use a silicone column for purification. The silicone column is composed of sewage sodium sulfate, acidic, alkaline, and neutral silicone, as well as neutral alumina and degreased cotton stacked from top to bottom to remove impurities in the sample. The purified solution is eluted with n-hexane. The eluent is concentrated again to 0.5mL using a rotary evaporator. The PUF concentration is represented by equation (1).

$$C_{PUF} = K_{PUF-A} C_A \left\{ -\exp\left[-\left(A_{PUF} / V_{PUF} \right) \cdot \left(k_A / K_{PUF-A} \right) \cdot t \right] \right\} \quad (1)$$

In equation (1), C_{PUF} refers to the pollutant concentration in the PUF membrane. K_{PUF-A} means PUF pollutant's air distribution coefficient. A_{PUF} means PUF's membrane area. V_{PUF} means the PUF membrane's volume. C_A means the pollutant concentration in the gas phase. k_A means a transmission coefficient. t means sampling time. Equation (2) is the relationship between pollutants' K_{PUF-A} and their n-octanol air distribution coefficient K_{OA}.

$$\log\left(K_{PUF-A} \right) = 0.6366 \log K_{OA} - 3.1774 \quad (2)$$

In equation (2), K_{OA} represents the octanol air distribution coefficient. Each sample is composed of three parallel sub samples. After freeze-drying, the sample is ground using a drug grinder. Add 20 milligrams of polychlorinated biphenyl 30 and polychlorinated biphenyl 198 as alternative standards to the dry sample (approximately 20 grams, mixed with anhydrous sodium sulfate). Perform Soxhlet extraction with a compound of dichloromethane (DCM) or acetone and n-hexane (1:3) for 48 hours, respectively. Exchange the extract solvent into n-hexane. Reduce the volume to approximately 1mL using a rotary evaporator. Then, clean from top to bottom on an acidic silica gel alumina column with a diameter of 7mm.The column contains anhydrous sodium sulfate (1cm), 50% silica gel sulfate (3cm), neutral silica gel (1cm, 3% deactivated), and neutral alumina (3cm, 3% deactivated). The chromatographic column was eluted with 40mL of hexane/DCM (1:1) to obtain a polychlorinated biphenyl fraction. Add 6g Bio Beads S-X3 to a 15cm glass column (inner diameter 2cm) to create a GPC

column. Load FR and elute with 45mL of DCM/n-hexane (1:1). Discard the first 15mL, collect the last 30mL fraction containing polychlorinated biphenyls, and concentrate under mild N2 flow. The PUF disk extract is purified using only multi-layer columns and subjected to N2 concentration. Before instrument analysis, inject 10ng 13C labelled PCB 141 into each sample as an internal standard.

2.3. Sample extraction and analysis

Table 1 shows the used instruments.

Table 1. Experimental key instruments

Instrument Name	Model Number	Manufacturer
Precision balance	FA2104 (d=0.001g)	Shanghai Precision Instrument Co.
Multi-function nitrogen blower	BCY-24	Beijing Bochuang Instrument and Equipment Co.
Continuous extractor	SXZ-06	Guangzhou Hengxin Instrument Co.
Vacuum evaporator	RE-2000A	Zhengzhou Kewei Instrument Co.
Solid phase extraction system	SPE-300	Beijing Jinpu Instrument Co.
Circulating water vacuum pumps	SHZ-95B	Chengdu Jingcheng Technology Co.
Gas chromatograph	7890A/5975C	Agilent Technologies, Inc.

When using gas chromatography-mass spectrometry, a capillary chromatography column with a length of 16m, an inner diameter of 0.26mm, and a film thickness of 0.11μm (model: HP-5MS, Thermo Fisher Scientific, USA) is utilized. Ten PBDE homologues (including BDE28, 47, 99, 100, 153, 154, 183, 209, 207, and 208) and six novel non-BFRs (TBPH, PBEB, HBB, TBE, DBDPE, and BTBPE) are determined using gas chromatography-chemical ionization mass spectrometry (GC-CI-MS, Thermo Trace 1300 in combination with ISQ7000) equipped with a capillary chromatography column with a length of 16 meters, an inner diameter of 0.26 millimeters, and a film thickness of 0.11 micrometers (model: HP-5MS, Thermo Fisher Scientific, USA). Firstly, inject 2μL of sample in a split flow mode, using high-purity helium gas for circulation. The flow rate of high-purity helium (purity 99.999%, provided by Harbin Xinrui Gas Co., Ltd.) is tentatively set at 1.2mL/min. In addition, methane, as a chemically ionized gas, has a transmission line temperature set at 290°C and an ion source temperature maintained at 240°C. The initial temperature of the gas chromatography oven is set to 120°C, maintained for 2min, then enhanced to 210°C, maintained for 2 minutes, and increased to 320°C for 15min.

2.4. Quality assurance/quality control

The gas chromatography-mass spectrometry's stability is verified by repeating the analysis of the standard solution daily. The instrument's Standard Deviation (SD) ranges from 3% to 12%. The Instrument Detection Limit (IDL) is three times that of SD, with a range of 0.09 to 0.36pg. The SD of blank samples ranges from 0.6% to 7.4%. IDL is calculated based on 3.36 times SD plus the blank sample's average value, ranging from 1.1 to 28.6pg/g, depending on the homolog. In addition, the instrument is calibrated daily using the standard solution's analytical results to ensure its accuracy and repeatability. Measurement errors are reduced by regularly checking and maintaining the status of the instruments. The blank samples analysis is also utilized to evaluate background interference and ensure the reliability of the results. To ensure data quality, the instrument is standardized before and after each experiment. All relevant data are recorded.

3. Result and discussion

3.1. Concentration of polybrominated diphenyl ethers

In Table 2 and Table 3, at different SRCCL stacking points, the concentration of Σ9PBDEs ranged from

0.26mg/kg to 119.15mg/kg. These results were comparable to other reports. Except SXST storage yard, the ∑9PBDEs PBDEs was higher compared to Spanish sediment (0.002 to 0.132mg/kg), soil and sediment of the c (PRD), soil and dust of China's electronic waste disposal area. However, the concentration of sewage sludge in PRD (1.277 to 64.599mg/kg), 30 households in the UK, 18 offices, and 20 cars (260, 31, and 340mg/kg) was relatively high. Eljarat et al. collected 13 different samples in Spain. The content of BDE 209 ranged from 0.002 to 0.132mg/kg. According to PBDEs' average concentration derived from 30 households, 18 working area, and 20 vehicles was 260, 31, and 340mg ΣPBDEs/kg, respectively. BDE's concentrations 47, 99, 100, and 154 in vehicles were significantly higher compared to residential and office areas. In the study of PRD and electronic waste treatment area in China, PBDEs, especially BDE209, were mainly contained in sediments and sewage sludge. This was consistent with the fact that the main industry in PRD was the manufacturing of electronic and electrical products.

Table 2. Concentration of polybrominated diphenyl ethers at different stacking sites (mg/kg)

BDE congener Sampling sites	BCLF	BCJL	SXST	YLVL	SHVL
T3BDE BDE28+BDE35	0.75	0.06	15.07	0.05	0.04
T4BDE BDE47	2.67	0.10	51.64	0.13	0.11
P5BDE BDE99+BDE100	1.58	0.07	36.83	0.10	0.11
H6BDE BDE153+BDE154	0.74	0.02	13.29	0.04	0.5
H7BDE BDE183	0.09	N.D.	1.10	0.01	0.03
D10BDE BDE209	0.08	0.01	1.22	0.07	1.47
∑9PBDEs	5.91	0.26	119.15	0.4	2.26

Table 3. Comparison of polybrominated diphenyl ether content in various samples (mg/kg)

BDE congener	TYPE	Concentrations	Location	Reference
D10BDE BDE209	Sewage sludge	1.277 to 64.599 dw	PRD, South China	Shi et al., 2009
D10BDE BDE209	Sediment	0.0327 to 2.015 dw		
D10BDE BDE209	Soil	0.0398 to 0.0952 dry wt		
D10BDE BDE209	Dust	1.736 to 4.408 dry wt	e-waste processing area, southern China	Shi et al., 2009
D10BDE BDE209	Soil	0.0215 to 0.179 dry wt		
ΣPBDEs	Dust	260, 31, and 340	30 homes,18 offices, and 20 cars in UK	Harrad et al., 2008
D10BDE BDE209	Sediment	0.002 to 0.132 dw	Spain	Eljarat et al., 2004

In Figure 3 and Table 4, natural adsorption of PBDEs from the CCL sorting residue was carried out by heating and dismantling the laboratory using PUF. During half a month of monitoring, BDE-28, BDE-154, and BDE-183's concentration changes were relatively small. BDE-47 and BDE-209's concentration changes were the largest. The concentration of BDE-209 increased from 0.013mg/kg on the second day to 0.018mg/kg on the 16th day, indicating its presence in the environment and effective adsorption by PUF. The concentration of BDE-47 increased from 0.015mg/kg on the second day to 0.023mg/kg on the 16th day, indicating a significant increase in adsorption capacity. BDE-47 was high in the air and was effectively captured by PUF. These data indicated that PUF as a sampling medium effectively captured different types of PBDEs. The longer the sampling time, the higher the concentration of PBDEs adsorbed. This result was consistent with the research results on PBDEs and NBFRs' concentrations in CCL sorting residues. This further confirmed that the SRCCL landfill was an important source of PBDEs and NBFRs emissions. Untreated residues would continue to release these pollutants, causing environmental pollution. Furthermore, over time, the PUF air distribution coefficients of each PBDE increased, indicating an enhanced adsorption capacity of pollutants in the PUF. BDE-47 and BDE-209 had the highest PUF-air distribution coefficients, reaching 5000 and 7000, respectively, indicating that these pollutants were more easily adsorbed by PUF. On the other hand, as time increased, the pollutants concentration in the gas phase also increased. This corresponded to the pollutants concentration in the PUF membrane, indicating that PBDEs' concentration in the environment was continuously increasing over time.

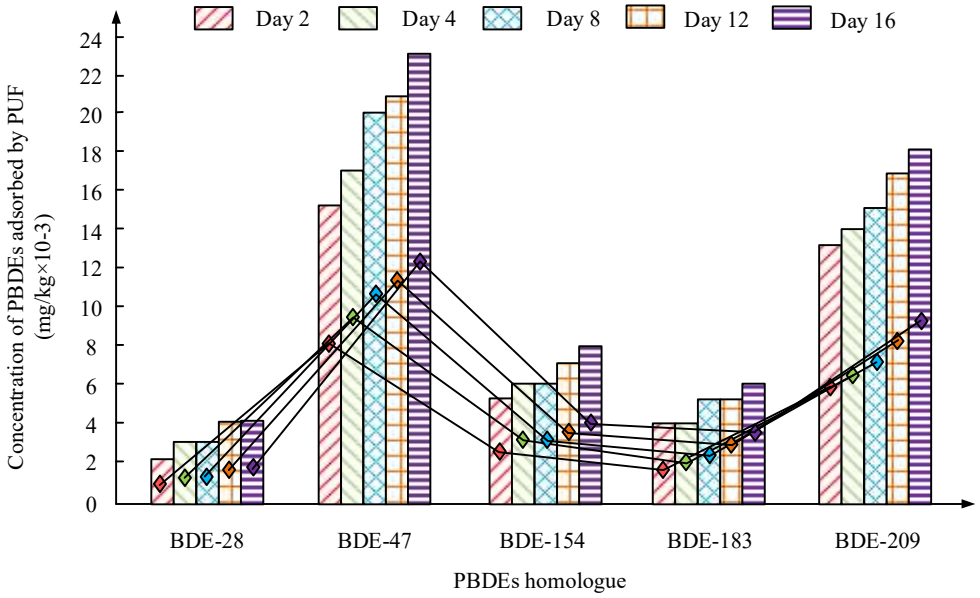

Fig. 3. Results of concentration changes in PBDEs adsorbed by PUF

Table 4. Comparison of PBDEs concentration in gas phase (mg/kg)

PUF homologs	Day	$\log K_{OA}$	$\log(K_{PUF-A})$	C_{PUF}	C_A
BDE-28	Day2	1000	3.000	0.002	0.001
	Day 4	1200	3.079	0.003	0.001
	Day 8	1500	3.176	0.003	0.001
	Day 12	1800	3.255	0.004	0.002
	Day 16	2000	3.301	0.004	0.002
BDE-47	Day 2	3000	3.477	0.015	0.005
	Day 4	3500	3.544	0.017	0.006
	Day 8	4000	3.602	0.020	0.007
	Day 12	4500	3.653	0.021	0.008
	Day 16	5000	3.699	0.023	0.009
BDE-154	Day 2	2000	3.301	0.005	0.002
	Day 4	2200	3.342	0.006	0.002
	Day 8	2500	3.398	0.006	0.002
	Day 12	2800	3.447	0.007	0.003
	Day 16	3000	3.477	0.008	0.003
BDE-183	Day 2	1500	3.176	0.004	0.002
	Day 4	1700	3.231	0.004	0.002
	Day 8	2000	3.301	0.005	0.003
	Day 12	2300	3.362	0.005	0.003
	Day 16	2500	3.398	0.006	0.004
BDE-209	Day 2	5000	3.699	0.013	0.004
	Day 4	5500	3.742	0.014	0.005
	Day 8	6000	3.778	0.015	0.006
	Day 12	6500	3.813	0.017	0.007
	Day 16	7000	3.845	0.018	0.008

3.2. NBFRs concentrations

In Table 5, the concentration range of ∑5NBFRs in different SRCCL stacking sites was between 2.45mg/kg and 6.52mg/kg. The concentration of ∑5NBFRs varied significantly among these sites, indicating that each site had different pollution. Especially, DBDPE accounted for the main component in ∑5NBFRs, with concentrations ranging from 1.17mg/kg to 6.08mg/kg, accounting for 31.79% to 93.25% of the total ∑5NBFRs. These data indicated that DBDPE was a major pollutant in the SRCCL site. In Table 6, this study had higher DBDPE concentrations than existed works. Sewage sludge is often utilized as an indicator for releasing hydrophobic pollutants into the environment in the technical circle. Compared with the sewage sludge collected in Shi's study, DBDPE was higher, indicating that SRCCL might be a potential source of pollution for NBFR.

Table 5. NBFRs concentrations at different sampling sites (mg/kg)

NBFRs congener Sampling site	BCLF	BCJL	SXST	YLVL	SHVL
TBPH	0.33	0.05	0.11	0.37	0.45
PBEB	0.04	N.D.	0.83	N.D.	0.02
HBB	0.02	0.01	1.47	0.06	0.07
BTBPE	0.02	N.D.	0.10	0.01	0.32
DBDPE	3.18	2.39	1.17	6.08	5.22
∑5NBFRs	3.59	2.45	3.68	6.52	6.08

Table 6. Comparison of DBDPE content in various samples (mg/kg)

NBFRs congener	TYPE	Concentrations	Location	Reference
DBDPE	Dust	0.27, 0.17, and 0.4	UK homes, offices, and cars	Harrad et al., 2008
DBDPE	Sewage sludge	0.266 to 1.995 (median 1.183) dw	PRD, South China	Shi et al., 2009
DBDPE	Sediment	0.0388 to 0.364 (mean 0.247) dw		
DBDPE	Soil	0.0281 dry wt		

The correlation analysis of Figure 4 and Table 7 reflected the distribution and migration patterns of NBFRs pollutants in both solid and gaseous environments. The PUF air distribution coefficients of TBPH, PBEB, HBB, and BTBPE were not significantly different, all ranging from 3000 to 5500. The concentration of all NBFRs in the PUF membrane increased over time, indicating that these pollutants were continuously released into the environment and effectively captured by the PUF membrane. The concentration changes in the gas phase also showed a similar trend, further confirming the sustained release and accumulation of these pollutants. In addition, compared to TBPH, PBEB, HBB, and BTBPE, DBDPE had the highest adsorption concentration of 0.065mg/kg in the concentration changes of NBFRs adsorbed by PUF. The concentration in its gas phase increased from 0.065mg/kg to 0.085mg/kg. Although DBDPE had low volatility, its persistence in the environment and high initial concentration led to a significant increase in its concentration. The high distribution coefficient and high gas-phase concentration of DBDPE indicated its strong persistence and migration ability in the environment, making it a potential source of environmental pollution.

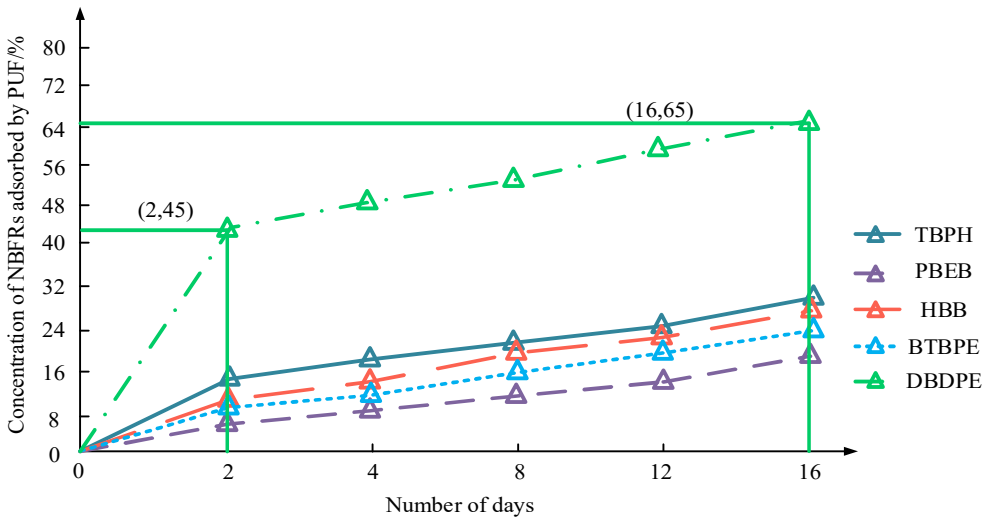

Fig. 4. Results of concentration changes in UF adsorbed NBFRs

Table 7. Comparison of NBFRs concentration in gas phase (mg/kg)

NBFRs	Day	$\log K_{OA}$	$\log(K_{PUF-A})$	C_{PUF}	C_A
TBPH	Day 2	4500	3.653	0.015	0.015
	Day 4	4700	3.672	0.018	0.017
	Day 8	5000	3.699	0.021	0.020
	Day 12	5300	3.724	0.025	0.022
	Day 16	5500	3.740	0.030	0.025
PBEB	Day 2	3000	3.477	0.007	0.013
	Day 4	3200	3.505	0.009	0.015
	Day 8	3500	3.544	0.012	0.017
	Day 12	3700	3.568	0.015	0.019
	Day 16	3900	3.591	0.018	0.021
HBB	Day 2	3500	3.544	0.012	0.014
	Day 4	3700	3.568	0.015	0.016
	Day 8	4000	3.602	0.019	0.019
	Day 12	4200	3.623	0.023	0.021
	Day 16	4400	3.643	0.028	0.023
BTBPE	Day 2	3200	3.505	0.010	0.013
	Day 4	3400	3.531	0.013	0.015
	Day 8	3700	3.568	0.016	0.017
	Day 12	3900	3.591	0.020	0.019
	Day 16	4100	3.613	0.025	0.022
DBDPE	Day 2	7000	3.845	0.045	0.065
	Day 4	7200	3.857	0.049	0.072
	Day 8	7500	3.875	0.053	0.076
	Day 12	7800	3.892	0.059	0.083
	Day 16	8000	3.903	0.065	0.085

3.3. PBDEs and NBFRs composition and profile analysis

Figure 5 shows the distribution of PDBEs and NBFRs homologues at different SRCCL stacking sites. The relative abundance of PBDEs and NBFRs varied and exhibited three patterns. The first mode came

from BCLF. In Mode 1, the contribution rate of T3+T4+p5BDE exceeded 50.56%, while the contribution rate of DBDPE was 32.15%. In Mode 2, regarding BCJL, YLVL, and SHVL, the contribution rate of DBDPE in ∑9 PBDEs and ∑5 NBFR was 62.59%-88.19%. On the contrary, in Mode 3 (i.e. SXST), the contribution rate of DBDPE was only 0.95%, while T3+T4+p5BDE accounted for 84.30%.

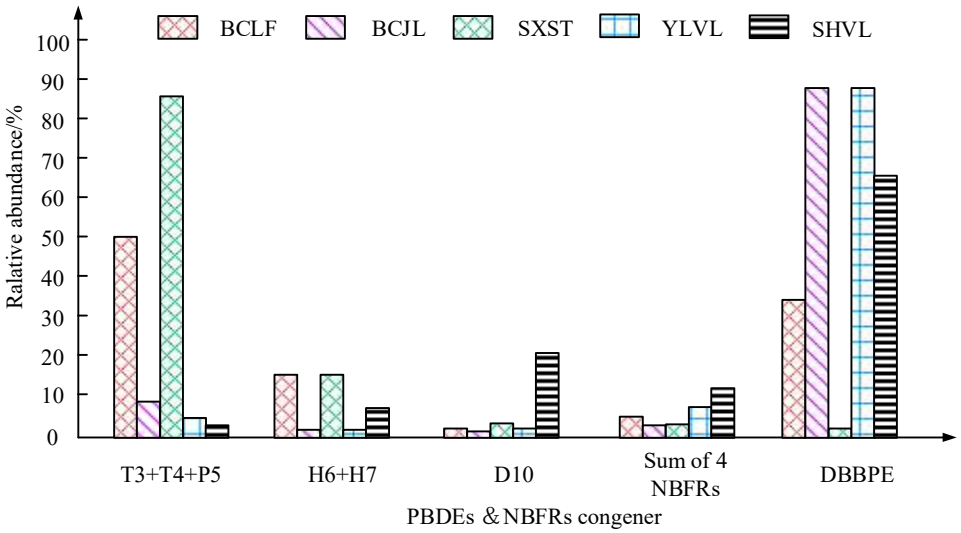

Fig. 5. PBDEs and NBFRs composition and profile analysis of different SRCCL stacking sites

There are two reasons for this difference. Firstly, previous studies confirmed that ultraviolet and sunlight could reduce the concentration of brominated PBDEs during the debromination of decabromodiphenyl ether. Ultraviolet light can convert it into tribromodiphenyl ether, while sunlight can convert it into tetrabromodiphenyl ether13. It is reasonable to believe that SXST is a relatively long stacking site, therefore decabromodiphenyl ether is debrominated to T3+T4+p5BDE, accounting for 84.30%. Secondly, the different patterns of relative abundance between PBDEs and PBDEs may indicate whether the source of electronic waste is imported or domestically produced. Therefore, the relatively high content of PBDEs in Mode 2 is due to the substitution of decabromodiphenyl ether by PBDEs abroad. As for another theory, namely BCJL, YLVL, and SHVL, the DBDPE storage yard may be imported. As for Mode 3, the SXST yard may be domestic waste, while the BCLF yard in Mode 1 may be a mixture of imported waste and domestic waste.

4. Conclusions

1) The concentration of ∑9PBDEs at different sampling sites ranges from 0.26 mg/kg to 119.15 mg/kg. At different SRCCL stacking points, the concentration of ∑5 NBFR ranges from 2.45 mg/kg to 6.52 mg/kg.
2) The content of PBDEs and BFR suggests that SRCCL may be a potential source of pollution.
3) The relative abundance of PBDEs and non BFR is different, and they exhibit three patterns. BCJL, YLVL, and SHVL storage points may come from imported waste. As for the SXST dumping site, it may be a mixture of domestic waste for a long time, while the BCLF dumping site may be a mixture of imported waste and domestic waste.
4) PUF as a sampling medium can effectively capture different types of PBDEs and NBFRs. With the extension of sampling time, the concentration of adsorbed pollutants significantly increases.
5) The persistence and migration ability of PBDEs and NBFRs in the SRCCL landfill sites are strong, which may affect a wider range of areas through atmospheric long-range drift.

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Data available with the paper

The author declares that data supporting the results of this study can be obtained in the paper. If readers need raw data files in other formats, they can request them from the corresponding authors. The original data are provided together with this article.

Conflict of Interest

The author declares that they have no conflict of interest.

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