Influence of plants on the distribution and composition of PBDEs in soils of an e-waste dismantling area: Evidence of the effect of the rhizosphere and selective bioaccumulation

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ABSTRACT

Rhizosphere effects on the distribution of PBDEs in e-waste contaminated soils were investigated. The geometric means of the PBDEs in the rhizosphere and non-rhizosphere soils were 32.6 ng/g and 12.2 ng/g, whereas the geometric means of the PBDEs in vegetable shoots and roots were 2.15 ng/g and 3.02 ng/g, respectively. PBDEs in soil at different distances from the root surface may first rise appreciably and then decrease to a non-rhizosphere level for long-term contaminated soils. Different PBDE compositions in roots and shoots indicated that PBDEs in shoots may be mainly taken up from the air. The ratios of BDE99/100 and BDE153/154 in plants and their corresponding soils were different. The bioaccumulations of BDEs 100 and 154 were much higher than those of BDEs 99 and 153, respectively. This indicated that the bioaccumulation was selective and influenced by the substitution pattern, with *ortho*-substituted isomers being more prevalent than *meta*-substituted isomers.

1. Introduction

Polybrominated diphenyl ethers (PBDEs) are a group of brominated flame retardants that are extensively used in electronic appliances and ubiquitously detected in environmental media (Wang et al., 2011b). Two major PBDE commercial products, penta- and octa-BDEs, have already been listed as persistent organic pollutants (POPs) by the Stockholm Convention and have been banned for their persistence, long-range transport, and potential toxicity to wildlife and humans (Zhang et al., 2011).

The bioavailability and bioaccumulation of POPs depend greatly on the physicochemical properties of the soil, especially for the rhizosphere soil (McCaskil et al., 1970). The rhizosphere is the narrow region of soil where the physical, chemical, and biological parameters are directly influenced by root secretions and associated soil microorganisms (Giri et al., 2005). The rhizosphere is most commonly distinguished from the bulk soil by a depletion of immobile nutrients and an elevated microbial activity based on readily available carbon derived from root exudations and other root depositions (Joner and Leyval, 2003). Rhizosphere processes, including the exudation of soluble compounds, nutrient mobilization by roots and microorganisms, and rhizosphere-mediated soil organic matter decomposition, are controlled or directly influenced by roots. Hence, the rhizosphere plays an important role in regulating organic matter decomposition. With the influence of roots, different concentrations of POPs in rhizosphere and bulk soils have also been discovered (Liste and Alexander, 2000; White et al., 2002; Joner and Leyval, 2003; Tao et al., 2004; Kaimi et al., 2006; Xu et al., 2010). However, most of the studies are conducted using pot experiments or artificial interference, while few of them are *in situ* field studies (Tao et al., 2004, 2005). Moreover, plants have demonstrated plenty of significant advantages in the dissipation of POPs. Specific plants can take up POPs from soil (Huang et al., 2010); certain plants can promote the growth and activity of pollutant-degrading bacteria via different mechanisms such as cometabolism, increased bioavailability, and improved aeration (Van Aken et al., 2004; Slater et al., 2011). In addition to absorption and uptake, plant roots can also adsorb POPs from the soil using their large specific surface area (Binet et al., 2000; Jiao et al., 2007).

Electronic-waste (e-waste) dismantling has caused serious environmental problems from the toxic contaminants released during the disposal procedures (Wu et al., 2008; Luo et al., 2011;

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Wang et al., 2011a, 2012) and is considered one of the important sources of these contaminants entering the environment (Wang et al., 2005; Bi et al., 2007; Leung et al., 2008; Chen et al., 2009). High levels of PBDEs and other contaminants have been frequently detected in the ambient environment around e-waste recycling sites, indicating severe pollution stresses and risks to the local ecosystem. Although many papers are concerned with PBDEs in environmental samples from e-waste recycling sites (Wong et al., 2007; Luo et al., 2007; Liu et al., 2008; Wu et al., 2008; Wang et al., 2011b; Tian et al., 2012), few studies have evaluated the effects of plants, especially in the rhizosphere, on the distribution or fate of PBDEs in soil, especially with respect to *in situ* field studies. The purpose of the present research is to investigate the effect of the plant rhizosphere on the distribution, composition, and bioaccumulation of PBDEs in this e-waste polluted area.

2. Materials and methods

2.1. Sampling

The sampling site is located in Longtang Town of Guangdong province, South China [23° 34′ N, 113° 0′ E]. Intensive uncontrolled e-waste dismantling operations left open burning sites scattered among agricultural fields, but cultivations of rice and vegetables are still ongoing. All samples were collected on June 7th, 2012. During the sampling, the plant was gently pulled from the soil, and the soil was mildly crushed and shaken to remove the bulk soil loosely attached to the roots. Soil strongly adhering to the roots was vigorously rubbed and collected as rhizosphere soil. Here, the "rhizosphere soil" included both rhizosphere soil and soil very close to the rhizosphere area (1–5 mm). Bulk soil located 10–20 cm from the corresponding plant was collected as "non-rhizosphere soil". The corresponding plant was also obtained, and its shoots and roots were separated. Each sample consisted of three subsamples. Plant samples were first washed with tap water and then subsequently rinsed three times with deionized water prior to storage. Soil and plant samples were freeze-dried, ground with agate mortar, and stored at −20 °C until further analysis. The sample information is listed in Table 1.
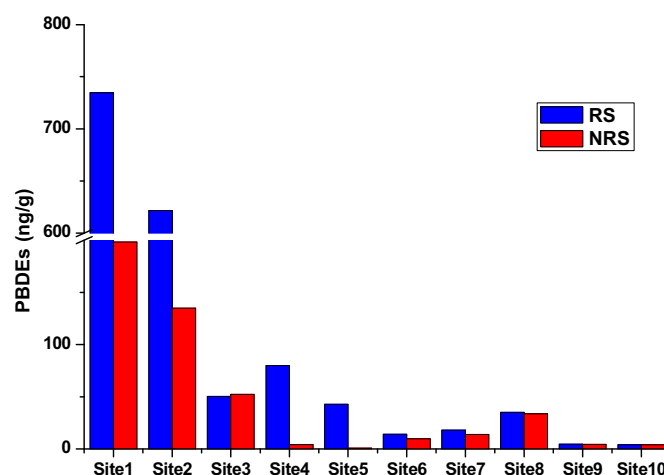
2.2. Sample extraction and cleanup

Briefly, about 10 g of a freeze-dried soil sample was Soxhlet extracted with dichloromethane (DCM) for 24 h. About 5 g of a plant sample homogenized with 5 g anhydrous sodium sulfate was Soxhlet extracted with n-hexane/acetone (3:1, v/v) for 48 h. All samples were spiked with a surrogate standard (PCB 209) before the Soxhlet extraction. The extracts were concentrated to about 0.5 mL after a solvent-exchange to hexane. The soil extract was cleaned by passing through a multi-layer column containing anhydrous Na_2SO_4, 50% (w/w) sulfuric acid silica gel, neutral silica gel (3% deactivated), and neutral alumina (3% deactivated) from top to bottom with an eluent of 20 mL hexane/DCM (1:1, v/v). The plant extract was first washed eight times with sulfuric acid and then purified with the multi-layer column. ^{13}C-PCB 141 was added as the internal standard.

2.3. Instrumental analysis

GC-ECNI-MS (Agilent GC7890 coupled with 5975C MSD) applied with a DB5-MS capillary column (30 m × 0.25 mm i.d. × 0.25 μm film thickness) was used for the determination of the seven PBDE congeners (BDEs 28, 47, 99, 100, 153, 154, and 183). The temperature program was from 110 °C (held for 5 min) to 200 °C (held for 3 min) at 15 °C/min, then to 295 °C (held for 12 min) at 5 °C/min. The GC injector temperature was maintained at 290 °C. The temperatures were set to 150 °C for the MSD source and quadrupole.

Fig. 1. Total PBDE concentrations in the rhizosphere and non-rhizosphere soils.

2.4. QA/QC

A procedural blank and a spiked blank consisting of all chemicals were run with each batch of 10 samples. The results showed no target compounds were detected in the laboratory blanks. The method detection limit (MDL) for the target PBDEs was 5.3 pg/g. The average surrogate recoveries for PCB 209 in the soil and plant samples were 80.3 ± 21.1% and 95.7 ± 13.2%, respectively. All of the results reported in the study are expressed on a dry weight basis (ng/g dry wt.) and are not corrected by the surrogate recovery ratios.

3. Results and discussion

3.1. Levels of PBDEs in the rhizosphere and non-rhizosphere soils

The total concentrations of the seven PBDE congeners (ΣPBDEs) in the rhizosphere and non-rhizosphere soils are presented in Fig. 1. ΣPBDEs in the rhizosphere soils ranged from 3.83 to 735 ng/g with a geometric mean of 32.6 ng/g, while ΣPBDEs in the non-rhizosphere soils were from 0.76 to 198 ng/g with a geometric mean of 12.2 ng/g. The highest PBDE concentrations for both soils were discovered in the samples from Site 1, which was quite close to an abandoned burning site. Meanwhile, the rhizosphere soil with the lowest PBDE concentration was from Site 10, and the non-rhizosphere soil with the lowest concentration was from Site 5.

Generally, the PBDE concentrations in the rhizosphere soils were higher than the non-rhizosphere soils, with several that were similar to the non-rhizosphere concentrations. The geometric means of the PBDE congeners in the rhizosphere soils were significantly different from those in the non-rhizosphere soils ($p = 0.006$). The ratio C_R/C_N represents the difference between the PBDE concentrations in the rhizosphere and non-rhizosphere soils. The results showed that C_R/C_N ranged from 0.96 to 56.6, with the highest value belonging to the taro (*Colocasia esculenta* L. Schott)

Table 1
Sampling information.

Site	Plant species	Longitude	Latitude	Rhizosphere soil	Non-rhizosphere soil	Root	Shoot
1	*Ipomoea batatas* L.	23.559	113.034	RS1	NRS1	R1	Sh1
2	*Solanum melongena* L.	23.56	113.033	RS2	NRS2	R2	Sh2
3	*Zea mays* L.	23.562	113.032	RS3	NRS3	R3	Sh3
4	*Oryza sativa* L.	23.588	113.037	RS4	NRS4	R4	Sh4
5	*Colocasia esculenta* L. Schott	23.574	113.01	RS5	NRS5	R5	Sh5
6	*Ipomoea aquatica* Forsk.	23.577	113.018	RS6	NRS6	R6	Sh6
7	*Vigna unguiculata* (Linn.) Walp	23.576	113.019	RS7	NRS7	R7	Sh7
8	*Lactuca sativa* L.	23.584	113.031	RS8	NRS8	R8	Sh8
9	*Arachis hypogaea* L.	23.585	113.03	RS9	NRS9	R9	Sh9
10	*Gynura cusimbua* (D. Don) S. Moore	23.554	113.027	RS10	NRS10	R10	Sh10

soil from Site 5. The sites with high PBDE concentrations showed much bigger differences (higher C_R/C_N) than the sites with low PBDE concentrations, suggesting that the rhizosphere effect is more significant in the more heavily polluted areas. However, there was no significant correlation between C_R/C_N and the PBDE concentration in the soils ($p = 0.755$ for the rhizosphere soil and 0.507 for the non-rhizosphere soil), which is mostly because of the different species of vegetation collected.

Plants can enhance the disappearance of organic pollutants either through adsorption or uptake by roots (Mueller et al., 2006) or through biodegradation or rhizoremediation (Huang et al., 2010). On the other hand, biological processes such as microbial metabolism and plant growth can also significantly increase the bioavailability of the organic contaminants (Welsh et al., 2009). Therefore, studies on the effects of plants on the hydrophobic compounds in rhizosphere and non-rhizosphere soils are not always consistent. Degradation or adsorption may vary among plant species and varieties (Wiltse et al., 1998; Liste and Alexander, 2000); the different sampling procedures of the rhizosphere soil may also lead to results that cannot be directly compared. Javorska et al. (2009) and Ma et al. (2010) demonstrated that the effects of degradation and dissipation led to PCB and PAH concentrations in the rhizosphere soils that were lower than in the bulk soils. Joner and Leyval (2003) also assumed that the PAH concentrations decreased logarithmically while moving towards the root surface. On the contrary, Liste and Alexander (2000) indicated that the rhizosphere of several plant species grown in either sterile or non-sterile soil contained appreciably more phenanthrene or pyrene than unplanted soil. They also suggested that plants can not only facilitate the transport of hydrophobic compounds towards the roots, but can also enrich these compounds in the rhizosphere. Joner and Leyval (2003) discovered that PAH levels in strongly adhering soils were appreciably higher than those in loosely adhering soils when the PAHs were at a high level, but the difference between these two soils was not statistically significant. However, when the PAH level was low, the strongly adhering soils showed significantly lower concentrations than the loosely adhering soils. Welsh et al. (2009) suggested that plant growth can lead to more available PBDEs, especially in long-term contaminated soils. The present results also showed that the amount of PBDEs in the rhizosphere soils was higher than in the non-rhizosphere soils, especially in the highly contaminated areas.

3.2. A hypothetical mechanism of the rhizosphere effects based on the current results

According to previous studies, the effects of the roots on the amounts of compound surrounding the roots are bidirectional with coexisting enhancement and dissipation. Thus, soils at the different distances from the root surface can be divided into three types (Fig. 2). Soil closely adhering to the root surface, forming a very narrow scope, is considered as rhizoplane soil (<1 mm), where the root adsorption rather than rhizosphere effects is mainly responsible for the significantly decreased concentrations of nonbiodegradable compounds. Rhizosphere soil (1–5 mm) is significantly affected by rhizosphere effects. Non-rhizosphere soil (>5 mm) is similar to bulk soil, which is rarely influenced by the plant root. Of course, the extension of each area may fluctuate depending on various parameters, as the soil texture, structure, organic matter content, and hydrophobicity can influence the diffusion of exudates into the soil, as well as mineral nutrients and other compounds towards the roots (Nye and Tinker, 1977; Grayston et al., 1997; Joner and Leyval, 2003). "Available" in Fig. 2 means the original available compounds and those released or desorbed by the biological activities of plants and microbes. "Dissipated" stands for the dissipation of compounds through degradation and/or adsorption by the roots. "Residual" represents the remaining contaminants, which is equal to "Available" minus "Dissipated".

The dissipation of compounds is mainly controlled by root adsorption, root uptake, and rhizospheric microbial biodegradation. The adsorption and uptake by the roots decrease sharply moving away from the root surface. The microbial degradation of highly persistent organic pollutants is difficult, and its contribution is very tiny or negligible. Moreover, the amount of naturally occurring, efficiently degrading rhizosphere microorganisms also decreases moving away from the root, which weakens the biodegradation of contaminant compounds. The availability of compounds is mainly affected by root exudates, which act as desorption or solubilization agents for the adhered compounds. Most of the exudates are water-soluble and can transfer freely in the soil solution, which leads to the activation or desorption influencing a larger area. If the amount of available compound decreases more rapidly than that of the dissipated compound, it results in a direct increase of the amount of "residual" compound to a normal level away from the roots. This situation is Case 1 (Fig. 2a). Case 1 is suitable for newly contaminated soil, which
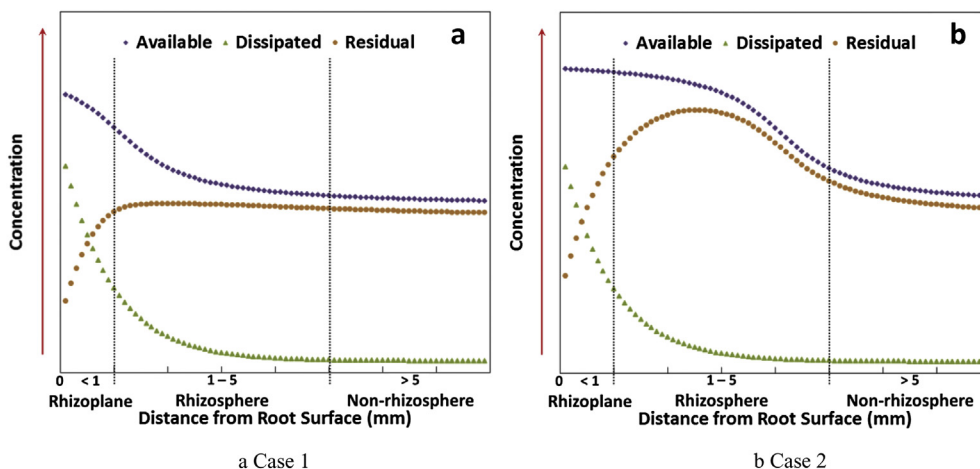

a Case 1   b Case 2

Fig. 2. Assumed relationship between the rhizosphere effects and the distance from the root surface. ("Available" means the concentration of available compound; "Dissipated" means the concentration of dissipated compound; "Residual" means the concentration of residual compound, Residual = Available − Dissipated.).
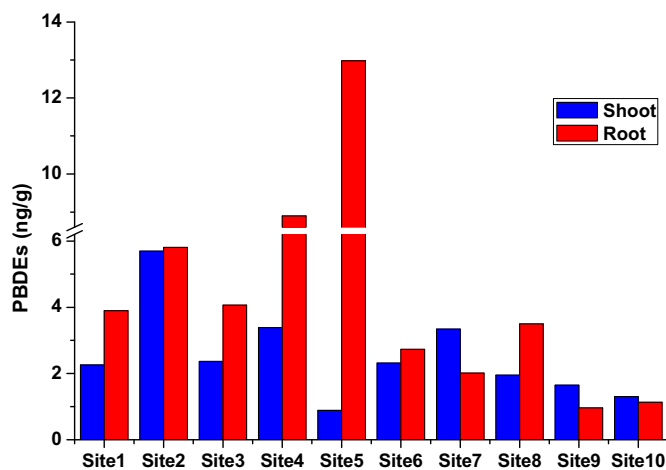
Fig. 3. Total PBDE concentrations in the different plant shoots and roots.

shows fewer adherences to contaminants. The available compounds in this type of soil account for a larger percentage than in long-term contaminated soil, and the effect by the roots is relatively limited. If the amount of dissipated compound decreases more rapidly than that of the available compound, it causes the amount of "residual" compound to first increase appreciably and then decrease rapidly away from the root surface. This situation is Case 2 (Fig. 2b). Case 2 is appropriate for long-term contaminated soils, which have abundant adhered compounds to be released. Thus, the activation or desorption is more active in aged or long-term contaminated soils (Case 2) than in newly contaminated soils (Case 1). Because the rhizosphere soils in the present study were 1–5 mm from the root surface (i.e., not the rhizoplane soils), available contaminants are more abundant than dissipated, which reasonably results in an enrichment of PBDEs in the rhizosphere soils (See Case 2).

In conclusion, without regard to the differences in the plant species or soil properties, the aging time of soil contaminants may also lead to dissimilar results. Contaminants may strongly bond to the soil particles, forming a strong affinity and an irreversible adsorption after a long-term aging or contamination period, which could significantly decrease the availability or bioavailability of the contaminants. Welsh et al. (2009) discovered that the recovery of PBDEs from aged soil dropped significantly over time.

3.3. Levels of PBDEs in plant roots and shoots

ΣPBDEs in the shoot samples were in the range of 0.88–5.69 ng/g with a geometric mean of 2.15 ng/g, while ΣPBDEs in the root samples were in the range of 0.97–13.0 ng/g with a geometric mean of 3.02 ng/g. The PBDE concentrations in the roots were generally higher than those in the shoots with a few exceptions, which were located at sites with low PBDE concentrations in the soil, indicating that the higher levels of PBDEs in the shoots of those plants may be attributed to the foliar uptake and adsorption from air. As shown in Fig. 3, the highest concentration in the roots was found at Site 5, followed by Site 4. The lowest concentration in the roots was observed at Site 9. For the PBDE concentrations in the shoots, the highest and lowest concentrations were discovered in samples from Sites 2 and 5, respectively. The high concentrations in the roots at Sites 4 and 5 may be responsible for the large difference between the corresponding rhizosphere and non-rhizosphere soils (see Figs. 1 and 3). Moreover, the rice and taro at Sites 4 and 5 belong to fibrous root plants with a large root surface area and high rooting density, which probably could secrete more root exudates.

3.4. PBDE congener profiles in soil and plant samples

In general, BDE 47 was the most abundant congener among the detected PBDEs in the plant and soil samples, followed by BDE 99 (Fig. 4). However, at Sites 5 and 8, the concentration of BDE 183 was extremely high in most of the samples except the shoot samples. Only a slight difference was discovered when comparing the congener profiles between the rhizosphere and non-rhizosphere soils (Fig. 4), although a significant difference ($p = 0.006 < 0.01$) was found for their geometric mean concentrations. However, the congener profiles of the roots were greatly different from those of the shoots, with a high percentage of high brominated PBDE congeners (such as BDEs 183 and 153) in the roots, especially at Sites 4, 5, 7, 8, and 10. These results suggest that low brominated PBDEs may be either more easily taken up by roots and translocated within plants, or they may be easier to evaporate from soil and more accessible for foliar uptake than high brominated PBDEs. High brominated PBDEs tend to adsorb on particles and are difficult to expel to the air or be absorbed by the root (ter Schure and Larsson, 2002; de Boer et al., 2003). Therefore, the high concentrations of "heavy" PBDEs in the roots suggest that they may be mainly from adsorption instead of uptake (see Fig. 4). Moreover, because the root uptake of PBDEs
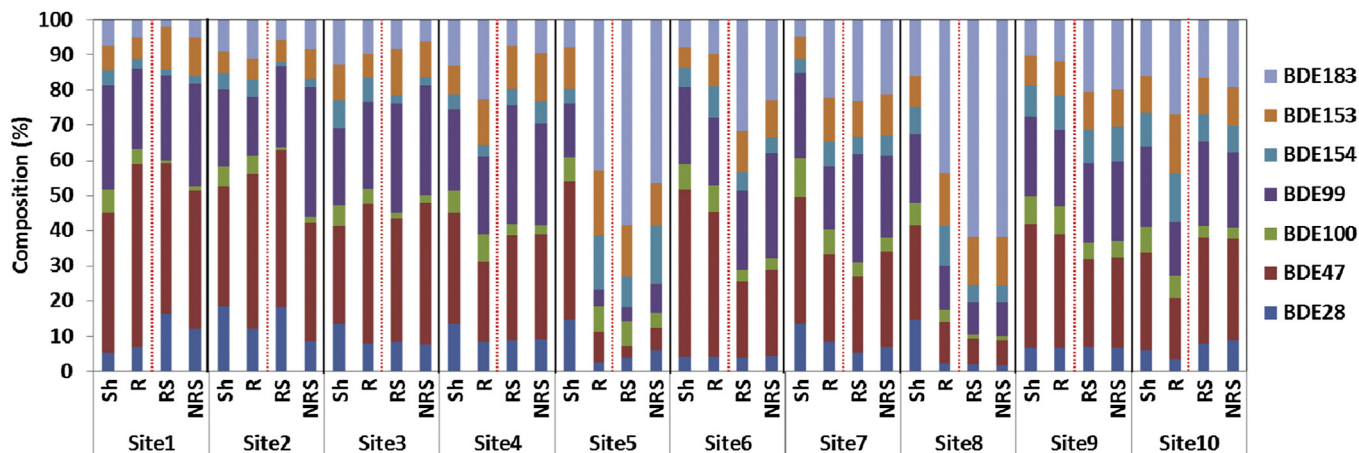

Fig. 4. Compositions of PBDE congeners in the different samples.

may be selective, similar congener patterns between the roots and the corresponding soils (both rhizosphere and non-rhizosphere) also confirmed that most of the heavy PBDEs in the roots may be directly from adsorption from soils. Meanwhile, the low proportion of "light" PBDEs and high proportion of "heavy" PBDEs in the roots compared with their associated soils at Sites 6 and 10 may imply the selective uptake of PBDEs by *Ipomoea aquatica* Forsk. and *Gynura cusimbua* (D. Don) S. Moore, respectively.

3.5. Evidence of the selective uptake of PBDE congeners

It has been proven that plants can reduce organic pollutants by root uptake (Mueller et al., 2006; Huang et al., 2010). Plant uptake is also believed to be selective, and low brominated PBDEs are more readily taken up by plants than high brominated PBDEs (Huang et al., 2010). However, reports on the bio-accumulations of congeners with same bromine content are very rare.

BDEs 99 and 100 and BDEs 153 and 154 are two pairs of structural isomers whose physical and chemical properties are very similar. The present results show that their bio-accumulations are dissimilar based on the different congener ratios of BDE 99/100 and BDE 153/154 in the shoots, roots, rhizosphere soils, and non-rhizosphere soils (Fig. 5). BDE 99 is much more abundant than BDE 100 in all of the samples, especially the soil samples (BDE 99/100 > 5). Assuming that the PBDEs in the roots were mainly from the corresponding soil, the congener ratios of 99/100 should be similar in the roots and rhizosphere soils if their bioaccumulations are comparable. However, the 99/100 ratios decreased sharply going from soils to plants (see Fig. 5a). First, this may suggest that BDE 99 experiences more difficulty in being accumulated or taken up by the roots than BDE 100, which led to the 99/100 ratio decreasing significantly in the roots compared with the corresponding soils. Zeng et al. (2013) also showed that the biomagnification factor of BDE 100 was much higher than that of BDE 99 in fish samples from an e-waste site. Second, another explanation may be that the degradation of BDE 99 is faster than that of BDE 100 within the root. Differences in the metabolic debromination efficiency have been discovered between BDE 99 and BDE 100 in fish (Roberts et al., 2011).

The low 99/100 ratio in the shoots may be attributed to three causes: first, most of the BDEs 99 and 100 in the shoots are probably translocated from the roots; second, the slightly different physicochemical properties of BDEs 99 and 100 may lead to different volatilities and a low ratio of 99/100 in the air, which may cause the ratio for the direct uptake by the leaves to also be low; moreover, the way that PBDEs enter the leaves may be selective as well. The scenario for BDEs 153 and 154 was similar, with the bioaccumulation of BDE 154 being higher than that of BDE 153 (see Fig. 5b).

Because the congener ratios in non-rhizosphere soils are hardly influenced by plants, they can be used to assess the difference in bioaccumulation for the individual congeners by comparing the congener ratios in the roots and soils. The congener ratios of 99/100 and 153/154 in the soils are 1.8—5.9 times and 1.2—4.0 times (except at Sites 4 and 5) higher than those in the roots, respectively. These results indicated that the bioaccumulation factors (BAFs) of BDE 100 and 154 were approximately 2—4 times higher those of BDE 99 and 153, respectively. BDE 99 and BDE 100 are known as 2, 2′, 4, 4′, 5-penta-BDE and 2, 2′, 4, 4′, 6- penta-BDE, respectively, whereas BDE 153 and BDE 154 are 2, 2′, 4, 4′, 5, 5′-hexa-BDE and 2, 2′, 4, 4′, 5, 6′-hexa-BDE, respectively. Hence, the compound with the bromine substitution at *ortho*- position 6 is
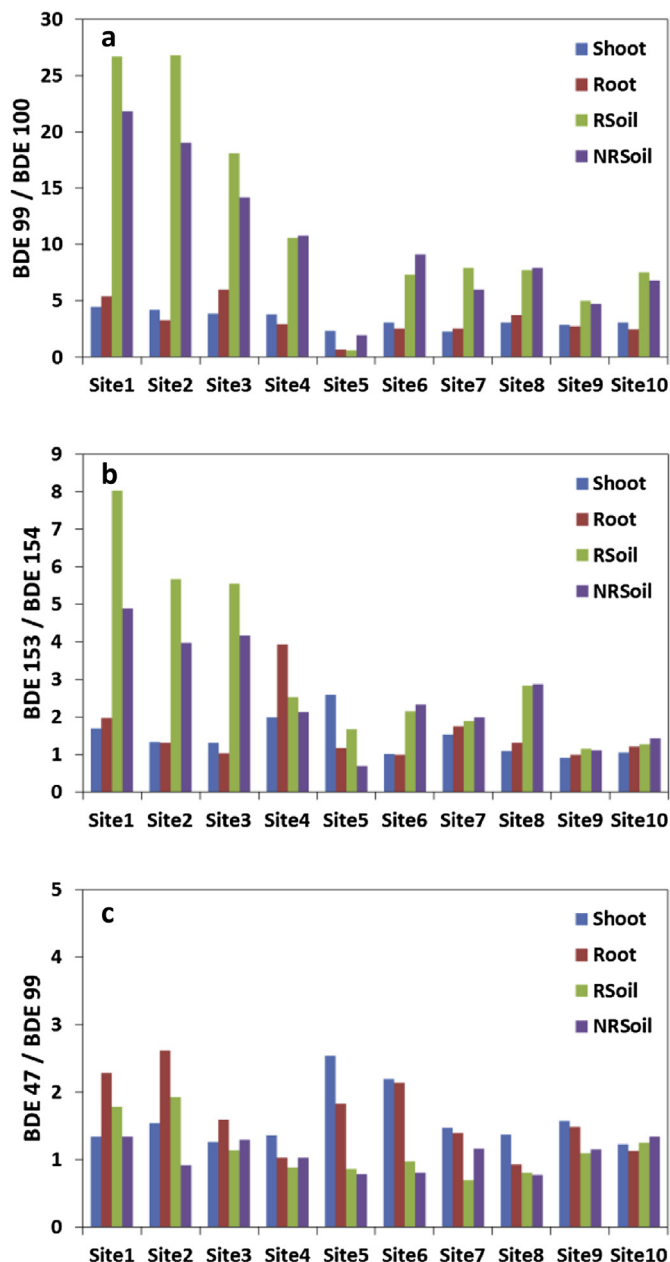

Fig. 5. Congener ratios of BDE99/100 (a), BDE153/154 (b); and BDE47/99 (c) in the shoots, roots, rhizosphere soils, and non-rhizosphere soils.

more easily bioaccumulated than that substituted at *meta*- position 5, which implies that the substituent may be mainly responsible for the different bioaccumulations of the isomers. However, the position of the bromine was not the only reason for the difference of the congener ratios; selective degradation can also influence this ratio (Meng et al., 2008). Because the degradation of BDE 99 to BDE 47 always occurs in biota samples (Roberts et al., 2011), we also calculated the ratio of BDE 47/99 (see Fig. 5c). The 47/99 ratios in the plant shoots, roots, rhizosphere soils, and non-rhizosphere soils were very similar to each other, suggesting that the degradation of BDE 99 to BDE 47 is insignificant or negligible within plant tissues. Thus, for short-term and seasonal vegetation, the substitution pattern instead of selective biodegradation was mainly responsible for the variation in the ratio of the different congeners.

4. Conclusion

Plant roots can influence the fate of PBDEs within the rhizosphere area. PBDEs in soils may be either first enriched appreciably and then decreased or increased directly to the non-rhizosphere level at a distance away from root surface that is probably dependent on the aging of the contaminated soil. The higher PBDE concentrations in the roots compared with the shoots suggested that the translocation of the PBDEs from the root to the shoot was quite low. The congener profiles also indicated that the PBDEs in the shoots mainly originated from foliar uptake from the air. The substituent effect was mostly responsible for the difference in the bioaccumulations between BDE 99 and 100 or BDE 153 and 154. The present results suggested that the bioaccumulations of the *ortho*-substituted PBDE congeners may be much higher than those of the *meta*-substituted congeners.

Acknowledgments

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