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Field study on the uptake pathways and their contributions to the accumulation of organophosphate esters, phthalates, and polycyclic aromatic hydrocarbons in upland rice

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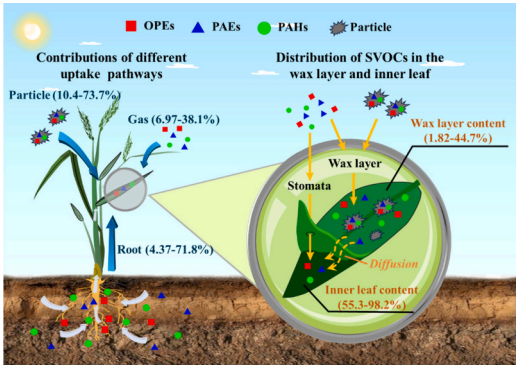
HIGHLIGHTS

- A field study was conducted to elucidate the main uptake pathway of SVOCs by rice.
- We develop a field-deployable chamber to isolate rice from different forms of SVOCs.
- Contribution of different uptake pathways on the SVOCs in rice leaf was quantified.
- Root, foliar gas and foliar particle uptakes all contribute to the SVOC burdens in rice.

GRAPHICAL ABSTRACT



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ABSTRACT

Plant uptake of organic contaminants generally occurs through either root, gas-phase foliar, or particle-phase foliar uptake. Understanding these pathways is essential for food-system practitioners to reduce human exposures, and to clean contaminated-sites with phytoremediation. Herein, we conducted a field-based experiment using an improved specific exposure chamber to elucidate the uptake pathways of organophosphate esters, phthalates, and polycyclic aromatic compounds, and quantitatively assessed their contributions to organic contaminant accumulations in field-grown rice. For most target compounds, all three uptake pathways (root, foliar gas, and foliar particle uptakes) contributed substantially to the overall contaminant burden in rice. Compounds with lower octanol–water partition coefficients (K_{ow}) were more readily translocated from roots to leaves, and compounds with higher octanol–air partition coefficients (K_{oa}) tended to enter rice leaves mostly through particle deposition. Most compounds were mostly stored in the inner leaves (55.3–98.2 %), whereas the relatively volatile compounds were more readily absorbed by the waxy layer and then transferred to the inner leaves. Air particle desorption was a key process regulating foliar uptake of low-volatility compounds. The results

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can help us to better understand and predict the environmental fate of those contaminants, and develop more effective management strategies for reducing their human exposure through food ingestion.

1. Introduction

Organophosphate esters (OPEs), phthalates (PAEs), and polycyclic aromatic hydrocarbons (PAHs) are three classes of semivolatile organic compounds (SVOCs) that are persistent, toxic, and ubiquitous in the environment (Mojiri et al., 2019; Wang et al., 2019). OPEs and PAEs are widely used in several industrial and household products as additive plasticizers and flame retardants (Li et al., 2019b; Li et al., 2021; Xing et al., 2023). PAHs are by-products of incomplete combustion of organic matter during thermal decomposition processes (Singh et al., 2016; Veiga et al., 2014). Previous studies have revealed that some of these compounds exhibit neurotoxicity, carcinogenicity, reproductive and developmental toxicity, and can cause a range of ecological and human health problems (Jesus et al., 2022; Mu et al., 2015; Wang et al., 2020).

As an essential land cover type on the Earth's surface, plants play an important role in the global biogeochemical cycles. Organic contaminants such as OPEs, PAEs, and PAHs accumulate in plants, and, in turn, directly enhance human exposure through food consumption (Yang et al., 2017). Therefore, interest is growing in the fate of OPEs, PAEs, and PAHs in terrestrial ecosystems, and their uptake by terrestrial plants, especially their uptake mechanisms in crops (Li et al., 2023; Yang et al., 2017). Rice is one of the most widely planted food crops in the world, meeting the food supply of nearly 50 % of the world's population (Mu et al., 2019; Zhu et al., 2020a). China's rice planting area accounts for 18 % of the global rice planting area, while its yield accounts for 30 % of the global rice production (Pokharel et al., 2019; Zhang et al., 2020). However, OPE concentrations in rice fields are relatively higher than those in other fields (Zhang et al., 2022). Similarly, OPEs in rice grains are also higher than other grains, like corn, which may pose potential risks to humans via rice consumption (Zhang et al., 2022).

Plants can absorb, accumulate, and metabolize organic pollutants; therefore, humans become exposed to pollutants through food consumption (Poma et al., 2017; Wang et al., 2021b). Three primary pathways are associated with passive uptake of organic pollutants by plants: (1) Transportation via the soil–root–leaf pathway, where pollutants adsorbed by soil particles are desorbed into soil water and then reach the root epidermis through diffusion or advection of water. Some pollutant portions are immobilized by epidermal lipids, while the rest enter the epidermis by passive transport, reach the cortex and endothelium via the ectoplasmic or symplastic pathway, and are transported upward through transpiration in the xylem of vascular bundles (Collins et al., 2006; Li et al., 2022b; Miller et al., 2016; Zhang et al., 2021b). Pollutants in the atmosphere are also sorbed and taken up by plant leaves via the air–leaf–root pathway, in the following forms: (2) gaseous pollutants are adsorbed by the waxy layer on leaves through diffusion; and (3) particulate-bound pollutants (particles or dust) are deposited on the waxy layer on leaves or captured by leaves and then enter the leaves through diffusion, stomata, or water pores (Barber et al., 2002; Collins et al., 2006; Wang et al., 2022b; Yang et al., 2017). To develop interventions that reduce contaminant burdens in the food supply chain, we must understand the magnitudes that each of these pathways contribute to overall plant contaminant burdens.

Plant contaminant uptake and transport pathways have a major correlation with contaminant physicochemical properties and plant physiology (Liu et al., 2019; Sun et al., 2016; Zhang et al., 2021a). Cousins and Mackay (2001) suggested that neutral compounds with n-octanol–water partition coefficient, log $K_{ow} < 2.5$ and *n*-octanol–air partition coefficient, log $K_{aw} < -1$ (the dimensionless Henry's law constants) are predominantly absorbed by roots, whereas those with log $K_{oa} > 6$ and log $K_{aw} > -6$ are predominantly absorbed from the atmosphere by leaves. According to McLachlan (1999), compounds with $6 < \log K_{oa} < 8.5$ are almost entirely absorbed in the gaseous form by leaves, those with $8.5 < \log K_{oa} < 11$ are accumulated through kinetically limited dry gaseous deposition, and those with log $K_{oa} > 11$ are largely deposited in particulate forms through dry or wet particle-bound depositions. However, the uptake of compounds by plants is a complex combination of multiple factors that may vary with environmental factors, plant species, as well as SVOC levels and species.

Zhu et al. (2020b) conducted a controlled chamber experiment to investigate the accumulation and translocation of polybrominated diphenyl ethers (PBDEs) in wheat under three exposure scenarios (soil, air, and dust). The authors observed that PBDE levels in wheat were primarily enhanced by foliar uptake (>81 %), with PBDE contributions from gaseous and particulate phases being 0.75–56.6 % and 37.3–98.9 %, respectively, and the contributions were significantly correlated with their log K_{oa} values. Although laboratory-controlled experiments have the advantages of simplifying and decomposing various complex factors, they often occur at unrealistic environmental concentrations and cannot perfectly simulate the real environment, including factors such as the complex forms of pollutants and their non-equilibrium concentrations in the environmental media, thereby resulting in potential gaps or even inconsistencies in data obtained from laboratory and field experiments (Hubai et al., 2021; Li et al., 2019a; Wang et al., 2022a; Wang et al., 2021a; Zhang et al., 2021b).

Together, these results indicate that both soil and foliar uptakes can determine overall plant contaminant burdens, but the contribution of specific uptake pathways to SVOC accumulation in plants grown under field conditions remains poorly understood (Lao et al., 2024; Zhang et al., 2021b). This lack of understanding makes it difficult to develop interventions aimed at reducing pollutant levels in plants, since different interventions will be more or less effective for different uptake pathways. Therefore, it is imperative to conduct field studies in situ to investigate the uptake mechanisms of contaminants by plants.

Herein, we developed and applied novel field-deployable specialized isolation chambers (SICs) to conduct a comprehensive exposure experiment to elucidate root, gaseous foliar and particulate foliar uptakes of OPEs, PAEs, and PAHs in upland rice (*Oryza sativa* L.). The results may guide us to have a better understanding and prediction of the fate of SVOCs in the environment.

2. Material and methods

2.1. Specialized isolation chamber (SIC)

The field-deployable SIC is a polymethyl methacrylate chamber that is 30 cm long, 20 cm wide, and 110 cm high, with a sloping roof to allow water to condense and flow down the inner wall (Figs. 1 and S1, S3). We designed the chamber with polymethyl methacrylate as it has high transparency, good light transmission capacity, and high mechanical strength compared with glass, allowing it to endure harsh field conditions. The SIC has two ventholes (8 cm in diameter) on opposite sides of the device at different heights to maintain ventilation using the chimney effect.

We tested three configurations of the SICs (Fig. 1, Tests A, B, and C) to elucidate the specific uptake pathways of OPEs, PAEs, and PAHs. In Test A we only sheltered the plants from rain, exposing them during the test period to pollutants from the soil, gaseous and particulate phases. In Test B, we covered the SIC vents with two-layer pre-cleaned stainless steel meshes (200 mesh size, 75 μm) to block SVOCs with particles ~>75 μm and also maintain good ventilation for the SIC. During the experiment, rice plants in Test B SICs were therefore exposed to pollutants via soil and gaseous foliar uptake, and to particles <75 μm in

diameter. Finally, we covered the SIC vents in Test C with pre-cleaned PUF disks (8.2 cm in diameter and 2.5 cm in height) to prevent the passage of gaseous and all particle-bound SVOCs. This means that during the test period rice plants in Test C, SICs were only exposed to pollutants absorbed from the soil.

The growth stages of rice can be roughly divided into: seedling (duration: ~35 d), tillering (~30 d), jointing (~30 d), booting (~25 d), heading (~20 d), filling (~20 d), and maturity (~30 d) stages. Before the SICs were deployed, rice plants grew naturally in the paddy field. The SICs were deployed at the beginning of the booting stage (early stage of reproductive growth), when rice flag leaves elongate and unfold until heading. We chose to isolate the plants at the booting stage as it represents the early stage of reproductive growth, and the rice plants were too fragile to be covered with the SIC prior to the booting stage. We deployed two SICs for Tests B or C, with each SIC covering two clusters (~6–8 plants) of upland rice (Fig. S3c). The selected rice plants were all of a uniform size and grew under similar conditions in the paddy field.

Rice plants were collected once in a cycle of 21 days for a total of 4 cycles during the booting, heading, filling, and maturity stages of rice. The PUF and mesh coverings of the vents were replaced at every sampling event to avoid breakthrough or fouling. Polydimethylsiloxane-based passive air samplers (PDMS sheets, 5 × 5 cm) were deployed in and outside the SICs to monitor the air concentrations of SVOCs during filling and maturity stages of rice (Fig. 1). Superoxide dismutase (SOD), peroxidase (POD), and catalase activities (CAT) in different rice tissues were measured at each growth stage.

Overall, our analysis rests on the following assumptions: (1) the SIC effectively removed gaseous or particulate SVOCs of the specified size fractions, (2) the SIC had a negligible impact on the growth and physiological activities of rice; (3) contaminants undergoing each uptake pathway faced similar metabolism, translocation, and growth dilution processes, since rice plants used in this study were at the same growth stage (Collins et al., 2006); (4) the growth and uptake processes for the plants prior to the deployment of the SICs were similar, leading to similar starting concentrations across treatments; and (5) the rice plants were exposed to similar soil concentrations across the test period.

2.2. Sample information

The study was conducted in an upland rice field (N39°46′49″, E123°14′46″) without any obvious pollution sources in Qingduizi Town, Dalian, China. Four surface soil samples (0–10 cm soil layer), six PDMS sheets, and 54 rice samples at the booting (root sample in the test A was lost), heading, filling, and maturity stages of rice, were collected from July to October 2022. All samples were immediately transported to the laboratory in self-sealing polyethylene bags. Rice samples were initially rinsed with tap and ultrapure water, and then divided into roots, stems, leaves, grains (if any), and husks (if any). Duplicate rice samples from the same treatment were merged into one sample before analysis. Fresh rice samples were tested for antioxidant enzyme activity to determine whether the SICs had any significant effects on rice growth (Texts S1-S3) (Ahmad et al., 2022; Wang et al., 2022b). Fresh leaves were immersed in a solvent mixture of dichloromethane and ethyl acetate (1:1, v/v) for 15 s to obtain waxy epidermal samples (Beattie and Seibel, 2007). Soil, immersed leaf, and other rice tissue samples were freeze-dried, ground into powder, passed through a 60-mesh sieve, and stored at −20 °C until further processing.

2.3. Sample extraction and purification

The PDMS sheets were spiked with 12 deuterated surrogate standards (tris (2-chloroethyl) phosphate-d_{12} (TCEP-d_{12}), tris (2-chloro-isopropyl) phosphate-d_{18} (TCIPP-d_{18}), and triphenyl phosphate-d_{15} (TPHP-d_{15}) for OPEs; dimethyl phthalate -d_4 (DMP-d_4), diethyl phthalate-d_4 (DEP-d_4), di-*n*-butyl phthalate-d_4 (DNBP-d_4), and di-2-ethylhexyl phthalate-d_4 (DEHP-d_4) for PAEs; naphthalene-d_8 (NAP-d_8), acenaphthene-d_{10} (ACE-d_{10}), phenanthrene-d_{10} (PHE-d_{10}), chrysene-d_{12} (CHR-d_{12}), and perylene-d_{12} (PER-d_{12}) for PAHs), before they were immersed in ethyl acetate, shaken, and extracted for 24 h (120 r/min). Soil (~0.5 g) and rice samples (~0.2 g) were spiked with surrogate standards, ultrasonically extracted with a mixture of dichloromethane: hexane: acetone (2:2:1, v/v/v) for 20 min, and centrifuged at 4000 r/min for 10 min. The extraction process was repeated three times. The combined extracts were cleaned up using a silica gel column filled with 3 % deactivated silica gel (baked at 450 °C for 4 h), ethylenediamine-N-propyl (PSA), and anhydrous Na_2SO_4 (baked at 450 °C for 4 h) from bottom to top. The eluate was solvent exchanged to isooctane and then spiked with the internal standard before analysis.

2.4. Instrumental analysis

We analyzed the samples for 8 OPEs, 11 PAEs, and 16 PAHs using a Shimadzu GCMS-QP2020 equipped with an electron ionization (EI) source in the SIM mode and a SH-Rxi-5Sil MS capillary column (30 m × 0.25 mm × 0.25 μm). The targeted OPEs included tri-isobutyl phosphate (TIBP), tri-*n*-butyl phosphate (TNBP), TCEP, TCIPP, tris (1,3-dichloro-2-propyl) phosphate (TDCIPP), TPHP, tris (2-ethyhexyl) phosphate (TEHP), and tricresyl phosphate (TMPP). The targeted PAEs included DMP, DEP, di-isobutyl phthalate (DIBP), DNBP, butyl benzyl phthalate (BBzP), bis (2-ethylhexyl) adipate (DEHA), dicyclohexyl phthalate (DCHP), DEHP, di-*n*-octyl phthalate (DnOP), di (2-ethylhexyl) terephthalate (DEHT), and trioctyl trimellitate (TOTM). The targeted PAHs included naphthalene (NAP), acenaphthylene (ACY), acenaphthene (ACE), fluorene (FLU), phenanthrene (PHE), anthracene (ANT),

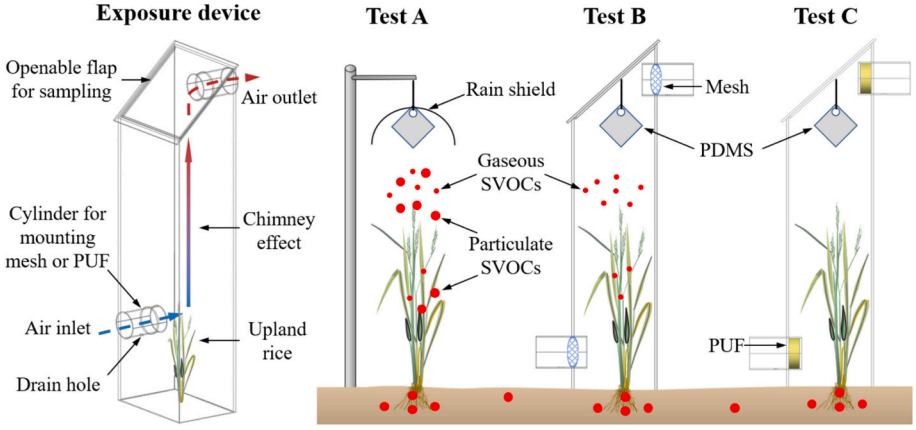

Fig. 1. Specific exposure devices and field exposure experiments of upland rice to OPEs, PAEs, and PAHs via soil, gas, and particle uptake pathways.

fluoranthene (FLA), pyrene (PYR), benzo[*a*]anthracene (BaA), chrysene (CHR), benzo[*b*]fluoranthene (BbF), benzo[*k*]fluoranthene (BkF), benzo[*a*]pyrene (BaP), indeno[123-*cd*]pyrene (IcdP), dibenz[*ah*]anthracene (DBahA), and benzo[*ghi*]perylene (BghiP) (Table S1). The carrier gas was 1.5 L/min of helium. Injector and transfer line temperatures were both 290 °C.

2.5. Quality assurance and quality control (QA/QC)

Glass products were calcined at 450 °C for at least 4 h and washed with the corresponding solvent three times before being used. A procedural blank and a spiked sample were performed every 10 samples. The method detection limit (MDL) was calculated as the mean concentration of the procedure blank plus 3 times the standard deviations (Table S2). The recoveries of TCEP-d_{12}, TCIPP-d_{18}, TPHP-d_{15}, DMP-d_4, DEP-d_4, DNBP-d_4, DEHP-d_4, NAP-d_8, ACE-d_{10}, PHE-d_{10}, CHR-d_{12} and PER-d_{12} deuterium surrogate standards in all plant samples were 89.5 ± 10.2 %, 91.7 ± 7.86 %, 94.1 ± 10.4 %, 75.0 ± 13.9 %, 85.3 ± 10.7 %, 95.7 ± 7.02 %, 99.4 ± 12.8 %, 54.0 ± 16.6 %, 74.4 ± 15.3 %, 90.5 ± 9.41 %, 96.6 ± 11.5 %, 103 ± 10.1 % (Table S3). All samples were blank- and recovery-corrected.

We investigated possible OPE, PAE, and PAH contamination from the SICs by wiping 10 cm^2 of the chamber surface using pre-cleaned cotton wipes soaked in 3 mL isopropyl alcohol. Relatively high concentrations of DEHP, DNBP, DEHA, DEHT, and TOTM were found in the surface wiping sample (Fig. S2), suggesting these PAEs have been added in the polymethyl methacrylate as plasticizers. Therefore, DEHP, DNBP, DEHA, DEHT, and TOTM were excluded from further analysis.

2.6. Data analysis and statistics

Concentrations (ng/g) in the waxy layer of leaves were calculated based on their epidermal wax weight. Concentrations in plant tissue and soil samples were calculated based on their dry weight (dw). Airborne concentrations (ng/m^3) were calculated based on the chemicals' mass in the passive PDMS samples and their sampling rates (0.2–0.228 m^3/d, Text S4). Statistical analysis was performed using IBM SPSS Statistics 26 and Microsoft Excel 2019. A p-value <0.05 was considered statistically significant.

2.7. Contribution rates of root uptake and foliar uptake via gaseous and particle phases

The contribution rates of root uptake (R_{soil}, including both root uptake and acropetal translocation) and foliar uptake via gaseous (R_{gas}) and coarse particle phases ($R_{particle}$) to the SVOC burdens in rice leaves can be defined as follows (Wang et al., 2023b).

$$R_{soil} = \frac{C_{leaf,C}}{C_{leaf,A}} \times 100\% \quad (1)$$

$$R_{gas} = \frac{C_{leaf,B} - C_{leaf,C}}{C_{leaf,A}} \times 100\% \quad (2)$$

$$R_{particle} = \frac{C_{leaf,A} - C_{leaf,B}}{C_{leaf,A}} \times 100\% \quad (3)$$

where $C_{leaf,\ A}$, $C_{leaf,\ B}$, and $C_{leaf,\ C}$ are the SVOC concentrations (ng/g dw) in leaves from the specific exposure experiments Tests A, B, and C, respectively. Due to the comparability of SVOC concentrations in rice leaves throughout the whole sampling period, the average concentrations of SVOCs in leaves at different stages were used in this calculation.

3. Results and discussion

3.1. Evaluation of experimental design

The concentrations of OPEs, PAEs, and PAHs in the rice leaves followed the order: Test A (no isolation) > Test B (isolated from coarse particle-bound SVOCs) > Test C (isolated from both gas and particle SVOCs), suggesting that the SICs successfully isolated the rice plants from different SVOC uptake pathways (Fig. 2, left panels). The results showed a substantial concentration gradient between treatments A, B and C, indicating that coarse particles (>75um) contributed substantially to overall plant contaminant burdens. The much lower air concentration observed in Test C, isolated from gas and particle SVOCs, also reinforces that the SIC successfully isolated the rice from airborne gas and particle phase contaminants (Fig. 2, middle panels), particularly since the performance reference compound-derived uptake rates were similar for all three treatments. For Test B, the intermediate air concentration between A and C suggests that the plants in the chamber may be isolated from particles, as intended. Although PDMS is known to capture fewer particles than PUF (Okeme et al., 2018), passive samplers are known to capture some particles and have been shown to scavenge both coarse and fine particles from the atmosphere (Markovic et al., 2015). Concentrations in the roots did not show this trend (Fig. 2), confirming that the chambers are effectively isolating the plants from atmospheric influences, although it remains possible that the lower air and leaf concentrations observed in Test B were due to a decrease in bulk air concentrations and not to a decrease in particles in particular. Finally, the rice antioxidant enzyme activities during different growth stages (except for the filling stage) showed no significant differences, suggesting the SICs had little impact on rice growth (Fig. S4).

3.2. Concentrations of OPEs, PAEs, and PAHs in different matrices and tests

The average concentrations of OPEs, PAEs, and PAHs in all rice tissues from different seasons were 96.09 ± 34.31, 923 ± 496, and 124 ± 56.1 ng/g for Test A; 81.8 ± 24.9, 805 ± 456, and 115 ± 50.2 ng/g for Test B; 78.2 ± 26.6, 691 ± 404, and 97.3 ± 45.7 ng/g for Test C, respectively. The observations were verified by the variations in the compositions of OPEs, PAEs, and PAHs in different rice tissues in Tests A, B, and C (Fig. S5).

The air concentrations in the three tests followed the pattern of Test A > B > C (Fig. 2). The average air concentrations in Test A, B, and C were 3.13 ± 0.05, 2.78 ± 0.12, and 2.29 ± 0.10 ng/m^3 for OPEs, 20.7 ± 0.70, 8.68 ± 2.17, and 4.82 ± 1.76 ng/m^3 for PAEs, and 20.8 ± 4.75, 3.31 ± 0.65 and 2.08 ± 0.36 ng/m^3 for PAHs, respectively. The SVOC concentrations in the soil fluctuated slightly during the four growth stages of rice. The ΣOPEs, ΣPAEs, and ΣPAHs in the soil were in the range of 43.8–54.1 ng/g, 662–740 ng/g, and 53.8–72.6 ng/g. Notably, SVOC concentrations in rice roots at the same growth stage among Tests A, B, and C were comparable, suggesting that the influence of SIC on uptake of the compounds by rice roots was negligible (Fig. 2).

The accumulation patterns of OPEs, PAEs and PAHs in rice tissues followed the order: leaf (139 ± 37.2, 1184 ± 601 and 167 ± 68.4 ng/g, respectively) > root (94.9 ± 29.3, 1097 ± 449 and 123 ± 26.7 ng/g, respectively) > stem (86.7 ± 25.2, 924 ± 534 and 121 ± 87.6 ng/g, respectively) > grain (75.9 ± 12.2, 656 ± 283 and 113 ± 21.2 ng/g, respectively) > husk (73.0 ± 15.7, 606 ± 536 and 83.8 ± 17.8 ng/g, respectively). Overall, the concentrations of the three types of SVOCs in rice leaves and roots initially increased and subsequently decreased with the advancement of the growth stages of rice in all the three tests (except for the roots in Test B) (Fig. S6). The concentrations of SVOCs in rice stems exhibited fluctuating decreases with the advancement of the growth stages of rice, and a considerable variation in SVOC concentrations was observed between growth stages in Tests A, B, and C (Fig. S6). SVOC concentrations in rice leaves were in the order of Test A > B > C

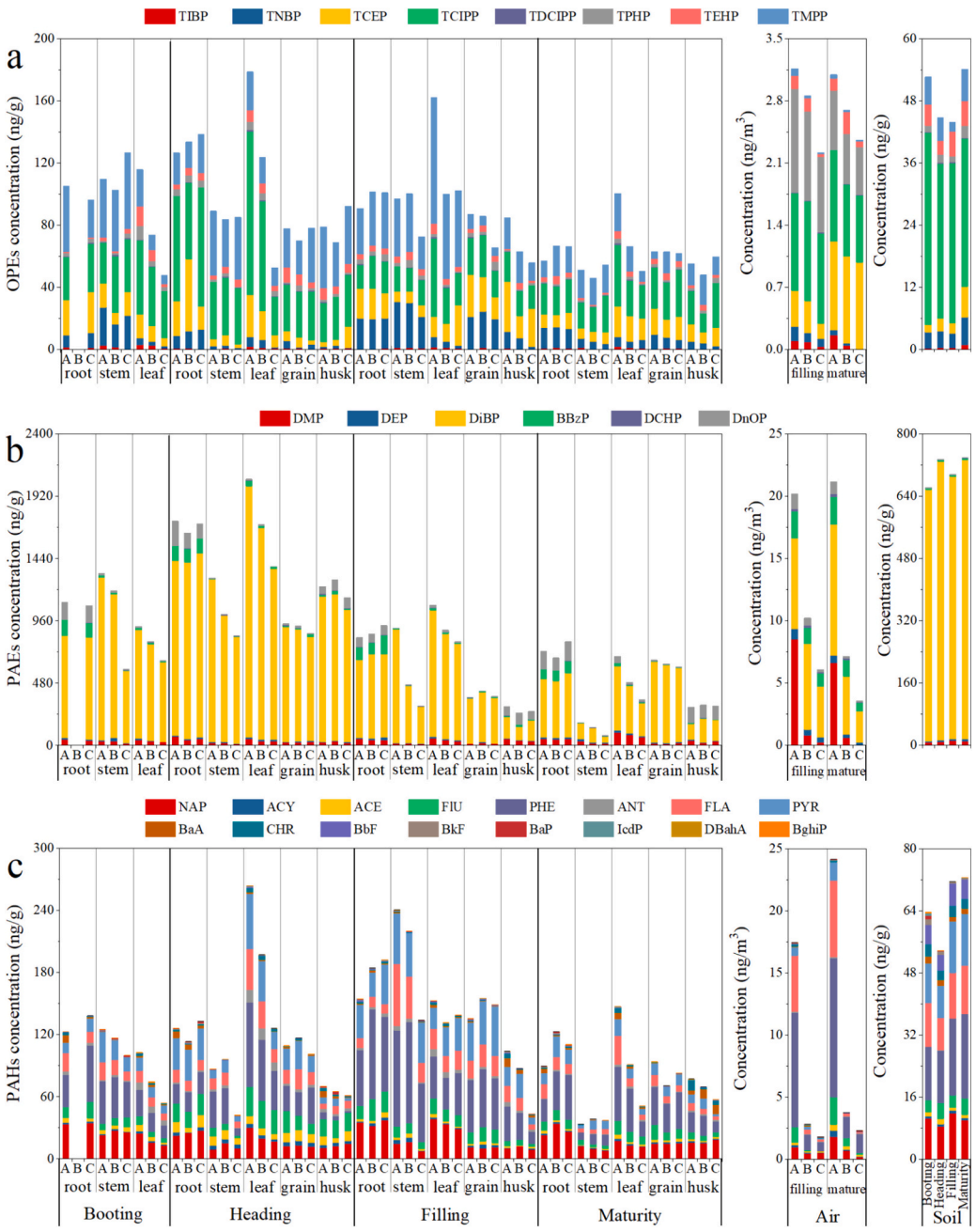

Fig. 2. Concentrations of OPEs (a), PAEs (b), and PAHs (c) in upland rice, air, and soil at different growth stages of rice (A: Test A, B: Test B, and C: Test C).

(Fig. 2), suggesting that rice with different exposure pathways (Tests A, B, and C) exhibited variations in contamination levels in leaves. Despite significant differences of SVOC levels in rice leaves among different treatments, no accumulation patterns in rice grains and husks over time and no differences between tests probably because SVOCs in rice grains may be controlled by multiple sources, including translocations from root uptake, leaf absorption, and rice husk absorption. However, the translocations of SVOCs from other tissues to rice grains may be relatively slow.

3.3. Contributions of different uptake pathways to SVOCs in rice leaves

At each growth stage, the concentrations of every individual OPE, PAE, and PAH congeners and their total concentrations in rice leaves followed the order of A (all uptake pathways) > B (isolated from coarse particle uptake) > C (isolated from gas and particle uptake) (Fig. 2). Icdp, DBahA, and BghiP were excluded due to their relatively low concentrations.

The three uptake pathways were all major contributors to overall SVOC levels for various compounds, with R_{soil}, R_{gas}, and $R_{particle}$ values of the targeted SVOCs in the whole leaves ranging from 4.37–71.8 % (mean: 39.8 ± 16.1 %), 6.97–38.1 % (mean: 24.0 ± 8.12 %), and 10.4–73.7 % (mean: 36.2 ± 14.7 %), respectively (Fig. 3a; details in Table S4). The results differed from those of some previous studies that investigated the uptake and translocation of OPEs, PAHs, and PBDEs using hydroponic or potted plant experiments. Zhu et al. (2016) found that 3 hexabromocyclododecane diastereo isomers (α-, β-, and γ-HBCDs) could be absorbed by wheat root and leaves; however, the root–to–leaf translocation of HBCDs in wheat was limited (14.4–29.8 %). Other studies have revealed that compounds absorbed by plant roots were mainly stored in the roots and their acropetal translocation was limited (Li et al., 2022a; Lin et al., 2006; Vrkoslavova et al., 2010; Wang et al.,

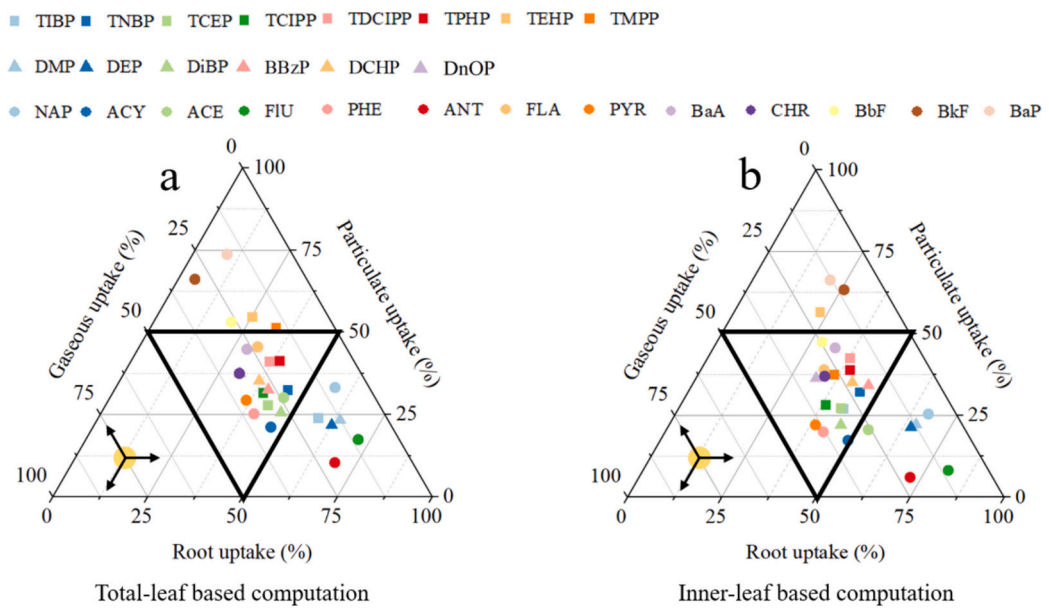

Fig. 3. Contributions of root uptake and foliar uptakes via gas and particle phases to the total burdens of OPEs, PAEs, and PAHs in the upland rice leaf.

2011); that is, the compounds were translocated from the roots to the leaves and their levels in leaves were insignificant. Our results suggest that all the three uptake pathways contributed to the accumulation of SVOCs in upland rice. The observation is consistent with the results of Wan et al. (2016), who found that OPEs in the aboveground parts of wheat under field conditions were associated with translocation from roots and foliar uptake from the air. Trapp et al. (Trapp, 2007). showed that root structure differences between hydroponically and soil grown can lead to large changes in plant uptake, which could explain some of this difference.

Previous field studies have shown that air–leaf exchange was the primary pathway for PBDE uptake by indica rice (*O. sativa* L. subsp. indica) grown in a subtropical region (Wang et al., 2015), and the soil–root–leaf pathway did not substantially contribute to PBDEs accumulated in tree leaves (Ding et al., 2014). The contrasting results obtained from this study could be attributed to the complex nature of the field environment, such as varying soil properties, co-contaminants, and the impacts of different plant species or compounds (Brunetti et al., 2021; Deng et al., 2018; Hu et al., 2021; Zhang et al., 2017). In this study, the contribution of SVOCs by foliar uptake in the gaseous phase was relatively low when compared to that in the particulate phase. The finding is consistent with that of a previous study conducted by Zhu et al. (2020b), which showed that most PBDEs in wheat leaves were from the air, with the ranges of contributions from particles and gases being 37.3–98.9 % and 0.747–56.6 %, respectively. However, since the 200-mesh screen cannot prevent fine particles, especially those <10 μm, it may, to some extent, underestimate the contribution of foliar uptake via particle phase, but overestimate the contribution of foliar uptake via gas phase.

To determine whether the SVOC concentrations of whole leaves was biased because of the presence of a relatively variable waxy layer, which may not be translocated to the rest of the plant tissues, the contributions of different uptake pathways based on the inner leaf were calculated (Fig. 3b; details in Table S5). By contrast, the inner leaf concentration refers to the amount of contaminants already present in the plant, and may therefore, better represent foliar uptake. The R_{soil}, R_{gas}, and $R_{particle}$ values in inner rice leaves were 20.6–81.1 % (mean: 43.8 ± 15.2 %), 7.38–49.1 % (mean: 24.0 ± 10.2 %), and 6.17–66.4 % (mean: 32.2 ± 15.8 %), respectively. Overall, no significant differences were observed for the contributions of the three uptake pathways between the inner and whole leaves, suggesting that using the whole leaf may adequately represent the contributions of different uptake pathways involved in foliar accumulation for most chemicals. This may be due to the relatively low quality of wax on the leaves. However, slight differences in the contributions of different uptake pathways were observed for compounds with high log K_{oa} values, where particle uptake was dominant. Therefore, further studies are required to determine whether the inner leaf could provide a better representation of foliar uptake for compounds with high log K_{oa} values.

Overall, our results suggested that management strategies aimed at reducing exposure through food ingestions should be developed taking into consideration the different uptake pathways in plants, which may depend on the specific contaminants being managed. For some compounds, strategies, such as locating agricultural fields away from pollution sources or ensuring that irrigation water does not contain contaminants, could be effective. Regarding other compounds, interventions of no single pathway could have outsized the impacts; therefore, strategies aimed at reducing overall emissions could effectively.

3.4. *Influence of compound properties on uptake by rice*

The contribution rates of the three uptake pathways to SVOCs in leaves varied considerably due to variations in physicochemical properties of the compounds. A significant negative correlation was observed between the R_{soil} values of SVOCs and their log K_{ow} values ($R^2 = 0.399$, $p < 0.001$; Fig. 4a). The results suggest that, as expected, hydrophilic compounds were more readily translocated from roots to leaves, as they were less likely to sorb onto plant materials, and were therefore transported to the leaves through the transpiration stream (Trapp, 2002; Trapp, 2007). The effect was more pronounced in PAHs, where root contribution exhibited a sharp decrease (from 70 % to <5 %) in the contributions of compounds with log K_{ow} values of 4–6. The OPEs and the PAEs exhibited similar trends, with a general linear trend being observed across the measured range, although the slope for PAEs was steeper than that for OPEs. A previous study investigating the uptake pathways of PAHs in white clover (*Trifolium repens* L.) showed that the movement of hydrophilic (water-soluble) PAHs from the roots to shoots was mainly driven by the water potential gradient, created throughout the plant during transpiration (Gao and Collins, 2009). SVOCs absorbed by plant roots must penetrate the root epidermis, cortex, endodermis, and pericycle to reach the xylem and being transported upward, during

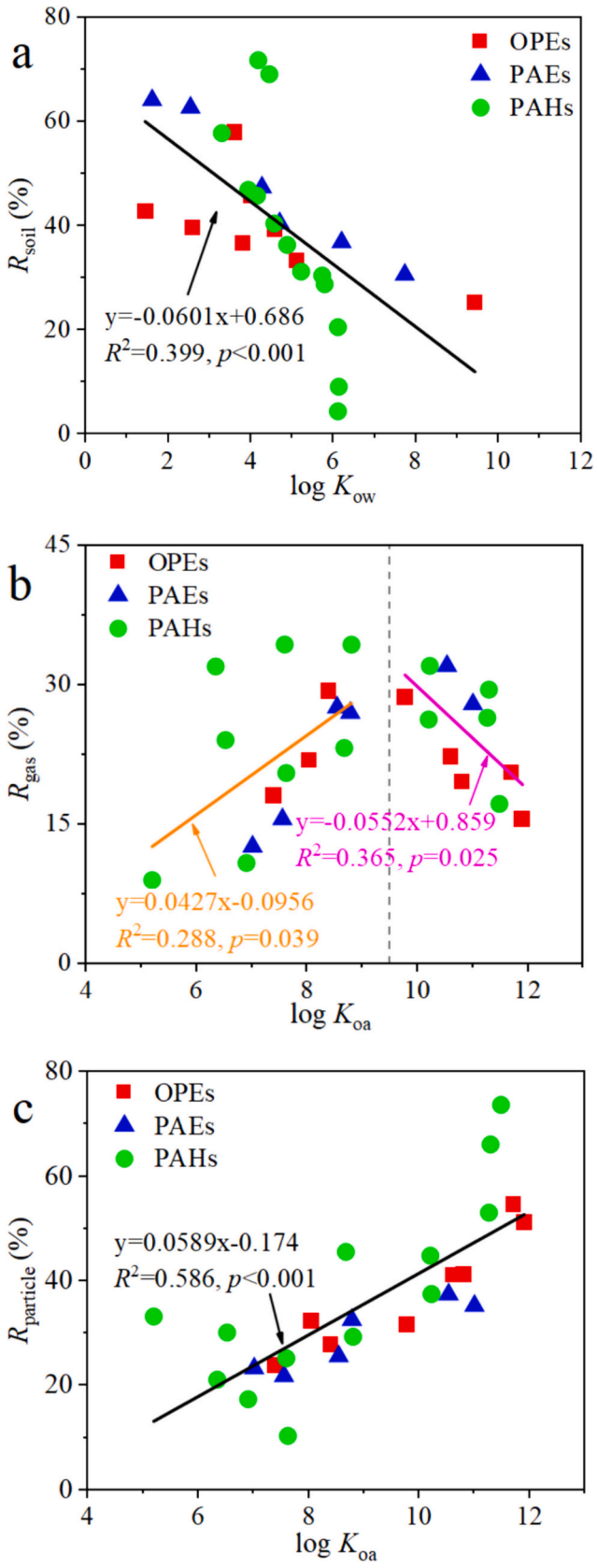

Fig. 4. Correlations between R_{soil} and log K_{ow} (a), R_{gas} and log K_{oa} (b), and $R_{particle}$ and log K_{oa} (c) for OPEs, PAEs, and PAHs.

which SVOCs must pass through at least one cell membrane (Collins et al., 2006). Therefore, the uptake of compounds by plant roots and subsequent transport to plant shoot are determined by a combination of the compound solubility in water and within cell membranes (rich in lipids) (Zhang et al., 2017; Zhu and Kannan, 2019). Furthermore, besides the physicochemical properties of compounds, the root uptake and acropetal translocation within plants may also be affected by plant inter−/intra-species variation and their physiological status, e.g., lipophilic solids content, plant transpiration streamflow rate, and root exudates (Collins et al., 2006; Pan and Chu, 2017). According to a previous field study on the accumulation and translocation of polyhalogenated compounds (PHCs) in rice grown in paddy soils, the logarithm of the stem-to-leaf transport factor (log $TF_{stem\text{-}leaf}$) of PHCs is negatively correlated with log K_{ow} when the soil concentrations of PHCs are low, whereas log $TF_{stem\text{-}leaf}$ is positively correlated with log K_{ow} when the soil concentrations of PHCs are sufficiently high (Zhang et al., 2015). This may be attributed to the form and bioavailability of contaminants, which requires further research.

The correlation between R_{gas} and log K_{oa} had two key segments (parabola) (Fig. 4b). For SVOCs with log K_{oa} value of 5.19–8.8, R_{gas} was positively correlated with log K_{oa} ($R^2 = 0.288$, $p = 0.039$), whereas for SVOCs with log K_{oa} value of 9.77–11.9, R_{gas} was significantly negatively correlated with log K_{oa} ($R^2 = 0.365$, $p = 0.025$). The results can be explained by McLachlan's framework as follows. Compounds with low log K_{oa} < 8.5 are almost entirely derived from foliar uptake in the gaseous form. Compounds with log K_{oa} of 8.5–11 are derived from foliar uptake that may be K_{oa} independent, but are still dominated by kinetically limited gaseous deposition, which depends on gas concentrations, plant age, wind speed, atmospheric stability, canopy structure, and leaf surface roughness. Nevertheless, compounds with high log K_{oa} (>11) are primarily derived from foliar uptake in the particulate form (McLachlan, 1999). A previous study showed that the gaseous phase of PBDEs contributed to PBDE accumulation in leaves and their concentrations decreased with increasing log K_{oa}. However, the log K_{oa} of the target PBDEs was >9.4, which is consistent with the observation made in the second segment (log K_{oa} > 9.77) (Zhu et al., 2020b). $R_{particle}$ was significantly positively correlated with log K_{oa} ($R^2 = 0.586$, $p < 0.001$, Fig. 4c). The observation could be attributed to gas–particle partitioning effects, with compounds having high log K_{oa} being readily adsorbed onto particles, and deposited and desorbed from the particles into the leaves (Collins et al., 2006).

Similarly, the correlations among R_{soil}, R_{gas} (except for BBZP, TMPP), and $R_{particle}$ of OPEs, PAEs, and PAHs with log K_{ow} and log K_{oa} of compound in the inner leaves were consistent with the correlations observed for whole leaf contributions (Fig. S7). The outliers could be explained by the deposition of compounds with high log K_{oa} values and in the particulate phase on the waxy layer of leaves, in addition to limited diffusion of compounds into the inner leaves. The significant correlations between the contributions of the three uptake pathways to SVOC accumulation in leaves and their log K_{ow} and log K_{oa} values supports the generalization of the results of this study to other SVOCs based on their physicochemical properties, which in turn, facilitates the prediction of the actual bioaccumulation coefficients of SVOCs in soil–plant–air systems.

This study had several inherent limitations. (1) The study was conducted under field conditions; therefore, variations in meteorological conditions during the sampling period could have led to fluctuations in the concentrations of SVOCs and subsequently affected the uptake of SVOCs in leaves. (2) The tested rice plants had already absorbed a certain amount of SVOCs before the exposure experiment. SVOCs in the soils inside the chamber can be evaporated into the air. These two situations can both overestimate the contribution of root uptake. (3) The mesh screen cannot prevent fine particles, which may, underestimate the contribution of foliar uptake via particle phase, but overestimate the contribution of foliar uptake via gas phase. (4) This study presumed that the variations in leaf SVOC concentrations under the three tests were

caused by the different uptake pathways, SICs had minimal impacts on the physiological, biochemical, and growth characteristics of rice, and the metabolism and degradation of SVOCs between the different treatments was similar. (5) Ventilation of SICs was based on the chimney effect. External wind speed may have affected the rate of ventilation, as well as the temperature and relative humidity inside the SIC, which may have affected transpiration in rice.

3.5. OPEs, PAEs, and PAHs on the waxy layer and inner leaf

The compositions of OPEs, PAEs, and PAHs on the waxy layer, inner leaf of rice, and air in Test A are shown in Fig. 5a. The compositions of SVOCs in both the waxy layer and the inner leaf differed from that in the air. The observation could be because SVOCs occur in both gaseous and particulate phases in the air, and the compounds cannot diffuse through the waxy layer into the inner leaf at the same rate. The composition proportions of SVOCs with relatively high volatility in the inner leaves were higher than those in the wax layers. The results indicated that more volatile compounds can be more likely to desorb from particles and enter the leaves faster through the wax layer than the less volatile compounds. McLachlan (1999) demonstrated that air-side and plant-side resistances are key factors that influence the overall mass transfer rate of compounds into plant leaves. SVOCs with low log K_{oa} values are more likely to occur in the gaseous phase and their diffusion into plant leaves is directly driven by the gradient in chemical potential between the atmosphere and the cuticular waxes. In contrast, SVOCs with high log K_{oa} values predominantly exist in the particulate phase and their absorption by leaves is driven by a combination of particle erosion and diffusion, which are relatively slow processes (Zhu et al., 2016; Zhu et al., 2020b). The finding is consistent with that of a study conducted by Horstmann and McLachlan who observed that rapid deposition of volatile compounds on the leaf cuticle lead to their rapid accumulation kinetics, which result in high trans-cuticular diffusion (Horstmann and McLachlan, 1998).

The distribution of SVOCs on the plant surface or within a plant can significantly affect the fate of such compounds. SVOCs in the inner leaves are more readily translocated within the plant, whereas SVOCs in the waxy layer can be photolyzed or revolatilized and reemitted into the atmosphere (Wild et al., 2005). We calculated the proportion of SVOCs in the waxy layer to those in the whole leaf (wax & inner leaf). According to the results, OPEs, PAEs, and PAHs in the waxy layer accounted for 1.82–23.4 % (mean: 13.1 ± 7.53 %), 4.82–35.1 % (mean: 22.3 ± 11.7 %), and 9.45–44.7 % (mean: 19.6 ± 9.73 %) of their total burdens accumulated in the whole leaf, respectively (Fig. 5b, details see Table S6, SI). The results showed that the wax layer only stored a limited amount of SVOCs (mean: 18.3 ± 9.92 %), which may be due to the limited mass fraction of the wax layer (10.0–12.7 %, dw/dw). With the increase of rice age, the proportion of the wax on the leaves significantly decreased, especially for the mature stage (Fig. S8, SI), which may, to some extent, affect the concentration and distribution of SVOCs in the wax layer. The proportions of OPEs, PAEs and PAHs in the waxy layer to the whole leaf were significantly positively correlated with log K_{oa} ($R^2 = 0.301$, $p = 0.003$). The results suggest that compounds bound to particles are readily immobilized by the waxy layer and their partitioning into the inner leaf and waxy layer may be influenced by the type of compound. In general, more volatile compounds are readily transferred into the inner leaf, probably due to their rapid diffusion within the waxy layer or through the stoma. A previous study on the retention and transport of PCDD/Fs and PAHs in maize revealed that volatile compounds are often distributed in the inner leaves of maize, whereas compounds bound on particles are mainly present in the waxy layer. Desorption of the compounds from particles is a crucial process that regulates the uptake of low volatile organic contaminants in maize leaves (Kaupp et al., 2000).

3.6. Translocation of OPEs, PAEs, and PAHs in rice grain

Most of the OPEs, PAEs, and PAHs were stored in the inner leaves. Generally, compounds in the inner leaves are more readily translocated within the plant when compared to those in the waxy layers. Several studies (Punshon et al., 2018; Sperotto, 2013; Sperotto et al., 2012) have demonstrated the importance of redistribution of nutrients from vegetative organs to seeds. The root-to-inner leaf ($TF_{root\text{-}inner\ leaf}$), root-to-grain ($TF_{root\text{-}grain}$), inner leaf-to-grain ($TF_{inner\ leaf\text{-}grain}$), and inner leaf-to-husk ($TF_{inner\ leaf\text{-}husk}$) translocation factors (*TF*) were calculated based on the concentrations in rice roots, inner leaves, grains, and husks in Test A. A significant negative correlation was observed between $TF_{root\text{-}inner\ leaf}$ (except for TPHP, TEHP, ANT, CHR and BbF) and log K_{ow} ($R^2 = 0.255$, $p = 0.017$, Fig. 6a; details see Table S7), suggesting that hydrophilic and water-soluble compounds were more readily translocated from rice roots to the inner leaves by the transpiration stream, which is consistent with the observations made in Section 3.3. The result was also consistent with the findings of a previous study, which suggested that a significant negative correlation was observed between *TF* (leaf/root) values and log K_{ow} values of OPEs in rice ($R^2 = 0.834$, $p = 0.03$) (Wang et al., 2023a).

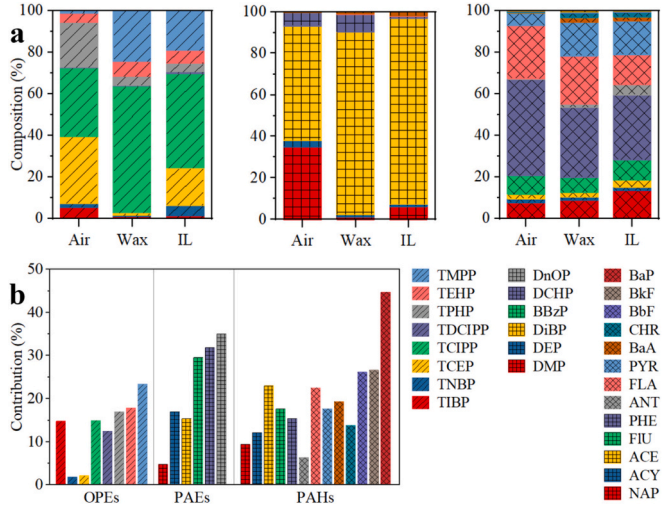

Fig. 5. Compositions of OPEs, PAEs and PAHs in the air, wax layer, and inner leaf (a) and proportion of SVOCs in the waxy layer to their burdens in the whole leaf (b).

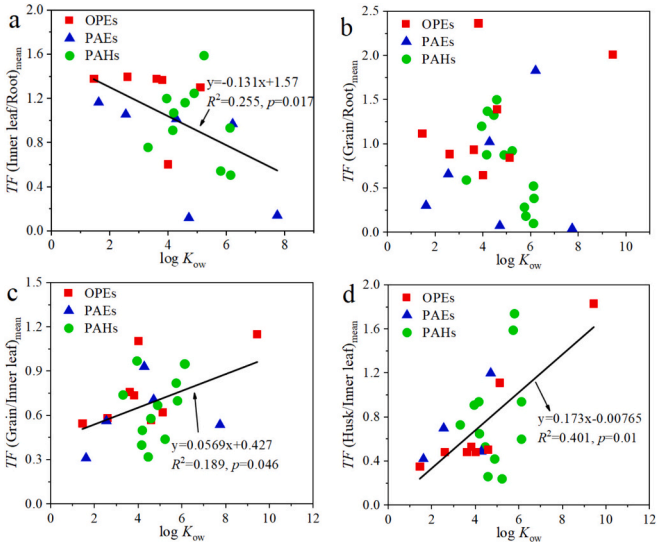

Fig. 6. Correlations between *TF* values of OPEs, PAEs, and PAHs in the upland rice and the log K_{ow}.

$TF_{root\text{-}grain}$ of SVOCs varied widely, and no significant correlation was found between the $TF_{root\text{-}grain}$ and their log K_{ow} values (Fig. 6b), indicating that besides root uptake, foliar uptake via both gas and particle phases may also influence the SVOCs in grains. Moreover, a significant positive correlation was found between $TF_{inner\text{-}leaf\text{-}grain}$ (except DCHP, and BkF) and log K_{ow} ($R^2 = 0.189$, $p = 0.046$) and $TF_{inner\text{-}leaf\text{-}husk}$ (except DCHP, DnOP, and BaP) and log K_{ow} ($R^2 = 0.401$, $p = 0.01$, Fig. 6c and d), suggesting that hydrophobic compounds were more susceptible to be translocated from the inner leaves to the grains. The observation could be because highly hydrophobic compounds bind more readily to the major latex-like proteins in the sap, and in turn, enter the grain mainly through the nutrient pathway (Inui et al., 2013). Furthermore, the observation made could be ascribed to the variations in water-to-lipid ratios in rice tissues (Fig. S10). Typically, the water-to-lipid ratio is higher in leaves than in grains, which could explain the negative correlation observed between $TF_{root\text{-}inner\ leaf}$ and log K_{ow} values, and the positive correlation observed between $TF_{inner\ leaf\text{-}grain}$ and log K_{ow} values (Jandrić et al., 2013).

4. Conclusion

Here, we developed a novel specific exposure chamber with field applicability and conducted a field-based experiment to elucidate the uptake pathways of typical SVOCs, and quantitatively assessed their contributions to organic contaminant accumulations in field-grown rice. Our findings revealed that SVOCs in rice plants are derived from a combination of root uptake and foliar absorption via the gaseous and particle phases, with the particle phase having the highest contribution; but their contributions are parameter-depended, such as chemical and plant properties, environmental factors, environmental concentrations, etc. The results suggested that management strategies aimed at reducing exposure through food ingestions should be developed considering the different uptake pathways in plants, which may depend on the specific contaminants being managed. Our results also implied that most SVOCs in rice leaves are stored in the inner leaves, and hydrophobic compounds in the inner leaves may be more inclined to be translocated to the grains. However, further in-depth studies are needed to obtain substantial evidence regarding the translocation of SVOCs within plants.

CRediT authorship contribution statement

Yan Wang: Writing – review & editing, Supervision, Resources, Methodology, Funding acquisition, Conceptualization. **Fei Gao:** Writing – original draft, Investigation, Data curation. **Yue Xu:** Writing – review & editing, Resources, Methodology. **Timothy F.M. Rodgers:** Writing – review & editing, Methodology, Data curation. **Feng Tan:** Writing – review & editing, Resources.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.scitotenv.2024.174205.

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