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Contribution and loading estimation of organochlorine pesticides from rain and canopy throughfall to runoff in an urban environment

Wei Zhang, Youbin Ye, Yindong Tong, Langbo Ou, Dan Hu, Xuejun Wang*

College of Urban and Environmental Sciences, Peking University, Beijing 100871, China

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ABSTRACT

Concentrations of OCPs in rain, canopy throughfall, and runoff water were measured in the Beijing metropolitan area during the rainy seasons from 2006 to 2007. This study was conducted to calculate the fluxes of OCPs in rain and canopy throughfall, as well as their contributions to runoff. At urban sites, the contribution of HCB and ΣHCHs from rainfall accounted for approximately 50% of the mass in runoff. At the site with significant coverage of landscaping trees, the HCB, ΣHCHs, and ΣDDTs from the net canopy throughfall accounted for approximately 10% of the mass in the runoff. Based on the data obtained in this study, loadings of OCPs (in μg) in rain, net canopy throughfall, and runoff water were calculated. The input of OCPs from rain and canopy throughfall water accounted for a significant portion of urban runoff. In cities undergoing rapid urban sprawl, monitoring and control of the transport of OCPs in urban runoff are essential for effective control of environmental hazards in surface water bodies.

1. Introduction

Cities are an important ecological unit in which the relationship between people and the environment is strong. With the acceleration of urbanization and urban sprawl, degradation of urban environmental quality has become a concern for both academic and administrative sectors [1–3]. In particular, persistent organic pollutants (POPs) in urban environments have recently gained attention [4]. Organochlorine pesticides (OCPs) are the most important species of POPs due to their chronic toxicity, persistence, and bioaccumulation. These compounds may cause great threats to ecosystems and human health. Long residence times and widespread use of these pesticides has resulted in continuously high levels of OCPs in the environment [5–8]. Surface water bodies are subject to pollution by OCPs globally, especially in developing countries, where the safety of surface water bodies is closely related to human health.

Pollutant loading from storm runoff is considered to be an important component of non-point source pollution in urban areas. Urban storm runoff has become a major cause of degradation of the quality of urban water bodies [9,10]. Understanding the magnitude and characteristics of urban runoff pollution, particularly the transfer, mobilization and loading of OCPs in urban environments, is crucial for improving aquatic environmental quality. Urban ground surfaces can be classified as either impervious surfaces or pervious surfaces. Pervious surfaces are usually characterized by landscaping and minor runoff erosion, while impervious surfaces are primarily constructed surfaces such as rooftops, sidewalks, roads, and parking lots, which are covered by impenetrable materials such as asphalt. On impervious surfaces, intense storms may quickly generate large volumes of runoff, followed by dry conditions a short time later. Runoff from urban impervious surfaces may introduce both organic and inorganic pollutants into water bodies, including polycyclic aromatic hydrocarbons, pesticides, halogenated phenols, metals, and de-icing salts [11,12]. However, few studies have focused on organochlorine pesticides contamination in urban runoff.

Urban runoff consists of a collection of pollutants from several input sources, including rain and canopy throughfall from landscaping trees, etc. Thus, an understanding of the transport and fluxes of OCPs in various media is essential for control of surface water quality in urban regions. This study was conducted to estimate the fluxes of OCPs in rain and canopy throughfall and their contributions to runoff. Hexachlorobenzene (HCB), hexachlorocyclohexanes (HCHs), and dichlorodiphenyltrichloroethanes (DDTs) were selected for this analysis because they have historically been used on a large scale, and fresh signatures were observed, indicating possible ongoing use and production [13,14]. The pollution features of a megacity such as Beijing, which is subject to rapid urbanization, are different from those of developed cities. Large cities in developing countries are generally subject to similar development processes and face similar urban environmental problems. From this aspect, the methods employed in this study and the results presented can be generalized to other cities in developing countries.

* Corresponding author. Tel.: +86 10 62759190; fax: +86 10 62759190.
E-mail address: xjwang@urban.pku.edu.cn (X. Wang).

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2. Materials and methods

2.1. Sampling site and procedure

Two years of field sampling was conducted in 2006 and 2007 in Beijing, China. Storm runoff water was collected during ten rainfall events. Runoff sampling was conducted using an automatic sampler (BC-9600, GRASP Technology, Beijing, China) equipped with pre-cleaned 3-L glass bottles. The runoff sampler and flow meter were deployed at the gully of the storm sewer system at three sites, Haidian road (HD), Chengfu vehicle road (CV) and Chengfu bicycle lane (CB). The site locations are depicted in Fig. S1 of the supplementary materials. These sites featured different urban traffic conditions and population densities. Detailed information regarding the sampling sites was provided in our previous study [15]. The target area is a composite residential and commercial catchment developed during the urban sprawling process over the past 15 years. This area is an epitome of the growing Beijing metropolitan area, and subjected to the impact of both primary and secondary emissions of OCP compounds. The drainage areas of CV, CB, and HD are 300 m^2, 140 m^2, and 180 m^2, respectively. The ground surface is fully paved with asphalt. The hydrologic data for each rainfall event can be found in Table S1. According to the rainfall intensity, the sampling was conducted at 5–15 min intervals in the first hour upon the generation of runoff, and at 15–30 min intervals during the second and third hours. The event mean concentration was calculated to evaluate the OCPs contamination of each event. Meteorological data, including the date of rain, rainfall volume, rainfall intensity, duration, and antecedent dry period, were recorded during each rainfall event and between events.

Rain and canopy throughfall water were sampled simultaneously with runoff. Rainwater was collected in aluminum containers and canopy throughfall samples were collected using funnel-shaped stainless collectors with a diameter of 50 cm and drained into brown glass bottles. The collectors were placed 1 m above ground level under landscaping trees (*Fraxinus pennsylvanica*) at the HD site. At the sampling location, the canopy area of the landscaping trees is 54 m^2.

2.2. Laboratory analysis

Samples of runoff, rain, and canopy throughfall water were filtered through pre-baked (450 °C for 4 h) glass fiber filters (142-mm diameter and 0.7-μm pore size) in a stainless-steel filter holder within 24 h of sampling. The filters were then freeze-dried for at least 72 h.

The freeze-dried filters and dust samples were extracted for 20 h with 100 mL dichloromethane in a Soxhlet apparatus. The extracts were then concentrated using a rotary evaporator, solvent-exchanged with hexane, and passed through a chromatography column (30 cm × 10 mm i.d.) filled with silica gel. Next, the column was eluted with 20 mL hexane and then a 50 mL mixture of 3:2 hexane:dichloromethane (v/v) to fractionate OCPs from other interfering materials. Finally, the eluate was evaporated under reduced pressure at 35 °C in a water-bath using a rotary evaporator.

The dissolved OCPs were recovered by passing the filtrate sample through a C18 extraction cartridge (6 mL, 0.5 g, Supelco). The cartridge was conditioned prior to use by washing five times with 10 mL dichloromethane, activating three times with 6 mL methanol, and then washing twice with 10 mL deionized water. The samples were then passed through the cartridges under vacuum, after which the trapped compounds were extracted from the sorbent cartridge with 10 mL dichloromethane. The final extracts were further concentrated using the rotary evaporator according to the method described above.

The final extracts were adjusted to about 1 mL under a gentle stream of nitrogen, after which known quantities of internal standard 4,4′-dichlorobiphenyl were added and then transferred to a sample vial for GC–MS analysis. Next, the samples were analyzed on an Agilent 6890 GC equipped with a ^{63}Ni electronic capture detector (ECD), an automatic injector, and a HP-5 column (30 m × 0.32 mm i.d. × 0.25 μm film thickness). The GC system was operated in splitless mode with a venting time of 0.75 min. The injector and detector temperature was 220 °C and 280 °C, respectively. The GC operating procedure was as follows: temperature program 50–150 °C at 10 °C/min, followed by 150–240 °C at 3 °C/min, where it was held for 15 min. Pure nitrogen (>99.999%) was used as both the carrier (1.0 mL/min) and the makeup (60 mL/min) gas. The target compounds were identified based on their retention time (previously confirmed with GC–MS) and quantified using an internal standard. A mixed working standard was used for calibration.

2.3. Quality assurance and control

Two procedural blanks were determined simultaneously for each batch of sample analysis by going through the same extraction and cleanup procedures. The measured procedure blanks were one order of magnitude lower than most of the sample measurements. The possible degradation of p,p′-DDT in the injector was routinely checked by running the standard solution. Two duplicates were analyzed for randomly selected samples in each batch to check for reproducibility. The relative percent difference for individual OCPs identified in duplicate samples was <15%. Surrogate (TCMX) recoveries were 92 ± 19% with water filtrate samples and 76 ± 13% with water filter samples. The limit of detection of the method (LOD (ng)) was defined as the mean blank mass plus three times the standard deviation of method blanks (LOD = mean blank value + 3SD). The LODs for OCPs ranged from 0.006 to 0.08 ng for filtrates and 0.01 to 0.2 ng for filters. Sample quantities exceeding the LOD were quantified and blank-corrected by subtracting the mean blank amount from the sample amount.

The extraction and elution solvents including hexane, acetone, and dichloromethane were of analytical grade (Beijing Chemical Reagent Co., China) and were purified by distillation prior to use. Silica gel (60–80 mesh, Beijing Chemical Reagent Co., China) was heated at 450 °C for 4 h, kept in a sealed desiccator, and reactivated at 130 °C for 16 h immediately prior to use. The working standard solution was prepared by diluting a commercial mixed stock (J&K Chemical Ltd., USA) with hexane. 2,4,5,6-tetrachloro-m-xylene (TCMX) and 4,4′-dichlorobiphenyl (J&K Chemical Ltd., USA) were used as a surrogate and internal standard, respectively.

2.4. Unit pollutant loading rate

The unit pollutant loading rate was calculated using the following equation [16]:

$$r = \frac{\sum C_i \times \Delta Q_i \times \Delta t_i \times R}{\sum I_i \times \Delta t_i \times A} \times 10^{-3} \quad (1)$$

where, r is the unit pollutant loading rate (g/km^2/year); C_i is the time variable concentration (ng/L); ΔQ_i is the time variable runoff rate (L/min); Δt_i is the sampling time interval (min); I_i is the rainfall intensity (mm/min); R is the annual total rainfall (mm/year); and A is the runoff flushing area (m^2).

3. Results and discussion

3.1. Flux of OCPs in rain and canopy throughfall

During the sampling campaign, rain samples were collected during ten rainfall events, with canopy throughfall water being simultaneously sampled under the landscaping trees at the HD site during eight of these events. These rainfall events covered a variety of rainfall conditions including heavy and poor rainy weeks. The wet flux of OCPs in rain and canopy throughfall was presented in ng/m^2 and calculated by the following equations:

flux of OCPs in rain

$$= (\text{OCP concentration in rain}) \times (\text{rainfall volume}) \quad (2)$$

flux of OCPs in canopy throughfall

$$= (\text{OCP concentration in canopy throughfall}) \times (\text{rainfall volume}) \quad (3)$$

The fluxes of the measured OCPs in the rain and canopy throughfall are listed in Table S2 of the supplementary materials. The fluxes of HCB, ΣHCHs (sum of α-, β-, γ-, and δ-HCH), and ΣDDTs (sum of o,p′-DDT, p,p′-DDT, o,p′-DDD, p,p′-DDD, o,p′-DDE, and p,p′-DDE) in rain are shown in Fig. 1a. For most events, the flux of ΣHCHs in rain was generally larger than that of HCB and ΣDDT. It is interesting to note that extremely large rain fluxes of HCB and ΣDDTs were observed in June 27, 2007. This event occurred only three days after a previous rainfall event in June 24, 2007, and the rain fluxes of HCB and ΣDDTs in June 24, 2007 were not large. Therefore, the large fluxes observed in June 27, 2007 may indicate an abnormal emission during the period between June 24 and 27, 2007. Canopy throughfall can be used as an indicator of the atmospheric deposition processes occurring in the canopy [17]. The fluxes of HCB, ΣHCHs, and ΣDDTs in canopy throughfall water are shown in Fig. 1b. It should be noted that the canopy throughfall fluxes for HCB and ΣDDTs were also high in June 27, 2007, indicating a possible emission to air during this period.

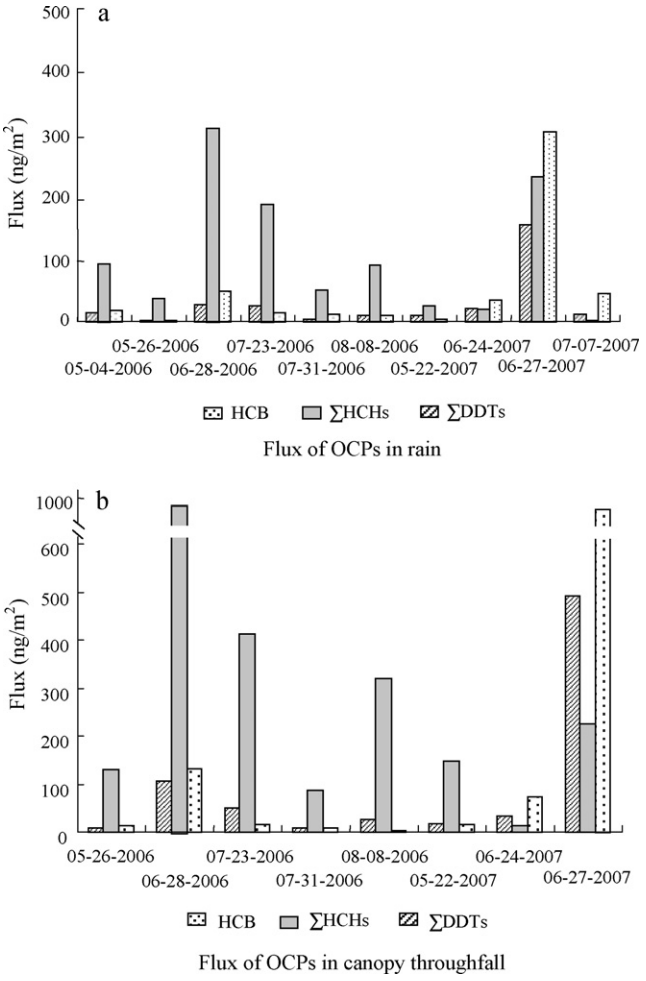

Fig. 1. Estimated fluxes of HCB, ΣHCHs and ΣDDTs in rain and canopy throughfall (ΣHCHs is the sum of α-, β-, γ-, and δ-HCH; ΣDDTs is the sum of o,p′-DDT, p,p′-DDT, o,p′-DDD, p,p′-DDD, o,p′-DDE, and p,p′-DDE).

Canopy throughfall includes wet deposition and the portion of dry deposition that is washed from the canopy. Assuming that the pollutant deposited on leaves during the preceding dry days is thoroughly washed off by rain water, net canopy throughfall, which is the difference between the canopy throughfall and wet deposition, is a more preferable parameter to measure the atmospheric deposition on leaves. The flux of OCPs in the net canopy throughfall is defined as the differences in canopy throughfall flux and rain flux, and can be calculated as:

flux of OCPs in net canopy throughfall

$$= \text{flux of OCPs in canopy throughfall-flux of OCPs in rain} \quad (4)$$

The flux of the measured OCPs in the net canopy throughfall was calculated for each event, and summarized in Table S2. The result for HCB, ΣHCHs, and ΣDDTs are shown in Fig. 2. In the rainfall event on June 27, 2007, large fluxes of HCB and ΣDDTs in the net canopy throughfall were noted. This is further evidence of the presence of some abnormal emissions of HCB and ΣDDTs during the period between June 24 and June 27, 2007, but further source information was not determined. In this study, negative values of the net canopy throughfall fluxes were observed for HCB on July 31 and August 8, 2006 and for the ΣHCHs on June 24 and June 27, 2007 (Fig. 2a and b). These negative values indicated that the mass in rain was higher than the mass in the canopy throughfall. This may occur when the canopy surfaces are relatively clean and the pollutants in rain water may be intercepted on the surfaces of leaves. The following interactions between the canopy surfaces and the rain water passing over them may occur: (1) pollutants deposited on canopy surfaces may be washed off and positively contribute to the mass in canopy throughfall water; (2) canopy surfaces may also intercept chemicals from rain water and abate the mass in canopy throughfall water. The net result of the interactions should be determined by factors such as the retention capacity of leaves, the clean level that may be affected by atmospheric deposition and antecedent dry days, and the flushing power, which may be influenced by rainfall intensity. These factors can be investigated in future studies.

3.2. Contribution of rain and canopy throughfall to runoff

The contribution of rain and canopy throughfall to runoff was calculated based on the fluxes and the mass loadings in runoff. The results for rain and net canopy throughfall are summarized in Tables S3 and S4, respectively. The mass contribution from rain to runoff at the CB and CV sites are shown in Fig. 3a and b, respectively. The average contribution percentages were 62% (HCB), 56% (ΣHCHs), and 35% (ΣDDTs) at the CB site, and 51% (HCB), 49% (ΣHCHs), and 31% (ΣDDTs) at the CV site. Generally, rainfall scavenging of pollutants from the atmosphere accounts for a significant portion of HCB and ΣHCHs in runoff, resulting in a percentage contribution of approximately 50% to the mass in runoff. These

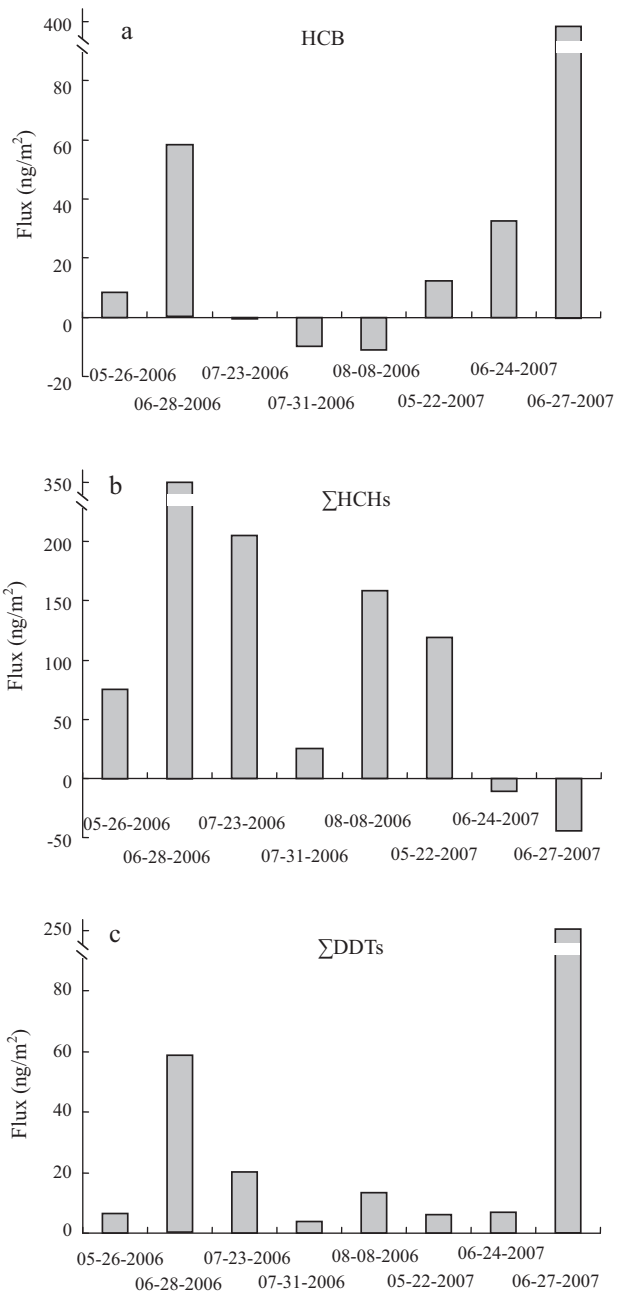

Fig. 2. Fluxes of HCB, ΣHCHs and ΣDDTs in the net canopy throughfall (ΣHCHs is the sum of α-, β-, γ-, and δ-HCH; ΣDDTs is the sum of o,p′-DDT, p,p′-DDT, o,p′-DDD, p,p′-DDD, o,p′-DDE, and p,p′-DDE).

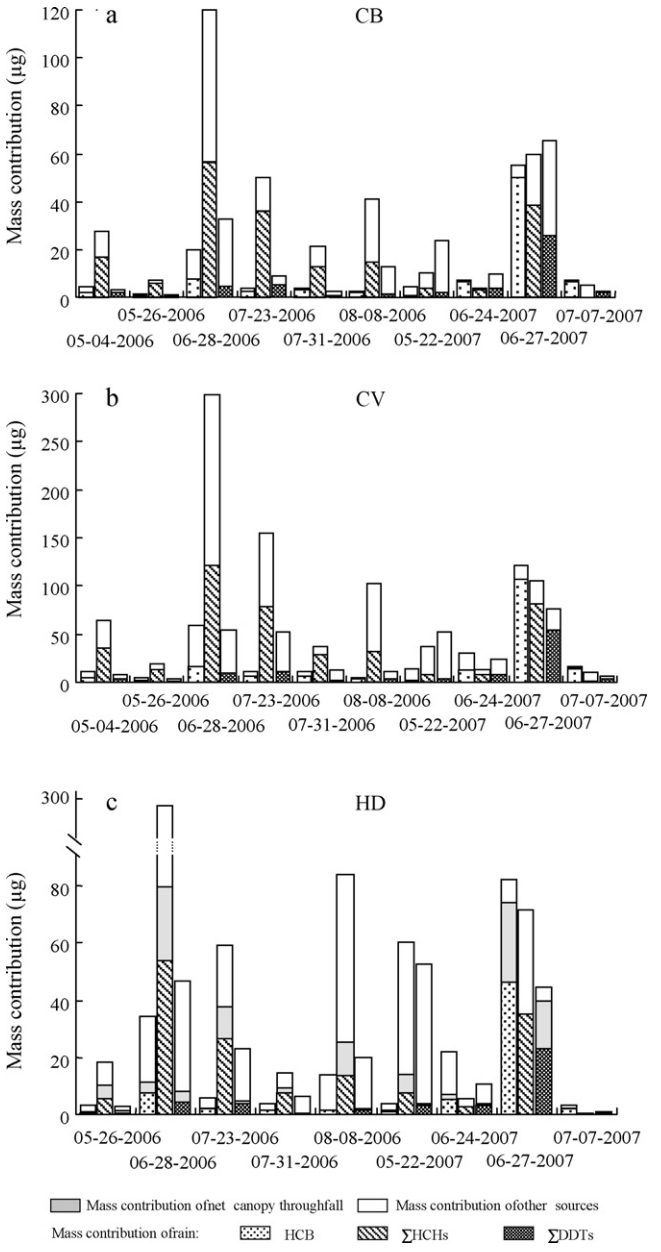

Fig. 3. Mass contributions of rain and net canopy throughfall to runoff at the CB, CV and HD sites. From left to the right is HCB, ΣHCHs and ΣDDTs (ΣHCHs is the sum of α-, β-, γ-, and δ-HCH; ΣDDTs is the sum of o,p′-DDT, p,p′-DDT, o,p′-DDD, p,p′-DDD, o,p′-DDE, and p,p′-DDE).

findings indicate that wet deposition should be a major source of HCB and ΣHCHs in urban runoff. Conversely, for ΣDDTs, the rain flux accounted for a lower percentage (approximately 30%) of the mass found in runoff. In our previous study [18], comparison of OCP profiles revealed that DDT related substances showed a higher affinity to dust. In urban environments, dust that has accumulated on impervious surfaces primarily originates from atmospheric dry deposition. Thus, atmospheric dry deposition is a key source of DDTs transported to urban runoff.

At urban sites with significant coverage of landscaping trees, canopy throughfall water may deliver a cumulative amount of pollutants that have been washed off from canopy surfaces. Thus, the contribution of OCPs in canopy throughfall water cannot be neglected. According to the field observations, the HD site was covered with a canopy of landscaping trees (*F. pennsylvanica*), and the canopy area was estimated to be 54 m^2 at the drainage area of the sampling location. The contribution of the net canopy throughfall to runoff water at the HD site is shown in Table S4. The mass contribution of HCB, ΣHCHs, and ΣDDTs was plotted using a bar graph and is presented in Fig. 3c. At the HD site, the net canopy throughfall accounted for 11% (HCB), 11% (ΣHCHs), and 9% (ΣDDTs) of the mass in runoff, and the average contribution of rain was 44% (HCB), 46% (ΣHCHs), and 28% (ΣDDTs). Although the contribution of net canopy throughfall was lower than that of rain, it still accounted for a significant percentage of OCPs input to urban runoff.

3.3. Loadings of OCPs in urban runoff, rain, and canopy throughfall

Because the urban storm runoff is directly discharged into surface waters in Beijing, it is important to assess the environmental

Table 1
Annual unit loading rate of OCPs in runoff, rain and net canopy throughfall in Beijing urban areas (g/km^2).

OCPs	Runoff				Rain	Net canopy throughfall
	CB	CV	HD	Mean		
α-HCH	9.8	13.4	18.3	13.8	1.5	2.4
β-HCH	10.8	8.5	19.3	12.9	2.2	2.6
γ-HCH	16.8	18.1	17.2	17.4	1.8	2.5
δ-HCH	13.2	16.3	14.6	14.7	2.0	1.3
o,p′-DDE	4.3	2.7	4.7	3.9	0.1	2.0
p,p′-DDE	3.6	3.8	4.0	3.8	0.3	1.3
o,p′-DDD	3.3	4.4	4.1	4.0	0.7	0.0
p,p′-DDD	5.1	3.3	3.7	4.1	0.4	0.0
o,p′-DDT	1.4	2.4	2.4	2.1	0.2	0.0
p,p′-DDT	4.1	3.6	4.6	4.1	0.1	0.4
HCB	15.0	18.1	20.0	17.7	3.3	5.0
ΣHCHs	50.6	56.3	69.5	58.8	7.5	8.8
ΣDDTs	22.0	20.2	23.5	21.9	1.9	3.8

Table 2
Estimated annual OCPs loading in runoff, rain and net canopy throughfall in 2002 and 2020 in Beijing urban areas (kg).

OCPs	Runoff		Rain		Net canopy throughfall	
	2002	2020	2002	2020	2002	2020
α-HCH	4.3	5.8	1.2	1.8	0.1	0.3
β-HCH	4.0	5.4	1.6	2.4	0.2	0.4
γ-HCH	5.4	7.3	1.7	2.6	0.2	0.4
δ-HCH	4.6	6.2	1.4	2.2	0.1	0.2
o,p′-DDE	1.2	1.6	0.1	0.2	0.3	0.5
p,p′-DDE	1.2	1.6	0.2	0.3	0.1	0.2
o,p′-DDD	1.2	1.7	0.4	0.6	0.02	0.04
p,p′-DDD	1.3	1.7	0.2	0.3	0.04	0.07
o,p′-DDT	0.6	0.9	0.1	0.2	0.01	0.03
p,p′-DDT	1.3	1.7	0.05	0.1	0.04	0.07
HCB	5.5	7.4	3.0	4.6	0.5	0.9
ΣHCHs	18.2	24.7	5.8	9.1	0.6	1.1
ΣDDTs	6.8	9.2	1.0	1.6	0.5	1.0

impact of this amount of OCPs. The annual unit loading rate of OCPs in urban runoff from the three sampling sites was calculated according to Eq. (1), and is summarized in Table 1. Briefly, the average unit loading rates for HCB, ΣHCHs, and ΣDDTs were 17.7 g/km^2/year, 58.8 g/km^2/year, and 21.9 g/km^2/year, respectively. The loading rate of OCPs in rain and net canopy throughfall per unit area was calculated assuming that the annual rainfall volume is 500 mm and listed in Table 1. Because the study area is representative of the Beijing urban region, it is reasonable to extend the unit loading rate of the subject area to the entire urban region of Beijing. Based on this assumption, the average unit loading rate in this study can be generalized to approximately estimate the annual OCPs loadings from runoff, rain, and net canopy throughfall in the Beijing urban area.

According to the Urban Planning of Beijing (2004–2020), the urban area of Beijing was 500 km^2 at the end of 2002, and is expected to be 778 km^2 in 2020. Based on data provided by the Landscaping Planning of Beijing (2005–2020), at the end of 2002, the impervious area accounted for 62% of the Beijing urban region, and the area covered by landscaping trees accounted for 23% of the total urban area. In 2020, the impervious surfaces will account for 54% of the total urban area, and the area coverage of landscaping trees will increase to 27%. According to these planning data, as well as the unit loading rate in runoff, rain, and net canopy throughfall in Table 1, the OCPs mass loading in runoff, rain, and net canopy throughfall water was calculated and summarized in Table 2. The mass loading of OCPs in runoff from impervious areas in Beijing was estimated to be 30.5 kg in 2002, and 41.3 kg in 2020. The loading estimate was obtained by summing the loadings of HCB, HCH related substances, and DDT isomers. Based on the planning data and assuming a constant annual rainfall volume of 500 mm, the mass loading of OCPs in rain that contributes to the runoff from urban impervious areas was estimated to be 9.8 kg in 2002 and 15.3 kg in 2020. For the area planned to be covered with landscaping trees, the mass loading of OCPs in net canopy throughfall was calculated to be 1.6 kg in 2002, and 3.0 kg in 2020. These loadings will also be transported to urban runoff. It should be noted that these calculations were based on an assumption of no changes in OCP levels and emission to air and constant meteorological parameters such as annual rainfall volume. Since the usage of organochlorine pesticides has been banned in China, the assumption of constant OCP levels may lead to an over estimate of the OCPs loading in 2020. However, our previous study indicated there may be possible ongoing local or regional emission sources of OCPs in Beijing area [18], and other studies also reported little sign of any declining trend in OCP concentrations in other areas in China [6,8,13]. Accordingly, such estimations may provide a reference for authorities to understand the magnitude of the loading of OCPs from rain, net canopy throughfall, and runoff, and will help in effective control of urban water quality.

4. Conclusions

Fluxes of OCPs in rain and net canopy throughfall, and their contributions to runoff were calculated in this study. The loadings of OCPs from runoff, rain, and canopy throughfall were also estimated for urban areas. These estimates suggest that the input of OCPs from rain and canopy throughfall water contributed a significant portion to the total in urban runoff. In cities of developing countries that are currently undergoing rapid urban sprawl, runoff from highly urbanized watersheds can carry a substantial amount of OCPs and be potentially dangerous to urban water systems. Therefore, transport of OCPs in urban runoff and its related media needs to be considered and reduced. It is our suggestion that toxic substances

in multiple environmental media, including OCPs, should be monitored systematically, especially in large urban agglomerations in developing countries.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.jhazmat.2010.09.091.

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