

When the particle radius is much smaller than the adsorbate penetration distance, as is the case for SPAC, the adsorption capacity is independent of particle size, and the chemistry of the pore surface is the main determinant of adsorption capacity. For SPAC adsorption of hydrophobic compounds such as geosmin, adsorption capacity per unit BET surface area is correlated with carbon hydrophilicity, as indexed by oxygen content. Selection of a less-hydrophilic carbon is therefore recommended for removal of hydrophobic compounds with a SPAC. In contrast, for a PAC, hydrophilicity may not be a straightforward index of adsorption capacity; the reason is that only part of the interior region of the carbon particle may be utilized because of the limited penetration of the adsorbate molecules, even though the pore surface has a strong affinity for the adsorbate.

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Appendix A. Supplementary data

Supplementary data related to this article can be found at <http://dx.doi.org/10.1016/j.watres.2015.08.017>.

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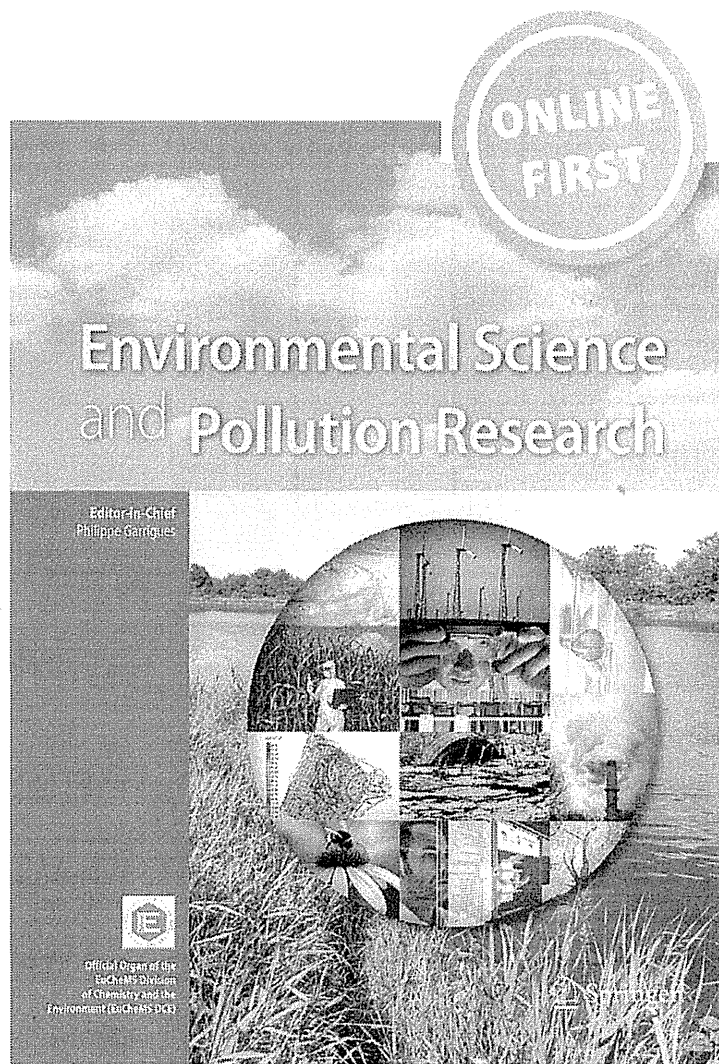
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RESEARCH ARTICLE

Groundwater screening for 940 organic micro-pollutants in Hanoi and Ho Chi Minh City, Vietnam

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Abstract Groundwater is the main source of drinking water for more than half of the residents of Hanoi (HN). It also provides about one third of the total water demand for residents of Ho Chi Minh City (HCM). However, due to rapid urbanization and frequent discharges of untreated urban wastewater to surface water, freshwater is widely contaminated by man-made chemicals, which may result in groundwater pollution. As part of an ongoing campaign to collect baseline information on the occurrence of organic micro-pollutants (OMPs) in the aquatic environment in Vietnam, 43 water samples were collected from 26 groundwater wells in HN (22) and

HCM (4) in September 2013 and August 2014. Samples were analyzed by gas chromatography-mass spectrometry (GC-MS) and the resulting chromatograms were screened for 940 OMPs by an automated identification and quantification system (AIQS) within a GC-MS database. A total of 74 compounds were detected, with between 4 and 43 (median 18) compounds found at each site. Overall, contamination levels were low, with over 89 % of the detected concentrations that were less than $0.5 \mu\text{g L}^{-1}$. Results suggest that most of the sampled aquifers have been impacted by non-point source pollution. Most of the contaminants detected are either currently not regulated in drinking water or are present at low levels. A health risk assessment for detected contaminants implied that there were no risks to humans. Since this study was based on a limited number of samples, especially in HCM, further, more detailed studies on the occurrence of OMPs in groundwater in HCM and a full risk assessment of detected contaminants should be prioritized.

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Keywords GC-MS · Organic micro-pollutants · Comprehensive analysis · Groundwater · Occurrence

Introduction

Groundwater is an important source of water for industrial production and domestic use that has been a key driver for the geographical location and development of cities. Hanoi and Ho Chi Minh City, the two largest cities and most highly industrialized and urbanized areas in Vietnam, rely on groundwater to meet the high demand for freshwater. Huge amounts of groundwater are abstracted to meet the demands of residents, services, and industries. UNICEF (2001) reported that there were three types of groundwater exploitation in HN: (1) publicly exploited wells (large diameter) that are managed

by the Hanoi Clean Water Business Company and about eight major public exploitation wells that supply water to city treatment facilities, and which process 400 m³ of water per day; (2) private wells (500 medium diameter wells) that belong to businesses and factories and have a pumping rate of 120 m³ day⁻¹; and (3) 10,000 small diameter wells in four suburban districts sponsored by UNICEF that have a pumping rate of 30 m³ day⁻¹. The rural population of HN uses groundwater that is pumped from individual private (family based) tube wells as their source of drinking water. In HCM, high rates of industrialization and urbanization have resulted in a sharp increase in the demand for water. Out of the three sources of water (river water, groundwater, rainwater), groundwater use accounted for 34 % of the total water demand for HCM in 2005 (IGES 2007). Water is abstracted from over 150,000 wells or boreholes in HCM, demonstrating that groundwater makes an important contribution to the city's water supply (IGES 2007). In 2005, groundwater exploitation was estimated at about 611,000 m³ day⁻¹, of which 346,000 m³ day⁻¹ and 265,000 m³ day⁻¹ were used for domestic activities (residents, public works, and services) and industry, respectively (Nga 2006).

Just as vast quantities of freshwater are consumed, correspondingly large quantities of wastewater are produced that may, through various transport pathways, have an impact on the groundwater. Groundwater may suffer pollution from many sources. For example, groundwater in urban areas is likely to be impacted by pollutants from sewage, industrial activities, and diffuse leakages from reticulated sewerage and septic systems (Ellis 2006), while the main contributors to groundwater pollution in rural areas are probably fertilizers, agrochemicals, and veterinary medicines related to agriculture and animal waste (Boxall et al. 2004; Vázquez-Suñé et al. 2010). There have been many studies of heavy metals (especially dissolved iron(II), arsenic, and manganese), nitrogen compounds (nitrate and ammonium), and pathogens (coliforms) in groundwater of HN and HCM (Berg et al. 2001; Nga 2006; Buschmann et al. 2008; IGES 2007; Takizawa 2008). In these studies, the concentrations of some of the detected contaminants exceeded the Vietnamese Groundwater Standards and the World Health Organization (WHO) guidelines (IGES 2007; Buschmann et al. 2008).

Eighty thousand man-made chemicals are currently used in vast quantities in today's society (Schaidler et al. 2014) for a wide range of purposes including the production and preservation of food, industrial manufacturing processes, and for human and animal health care (Lapworth et al. 2012). In recent decades, there has been a growing interest in the occurrence, environmental fate, and potential toxicity of these so-called organic micro-pollutants in the terrestrial and aquatic environment (Kümmerer 2009; Carrara et al. 2008; Kishida et al. 2007; Swartz et al. 2006; Kolpin et al. 2002; Nhan et al. 2001; Daughton and Ternes 1999). These OMPs can make

their way into the aquatic environment by a variety of pathways such as direct discharges from wastewater treatment plants and landfills and application of human and animal waste to farmland (Barnes et al. 2008). However, while there is growing concern about groundwater contamination by OMPs, their fate and transport in groundwater remains relatively poorly understood compared with other types of freshwater pollution. Further, even with this concern, no comprehensive survey has been carried out in Vietnam to determine the extent of contamination by OMPs in groundwater.

In a previous study, we evaluated the occurrence of a range of OMPs (940) in surface waters and sediments in Vietnam (Duong et al. 2014a, b), including pharmaceuticals and personal care products (PPCPs); endocrine disrupting chemicals (EDCs); lifestyle compounds; sterols; persistent organic pollutants (POPs) such as organochlorine pesticides (OCPs) and polychlorinated biphenyls (PCBs); pesticides; polycyclic aromatic hydrocarbons (PAHs); plasticizers; fire retardants; antioxidants; as well as numerous other compounds that originate from industrial and domestic sources. Two hundred and thirty-five and 185 analytes were detected in surface waters and sediments, respectively, indicating that the Vietnamese aquatic environment was contaminated by a wide range of OMPs. The findings suggested that the fate and transport of OMPs originating from households, such as sterols, EDCs, PPCPs, and pyrethroid insecticides in the environment, deserved greater attention from a research perspective. EDCs (such as bisphenol A (BPA), 4-tert-octylphenol (OP), and nonylphenol (NP)) have confirmed estrogenic potential even at low concentrations. In particular, elevated concentrations of OP in surface water may be hazardous as it can cause estrogenic effects in fish and other aquatic organisms. There is also concern about legacy POPs because of their persistence, accumulation in sediments, high bioaccumulation potential, and harmful biological effects. Some banned OCPs and PCBs remain at high concentrations in sediments. Concentrations of some plasticizers (such as phthalate compounds that are widely used in industrial production) and PAHs in sediments exceeded the standard values for protecting aquatic life, demonstrating that there may be toxicological stress on aquatic biota. In addition, Hoai et al. (2010) reported that 95 % of HN's wastewater effluents are discharged into its inner rivers without treatment. Hence, a large number of contaminants detected at elevated levels in Vietnamese surface waters and sediments may cause groundwater pollution. In spite of this, to date, there have been no comprehensive studies on the occurrence of OMPs in groundwater in Vietnam.

To address this knowledge gap, we undertook a comprehensive study of 940 OMPs, representing a wide variety of uses and origins, in groundwater from 26 wells in the two biggest cities in Vietnam, where previous research had demonstrated that surface water was heavily polluted by chemicals that originated from urban sewage discharges. The objectives

of this study were to (1) gain an overview of the occurrences and levels of OMPs in groundwater in Vietnam, (2) find potential sources of detected contaminants, and (3) to assess the potential risk of detected contaminants.

Materials and methods

Reagents and materials

All solvents used (acetone, dichloromethane, methanol, and n-hexane) were of pesticide residue quality and were purchased from the Kanto Chemical Co. (Tokyo, Japan). Reagents used for surrogate compounds and internal standards (IS) were purchased from the Kanto Chemical Co., Wako Pure Chemical Industries (Osaka, Japan), Wellington Laboratories (Ontario, Canada), and Sigma-Aldrich Japan K.K. (Tokyo, Japan) and were used in a 10 µg mL⁻¹ acetone solution or in a 10 µg mL⁻¹ hexane solution (IS). Sodium sulfate, disodium phosphate, and monosodium phosphate (99 % grade) were supplied by the Kanto Chemical Co. and were used to prepare a buffer solution of 1 mol L⁻¹ NaH₂PO₄-Na₂HPO₄ (pH 7.0). Empore SDB-XD SPE disks (SDB polymer, 47 mm, XD) and Empore Activated Carbon SPE disks (47 mm, AC) were purchased from 3M Co. (St. Paul, MN, USA). Whatman GMF 150 glass fiber filters (47 mm) were purchased from GE Healthcare UK LTD. (Buckinghamshire, UK).

Water sampling

Samples were collected from groundwater wells in densely populated urban and suburban areas in HN and HCM. The wells were not necessarily used for drinking water but were thought to be contaminated from either unsewered household or industrial wastewaters, which suggested that the water may have been subject to organic pollution. Samples were collected twice in HN in the rainy seasons of 2013 and 2014 to examine if there was any variability in the occurrence and concentrations of the OMPs in the groundwater at different times of the year. A total of 22 samples were collected from 18 wells in HN and 4 wells in HCM in September 2013, while 21 samples were collected from 21 wells in HN (Fig. 1, Table S1) in August 2014. Overall, the samples were collected twice from 17 wells in HN. A bioassay test in September 2013 showed that samples from wells HN14 and HN3 had elevated concentrations and high toxicity values, so, in August 2014, samples were collected from four additional wells (HN3A, HN3B, HN14A, HN14B), located close to HN3 and HN14 (Fig. 1), and were investigated for OMPs as well as possible pollution sources. Samples were only collected once (September 2013) from one well in HN (HN3) and four wells in HCM (HCM1-4) because the wells were closed. Detailed

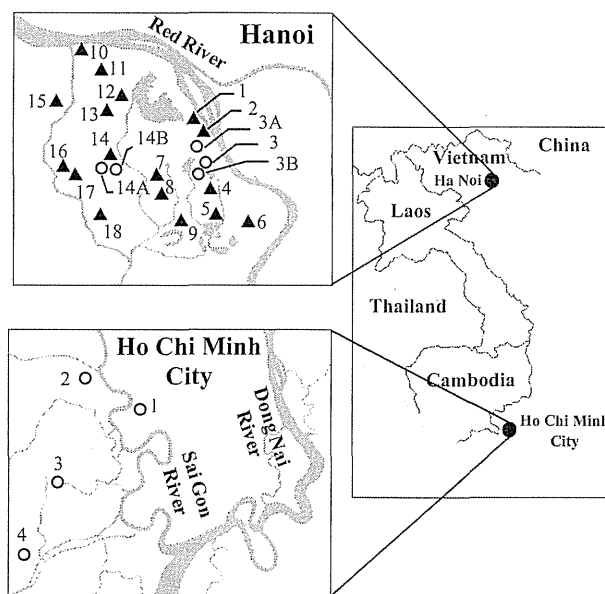


Fig. 1 Locations of the 26 wells sampled in Hanoi and Ho Chi Minh City. Two samples were collected (triangle) and only one sample was collected (circle). A detailed description of each well is provided in Table S1

information about the sampling locations is provided in Table S1 in the supplementary data.

Each well was pumped for 5 min before sampling to ensure that the water in the well was representative of the aquifer, and then, 1 L of a water sample was collected in a 1-L glass bottle, previously washed with acetone and purified water. Once collected, samples were labeled and stored in ice boxes and transported to a laboratory in the Institute of Environmental Technology (IET), Vietnam, where they were then refrigerated for 1–2 days until they were processed. Solid-phase extraction (SPE) of samples was carried out at the laboratory, and samples were kept in loaded SPE disks during transportation to Japan for further analysis.

Sample extraction and analysis

The groundwater samples were analyzed for 940 OMPs (Duong et al. 2014b) using the method of Jinya et al. (2013). Briefly, after addition of surrogates (1 µg) to 1 L of a water sample, the pH of the sample was adjusted to 7.0 with a phosphate buffer. The sample was loaded onto disks that had been previously pre-conditioned with dichloromethane, acetone, methanol (10 mL each), and water (20 mL) in the order: GMF 150, XD, and AC. After extraction, the disks were dried by applying a vacuum for 30 min. The disks were then eluted separately with 10 mL of acetone, 5 mL of dichloromethane (XD), and 10 mL of acetone (AC). The eluates were combined and finally concentrated to 1 mL under a gentle nitrogen stream and then was spiked with 100 µL of IS solution (10 µg mL⁻¹) prior to instrumental analysis (GC-MS selected ion monitoring (SIM)/total ion monitoring (Scan) (QP-2100

Plus, Shimadzu, Kyoto, Japan) and GC-MS/MS selected reaction monitoring (SRM) (TSQ Quantum XLS, Thermo Fisher Scientific, Yokohama, Japan)). Measurement conditions for both instruments are described by Duong et al. (2014b). Total ion chromatograms from the GC-MS Scan were processed by an identification and quantification system in a GC-MS database (AIQS-DB) (Kadokami et al. 2005) that was able to determine the concentrations of the 940 OMPs. Substances targeted by SIM and SRM were quantified by the internal standard method as reported by Duong et al. (2014b). If samples were measured by multiple methods (Scan, SIM, and/or SRM), we preferentially used the results from SRM, followed by SIM, and finally, Scan. The method detection limits (MDLs) of PAHs, OCPs, sterols, and PCBs measured by SIM were 1, 2, 8–320, and 0.4–1.6 ng L⁻¹, respectively, while the MDL of OCPs and PCBs measured by SRM ranged from 0.1 to 0.4 ng L⁻¹. The MDL of the remaining compounds measured by TIM were between 5 and 500 ng L⁻¹.

Quality control

Quality controls consisted of blank analysis, duplicate analysis, and recovery tests of 13 surrogates (deuterium-labeled internal standards) that were spiked prior to extraction. These surrogates had a similar range of physicochemical properties as were found among the OMPs measured. Good recoveries (69–115 %) were obtained for 10 out of the 13 surrogate compounds, except for highly polar and/or volatile compounds such as 2-fluorophenol. Tris(2-ethylhexyl)phosphate-d₅₁ showed high recovery rates (140 %), probably due to the matrix effect (Kadokami et al. 2012). Relative standard deviations of surrogates were mostly less than or equal to 20 % (Table S2), confirming that sample analyses were acceptably precise. Blank samples were processed regularly for every set of five samples using 1 L of purified water previously passed through SPE disks. When reporting data, blank concentrations were subtracted from sample concentrations. Duplicate analyses were performed on the sample from well HN14 (Table S3), and the relative average deviations for 10 of the 13 detected substances were below 20 %, indicating that the analyses gave results with good reproducibility and that the precision was sufficient for environmental surveys.

Results and discussion

We have mainly based our discussion on the results from HN because the number of samples and sampling sites were limited in HCM, where samples were collected once at four sites, compared with HN, where samples were collected twice at 22 sites. These initial findings about OMPs in groundwater in HCM will serve as reference values and will be the basis of

further intensive studies on the occurrence of OMPs in groundwater in HCM.

Occurrence of organic micro-pollutants

Of the 940 analytes, 74 that represent a variety of uses and origins were detected at least once (Table 1, Table S3). The maximum and the median number of compounds detected per well were 43 and 16, respectively. None of the samples were free of analytes; however, five of the samples contained less than 10 compounds. Of the 43 samples examined, 20 had total detectable concentrations that were less than 3.0 µg L⁻¹, while 17 had values between 3 and 7 µg L⁻¹. OMPs occurred more frequently in groundwater samples collected in the first sampling round (62 compounds, Sep 2013) than in the second sampling round (37 compounds, Aug 2014), with seven (Sep 2013) and three compounds (Aug 2014) detected in 100 % wells, respectively. The total concentrations in the first sampling round were highest at HN1 and HN14 (8.0 and 16 µg L⁻¹, respectively), while the total concentrations in the second round were highest at HN6 (15 µg L⁻¹) and HN16 (10 µg L⁻¹). The OMPs with the highest concentrations (greater than or equal to 0.7 µg L⁻¹) were not necessarily among the most frequently detected compounds (Fig. 2). For example, although several compounds, such as di-n-butyl phthalate (DBP) and 4-cymene, were detected infrequently, their maximum concentrations exceeded 0.7 µg L⁻¹ (Fig. 2, Table 1). Previous studies (Kolpin et al. 2002; Loos et al. 2010) have also demonstrated that the compounds that occur most frequently are not always those with the highest concentrations. The measured concentrations of individual chemicals were generally low, and 89 % of concentrations were less than or equal to 0.5 µg L⁻¹. Bis(2-ethylhexyl)phthalate (DEHP), benzyl alcohol, cholesterol, DBP, and stigmasterol (Fig. 2) were detected in the samples 27, 19, 9, 5, and 3 times, respectively, at concentrations exceeding 0.5 µg L⁻¹.

Sterols and caffeine

The samples were screened for 10 sterols, of which campesterol, cholestanol, cholesterol, beta-sitosterol, stigmasterol, and ergosterol were detected. Cholesterol was observed in 100 % of wells, followed by beta-sitosterol (21 %) and stigmasterol (16 %), while cholestanol, ergosterol, and campesterol were observed in only one, one, and two wells, respectively (Table 1). Sterol appearances and concentrations in groundwater were highly variable across wells. Out of 43 samples investigated (both rounds), 33 were contaminated with only one sterol at detectable concentrations of less than 0.62 µg L⁻¹ except for HCM3 (1.0 µg L⁻¹), while two, seven, and one samples were polluted with two, three, and five sterols, respectively (Table S3). The highest total concentrations of sterols were observed in HN2 (2.1 µg L⁻¹), HN1

Table 1 Summary of analytical results of groundwater wells sampled for 940 OMPs in Hanoi and Ho Chi Minh City

No.	Name	CAS number	LOD ($\mu\text{g L}^{-1}$)	Number of detections		Maximum concentration ($\mu\text{g L}^{-1}$)		Drinking water standards and health advisories ($\mu\text{g L}^{-1}$)	Origin/source	Detector
				Sep 2013 (n=22)	Aug 2014 (n=21)	Sep 2013 (n=22)	Aug 2014 (n=21)			
Sterol										
1	Campesterol	474-62-4	0.01	0	2	0	0.16	–	Phytosterol	GC-MS/SIM
2	Cholesterol	80-97-7	0.01	0	1	0	0.35	–	Animal sterol	GC-MS/SIM
3	Cholesterol	57-88-5	0.01	22	21	1.0	0.88	–	Animal sterol	GC-MS/SIM
4	beta-Sitosterol	83-46-5	0.01	7	2	0.62	0.53	–	Phytosterol	GC-MS/SIM
5	Stigmasterol	83-48-7	0.01	7	0	5.8	0	–	Phytosterol	GC-MS/SIM
6	Ergosterol	57-87-4	0.01	0	1	0	0.34	–	Phytosterol	GC-MS/SIM
Pharmaceuticals and Personal Care Products (PPCPs)										
7	L-Menthol	2216-51-5	0.01	18	4	0.06	0.02	–	PPCPs	GC-MS/TIM
8	Squalane	111-01-3	0.01	14	0	0.14	0	–	PPCPs	GC-MS/TIM
9	Dichthyltoluamide	84-66-2	0.01	3	4	0.04	0.40	–	PPCPs	GC-MS/TIM
Lifestyle compounds										
10	Caffeine	58-08-2	0.01	2	0	2.7	0	–	PPCPs	GC-MS/TIM
Phthalates										
11	Diethyl phthalate (DEP)	84-66-2	0.01	22	15	0.09	0.71	³ 0.8; ⁴ 30,000	Plasticizer	GC-MS/TIM
12	Diisobutyl phthalate (DIBP)	84-69-5	0.01	22	0	0.80	0	–	Plasticizer	GC-MS/TIM
13	Dimethyl phthalate (DMP)	131-11-3	0.01	4	3	0.02	0.04	–	Plasticizer	GC-MS/TIM
14	Di-n-butyl phthalate (DBP)	84-74-2	0.01	22	16	0.39	10	³ 0.1 ⁴ 4000	Plasticizer	GC-MS/TIM
15	Di(2-ethylhexyl)phthalate (DEHP)	117-81-7	0.01	22	8	3.6	6.2	¹ 6.0; ³ 0.02; ⁴ 700; ⁵ 300; ⁷ 8.0	Plasticizer	GC-MS/TIM
16	Butyl benzyl phthalate	85-68-7	0.01	18	9	0.10	0.11	³ 0.2; ⁴ 7,000	Plasticizer	GC-MS/TIM
Other OMP compounds										
17	3- and 4-tert-Butylphenol	585-34-2 and 98-54-4	0.01	1	0	0.04	0	–	Antioxidant	GC-MS/TIM
18	Acetophenone	98-86-2	0.01	17	7	0.12	0.07	³ 0.1	Cosmetics/fragrance	GC-MS/TIM
19	alpha-Terpineol	10482-56-1	0.01	1	0	0.65	0	–	Cosmetics/fragrance	GC-MS/TIM
20	Benzyl alcohol	100-51-6	0.01	15	21	3.6	0	–	Cosmetics/fragrance	GC-MS/TIM
21	Phenylethyl alcohol	60-12-8	0.01	0	3	0	0.02	–	Cosmetics/fragrance	GC-MS/TIM
22	Methyl palmitoleate	1120-25-8	0.01	0	1	0	0.13	–	Fatty acid methyl ester	GC-MS/TIM
23	Tributyl phosphate	126-73-8	0.01	11	1	0.50	1.4	–	Fire retardant	GC-MS/TIM
24	4-Chloro-3-methylphenol	59-50-7	0.01	0	1	0	0.30	–	Fungicide, paint	GC-MS/TIM
25	epsilon-Caprolactam	105-60-2	0.01	0	19	0	0.11	³ 0.5	Intermediate for fiber	GC-MS/TIM
26	Bisphenol A	80-05-7	0.01	5	0	0.04	0	³ 0.05	Intermediate for resin	GC-MS/TIM
27	4-Chloro-2-nitroaniline	89-63-4	0.01	18	0	1.9	0	–	Intermediate in organic synthesis	GC-MS/TIM
28	3,5-Dimethylphenol	108-68-9	0.01	0	21	0	0.38	–	Intermediate in organic synthesis	GC-MS/TIM
29	Diphenylamine	122-39-4	0.01	0	5	0	0.07	³ 0.025		GC-MS/TIM

Table 1 (continued)

No.	Name	CAS number	LOD ($\mu\text{g L}^{-1}$)	Number of detections		Maximum concentration ($\mu\text{g L}^{-1}$)		Drinking water standards and health advisories ($\mu\text{g L}^{-1}$)	Origin/source	Detector
				Sep 2013 (<i>n</i> =22)	Aug 2014 (<i>n</i> =21)	Sep 2013 (<i>n</i> =22)	Aug 2014 (<i>n</i> =21)			
30	Nitrobenzene	98-95-3	0.01	0	1	0	0.10	³ 0.002	Intermediate in organic synthesis	GC-MS/TIM
31	Safrole	94-59-7	0.01	1	0	0.69	0	–	Intermediate in organic synthesis	GC-MS/TIM
32	2(3H)-Benzothiazolone	934-34-9	0.01	1	1	0	0.39	–	Intermediate in organic synthesis/preservative	GC-MS/TIM
33	2-(Methylthio)-benzothiazol	615-22-5	0.01	5	1	0.11	0.09	–	Leaching from tire	GC-MS/TIM
34	Benzothiazole	95-16-9	0.01	12	4	0.04	0.13	–	Leaching from tire	GC-MS/TIM
35	Ethanol, 2-phenoxy-	122-99-6	0.01	22	5	0.30	0.04	–	Leaching from tire	GC-MS/TIM
36	4-tert-Octylphenol	140-66-9	0.01	2	0	0.06	0	–	Leaching from tire	GC-MS/TIM
37	Longifolene	475-20-7	0.01	4	0	0.09	0	–	Nonionic detergent metabolite	GC-MS/TIM
38	2-Ethyl-1-hexanol	104-76-7	0.01	21	0	0.32	0	–	Other	GC-MS/TIM
39	Bis(2-ethylhexyl) sebacate	122-62-3	0.01	2	1	0.02	0.13	–	Plasticizer	GC-MS/TIM
40	Di(2-ethylhexyl)adipate	103-23-1	0.01	22	17	0.1	0.95	¹ 400; ² 400; ³ 0.6; ⁴ 20,000; ⁵ 3,000	Plasticizer	GC-MS/TIM
41	4-Cymene	99-87-6	0.01	5	0	6.8	0	–	Plasticizer	GC-MS/TIM
42	trans-Decahydronaphthalene	493-02-7	0.01	21	0	0.34	0	–	Solvent	GC-MS/TIM
43	Isophorone	78-59-1	0.01	5	8	0.02	0.20	² 100; ³ 0.2; ⁴ 7,000; ⁵ 4,000	Solvent	GC-MS/TIM
	Organochlorine pesticides ^a									
44	trans-Chlordane	5103-74-2	0.4	1	0	3.0	0	–	Solvent/paint	GC-MS/TIM
45	cis-Chlordane	5103-71-9	0.4	1	0	1.7	0	–	Insecticide	GC-MS-MS/SRM
	Σ Chlordane							¹ 2.0; ² 4.0; ³ 0.0005; ⁴ 20; ⁵ 10; ⁷ 0.2	Insecticide	GC-MS-MS/SRM
46	trans-Nonachlor	5103-73-1	0.4	1	0	0.72	0	–	Insecticide	GC-MS-MS/SRM
47	Endrin	72-20-8	0.4	4	7	1.3	1.7	¹ 2.0; ² 2.0; ³ 0.0003; ⁴ 10	Insecticide	GC-MS-MS/SRM
48	Dieldrin	60-57-1	0.4	2	0	2.8	0	³ 0.00005; ⁴ 2.0; ⁵ 0.2; ⁶ 0.03	Insecticide	GC-MS-MS/SRM
49	Hexachlorobenzene	118-74-1	0.1	2	0	0.35	0	¹ 1.0; ³ 0.0008; ⁴ 30; ⁵ 2.0	By-product	GC-MS-MS/SRM
50	Heptachlor epoxide (B)	1024-57-3	0.4	0	8	0	5.9	¹ 0.2; ³ 0.00001; ⁴ 0.4; ⁵ 0.4; ⁶ 0.03	Insecticide	GC-MS-MS/SRM
51	a-HCH	319-84-6	0.4	1	0	1.9	0	–	Insecticide	GC-MS-MS/SRM
52	g-HCH	58-89-9	0.4	1	0	1.3	0	¹ 0.2; ³ 0.005; ⁴ 200	Insecticide	GC-MS-MS/SRM
53	p,p'-DDT	50-29-3	0.1	6	11	3.6	12	³ 0.0005; ⁷ 1.0 ^b	Insecticide	GC-MS-MS/SRM
54	p,p'-DDE	72-55-9	0.1	1	1	0.11	0.33	–	Insecticide	GC-MS-MS/SRM
55	p,p'-DDD	72-54-8	0.1	2	4	0.46	0.55	–	Insecticide	GC-MS-MS/SRM
56	o,p'-DDT	789-02-6	0.2	2	2	0.38	1.3	–	Insecticide	GC-MS-MS/SRM

Table 1 (continued)

No.	Name	CAS number	LOD ($\mu\text{g L}^{-1}$)	Number of detections		Maximum concentration ($\mu\text{g L}^{-1}$)		Drinking water standards and health advisories ($\mu\text{g L}^{-1}$)	Origin/source	Detector
				Sep 2013 (<i>n</i> =22)	Aug 2014 (<i>n</i> =21)	Sep 2013 (<i>n</i> =22)	Aug 2014 (<i>n</i> =21)			
57	<i>o,p'</i> -DDD Polychlorinated biphenyls ^a	53-19-0	0.1	16	8	2.7	6.4	–	Insecticide	GC-MS-MS/SRM
58	PCB #8	34883-43-7	0.1	3	0	0.35	0	–	PCB	GC-MS-MS/SRM
59	PCB #74	32690-93-0	0.1	2	0	0.13	0	–	PCB	GC-MS-MS/SRM
60	PCB #70	32598-11-1	0.1	2	0	0.11	0	–	PCB	GC-MS-MS/SRM
61	PCB #66	32598-10-0	0.1	2	0	0.12	0	–	PCB	GC-MS-MS/SRM
62	PCB #60	33025-41-1	0.1	5	7	1.1	0.33	–	PCB	GC-MS-MS/SRM
63	PCB #52	35693-99-3	0.1	2	0	0.46	0	–	PCB	GC-MS-MS/SRM
64	PCB #44	41464-39-5	0.1	2	0	0.25	0	–	PCB	GC-MS-MS/SRM
65	PCB #41	52663-59-9	0.1	2	0	0.34	0	–	PCB	GC-MS-MS/SRM
66	PCB #4 and 10	13029-08-8 and 33146-45-1	0.1	2	0	0.29	0	–	PCB	GC-MS-MS/SRM
67	PCB #37	38444-90-5	0.2	1	0	0.20	0	–	PCB	GC-MS-MS/SRM
68	PCB #33	38444-86-9	0.1	3	0	0.40	0	–	PCB	GC-MS-MS/SRM
69	PCB #28	7012-37-5	0.1	4	0	1.8	0	–	PCB	GC-MS-MS/SRM
70	PCB #22	38444-85-8	0.2	2	0	0.30	0	–	PCB	GC-MS-MS/SRM
71	PCB #19	38444-73-4	0.1	1	0	0.15	0	–	PCB	GC-MS-MS/SRM
72	PCB #18	37680-65-2	0.1	4	0	0.78	0	–	PCB	GC-MS-MS/SRM
73	PCB #15	2050-68-2	0.1	2	0	0.19	0	–	PCB	GC-MS-MS/SRM
74	PCB #1	2051-60-7	0.1	1	0	0.11	0	–	PCB	GC-MS-MS/SRM
	Σ PCBs							¹ 0.5; ⁵ 10		GC-MS-MS/SRM

LOD limit of detection, – not available

^a Compounds presented concentrations in ng L^{-1}

^b Total DDT and metabolites concentration ($\mu\text{g L}^{-1}$)

Drinking Water Standards and Health Advisories:

¹ US EPA Maximum Contaminant Levels (MCL) ($\mu\text{g L}^{-1}$)

² US EPA Lifetime Health Advisory ($\mu\text{g L}^{-1}$)

³ US EPA Reference Dose (RfD) ($\text{mg kg}^{-1} \text{ day}^{-1}$)

⁴ US EPA Drinking Water Equivalent Level (DWEL) ($\mu\text{g L}^{-1}$)

⁵ US EPA 10^{-4} Cancer Risk ($\mu\text{g L}^{-1}$)

⁶ European Health-Based Chemical Standards ($\mu\text{g L}^{-1}$), http://www.doeni.gov.uk/nica/european_and_national_drinking_water_quality_standards_-_october_2011.pdf.

⁷ WHO Guidelines for Drinking-water Quality ($\mu\text{g L}^{-1}$)

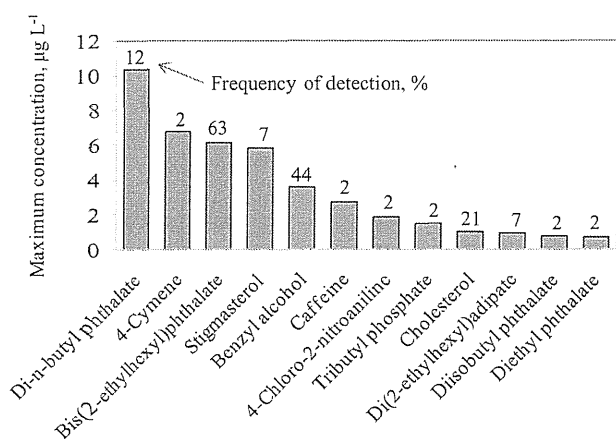


Fig. 2 Maximum concentrations and detection frequencies of compounds that were detected at concentrations greater than $0.7 \mu\text{g L}^{-1}$

($6.6 \mu\text{g L}^{-1}$), and HN9 ($1.1 \mu\text{g L}^{-1}$) in the first sampling round, while HN6 was contaminated with high sterol concentrations in both rounds (Table S3). Stigmasterol and cholesterol were the only two sterols detected at concentrations greater than $1 \mu\text{g L}^{-1}$ (Table 1), whereas the other four remaining sterols were identified at concentrations that were lower than $0.88 \mu\text{g L}^{-1}$. These results may indicate a high degree of heterogeneity in loadings from local sources and/or spatial differences in hydraulic conductivity and biogeochemical conditions, as mentioned by Schaidler et al. (2014).

Six sterols that were detected in groundwater were also abundant in river waters and sediments collected in the same study areas (HN and HCM) in previous studies (Duong et al. 2014a, b). However, a fecal sterol (coprostanol) that was detected most often and at extremely high concentrations in river waters and sediments due to sewage contamination (Duong et al. 2014a, b) was not found in groundwater samples.

Caffeine was one of the most frequently detected compounds in previous groundwater studies (Barnes et al. 2008; Focazio et al. 2008) and was detected in nearly 100 % of river waters in HN and HCM (Duong et al. 2014a). In this study however, it was observed in only two (HN14 and HCM3) of the 43 groundwater samples. The maximum concentration that was detected in this study ($2.7 \mu\text{g L}^{-1}$) is about 20 times higher than the maximum detected in groundwater in the USA ($0.13 \mu\text{g L}^{-1}$, Barnes et al. 2008), 14 times higher than the maximum concentration detected in groundwater in Europe ($0.19 \mu\text{g L}^{-1}$, Loos et al. 2010), and 10 times higher than the maximum value in untreated drinking water sources in the USA ($0.27 \mu\text{g L}^{-1}$, Focazio et al. 2008). The presence of caffeine in shallow drinking water wells has been suggested as an indicator of wastewater impacts (Seiler et al. 1999), and it is thought that it is attenuated in oxic subsurface conditions (Schaidler et al. 2014). Moreover, it persists in anaerobic conditions in groundwater (Ying et al. 2003). Therefore, the presence of caffeine in two wells probably reflects direct entry of

leachate from surface water, leaks in sewage canals, or underground septic tanks.

Emerging chemicals

Of the 14 PPCPs analyzed, N,N-diethyl-m-toluamide (DEET), L-menthol, and squalane were detected in groundwater samples at maximum concentrations of 0.40 , 0.06 , and $0.14 \mu\text{g L}^{-1}$, respectively (Table 1, Table S3). L-menthol and DEET, which are well-known molecular markers of sewage contamination (Nakada et al. 2008), were detected in 22 and 7 groundwater samples (in both sampling rounds), respectively, while squalane, which is used in numerous vaccine and drug delivery emulsions (Fox 2009), was only detected in 14 out of 22 groundwater samples in the first sampling round (Table 1, Table S3). DEET was the most frequently detected compound in groundwater in the USA at maximum concentrations of $13.5 \mu\text{g L}^{-1}$ (Barnes et al. 2008), 34 times higher than those of this study. However, the maximum levels of DEET in this study were six times higher than those ($0.07 \mu\text{g L}^{-1}$) in groundwater from the metropolitan area of Tokyo (Nakada et al. 2008).

The three detected PPCPs in groundwater samples were the most abundant in surface water of HN and HCM that was contaminated with sewage (Duong et al. 2014a), and two of them (L-menthol and squalane) were detected in sediments at the same study areas, which demonstrates the ubiquitous use of these PPCPs; the fact that they exist in the water environment in Vietnam reflects the lack of adequate wastewater treatment facilities. Nakada et al. (2008) reported that DEET and other PPCPs were also detected in groundwater in Tokyo as the consequence of sewage leakage from decrepit sewers. Therefore, the presence of PPCPs in groundwater in this study is probably the result of a combination of leaking underground septic tanks, infiltration of untreated wastewater through old sewer canal systems, and urban storm water recharge/runoff. Furthermore, because a large number of PPCPs are known to be found in domestic wastewater, it is thought that groundwater is polluted by many PPCPs. However, the number of PPCPs (14) registered in the AIQS database is very limited, so a more detailed survey on PPCPs should be carried out after new PPCPs are registered in the database.

Two phenolic EDCs, BPA and OP, deserve particular attention because of their estrogenic activity and widespread application and ubiquity in the environment (Kuch and Ballschmiter 2001; Meesters and Schroder 2002). There is evidence that BPA has estrogenic potential even at low concentrations (Jin et al. 2004) and OP can cause estrogenic effects in fish and other aquatic organisms (Jobling et al. 1996). BPA was detected in 5, while OP was detected in 2 of the 22 wells sampled in the first round at concentrations that ranged from 0.02 to $0.04 \mu\text{g L}^{-1}$ and 0.02 to $0.06 \mu\text{g L}^{-1}$, respectively (Table S3). The maximum BPA concentrations were 64 and

57 times lower than those in groundwater in the USA ($2.55 \mu\text{g L}^{-1}$, Barnes et al. 2008) and in Europe ($2.3 \mu\text{g L}^{-1}$, Loos et al. 2010), respectively, and the maximum OP concentration was six times lower than those reported in Oxford, England ($0.83 \mu\text{g L}^{-1}$, Stuart et al. 2014) and 1.5 times higher than those in European groundwater ($0.04 \mu\text{g L}^{-1}$, Loos et al. 2010). The concentrations of BPA detected in this study are much lower than the human health-based guideline values ($100 \mu\text{g L}^{-1}$) for exposure to BPA in groundwater proposed by the Minnesota Department of Health (MDH 2013).

Of the six phthalate compounds detected, diethyl phthalate (DEP), DBP, and DEHP were detected in over 70 % of the sampled wells with maximum concentrations of 0.71, 10, and $6.2 \mu\text{g L}^{-1}$, respectively (Table 1, Table S3). These compounds have been detected in groundwater in England; however, the concentrations in this study were more than 2.5 times lower than those reported in Boxford but more than 1.5 times higher than those reported in Oxford (Stuart et al. 2014). Dimethyl phthalate (DMP) was found in scattered wells in this study at trace concentrations of less than $0.04 \mu\text{g L}^{-1}$ and was detected only once in groundwater from Maresme, Spain, at a concentration of $0.12 \mu\text{g L}^{-1}$ (Sánchez-Avila et al. 2009). The highest concentration of DEHP ($6.2 \mu\text{g L}^{-1}$), an EDC (Akingbemi et al. 2004), was comparable with the concentration detected in the Llobregat River aquifers, Spain ($5.67 \mu\text{g L}^{-1}$, López-Roldán et al. 2004).

Organochlorine pesticides and polychlorinated biphenyls

Pollution from OCPs and PCBs in surface waters and sediments has been extensively researched in Vietnam (Nhan et al. 1998, 2001; Minh et al. 2007; Hoai et al. 2010). However, they are of little concern as groundwater contaminants because they are relatively insoluble in water and are retained strongly by soil. Our previous studies (Duong et al. 2014a, b) showed that PCBs and OCPs still remain in the aquatic environment and most of the banned OCPs can still be found in high concentrations in the environment throughout Vietnam (e.g., Kishida et al. 2007; Minh et al. 2007; Hoai et al. 2010). In addition, Belfroid et al. (1998) demonstrated that many pesticides entered the aquatic environment via soil percolation, air drift, or surface runoff and eventually ended up in groundwater, where their transformation products could remain for years.

In this study, 14 OCPs and 17 PCBs were detected, and the detection frequency was higher in the first sampling round than in the second round (Table 1, Table S3). Of the 43 samples, 17 and 33 contained at least one of the OCP and PCB congeners, respectively. *o,p'*-DDD, *p,p'*-DDT, and endrin were the most frequently detected OCPs with maximum concentrations of 6.4, 12, and 1.7 ng L^{-1} , respectively. *trans*-Chlordane was detected in one well, and dieldrin was detected in two wells, and their concentrations reached around 3 ng L^{-1} (Table 1, Table S3). As was observed for PCBs, none of the

PCBs were detected in the groundwater samples from the second sampling round except for PCB #60, which was the most abundant PCB congener and had the highest occurrence in both rounds. The highest concentrations of individual PCB congeners were generally less than 0.5 ng L^{-1} except for PCB #28 (1.8 ng L^{-1}), followed by PCB #60 (1.1 ng L^{-1}) and PCB #18 (0.78 ng L^{-1}) (Table S3). Well HCM2 was seriously polluted by PCBs and OCPs with a total concentration of 20 ng L^{-1} , while HN3A, HN4, and HN5 were heavily polluted by OCPs and had total detected concentrations of 16, 14, and 9.4 ng L^{-1} , respectively (Table S3). This is not surprising, as the surface waters and sediments collected at these sites were also extremely polluted by elevated levels of OCPs and PCBs (Duong et al. 2014a, b). OCPs such as α -, β -, γ -, δ -hexachlorocyclohexane (HCH), aldrin, dieldrin, heptachlor, and DDT and its metabolites have been detected in groundwater in India (Sankararamkrishnan et al. 2005; Shukla et al. 2006; Lari et al. 2014); Syria (Jamal 2011); and in Beijing, China (He et al. 2011). However, the pathways by which these contaminants can make their way into the groundwater are still unclear. For example, Jabbar et al. (1993) demonstrated that the principle mechanism by which OCPs are transported from soil to groundwater is via downward percolation of water containing dissolved pesticides, while Cerejeira et al. (2003) indicated that because of drift, runoff, drainage, and their leaching potential, OCPs caused contamination of surface and groundwater. However, more detailed studies need to be carried out to gain an improved understanding of the leaching behavior of OCPs and PCBs in groundwater.

Organic micro-pollutant compound groups

The 74 OMPs (or semi-volatile organic compounds, SVOCs) can be divided into 18 groups based on general use category or type of compound (Fig. 3). *n*-Alkanes were excluded from the pollution profile because of their widespread existence in environmental samples. However, it should be noted that the uses of any given compound can vary widely; therefore, the tabulated use categories are presented for illustrative purposes and may not be all-inclusive. The frequency of detection was calculated as the ratio of the measured detection to the total measurement. Although these groupings are composed of unequal numbers of compounds, the detection frequency of the compound group is not controlled by the number of compounds in the group. The phthalate group, consisting of six compounds, had the greatest detection frequency and accounted for the highest percentage of the total concentrations in the first round, while the fiber group contained only *ε*-caprolactam, which was detected in 19 out of 21 samples in the second sampling round at relatively low concentrations. A total of 9 out of 16 groups in the first sampling round had a detection frequency exceeding 40 %, and two groups (phthalates and sterols) accounted for over 60 % of the total

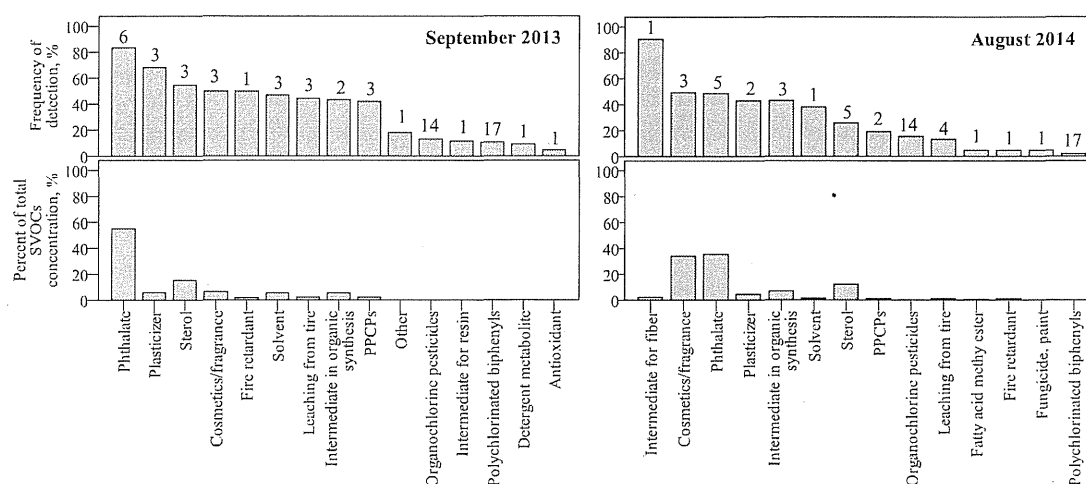


Fig. 3 (1) Detection frequency of SVOCs by general use category and (2) percent of total concentration of SVOCs by general use category in the first round (Sep 2013) and the second round (Aug 2014). Number of compounds in each category are shown above the bar

concentrations. While detection frequencies that exceeded 40 % were reported for five groups in the second sampling round, three groups (cosmetics/fragrance, phthalate, sterol) accounted for 80 % of the total concentrations (Fig. 3).

General comparison with the surface water study

Data collected for groundwater in this study can be qualitatively compared with data collected for the surface waters in our previous study (Duong et al. 2014a). This is a valid comparison because the same analytical method was used for this study and the previous surface water study, although fewer surface water samples were collected (26 surface water samples compared with 43 groundwater samples). Because a large number of chemicals that originated from domestic wastewater were detected in surface waters (Duong et al. 2014a), it was thought that groundwater would also be contaminated with many chemicals. Therefore, the groundwater sampling sites were close to the surface water sampling sites. Overall, fewer contaminants were detected in groundwater, and only 74 of the 940 analytes were detected in groundwater, while 235 were detected in surface waters (Duong et al. 2014a). All of the compounds that were detected in groundwater samples were also detected in the surface water samples. Although similar compounds were detected in the groundwater, the number of compounds having a detection frequency exceeding 70 % in groundwater (six compounds) was much smaller compared with surface water (42 compounds). The concentrations of pollutants in surface water were higher than those in groundwater. Total concentrations of the detected pollutants in 12 out of the 43 groundwater wells exceeded $5 \mu\text{g L}^{-1}$, with three wells having total concentrations higher than $10 \mu\text{g L}^{-1}$. The total concentrations in 50 % of the surface waters were greater than $120 \mu\text{g L}^{-1}$ (from 120 to $440 \mu\text{g L}^{-1}$) while the levels were below $10 \mu\text{g L}^{-1}$ in only six samples. Coprostanol

and coprostanone were detected at extremely high concentrations in all surface water samples in HN and HCM but were not detected in groundwater samples. While surface waters in HN and HCM were heavily contaminated by permethrin and carbamate insecticide (fenobucarb), these compounds were not detected in any of the groundwater wells. This is most likely because of their hydrophobic properties (K_{ow} log P 6.1 and 2.79, respectively) and their tendency to adsorb onto organic matter and sediment, meaning it is difficult for them to reach the aquifer. In addition, Sharom and Solomon (1981) and Kim et al. (2014) demonstrated that permethrin and fenobucarb are biodegraded in aqueous systems and soils, so they were not present in aquifers.

Identification of potential sources of contamination

Information about the aquifer conditions, such as hydrogeological processes, aquifer characteristics (gravel, chalk, hyporheic zone), kinetics, groundwater flow velocity, and information on the well depth, was not available for all the wells sampled. Therefore, an evaluation of the characteristics of chemicals detected in groundwater in this study may provide a useful insight into the factors that control the presence of organic contamination in the aquifers of HN and HCM. The sampled groundwater wells were close to the surface water and sediment sampling sites. As such, heavily polluted surface waters may influence aquifers via numerous processes, including concentrations at the source, dilution, adsorption, and degradation (Jurado et al. 2012). The largest number of contaminants (27 compounds) and the highest total concentration ($18 \mu\text{g L}^{-1}$) were detected at HN14. In addition, 4-cymene, caffeine, and DEHP were the main contributors to pollution at this well. Out of these, caffeine, an indicator of wastewater impacts in shallow drinking water wells (Seiler et al. 1999), was only detected in 2 out of 43 wells. Therefore, it is thought that this well may be impacted by

point sources, such as direct influences from the surface or leaks from underground septic systems. Three other wells (HN13, HN16, and HN17), located close to the most polluted canals in HN, had elevated total concentrations of 9.0, 9.7, and 8.3 $\mu\text{g L}^{-1}$, respectively, and a similar range of chemicals contributed to the high total concentrations at each of these sites. The leakage of surface water contaminated by sewage from the decrepit sewer canals to the aquifers is probably the cause of the pollution at these wells. The total concentrations in the remaining wells were less than 6 $\mu\text{g L}^{-1}$. At 20 of the sites, the total concentrations were lower than 3 $\mu\text{g L}^{-1}$ and fewer than 20 compounds were detected; these sites were possibly influenced by non-point source pollution. There are multiple possible sources of non-point pollution to aquifers, such as storm water and urban runoff, leakages from urban sewerage systems, diffuse aerial deposition (Nakada et al. 2008; Buerge et al. 2011; Vulliet et al. 2008), or intentional and unintentional recharge of wastewater sources (Drewes 2009). However, the possibility of contaminant leakages from septic systems is not excluded because it was reported that about 32 % of HN's population is served by septic tanks (World Bank 1996). In addition, several case studies in the USA and Canada have reported a range of emerging organic contaminants in groundwater impacted by septic tanks (Carrara et al. 2008; Swartz et al. 2006). Hence, further research is needed to clarify the major source of detected contaminants as well as to determine the primary fate, degradation, transformation, and transport processes in impacted aquifers.

Risk assessment

Well water sampled in this study is not used for drinking purposes; however, more than half of the population of HN get their tap water from groundwater sources. Hence, the presence of contaminants in HN and HCM's groundwater raises human health concerns even though most of the detected contaminants are not currently regulated in drinking water or were present at low levels. Therefore, in this context, we were not able to evaluate the potential health implications of all the detected contaminants because of a lack of toxicity information for many organic pollutants (Stephenson 2009) and limited testing requirements (Schaidler et al. 2014). Further, while this study included a diverse list of 940 organic pollutants, there are about 80,000 chemicals currently in use (Schaidler et al. 2014), suggesting that other organic pollutants, as well as their metabolites, were also present in our samples. Consequently, we have assessed the health risk for 19 out of the 74 detected contaminants, for which health-based guidelines are available, by comparing the measured concentrations with the drinking water standards or health advisories (Table 1) proposed by the US Environmental Protection Agency (USEPA), European Commission (EU), and the WHO. None of the detected concentrations exceeded the maximum contaminant levels, drinking water equivalent levels or

lifetime health advisory proposed by the USEPA (Table 1), or the health-based chemical standards and guideline values for drinking water issued by EU and WHO, respectively. A non-carcinogenic assessment of some chemicals was carried out based on the exposure of a 50 kg adult consuming 2 L of water per day by dividing the reference dose (USEPA). The risk quotients for the 18 investigated pollutants were much smaller than 1, which demonstrates that there is no risk to humans from these detected contaminants.

Conclusions

This is the first comprehensive study of OMPs in groundwater in Vietnam, and as such, this study provides new baseline knowledge about the occurrence and levels of organic contaminants in groundwater. Key findings are as follows: (1) Out of 940 analytes, 74 were detected, which represented about one third of the number of contaminants that were detected in surface waters. (2) Cholesterol, di(2-ethylhexyl)adipate, DBP, DEP, DEHP, and benzyl alcohol were the most frequently detected compounds and were found in over 80 % of samples. The total concentrations in 75 % of the groundwater samples were less than 5.0 $\mu\text{g L}^{-1}$. (3) Many of the wells and aquifers are probably influenced by non-point source pollution, such as leaks from the urban sewerage system or recharge by wastewater. (4) There is concern for human health related to the presence of pollutants in groundwater in HN and HCM, even though most detected contaminants are not currently regulated in drinking water or were present at low levels. A health risk assessment for 19 detected contaminants showed that there were no risks to humans from these contaminants.

This study was carried out over a limited time period (Sep 2013 and Aug 2014) with a limited number of sampling events. The number of sampling sites was particularly limited in HCM, which means that we only have limited information on the occurrence of OMPs in groundwater in HCM. In spite of the limited information, these initial results can be used as baseline data and will form the basis for further intensive studies on the occurrence of OMPs in groundwater in HCM and on the primary fate, degradation, and transport processes of OMPs in impacted aquifers. A full evaluation of the potential risks of detected contaminants should also be carried out.

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