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Phthalates and pharmaceuticals in soil, groundwater, and surface water downgradient of a wastewater soil infiltration system

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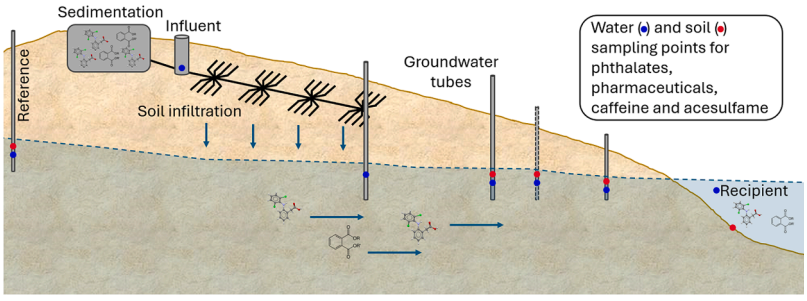
HIGHLIGHTS

- Micropollutant removal predominantly occurred in the unsaturated vadose zone.
- Significant attenuation observed for caffeine, carbamazepine, losartan, phthalates.
- Diclofenac and ibuprofen exceeded ecological risk thresholds in surface waters.
- Phthalate removal efficiency comparable to conventional wastewater treatment plants.
- Soil infiltration systems have limits for persistent drugs such as diclofenac.

GRAPHICAL ABSTRACT



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ABSTRACT

This study investigates the occurrence, attenuation, and ecological risks of phthalates and pharmaceuticals in a long-operating wastewater soil infiltration system in northern Sweden. Concentrations of 15 phthalates, 67 pharmaceuticals, caffeine, and acesulfame K were measured in influent wastewater, groundwater, soil, and a downgradient pond across multiple seasons. Results showed that most micropollutant removal occurred in the unsaturated soil zone prior to groundwater recharge, possibly due to processes such as biodegradation and sorption. Substantial reductions were observed for caffeine (>99 %), carbamazepine (>96 %), losartan (>99 %), and phthalates (51 ± 72 % and 92 ± 5 %), with the higher attenuation for phthalates comparable to conventional activated sludge treatment. In contrast, compounds such as metoprolol exhibited moderate reductions (>71 %), while others showed low or even negative attenuation, including diclofenac (46 % and −180 %) and ibuprofen (33 % and −11 %). After groundwater recharge, only ibuprofen showed attenuation beyond dilution, although the mechanisms for this remains unknown. Several pharmaceuticals, including metoprolol, irbesartan, and metformin, were detected in soil samples, though it is unclear whether they were sorbed to the soil matrix or present in porewater. In downgradient surface water, diclofenac and ibuprofen exceeded risk quotient thresholds, while oxazepam surpassed the lowest predicted no-effect concentration (PNEC) in one sample, indicating ecological risks. Overall, the findings highlight both the strengths and limitations of soil infiltration systems in

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mitigating micropollutant contamination, emphasizing the importance of vadose zone processes while underscoring uncertainties in sorption and degradation mechanisms

1. Introduction

In Sweden and elsewhere, many small communities rely on local wastewater treatment systems. This often involves small conventional wastewater treatment plants; however, treatment can also be conducted using nature-based treatment systems, such as wetlands and soil-based systems [4]. A common nature-based system includes a septic tank for sludge separation and a soil infiltration system. In this approach, wastewater is dispersed into the ground using underground distribution pipes, allowing it to be treated as it percolates through the soil layers. The system utilizes natural mechanisms in the soil, such as biologically driven degradation of organic matter and bacteria, transformation of nitrogen, and adsorption of phosphorus, to treat the wastewater. However, wastewater typically also contains a diverse array of micropollutants [28], which soil infiltration systems are generally not designed to treat, posing a potential risk to the surrounding environment. Untreated micropollutants may not only negatively impact aquatic ecosystems [32], but also contaminate sources of drinking water [20,37], posing potential health risks to humans. Therefore, the European Union's urban wastewater directive demands the treatment of micropollutants at large wastewater treatment plants [10]. This, together with the complexity of assessing the fate and potential environmental risks associated with micropollutants, justifies further research on the occurrence of micropollutants and their attenuations in soil infiltration systems.

A relatively small number of studies have reported on the occurrence and fate of micropollutants released from septic tanks into soil infiltration systems [12,16,3,7,9]. These studies reported large variability in the occurrence and concentration of different compounds in the wastewater across sampling events [16] and study sites [12,7,9], as this depends on the number and lifestyle of the served inhabitants and the presence of industrial flow in the area. Furthermore, attenuation rates within the infiltration area and surrounding soil have also been found to vary significantly [12,3], as they depend on several interconnected factors such as the characteristics and concentration of different compounds, design and age of the infiltration system, soil type and properties, length of the vadose zone, and climate and hydrological conditions. Laboratory studies with vegetated biofilters have also been conducted to assess micropollutant removal in soil [42], which has showed that e.g., accumulation of several micropollutants is higher in the saturated zone compared to the unsaturated zone

Phthalates, industrial compounds used as plasticizers in common items such as toys, detergents, cosmetics, and PVC-based building materials [13], have not been evaluated in wastewater treated in soil infiltration systems. Their presence has only been studied in similar treatment systems such as biofilters and trickling filters [1,27]. This raises concerns because phthalates are known to disrupt endocrine and reproductive functions in both humans and animals [21,29]. Despite the European Union's ban on four phthalates deemed harmful to reproduction in toys (REACH Annex XVII, entries 51 and 52), as well as in clothing, textiles, and footwear [11], these substances continue to be present in wastewater due to their widespread use in the urban environment. Significant levels have been detected in the discharge from conventional wastewater treatment facilities [36]. Conversely, another study indicates that wastewater treatment generally serves as an effective barrier, preventing phthalates from entering the environment [39], indicating that the levels of phthalates in processed wastewater can fluctuate.

Additionally, pharmaceutical residues are among the most frequently detected micropollutants in wastewater [40], originating from human excretion or entering the wastewater through other means. This can impact the environment, as numerous compounds, for example carbamazepine, diclofenac, and metoprolol exhibit toxic effects on aquatic life [18,31,38].

However, only few of the mentioned studies on soil infiltration has investigated the retention of pharmaceuticals in the soil, and research on retention of micropollutants in soil after long-term wastewater infiltration is limited. Consequently, because there is a scarcity of studies on phthalates and pharmaceuticals in soil infiltration systems, the aim of this study was to investigate the presence of these substances in the soil, groundwater, and surface water surrounding a 40-year-old soil infiltration system. A total of 15 phthalates and 67 pharmaceuticals, and two compounds commonly found in household-derived wastewater (caffeine and the artificial sweetener acesulfame K) were analysed at different distances from the infiltration system and during different seasons with fluctuating groundwater levels. The aim was also to determine the extent to which different micropollutants were removed, whether they accumulated in the soil, if their concentrations decreased in the groundwater, and whether there were ecological risks to the downgradient surface water.

2. Methods

2.1. Study site

The studied soil infiltration system (Fig. 1), which had been in operation since 1981, received primarily treated wastewater from approximately 90 residents in a northern Swedish village. The regional climate was subarctic, characterized by long, snowy winters, with average temperatures of −10 °C in winter and + 14 °C in summer, and annual precipitation ranging from 600 to 800 mm [34]. The system was located in an area of forest and farmland, adjacent to a river and within a natural sand deposit sloping toward a pond that was partially connected to the river downstream. Wastewater was intermittently pumped from a sludge separator in the village to a main well situated at the highest point of the infiltration system. From there, it was distributed by gravity to four distribution wells serving four sections of the system, which together covered a drain area of approximately 2700 m^2 (40 × 70 m). Each distribution well contained eight pipes buried 1.5 m below the ground surface to ensure even dispersion. The original design intended for only two sections to operate simultaneously while the other two rested, with manual valve adjustments twice per year. However, observations indicated that three sections were active continuously without rotation, resulting in 75 % of the total drain area being used at all times. Although inflow rates were not recorded, the system had been designed for 300 population equivalents (PE) and was therefore operating below its maximum capacity. During the study period, groundwater levels at point A (Fig. 1) ranged from 2.5 to 3 m below the ground surface, corresponding to an infiltration depth of approximately 1–1.5 m.

2.1.1. Groundwater flow

Several historical groundwater monitoring tubes were identified within and around the infiltration system; however, only one (point A) was suitably located, functional, and therefore used in this study. To supplement this, new stainless-steel monitoring tubes were installed in November 2020 in the surrounding area: three tubes positioned north of the infiltration system (points B–D), where groundwater was expected to be influenced by treated wastewater based on previous investigations, and one reference tube (REF) in an area where contamination was considered unlikely. Groundwater levels were measured in these tubes on five occasions between March and November 2021, and approximate

flow directions from points A and B (Fig. 1) were determined using triangulation to confirm that the locations were appropriate for monitoring wastewater-impacted groundwater. In addition, a point dilution test was conducted in autumn 2021 to provide a rough estimate of groundwater flow velocities between points B and C; details of this procedure are provided in the supplementary information.

2.2. Sampling

2.2.1. Soil sampling

Soil sampling was performed once, during the installation of groundwater monitoring tubes (B, C, D, and REF) in November 2020. Samples were collected from the drilling equipment as 0.5 m composite samples representing soil below the groundwater level. To prevent phthalate contamination, only stainless-steel equipment was used. Individual samples were homogenized prior to distribution into sample containers, and the drill was cleaned between samples using a natural fibre brush. Additionally, a pond-bottom sediment sample was collected from the ice cover in March 2021 using a sediment sampler, representing the upper 10 cm of the pond sediments. All samples were stored at + 5 °C in the field and subsequently frozen until analysis.

2.2.2. Water sampling

Influent wastewater (septic tank effluent), groundwater, and surface water were collected as grab samples during multiple sampling campaigns in 2021. Influent samples were taken in January, March, and June, with an additional sample in August for phthalates due to breakage of the January phthalate sample bottle during transport. Groundwater and surface water (pond) were sampled in March, June, and August, with an additional campaign in November to compensate for analytical issues that resulted in missing pharmaceutical data in August. The reference groundwater tube (REF) was sampled only in June and August because groundwater levels were too low for sampling in March. All water samples were collected on the same day during each sampling event.

Influent wastewater was sampled using a stainless-steel beaker at the influent point. Groundwater and surface water were collected with a stainless-steel bailer (with a fluorocarbon O-ring seal) or a stainless-steel beaker. Groundwater tubes were purged and recharged with three times their volume prior to sampling to ensure collection of representative groundwater. Surface water samples were obtained through the ice cover using the bailer in March and by wading into the pond and submerging the beaker below the water surface in June, August, and November.

Sampling equipment was rinsed at least twice with sample water at each site before use. Pharmaceutical samples were stored and transported in high-density polyethylene bottles, whereas phthalate samples were transferred directly into glass bottles by pouring from the bailer or stainless-steel beaker through a stainless-steel funnel, ensuring no contact with plastic. For ice-free conditions, surface water samples were collected by submerging the glass bottle directly into the pond. Cotton lab coats were worn during phthalate sampling to prevent contamination from plastic materials, and gloves were not used.

2.3. Micropollutant analyses

Water samples were analysed for the total fraction of micropollutants, comprising 15 phthalates, as well as 67 pharmaceutical substances across multiple therapeutic classes (including cardiovascular drugs, analgesics, allergy medications, antidepressants, antibiotics, antiparasitic agents, antiepileptics, antiplatelets, antifungals, anti-inflammatory drugs, hormones, and antidiabetic agents), caffeine, and acesulfame K. The latter two compounds were analysed together with the pharmaceuticals.

Samples were analysed for pharmaceuticals using LC-MS/MS (Acquity UPLC and Xevo TQ, Waters, Milford, MA, USA) and for phthalates using GC-MS/MS (Thermo Fisher Scientific, San Jose, CA, USA). Water samples were spiked with mass-labelled internal standards before extraction. Solid-phase extraction (SPE) was applied for the pharmaceutical samples: Oasis WAX 150 mg, 6 cc by Waters for acesulfame, ibuprofen, diclofenac and caffeine, and for the other pharmaceuticals Atlantic® HLB-M disk, 47 mm by Biotage for groundwater and surface water samples and Oasis HLB, 500 mg, 6 cc by Waters for wastewater samples. Phthalates were extracted from the water samples by liquid-liquid extraction using *n*-hexane followed by SPE (Strata®, 500 mg, 3 mL by Phenomenex). Water samples were not filtered before analysis, i.e. they were analysed including suspended solids.

Five soil samples were analysed for the same suite of micropollutants as the water samples. The samples were freeze-dried, spiked with mass-labelled internal standards, and subjected to solid–liquid extraction. For phthalate analysis, *n*-hexane was used as the extraction solvent; ethyl acetate was employed for acesulfame, ibuprofen, diclofenac, and caffeine; and McIlvaine buffer (pH 4), acetonitrile, and methanol were used for the remaining pharmaceuticals. Following extraction, sample

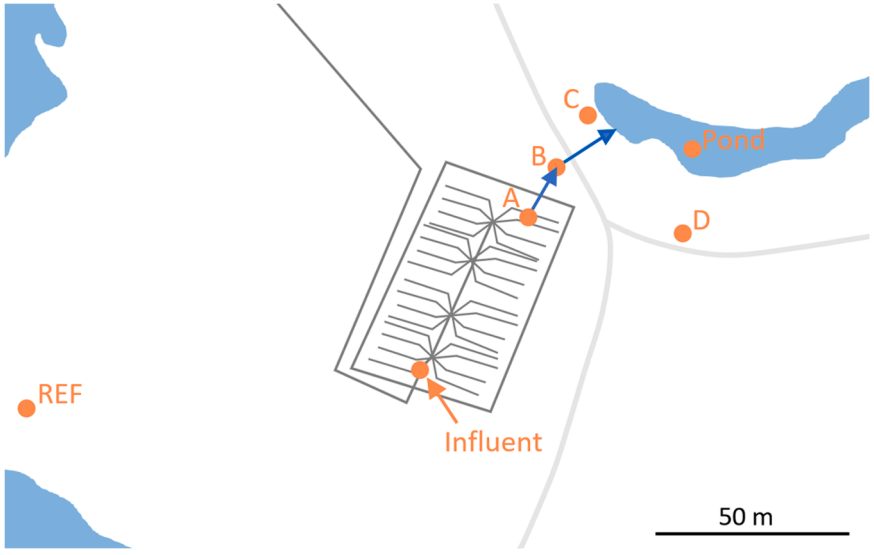

Fig. 1. The studied infiltration system and locations of sampling points: influent sampling point, groundwater monitoring tubes (A-D and REF), and pond surface water sampling point. The blue arrows show the approximate main groundwater flow directions from points A and B during the study.

extracts were purified using solid-phase extraction (SPE): Oasis MAX (150 mg, 6 cc; Waters) for pharmaceutical samples and Strata® (500 mg, 3 mL; Phenomenex) for phthalate samples.

2.3.1. Quality assurance and quality control

The quality control of sampling and analyses included control samples spiked with the studied chemicals and at least one method blank per sample set. The method blank covered all the background and interferences coming from the analytical procedure. For the phthalate samples, three method blanks for water samples and one method blank for solid samples were conducted per sample set to evaluate the background. Results from the method blanks were subtracted from the sample results. The pharmaceutical sample blanks were clean. The recoveries of the spiked control samples were 76–128 % for acesulfame, ibuprofen, diclofenac, and caffeine. For phthalates, the recoveries of spiked control samples varied between 72 % and 120 %. The absolute recovery range for the other pharmaceuticals was 25–120 %. The analysis results were corrected with the recovery of mass-labelled internal standards. For pharmaceuticals that had no mass-labelled analogue, the spiked control samples were used for recovery correction.

2.4. Other analyses

In addition to micropollutants, water samples were analysed for conventional water quality parameters, including chloride, ammonium and ammonia, nitrate, total and dissolved organic carbon (TOC and DOC), and biochemical and chemical oxygen demand (BOD_7 and COD). Soil samples from points B, C, and D were examined for pH, effective cation exchange capacity, total organic carbon, loss on ignition, and grain-size distribution. The pond sediment sample was analysed for loss on ignition. Detailed descriptions of analytical methods and grain-size distribution results (Table S1) are provided in the supplementary information.

2.5. Dilution and attenuation

To account for dilution by groundwater as wastewater migrated through the infiltration system, dilution factors (DF) were calculated by comparing chloride and acesulfame concentrations at each sampling location with those in the influent, using the geometric mean (Eq. 1):

$$DF_{X,\ Month} = \sqrt{\frac{C_{ACE_{X,\ Month}}}{C_{ACE_{Influent,\ Month}}} * \frac{C_{Cl_{X,\ Month}}}{C_{Cl_{Influent,\ Month}}}} \quad (1)$$

where DF is the dilution factor and C represents the concentration of acesulfame (ACE) or chloride (Cl) at the respective sampling point (X) or influent during a given month. This calculation was performed for March and June, when influent concentrations were available. Using two tracers provided a more robust estimate than relying on a single tracer, as both chloride and acesulfame K are recognized as reliable indicators of wastewater presence [19,33]. These substances are expected to attenuate primarily through dilution rather than degradation or sorption, and their attenuation patterns were similar despite differences in absolute concentrations. Micropollutant concentrations were subsequently adjusted for dilution using the DF values for each sampling point and event, as shown in Eq. 2:

$$Attenuation(\%) = (1 - \frac{C_{X,\ Month}}{DF_{X,\ Month} *\ \ C_{Influent,\ Month}}) * 100 \quad (2)$$

where attenuation is expressed as a percentage, and C denotes the concentration of each substance at the respective sampling point and time. When concentrations in groundwater or surface water were below the limit of quantification (LOQ), the LOQ was used as a conservative estimate to enable attenuation calculations. To support interpretation, the hydrophobicity of individual compounds was considered by evaluating their octanol–water partition coefficient (Log K_{OW}), obtained from the Norman Substance Database [25].

2.6. Risk quotient

Risk quotients (RQ) were calculated for substances whose annual average concentrations in surface water exceeded the lowest predicted no-effect concentration (PNEC), using the ratio between these two values [23]. The lowest PNEC for freshwater, based on the most sensitive species, was obtained from the Norman ecotoxicology database [26]. An RQ greater than 1 was interpreted as indicating a high environmental risk.

3. Results

3.1. Groundwater flow direction and velocity

The primary groundwater flow from point A, located north of the infiltration system, was consistently directed toward point B across all seasons (Fig. 1). From point B, the flow direction exhibited slight seasonal variability, alternating between point C and the pond sampling location. These observations confirm that the selected sampling points were appropriately positioned and that the collected groundwater samples represent the flow path originating from the infiltration system. In contrast, groundwater at point D appeared to receive minimal wastewater-contaminated flow from the lower section of the system (point A), as indicated by lower concentrations compared to points A, B, and C. However, point D may have been influenced by other parts of the infiltration system. The suggested groundwater velocities estimated from point dilution tests were 6.6 cm h^{-1} and 16 cm h^{-1} in tubes B and C, respectively. Based on the average of these velocities and the distance between the two points, the travel time from B to C would be approximately 5.4 days, assuming the shortest flow path. This suggests that wastewater required at least this duration to move beyond the infiltration system and reach the pond, given that point B was situated near but outside the system and point C was located close to the pond. Groundwater levels recorded during the different sampling events are presented in Fig. 2.

3.2. Estimation of dilution and treatment of conventional parameters

The estimated attenuation of chloride and acesulfame, along with their mean values at sampling points A, B, C, D, and the pond, is presented in Table 1. In general, attenuation increased with distance from the infiltration system, although this trend varied between the two sampling occasions. Instances of negative dilution observed at location A in March and location B in June—indicating concentration rather than dilution—can likely be attributed to the fact that influent, groundwater, and surface water samples did not originate from the same water mass, as they were collected simultaneously. Consequently, the dilution estimates provided should be regarded as approximations.

The removal of organic matter from the wastewater was highly efficient, as evidenced by the low biochemical oxygen demand (BOD) in groundwater at points A–D, frequently below the limit of quantification (1 mg L^{-1}). For instance, point A exhibited reductions of approximately 96 % compared to the influent in both March and June. The ratio of chemical oxygen demand (COD) to BOD increased from 2.2 to 3.3 in the influent—values typical for untreated wastewater [17] — to > 8.9 in groundwater. This shift indicates that biodegradable organic matter was preferentially removed relative to non-biodegradable fractions, suggesting that biodegradation was a key removal mechanism within the system. Influent concentrations of organic carbon ranged from 55 to 100 mg L^{-1} (TOC) and 32–73 mg L^{-1} (DOC), whereas groundwater concentrations were substantially lower, ranging from 2.5 to 45 mg L^{-1} (TOC) and < 1.0–12 mg L^{-1} (DOC), with averages of 15.2 ± 9.7 mg L^{-1} (TOC) and 6.5 ± 3.1 mg L^{-1} (DOC). Ammonium, the dominant nitrogen species in the influent, was largely transformed to nitrate

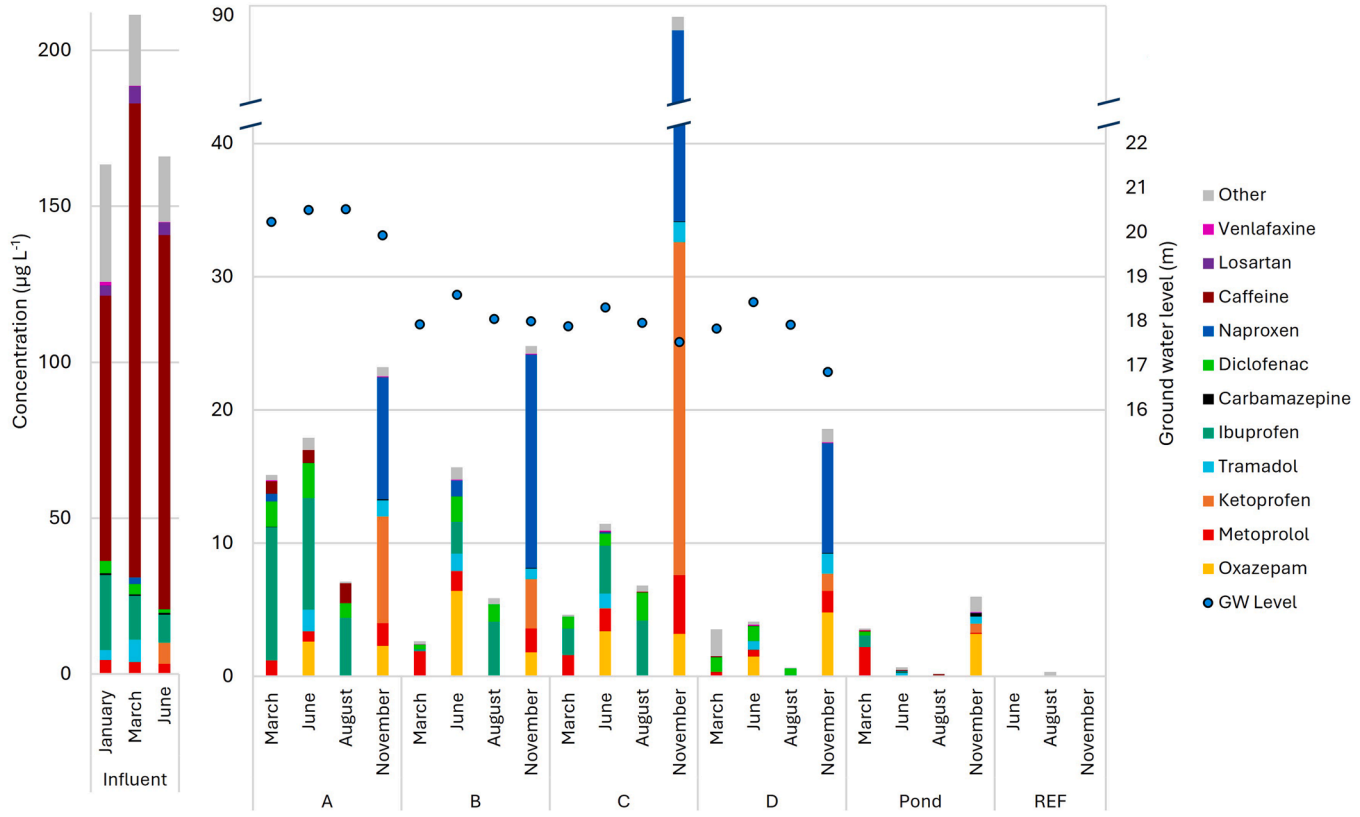

Fig. 2. Concentrations of micropollutants measured as grab samples in the influent, groundwater, and surface water as stacked bars, and groundwater levels as dots, corresponding to the second y-axis (meters above sea level). Concentrations are not adjusted for dilution. Eleven pharmaceuticals are shown by colour, and the remaining pharmaceuticals and phthalates are shown in grey as "other". Metformin and paracetamol were omitted from the figure as they were, with a few exceptions, only found in the influent and only in March, and at much higher concentrations (410 µg/L and 170 µg/L) compared to the other pharmaceuticals. Acesulfame K was omitted as it was found at high concentrations. Only diclofenac, ibuprofen, caffeine, acesulfame K, and phthalates were analysed in August, as analytical errors hindered analysis of remaining 65 pharmaceuticals. Supplementary sampling was carried out in November for these pharmaceuticals. The exact concentrations of all measured compounds can be seen in the data repository [14].

Table 1
Dilution expressed as 1-DF (dilution factor) at different water sampling points, calculated using concentrations of chloride (Cl) and acesulfame K (ACE) measured in March and June. Negative dilution means that the tracer was more concentrated.

Sampling point	Dilution (1- DF)					
	March (%)			June (%)		
	Cl	ACE	mean	Cl	ACE	mean
A	1.74	-15.00	-6.30	8.36	22.2	15.6
B	26.5	17.5	22.1	-18.2	-11.1	-14.6
C	36.6	20.0	26.6	21	25.0	23.0
D	63.9	57.5	60.8	51.4	41.7	46.8
Pond	42.2	30.0	36.4	84.9	82.8	83.9

through nitrification, as reflected by a marked decrease in ammonium and a corresponding increase in nitrate in groundwater. After adjusting for dilution, ammonium concentrations decreased by 87.1 ± 10.6 % and 73.7 ± 24.8 % in March and June, respectively, while nitrate increased from ≤ 0.05 mg L^{-1} in the influent to averages of 26.0 ± 6.1 mg L^{-1} and 23.0 ± 13.3 mg L^{-1} in March and June. Subsurface water temperatures averaged 5.9 ± 1.0 °C (March), 9.1 ± 0.7 °C (June), 9.8 ± 1.0 °C (August), and 4.0 ± 0.7 °C (November). Complete datasets for all water quality parameters, including conventional wastewater indicators, are available in the data repository [14].

3.3. *Micropollutants in influent, groundwater, and surface water, and their reductions*

Of the 84 substances screened in water and soil samples, 64 were detected in at least one matrix—either influent, groundwater, surface water, or soil. Four compounds (amlodipine, atorvastatin, paracetamol, and sertraline) were found exclusively in the influent wastewater. Overall, micropollutant concentrations in groundwater and surface water were substantially lower than in the influent (Fig. 2). However, concentrations at point C in November were markedly elevated (89 µg L^{-1}) compared to other groundwater samples (<25 µg L^{-1}), primarily due to exceptionally high levels of naproxen (54 µg L^{-1}) and ketoprofen (25 µg L^{-1}). For these compounds, the analytical method's quantitative range was exceeded, and the reported values should therefore be considered approximate. Comparison across sampling events suggests an inverse relationship between micropollutant concentrations and groundwater levels (Fig. 2). Lower groundwater levels theoretically imply reduced dilution, resulting in higher concentrations. This likely contributed to the elevated concentrations observed in November, when groundwater levels were lowest, although not all micropollutants were analysed during that sampling event.

The reduction in micropollutant concentrations within and beyond the infiltration system varied considerably among individual compounds. In March and June, when dilution effects could be estimated and accounted for, many substances were still efficiently removed from the wastewater plume, as indicated by substantially lower concentrations in groundwater and surface water compared to the influent (Table 2). Compounds exhibiting the highest removal included caffeine,

Table 2
Influent concentrations and attenuations of 42 compounds at the five sampling points (A, B, C, D, Pond) in March and June after adjusting for dilution. *Calculation based on the LOQ as a conservative estimation for the groundwater or surface water sample, as concentration was below the limit of quantification. (-) Either groundwater/ surface water was not analysed, or influent concentration was not analysed, or was below the limit of quantification. NA = not analysed due to analytical issues.

Compound	Inflow concentration (ng/L)		Attenuation (%) in groundwater (A-D) and surface water (Pond) compared to the influent concentration after adjusting for dilution using the DF									
	March	June	March					June				
			A	B	C	D	Pond	A	B	C	D	Pond
Diclofenac	3300	1100	46	86	64	15	86	-180	-51	-6	-88	84
Ibuprofen	14000	9000	33	99	81	> 99	90	-11	77	48	> 99	98
Amlodipine	280	< 5.4	> 99*	> 99*	> 99*	> 99*	99*	-	-	-	-	-
Atenolol	3300	4100	> 99	> 99	> 99*	> 99*	> 99*	99	97	94	-	98
Bisoprolol	730	670	> 99*	> 99*	99	> 99	> 99*	-	99	99	> 99*	> 99*
Carbamazepine	450	550	96	> 99*	> 99*	> 99*	> 99*	99	-	-	-	46
Cetirizine	920	1300	> 99	> 99*	> 99*	> 99*	> 99	94	> 99*	99	> 99*	> 99
Citalopram	3400	930	> 99*	> 99*	> 99*	> 99	> 99*	> 99*	> 99*	> 99*	> 99*	> 99*
Dipyridamole	34	NA	98*	97*	97*	95*	99*	-	-	-	-	-
Doxycycline	630	NA	> 99*	99*	99*	99*	98*	-	-	-	-	-
Enalapril	440	340	> 99*	99*	> 99*	> 99*	> 99*	-	98	> 99	> 99*	> 99*
Fexofenadine	64	460	> 99*	> 99*	> 99*	99*	> 99*	97	> 99*	99	> 99*	> 99*
Fluconazole	2	< 1.1	-68	-936	99*	-1021	98*	-	-	-	-	-
Gabapentin	< 2.2	1700	-	-	-	-	-	80	82	84	-	65
Irbesartan	7600	3500	> 99*	> 99*	> 99*	> 99*	> 99*	-	-	-	99	96
Ketoprofen	< 14	6800	-	-	-	-	-	-	> 99	> 99	> 99*	99*
Levetiracetam	460	440	> 99*	95	> 99*	97	92	-	-	-	-	-
Losartan	5600	4100	> 99*	> 99*	> 99*	> 99*	> 99	-	-	-	99	98
Metformin	410000	NA	> 99*	> 99*	> 99*	> 99*	> 99*	-	-	-	-	-
Metoprolol	3900	3300	71	37	44	78	11	72	60	33	71	95
Naproxen	2100	NA	> 99*	> 99*	> 99*	> 99*	> 99*	-	-	-	-	-
Paracetamol	170000	NA	> 99*	> 99*	> 99*	> 99*	> 99*	-	-	-	-	-
Quetiapine	25	< 120	> 99*	> 99*	> 99*	> 99*	> 99*	-	-	-	-	-
Ramipril	29	34	76	29	82	67	96	-	73	78	> 99*	93
Sertraline	410	170	> 99*	> 99*	> 99*	> 99*	> 99*	> 99*	> 99*	> 99*	> 99*	93*
Simvastatin	< 4.7	210	-	-	-	-	-	94	> 99*	99	> 99*	87*
Sulfadiazine	140	100	97	> 99*	> 99*	> 99*	> 99*	> 99*	> 99*	> 99*	> 99*	86*
Sulfamethoxazole	< 1.6	230	-	-	-	-	-	-	-	-	> 99*	> 99*
Testosterone	< 12	160	-	-	-	-	-	-	> 99*	-	> 99*	99*
Tetracycline	650	1500	> 99*	> 99*	> 99*	99*	99*	> 99*	-	-	> 99*	98*
Trimethoprim	31	110	> 99*	82	> 99*	99*	97	-	94	95	> 99*	62
Warfarin	790	1600	90	> 99*	> 99*	> 99*	> 99*	-	> 99*	> 99*	> 99*	> 99*
Venlafaxine	36	39	-48	18	-4	30	-24	-	-57	-137	-146	-32
Xylometazoline	19	10	> 99*	> 99*	99*	99*	98*	-	99*	99*	99*	85*
Bis(2-ethylhexyl) phthalate (DEHP)	290	689	94	92	95*	43	95*	53	88	98*	90	91*
Benzyl butyl phthalate (BBP)	180	197	86	81	82	60	79	94*	96*	93*	90*	69*
Bis(2-ethylhexyl) terephthalat (DEHT)	< 10	563	-	-	-	-	-	83	91	98*	82	89*
Dibutyl phthalate (DBP)	290	254	97*	96*	95*	91*	95*	90	93	94	75	49
Diethyl phthalate (DEP)	1470	1100	88	98	98	98*	99*	93	91	96	94	94*
Diisobutyl phthalate (DIBP)	640	708	99*	98*	98*	96*	98*	98	99*	98*	97*	81
Dimethyl phthalate (DMP)	31	12	70*	59*	56*	18*	49*	1*	27*	-8*	-182	-416*
Caffeine	152000	120000	> 99	> 99*	> 99*	> 99	> 99	99	> 99*	> 99	> 99	> 99

carbamazepine, and losartan. Caffeine concentrations declined by more than 99 % at groundwater sampling point A and in the pond surface water during both sampling events. Similarly, carbamazepine concentrations decreased by 96 % and 99 % at point A and by > 99 % and 46 % in the pond surface water in March and June, respectively. Losartan showed reductions exceeding 99 % at point A in March and > 99 % and > 98 % in the pond surface water during the same periods. Other compounds demonstrated moderate attenuation. For example, metoprolol concentrations decreased by 71 % and 72 % at point A and by 11 % and 95 % in the pond surface water in March and June, respectively. In contrast, some substances exhibited low or even negative attenuation, with considerable variability between sampling events. Diclofenac concentrations at point A decreased by 46 % in March but increased by 180 % in June relative to the influent, although reductions in the pond surface water remained high (86 % and 84 %). Ibuprofen displayed similar fluctuations, with decreases of 33 % and −11 % at point A and 90 % and 98 % in the pond surface water in March and June, respectively. Venlafaxine showed only negative attenuation (−48 % and −57 % at point A and −24 % and −32 % in surface water), indicating that it was not effectively treated within the system.

Seven of the fifteen targeted phthalates were detected in the water samples (Fig. 3). Concentrations were substantially lower in groundwater (0.02–0.455 μg L^{-1}) and surface water (0.024–0.047 μg L^{-1}) compared to the influent (2.577–3.523 μg L^{-1}), with one notable exception: groundwater at point D in March contained 1.993 μg L^{-1}, primarily due to bis(2-ethylhexyl) terephthalate (DEHT) at 1.9 μg L^{-1}. The cause of this elevated DEHT concentration remains unclear but may reflect spatial or temporal variability in groundwater composition, as the sampled water masses were not necessarily identical across locations. After accounting for dilution, average reductions in phthalate concentrations were 51 ± 72 % and 92 ± 5 % in groundwater for March and June, respectively, and 99 % and 92 % in surface water. These removal efficiencies are comparable to those reported for conventional wastewater treatment plants, including activated sludge systems, which typically achieve around 90 % removal [6]. Furthermore, the observed reductions exceeded those documented for other nature-based technologies, such as constructed wetlands [24]. Phthalates were also detected in groundwater at the reference point in August, with concentrations

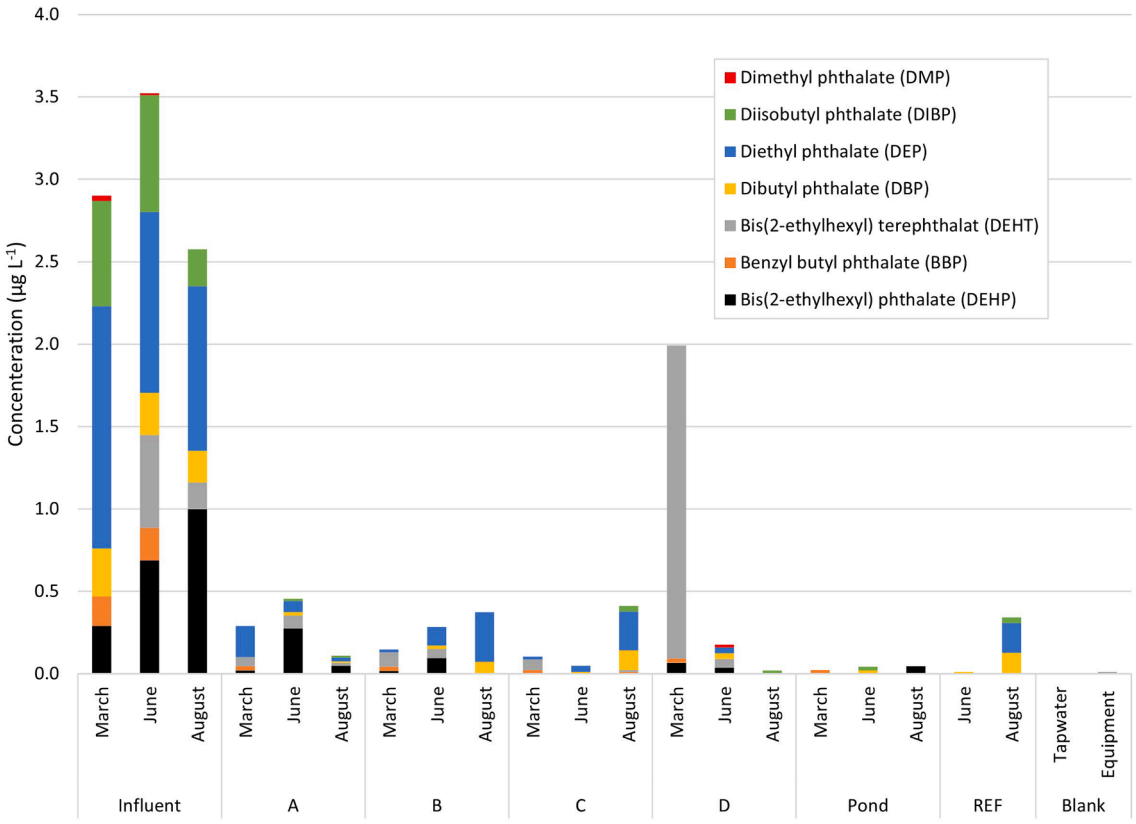

Fig. 3. Concentrations of seven phthalates detected in the influent, groundwater, surface water, and blank samples (without accounting for dilution). The exact concentrations of all measured compounds can be seen in the data repository [14].

similar to those at sampling points A, B, and C. A trace amount of DEHT (0.011 µg L^{-1}) was detected in the equipment blank and deemed negligible.

3.3.1. *Deviations from dilution in the groundwater*

When micropollutant concentrations were adjusted for dilution using the dilution factor (Eq. 1) and deviations from expected dilution were calculated (Eq. 2), the results indicated that attenuation in groundwater was primarily attributable to dilution along the flow path from point A to point C (Fig. 1). Three representative compounds illustrating this trend are shown in Fig. 4. Ibuprofen exhibited greater attenuation than could be explained by dilution alone, particularly between sampling points A and B. Diethyl phthalate showed attenuation only between the influent and point A; beyond this, its concentrations closely followed the dilution trend, although it was below the limit of quantification in the pond surface water. Diclofenac displayed inconsistent patterns between March and June but demonstrated a similar, albeit less pronounced, attenuation compared to ibuprofen between

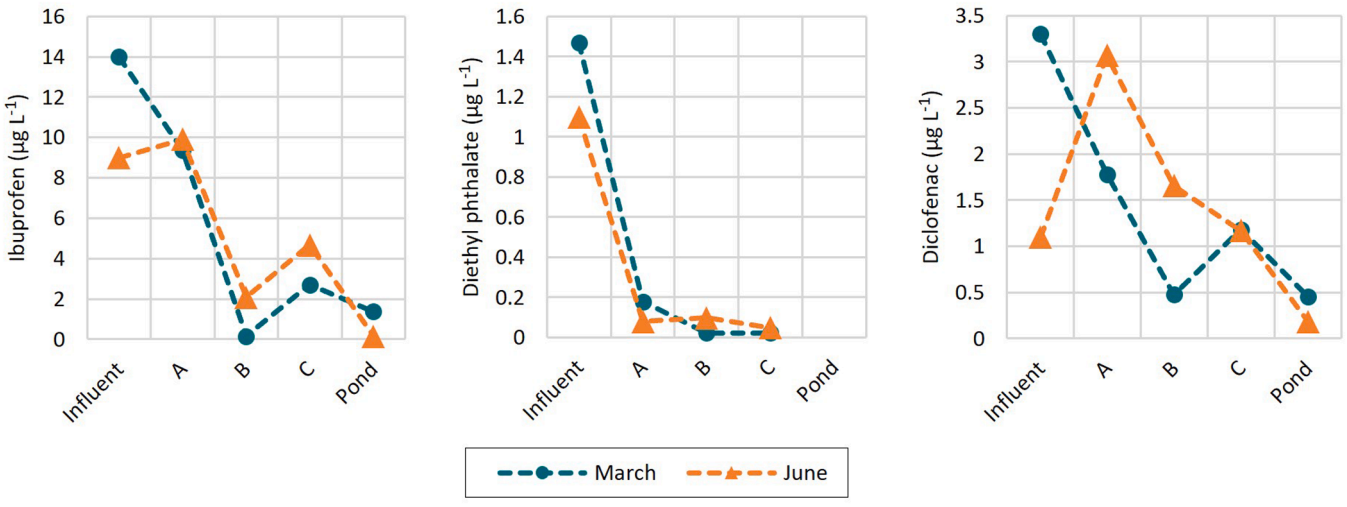

Fig. 4. Concentrations of ibuprofen, diethyl phthalate, and diclofenac adjusted for dilution, at the sampling points (influent, groundwater samples A, B and C, and pond) in March and June. Sampling point D was not included because it was not situated in the main groundwater flow path (Fig. 1).

points A and B. It should be noted that many substances could not be evaluated due to concentrations falling below the limit of quantification.

3.4. Micropollutants in soil and sediment

A total of 23 individual micropollutants were detected out of 87 analysed in soil samples collected from points B, C, D, the reference location, and pond sediments, with concentrations ranging from below the limit of quantification to 45 μg kg^{-1} dry weight (DW) (Fig. 5). Phthalates were below the limit of quantification in all solid samples. Concentrations at point B were lower than those at point C, which was situated closer to the pond (Fig. 1). Point D exhibited the lowest micropollutant concentrations, consistent with its greater distance from the infiltration system and its position outside the main groundwater flow path from point A. The reference sample contained disproportionately high concentrations of two compounds: the anti-inflammatory drug mesalazine (37 μg kg^{-1} DW) and the antibiotic ofloxacin (8.7 μg kg^{-1} DW), both of which were below the limit of quantification in other soil samples. Additionally, the reference sample included fenbendazole, ketoprofen, metformin, nebivolol, quetiapine, risperidone, sulfamethoxazole, and tramadol, all at concentrations below 1.1 μg kg^{-1} DW.

4. Discussion

4.1. Attenuation of micropollutants

Many of the investigated micropollutants were attenuated (retained or otherwise removed) from the wastewater as it percolated through the soil in the infiltration system, comparing the concentrations in the influent with those in the groundwater and surface water (Fig. 2). However, many compounds were already attenuated to a large extent when measured in groundwater sampling point A (Table 2), with the attenuations adjusted for dilution, indicating that a large part of the attenuation occurred in the unsaturated zone of the infiltration system (the vadose zone), between the distribution pipes and sampling point A. Treated wastewater may have, however, reached sample point A via the groundwater from other areas of the infiltration system, so that the water sampled there was a mixture of water. Removal of micropollutants primarily in the vadose zone of infiltration systems has been observed by e.g. Yang et al., [41], who found that the total concentration of several micropollutants decreased by more than 80 % in this zone. Gao et al. [12] also indicated that a thicker vadose zone could partially explain more efficient micropollutant removal in one infiltration system over another. Conn et al. [3] found that caffeine, triclosan, and nitrilotriacetic acid were mostly removed within the first 60 cm of the vadose zone, while other micropollutants, e.g., ethylenediaminetetraacetic acid (EDTA) and sulfamethoxazole, were still found in deeper parts of the vadose zone, indicating that some substances may not be removed regardless of the depth. This is consistent with the present study, where the depth of the vadose zone (1–1.5 m at sample point A) seemed to be adequate for the removal of many non-persistent substances, such as caffeine, while more persistent substances, such as diclofenac, were detected in the groundwater at mostly unaltered concentrations compared to the influent.

It is not known which processes were responsible for the attenuations in the vadose zone, but there could be several processes involved such as biodegradation, sorption, filtration, retardation, and chemical transformation. This may vary between compounds, depending on their physicochemical properties, and the properties of the environment. Biodegradation may be a major removal mechanism in the vadose zone as oxygen is often available there [5]. In this study, the low concentrations of BOD in groundwater, increased COD/BOD ratio and nitrate concentration, and the decrease in ammonium concentration in the groundwater compared to the influent, indicated that biological activity and nitrification occurred. This was seen both in March and June, although in March the groundwater temperature was lower (6°C) compared to in June (9 °C), indicating a lower biological activity. In general, cold and oligotrophic environments, which are often prevailing in northern Sweden, may slow down the degradation of pollutants, including e.g., phthalates [35].

The concentrations of TOC and DOC in the influent and the groundwater indicated that there was organic carbon present in the wastewater as it travelled through the vadose zone, which may have impacted how micropollutants were transported and how they behaved. Organic carbon may, during aerobic conditions, promote biodegradation of certain micropollutants, depending on their properties, but may also hinder the degradation of micropollutants due to the consumption of available oxygen [2].

When comparing the concentrations of individual micropollutants in the different groundwater tubes (A-D) and in the surface water (after adjusting for dilution), it was seen that the concentrations of most compounds did not decrease much after reaching the groundwater. An exception was ibuprofen (Fig. 4), whose concentration decreased

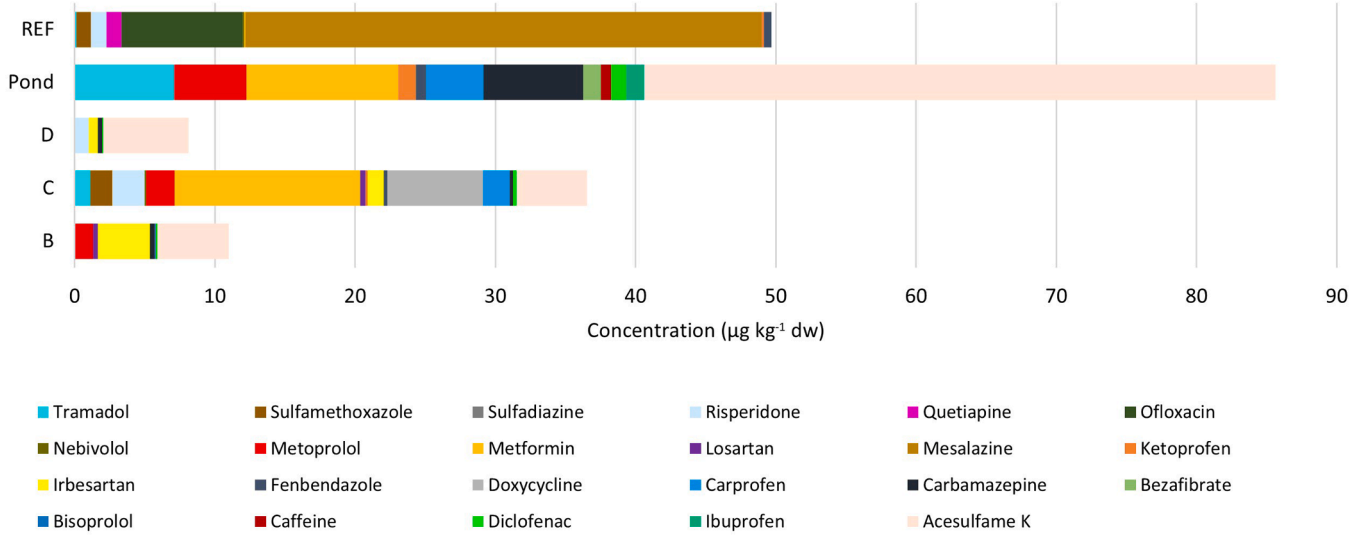

Fig. 5. Concentrations of micropollutants detected in soil samples (B-D and REF) and pond sediment (Pond). dw: dry weight. The exact concentrations of all measured compounds can be seen in the data repository [14].

between sample location A and B, but then was higher again in sample point C. This was seen both in March and June, implying that the higher concentration in point C was not due to variations in the groundwater, but possibly that point C received groundwater not only from B but from A or other parts of the infiltration system, so that the concentrations of ibuprofen there were elevated compared to point B. The observed attenuation of ibuprofen levels in groundwater was similar to the findings of Kreuzinger et al. [16], However, the cause of this attenuation remains unknown. Aerobic degradation is unlikely as oxygen should not be present in the groundwater in this context, and anaerobic degradation, which could potentially be a viable degradation mechanism, has not been identified as a significant pathway for ibuprofen breakdown [22,30].

4.2. Sorption of micropollutants to soil

In the present study, the properties of the soil, including high sand content, small fraction of organic material, and low cation exchange capacity (Table S1), did not seem to favour soil sorption processes. Cation exchange capacity measures the soil's ability to attract positively charged pollutants, and capacities below 10 cmol+ kg^{-1} are considered low [15], and here they were < 2.1. Sorption, however, also depends on other factors such as the hydrophobicity of the compound. The highest concentrations of micropollutants were found in the pond sediment sample, which may be due to its higher content of organic material, as indicated by the loss on ignition (Table S1). Acesulfame was present in the solid samples, although it is known to be a very water-soluble compound (log $K_{OW} = -0.57$) and therefore not expected to be found there. This may indicate that the results for the solid samples included compounds that were not fully sorbed but were possibly only present in porewater. Furthermore, it was not clear how long time substances could be sorbed to the soil particles or if they could be released when conditions are changed. Reversible sorption has been documented before, e.g., for carbamazepine [43].

Considering the attenuation of ibuprofen in the groundwater, and its hydrophobic character (log $K_{OW} = 3.74$), removal via sorption would be a plausible reason. Ibuprofen concentrations, however, were below the limit of quantification in all the soil samples, which does not support this theory. This may be attributed to analytical detection limits, spatial variability, sampling limitations, or adsorption-desorption dynamics, potentially leading to the release of previously sorbed ibuprofen.

At point C, the total concentration of micropollutants retained in the soil was higher than at point B (Fig. 5) because a larger number of micropollutants were detected there, including metformin, doxycycline, carprofen, and risperidone. Possibly, more wastewater-contaminated groundwater passed through point C than point B (Fig. 1), which was also suggested for the water samples. This was supported by that the loss on ignition of soil from points B and C was similar, meaning that their organic matter content was similar and should not imply different sorption capabilities. Sampling point D had the lowest total concentration of micropollutants in soil, which was expected since the groundwater flow path from point A was directed towards B.

The concentrations of micropollutants in the soil of the reference sample point (Fig. 5) were difficult to interpret. The sample contained ofloxacin that was not present in any other sample, and mesalazine and quetiapine that were not present in any other soil samples. Mesalazine was detected five times in groundwater and quetiapine two times in the influent and two times in the groundwater. These findings were unexpected but could be due to contamination from the river water, as the groundwater level in the reference tube seemed to be related to the river water level, although not measured. The fact that acesulfame was below the limit of quantification in the reference water sample (whereas it was clearly present in the wastewater-influenced samples) indicated that the reference point was not contaminated with wastewater from the infiltration system during the time of sampling.

4.3. Micropollutants in groundwater and surface water

Although the concentrations of many micropollutants decreased to a high degree in the infiltration system, and the concentrations measured in the pond surface water were generally lower than in the groundwater, several substances were present in the pond surface water at concentrations close to or exceeding the lowest PNEC. For example, the lowest PNEC for diclofenac in freshwater (0.04 μg/L) was exceeded in the pond surface water in both March and August, and approached the threshold in June with concentrations ranging from 0.029 to 0.29 μg/L. The annual average of diclofenac (0.12 μg/L) corresponds to a Risk Quotient of RQ= 3.1, indicating that diclofenac is considered an environmental risk in this case. Furthermore, diclofenac showed very little attenuation, aside from dilution, which aligns with its classification as a persistent substance [44]. This, together with the short travel time from the infiltration system to the pond, could explain its presence in the pond's surface water. The diclofenac concentrations in the groundwater were similar to those reported in [16] and were higher than the lowest PNEC for freshwater (0.04 μg/L). A special PNEC for diclofenac in groundwater does however not exist, but different scenarios for the environmental risk assessment in groundwater have been discussed [8]. Ibuprofen concentrations exceeded the lowest PNEC (0.011 μg/L) in the pond surface water at all sampling occasions (0.025–0.88 μg/L; not analysed in November). The annual average of the ibuprofen concentrations (0.31 μg/L) corresponds to a Risk Quotient of RQ= 28.6, indicating that ibuprofen should be considered an environmental risk in this case. Some substances, such as erythromycin, caffeine, and gemfibrozil were detected at concentrations approaching but not exceeding their respective lowest PNECs. Metoprolol, venlafaxine, carbamazepine, and losartan were detected in the surface water samples at concentrations well below their respective lowest PNECs. Phthalates did not exceed their respective lowest PNECs. Oxazepam exceeded the lowest PNEC for freshwater (0.37 μg/L) in several groundwater samples and one surface water sample. A risk quotient was however not calculated as only one surface water sample exceeded the lowest PNEC.

The fate of micropollutants in aquatic environments is complex, as their persistence, transformation, and potential risks depend on various environmental factors. In this study, the groundwater in question was not utilized as a drinking water source; however, in other contexts, it may serve this purpose. Additionally, wastewater-contaminated groundwater may have infiltrated the river's surface water from pathways beyond the assessed infiltration system, which was not evaluated in this study. The analysis of transformation products was not included, but it is plausible that substances removed during infiltration underwent partial transformation during treatment. Some transformation products may retain environmental toxicity, while others have the potential to revert to their parent compounds. Therefore, further investigation is warranted, particularly if there are potential risks for the environment and drinking water sources.

4.4. Limitations

Soil and pond sediments were sampled once, which limits the ability to capture seasonal dynamics or time-dependent contaminant retention. Furthermore, no soil samples could be taken from within or beneath the infiltration system, which is restricting conclusions about micropollutant retention there. The dilution was estimated in relation to the chloride and acesulfame K concentrations, and influent, groundwater, and surface water were sampled simultaneously without accounting for flow velocity through time-shifted sampling. The LOQ for micropollutants varied across water types and sampling times, typically being lower in groundwater than influent water. This variability was expected and limited comparisons of detected substances across water types and influenced the treatment efficiency calculations. Some substances in influent wastewater may remained undetected at low concentrations, while certain compounds identified in groundwater could not be

analysed in the influent, making direct comparisons challenging.

5. Conclusions

Micropollutant concentrations were analyzed in the influent of a wastewater soil infiltration system, in a downgradient pond, and in groundwater and soil located between the infiltration system and the pond. The results indicate that the majority of removal occurred within the unsaturated soil zone, prior to the wastewater reaching the groundwater. The infiltration system achieved substantial reductions for several compounds, including caffeine, carbamazepine, losartan, and phthalates, while reductions were more moderate for substances such as metoprolol. Some substances had minor or negative attenuation, for example diclofenac, ibuprofen and venlafaxine. Notably, the attenuation of phthalates was comparable to that observed in conventional wastewater treatment plants. In the downgradient groundwater, only ibuprofen exhibited attenuation beyond what could be explained by dilution between the infiltration system and the pond, although the underlying mechanism remains unclear. These findings suggest that, while the soil provides an effective barrier in infiltration systems, its properties may not be optimal for the sorption and removal of several micropollutants. Soil samples revealed the presence of several compounds, including metoprolol, irbesartan, and metformin. However, it remains uncertain whether these compounds were truly sorbed to the soil matrix or simply present in porewater. In the downgradient surface water, diclofenac and ibuprofen exceeded risk quotient thresholds, while oxazepam surpassed the lowest PNEC in one sample. These exceedances highlight potential environmental risks and possible toxic effects on aquatic organisms.

Environmental implication

Wastewater infiltration systems are widely used in many parts of the world, making it essential to understand their performance in removing micropollutants and their potential impact on the surrounding environment. There is growing concern about ecotoxicological risks to aquatic organisms, as well as the potential for contamination of drinking water sources. Our study shows that many substances are effectively removed during infiltration, but several persistent pollutants were detected in the surrounding environment with little or no attenuation. These findings indicate that wastewater infiltration systems can pose ecological risks, although these might be lower compared to some conventional wastewater management technologies.

CRediT authorship contribution statement

Rasmus Klapp: Writing – review & editing, Writing – original draft, Visualization, Methodology, Investigation, Data curation, Conceptualization. **Inga Herrmann:** Writing – review & editing, Supervision, Project administration, Methodology, Funding acquisition, Conceptualization. **Annelie Hedström:** Writing – review & editing, Supervision, Methodology, Conceptualization. **Noora Perkola:** Writing – original draft, Validation, Methodology, Formal analysis. **Marja Hagström:** Validation, Methodology, Formal analysis. **Hanna Niemikoski:** Validation, Methodology, Formal analysis. **Juho Kinnunen:** Supervision, Software, Resources, Methodology, Conceptualization. **Pekka Rossi:** Writing – review & editing, Supervision, Project administration, Funding acquisition, Conceptualization. **Elisangela Heiderscheidt:** Writing – review & editing, Supervision, Project administration, Funding acquisition, Conceptualization.

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.

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Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at doi:10.1016/j.jhazmat.2025.140840.

Data availability

The data is available at the data respository of the Swedish National Data Service, link is provided.

Pollutants at a wastewater infiltration system in Northern Sweden: measurements in influent wastewater, groundwater, surface water and soil (Researchdata.se)

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