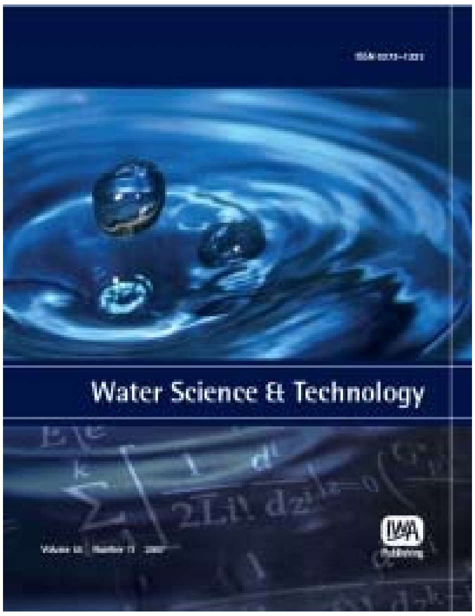
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Retention of pharmaceutical residues and microorganisms at the Altendorf retention soil filter

E. Christoffels, F. M. Mertens, T. Kistemann and C. Schreiber

ABSTRACT

A study has been conducted on a retention soil filter (RSF) to test its effectiveness in removing pharmaceutical residues and microorganisms from combined sewer overflows (CSOs). Efficient removal of solids, nutrients and heavy metals has already been proven. The possibility that organic micropollutants and microorganisms are also retained by the use of RSFs has been identified, but data are lacking. Results obtained in this study, in which testing for removal by a RSF of numerous micropollutant substances was performed, are most promising. The pharmaceuticals diclofenac and ibuprofen are presented in detail as examples of such micropollutants. Both showed a reduction in positive samples of more than 55% as well as a significant reduction in median and maximum concentrations. For microorganisms such as *Escherichia coli*, coliphages and *Giardia lamblia* (cysts), an average reduction in concentrations by three logarithmic steps (99.9%) was achieved. These results add to the evidence that using a RSF in the advanced treatment of wastewater from CSOs reduces the exposure of watercourses to pharmaceutical residues and microbial contamination.

Key words | combined sewer overflow, microorganisms, micropollutants, pharmaceuticals, retention soil filter, wastewater treatment

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INTRODUCTION

In addition to hydraulic impulsion loads on receiving watercourses, overflows from combined sewer systems induce significant peak loads (Christoffels 2008). Nevertheless, there are currently no mandatory regulations in Germany to limit such emissions. One reason may be that so far only a few systematic monitoring programmes for the evaluation of substances contained in combined sewer discharges have been established (Brombach & Fuchs 2003). In previous studies the number of parameters examined and the density of the collected data have often been insufficient, and organic micropollutants as well as microorganisms have hardly been taken into account.

The present study has been conducted as part of a research project funded by the German Federal State of North Rhine-Westphalia (NRW). The overall project entails examination of all relevant pathways for important substance groups, including selected micropollutants and microorganisms, in the catchment of a stream called the Swist, within the basin of the Erft river (a Rhine tributary). Within this project, a programme to monitor combined sewer overflows (CSOs) was initiated. It was found that CSOs represent a major source of micropollutants and microorganisms in watercourses (MUNLV 2010). It was thought that an appropriate measure to reduce such pollution would be advanced treatment using retention soil filters (RSFs). One RSF had already been installed in the Swist catchment as a means of advanced treatment of CSOs. The excellent cleaning capacity of the RSF had already been the subject of past studies, with nutrient retention rates of 75 and 98% reported (Frechen 2010; MUNLV 2003). Studies undertaken by the authors delivered similar results. The scope of this paper is thus limited to the methods applied and results obtained in the study of micropollutant and microorganism removal by a RSF.

RETENTION SOIL FILTER AND THE WATERCOURSE

RSFs are installed as a measure to treat wastewater from CSOs and thereby reduce hydraulic stress and pollutant load in a watercourse. The retention of combined sewage from direct discharge mitigates the hydraulic peak load for

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the receiving watercourse. The structure of a RSF is shown in Figure 1. In general, RSFs are planted with reeds to ensure a permeable filter surface. The filter body of sand (diameter 0.063–2 mm) has a layer thickness of 0.75 to 1 m. It is dewatered by a drainage system situated below the filter layer (filter gravel 2–8 mm diameter). Beneath the drainage layer the RSF is sealed against the ground with an impervious membrane. While the water percolates through the filter body, particulates and solutes are retained and reduced by filtration, adsorption and biochemical degradation. To avoid clogging in the filter, a combined sewage retention basin for reducing the load of particulate contents is situated upstream of the RSF (Dittmer 2006).

The RSF considered in this study is consistent with the general specifications stated above. It was built in 2005 and is located near the village of Altendorf, 15 km from the city of Bonn. The connected combined sewer system provides drainage for the villages of Hilberath and Altendorf, with a total of around 1,650 inhabitants. The runoff area is 18.8 ha. The sewer system serves a predominantly rural residential area with some farms. No significant commercial/industrial sites or facilities with special wastewater, such as hospitals, exist. A storage sewer with a volume of 143 m^3 lies upstream of the RSF. When the capacity of the sewer system and storage sewer is exceeded due to heavy precipitation, combined sewage flows into the RSF. The filter area of the RSF is 707 m^2, the volume 782 m^3. After treatment in the RSF, combined sewage is then discharged into a creek called the Altendorfer Bach, which flows into the Swist. The throttled discharge amounts to 10.5 L/s. The filtering performance is thus 0.015 L/(s m^2). The Altendorfer Bach is classified as a siliceous low mountain watercourse rich in fine material (MUNLV 2009). The mean water runoff upstream of the outflow of the RSF amounts to 26 L/s (result of rainfall–runoff simulation).

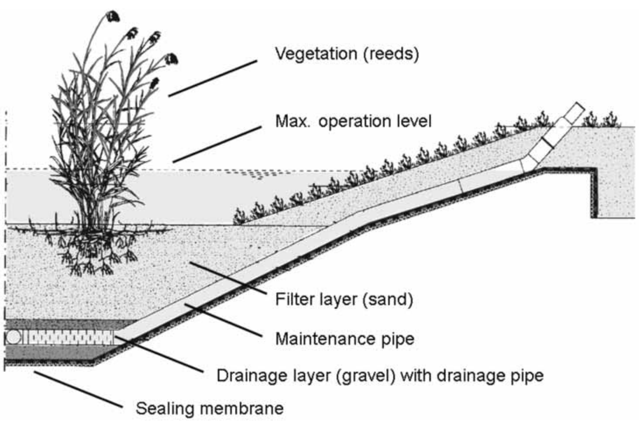

Figure 1 | Structure of a RSF, cross-section (MUNLV 2003).

SAMPLING SYSTEM AND ANALYSIS

At the inflow of the Altendorf RSF a water level sensor registers the beginning of the discharge from the storage sewer to the RSF. For the present study, a system was additionally installed to automatically detect RSF filling and trigger two sampling procedures, one for analysing physico-chemical parameters and the other for microbiological analysis. Sampling was performed at both the RSF inflow and outflow, at the inflow immediately when filling commences and at the outflow with a 1-hour time delay to ensure that all residual water from the previous filling event has been discharged.

For physico-chemical parameters, automatic sampling devices, each equipped with 11 bottles, were installed at both the inflow and outflow. The sampling procedure was controlled by a programmable logic controller, which allows individual programming of sample filling. For each RSF filling event, 12-minute composite samples of 2.9 litres over a maximum of 132 minutes were collected from the inflow and the outflow.

In addition to these spot samples used to assess physico-chemical parameters, large 200-litre composite samples were collected for microbiological analysis. A large sample volume is primarily necessary for acquiring sufficient knowledge on parasites because they generally occur in low population densities. The 200-litre samples were obtained first from the RSF inflow during the first 15 minutes of filling and second from the outflow over a 15-minute time span taking account of the aforementioned time delay. When sampling started, notification was automatically sent to staff via SMS to ensure rapid collection and analysis.

For each sample 35 pesticides and 10 pharmaceuticals were examined. The analyses were carried out by standard methods (HPLC–DAD (high-performance liquid chromatography with diode-array detection): DIN EN ISO 11369 (1997), GC-MS (gas chromatography–mass spectrometry): DIN EN ISO 15913 (2003)). The microbiological analyses included 12 parameters. The analyses of *Escherichia coli* were conducted according to the former EU bathing water directive 76/160/EEC; the analyses of the somatic coliphages according to the guideline DIN EN ISO 10705-2 (2002). Sampling and analyses of *Giardia lamblia* were performed according to the standards ISO 15553 (2006), EPA Method 1623 (2005); and Standing Committee of Analysts (1990). In the following, *E. coli* are indicated in MPN (most probable number) per 100 mL and coliphages in PFU (plaque forming units) per 100 mL. *Giardia lamblia* are specified as numbers of cysts present in each 100 litres.

RESULTS AND DISCUSSION

In the period from June 2010 to August 2011, using the sampling system described above, 33 RSF filling events with 343 individual samples were recorded. General physico-chemical parameters and micropollutant substances present at the RSF inflow and outflow were examined. Concentrations of bacteria and phages (virus with bacteria as host cell) were analysed from the RSF inflow and outflow for 25 filling events. Parasitological studies were conducted over 29 events.

Pharmaceutical residues

The findings for diclofenac and ibuprofen are considered to be representative of widely used pharmaceutical substances. These two pharmaceuticals are widely used as analgesics (painkillers) and to treat inflammation and fever. In Germany, the 2009 sales volume for diclofenac amounted to 85 tons, for ibuprofen 780 tons. Sales increased by 4% for the former and by 116% for the latter from 2002 to 2009 (Bergmann *et al.* 2011). Figure 2 shows the concentrations of these two substances for the inflow and the outflow of the Altendorf RSF. Median values and the 25th and 75th percentiles are depicted as boxes (interquartile range). The ends of the lines represent minimum and maximum values. For values below the limit of quantification (LOQ 0.1 μg/L), half the detection limit is used as a minimum. In addition, the proportions of the samples above the LOQ are given in the figure.

In the RSF inflow, diclofenac was detected in 68% of the samples and ibuprofen in more than 90%. Diclofenac was measured in concentrations up to 1.18 μg/L and ibuprofen up to 3.57 μg/L. The RSF retention capacity can be determined by comparing inflow and outflow concentrations. The median diclofenac concentration of 0.14 μg/L was reduced through RSF treatment to a level below the LOQ. Significant reduction in ibuprofen was also found, from the median concentration of 0.50 μg/L at the inflow to median values below the detection limit at the outflow. Likewise, maximum concentrations were decreased by 65% for diclofenac and 55% for ibuprofen. Furthermore, the percentage of positive findings at the outflow compared to the inflow was reduced from 68% to 9% for diclofenac and from 92% to 17% for ibuprofen.

The results show that the Altendorf RSF effectively retained pharmaceutical residues carried in combined sewage. Average concentrations and the number of positive findings for diclofenac and ibuprofen were substantially reduced. The significantly lower maximum concentrations at the RSF outflow also indicate that peak concentrations were capped.

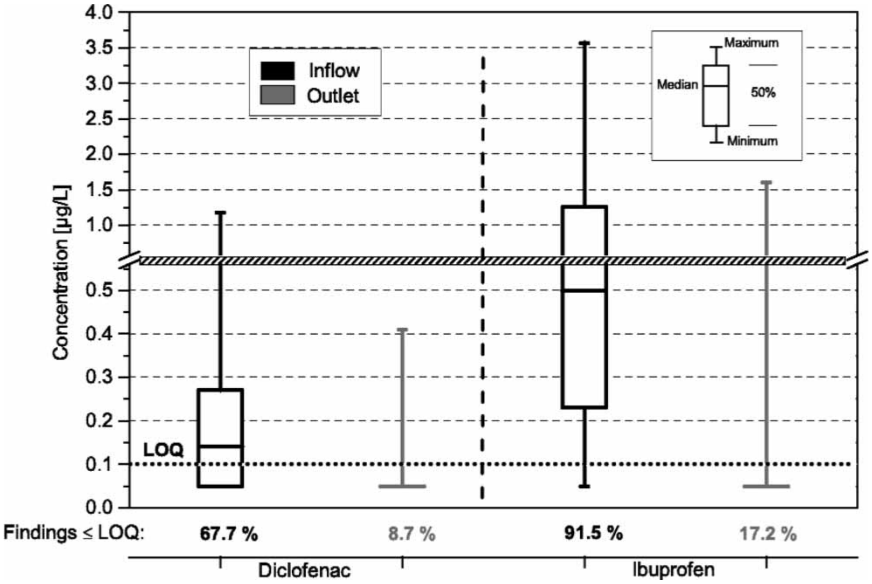

Figure 2 | Diclofenac and ibuprofen at the inflow and outflow of Altendorf RSF, measuring data from July 2010 to August 2011, 33 events with 343 individual samples, limit of quantification (LOQ) = 0.1 μg/L, assumption: values < LOQ equal to ½ LOQ.

For solids and substances adsorbed on solids, first flush effects (higher concentrations at the start of the event due to washout effects) have been described for CSOs (Christoffels 2008). Because of the high temporal resolution of the samples collected, statements about the temporal fluctuation of the concentration level during RSF feeding can be made. Figure 3 shows the median values and the 25th and 75th percentiles of pharmaceutical concentrations at the inflow and outflow of the Altendorf RSF during the loading phase.

At the entrance to the RSF, ibuprofen was observed in higher concentrations in the first 24 minutes of the examined events than in the further temporal course of the inflowing combined sewage. For the first 12 minutes, a median inflow concentration of 0.91 μg/L was obtained. For the next time span, the median value for ibuprofen held steady at 0.57 μg/L. After that, the median values of the overflow events fluctuated between 0.35 and 0.50 μg/L. The scatter of interquartile values fluctuated between 1.39 and 0.86 μg/L for ibuprofen in the inflow. In the RSF outflow, ibuprofen was detectable only sporadically in the first measurement cycle. The 75th percentile for the first measurement cycle, at 0.14 μg/L, stood just above the LOQ. For the remaining time window, all values were below the detection limit.

Although first flush effects were noticeable for ibuprofen in the inflow, no increased initial concentrations were detected for diclofenac. In the feed to the RSF, median concentrations for diclofenac ranged from 0.1 to 0.2 μg/L, a significantly lower level than for ibuprofen. Similarly, the scattering of the interquartile values, with a maximum of 0.3 μg/L, was less pronounced. In the outflow all percentile values were, without exception, below the LOQ. The results show that the RSF can effectively retain pharmaceutical residues present in combined sewage in a highly stable manner over the entire feed time.

Microorganisms

Three microbiological parameters were selected as representative examples of findings regarding the effectiveness of RSFs in reducing risks from waterborne pathogens. These included *E. coli* as a proxy for bacteria, coliphages as viral indicators, and cysts (permanent states) of the protozoa *Giardia lamblia* as representative of parasites. *E. coli* and phages represent indicator organisms of faecal contamination in water. *Giardia lamblia* are parasitic diarrhoeal pathogens.

Figure 4 shows median values for the three microorganisms, the 25th and 75th percentile, and the minima and maxima of observed concentrations from the inflow and outflow of the RSF. In addition, the percentage of positive findings is listed for each. Samples with values below the lower and above the upper LOQ are excluded from the calculation.

In the samples *E. coli* was detected in 96% of all cases in both the inflow and outflow of the RSF. Coliphages were also found in 96% of the inflow samples and 80% of the effluent samples. The studies of *Giardia lamblia* yielded positive findings in all feed samples but in only 43% of the

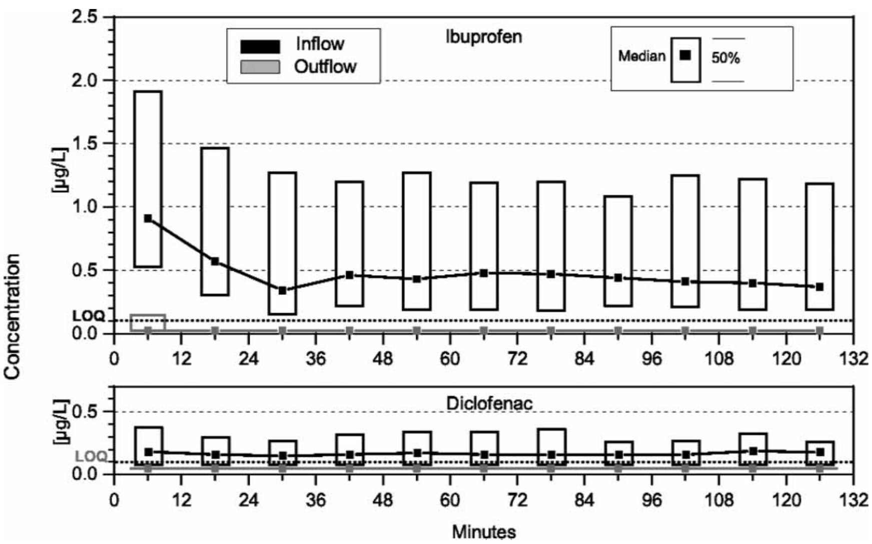

Figure 3 | Diclofenac and ibuprofen at the inflow and outflow of Altendorf RSF during the loading phase, from July 2010 to August 2011, 33 events with 343 individual samples, limit of quantification (LOQ) = 0.1 μg/L, assumption: values < LOQ equal to ½ LOQ.

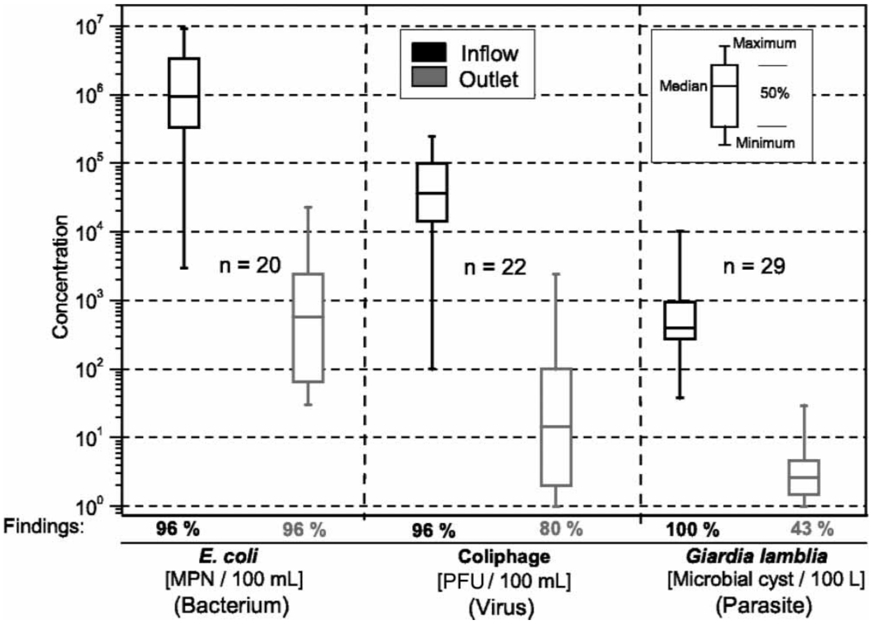

Figure 4 | *E. coli*, coliphages and *Giardia lamblia* in the Altendorf RSF inflow and outflow, from July 2010 to August 2011, maximum exploitable events = 29, readings with values above or below the limits of quantification were excluded, MPN = most probable number, PFU = plaque forming units.

effluent samples. Maximum inflow concentrations of 9.3×10^6 MPN per 100 mL for *E. coli*, 2.42×10^5 PFU per 100 mL for coliphages and 1.03×10^4 *Giardia lamblia* cysts per 100 L were detected. The determined median values in the feed for *E. coli* were at 9.30×10^5 MPN per 100 mL, for coliphages at 3.71×10^4 PFU per 100 mL and 400 *Giardia lamblia* cysts per 100 L.

In the outflow from the RSF, the concentrations of all three representative microorganisms were significantly reduced. Maximum concentrations reached 2.3×10^4 MPN per 100 mL for *E. coli*, 2.41×10^3 PFU per 100 mL for coliphages and 30 *Giardia lamblia* cysts per 100 L (Figure 4). Median values amounted to only 580 MPN per 100 mL for *E. coli*, 15 PFU per 100 mL for coliphages and three cysts per 100 L for *Giardia*. Compared with results for wastewater treatment plant outflows in the project area (MUNLV 2010), median RSF outflow concentrations of *E. coli* and *Giardia lamblia* cysts were an entire logarithmic step lower. The coliphage concentrations at the RSF outflow were at a level which can be measured in headwaters receiving no wastewater and draining an area where only extensive farming is practised (Franke *et al*. 2009).

Comparing concentrations at the inflow and outflow illustrates the RSF cleaning capacity (Figure 4). For *E. coli*, a reduction of 3.1 logarithmic steps expressed through the median values was observed, which corresponds to a decrease of 99.9%. Median concentration of coliphages also decreased through RSF treatment by 3.1 logarithmic steps. These results for the microbiological cleaning capacity of the Altendorf RSF are significantly better than the few benchmarks which exist for microbiological purification efficiency of RSFs in combined sewage systems (Hiekel *et al*. 2002; Frechen 2012). Differences in the detention time of combined sewage in the RSF, the properties of the filter material and the composition of the microbiota are possible reasons (Waldhoff 2008). In addition, the specific load at the RSF inflow plays a role. The feed concentrations at the Altendorf RSF are in a range which is typical for such facilities (Augenthaler & Huggenberger 2003; Frechen 2012).

With regard to *Giardia lamblia* cysts, the measured values in the RSF outflow were in many cases below the detection limit. For this reason, purification efficiency at the Altendorf RSF was calculated for *Giardia lamblia* using the detection limit value. The resulting median cleaning capacity under this assumption came to at least 2.1 logarithmic steps, which represents a 99.3% reduction in cysts. The effectiveness of RSF treatment is further underlined by the fact that positive results for *Giardia lamblia* cysts occurred in only 43% of cases. Given that survival rates of infectious permanent stages of parasites outside the host body are much higher than those of bacteria and phages, due to greater persistence, higher effluent concentrations of *Giardia lamblia* cysts were expected. However, a parasite such as *Giardia lamblia* is effectively retained in the filter material due to its size (up to nine times larger than bacteria (Frechen 2012)).

Based on a comparison of the cleaning capacity results for microorganisms at the RSF with results from studies using other wastewater treatment techniques (Carraro *et al*. 2000; Hiekel *et al*. 2002), it can be stated that the retention efficiency for microorganisms was on average at the same level as that achieved with mechanical–biological treatment steps. This is confirmed by comparison with results of in-house studies of wastewater treatment plants in the project area (MUNLV 2010). In addition it must be considered that the feed concentration at the Altendorf RSF is on average one logarithmic step lower than in the inflow of wastewater treatment plants; so achieving the same retention efficiency in the RSF is an excellent result. It thus appears that discharge of pathogens in rivers from CSOs can be reduced significantly through RSF operation.

DISCHARGE INTO THE WATERCOURSE

The average monthly runoff of the Altendorfer Bach at the level of the RSF outflow amounts to only 26 L/s. Without the RSF, the average volume of discharge would be at 100 L/s in the case of overflow from the combined sewage system. So, during overflow events, discharge of combined sewage amounts to nearly 80% of the total runoff of the Altendorfer Bach. The hydraulic peak load for the watercourse would also be far greater without the Altendorf RSF, since CSOs with discharge peaks up to 1,000 L/s have been observed. The throttle outflow of the RSF flowing into the Altendorfer Bach typically amounts to only 10.5 L/s. The total volume of discharge and the associated substance loads are greatly reduced through operation of the RSF. Without RSF operation, an average diclofenac concentration of 0.12 μg/L and ibuprofen concentration of 0.40 μg/L would become apparent downstream in the watercourse due to CSOs. In accordance with the European Environmental Quality Standard (EQS) for surface waters, diclofenac, with an EQS of 0.1 μg/L, will be included in a so-called ‘watch list’ of emerging pollutants, and could one day be placed on the priority list (European Commission 2012; European Parliament 2013). As median concentrations for diclofenac and ibuprofen in RSF outflow were below the detection limits, it is presumed that the RSF discharge does not contribute to possible pharmaceutical residues in the receiving watercourse.

Much higher concentrations of the microorganisms considered in the study would occur in the Altendorfer Bach if the RSF was not in service. In this case the concentration values would reach 7.4×10^5 MPN per 100 mL for *E. coli*, 2.9×10^4 PFU per 100 mL for coliphages and 320 *Giardia lamblia* cysts per 100 L. Given that the uptake of a single *Giardia lamblia* cyst is associated with a 2% probability of infection (Augenthaler & Huggenberger 2003), an increased risk of infection arising from CSO events would be expected without the RSF. In view of the reduction efficiency of over 99% recorded in this study, it is evident that infection by parasites in the Altendorfer Bach originating from RSF discharge is very unlikely.

CONCLUSIONS

Wastewater discharges from CSOs substantially contribute to pollution of watercourses with pharmaceutical residues and microorganisms. The present study shows that the substance load of pharmaceuticals and microorganisms in CSOs is significantly reduced by advanced treatment with RSFs; watercourses receiving treated combined sewage are noticeably relieved of pollutant loads and microorganisms. It is expected that increased application of RSFs will help to meet future EQSs for micropollutants and to reduce health risks associated with use of surface water.

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