Chapter 18

Field Study VI: The Effect of Loading Strategies on Removal Efficiencies of a Hybrid Constructed Wetland Treating Mixed Domestic and Agro-Industrial Wastewaters

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Abstract The treatment of domestic wastewater using constructed wetlands (CWs) is common in the Czech Republic; however, the treatment of agro-industrial wastewater is still at the beginning of its application. A hybrid CW consisting of two horizontal filters (HF1 and HF2), one vertical filter (VF), and three stabilization ponds (SP1–SP3) was put into operation in 2012 in Chrámce, Ústí region, Czech Republic. The hybrid system treats mixed household and agro-industrial wastewater mainly from sheep farms and wine and fruit juice production factories. The filters are planted with *Phragmites australis*, *Phalaris arundinacea*, *Iris pseudacorus*, *Iris sibirica*, *Glyceria maxima*, and *Lythrum salicaria*. In a fed-batch operation, the inflow values vary on the basis of wine processing seasons. In high season, it reaches 17,012 mg L^{-1} of chemical oxygen demand (COD), 1806 mg L^{-1} of biochemical oxygen demand (BOD_5), and 43,723 mg L^{-1} of total suspended solids (TSS) on average. Despite such high inlet concentrations, the removal efficiency (RE) for the

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three selected parameters reached up to 99% for all the parameters. In this work, the removal of four pharmaceuticals (diclofenac, ibuprofen, ketoprofen, and naproxen) in the hybrid system was studied as well. The CW performed high RE for all of the four pharmaceuticals when the RE of diclofenac, ibuprofen, ketoprofen, and naproxen was 81.1%, 93.2%, 96.7%, and 78.3%, respectively.

Keywords Agro-industrial wastewater · Hybrid constructed wetlands · Macrophytes · Pharmaceuticals · NSAIDs

18.1 Introduction

Constructed wetlands (CWs) is an efficient technology for treatment of wide range of wastewaters, which has been used for more than 50 years (Vymazal 2011a). Domestic and municipal wastewater is usually treated using CWs (Brix and Arias 2005; Kadlec and Wallace 2009), but other types of wastewater such as agro-industrial wastewater (Cronk 1996; Knight et al. 2000; Sultana et al. 2015) can be effectively treated by CWs as well. Constructed wetlands are also used for treating diffuse agricultural pollution (Kasak et al. 2018; Mendes et al. 2018) and they can be used for pesticide mitigation from non-point sources as well (Vymazal and Březinová 2015). In the Czech Republic, the last reported number of CWs for municipal wastewater treatment was 250 (Vymazal 2011b); however, we estimate that the number has increased up to 400 constructed wetlands recently. Municipal and agro-industrial wastewater might contain many different pharmaceuticals and personal care products (PPCPs) some of which can be considered endocrine disruptors (EDs). Degradation of such compounds in constructed wetlands was also tested in many studies (e.g., Ávila and García 2015; Hakk et al. 2018).

18.1.1 *Hybrid Constructed Wetlands*

Many different types of constructed wetlands such as free water surface CWs, subsurface flow CWs with upward flow, etc., were developed and used during the 50-year history of the CWs application. However, currently the most common ones are wetlands with subsurface horizontal (also called horizontal filters or horizontal flow constructed wetlands—HF CWs) (Vymazal 2011c) and subsurface vertical flow (also called vertical filters or vertical flow constructed wetlands—VF CWs) (Brix and Arias 2005). The difference of VF lies in filtration medium fractions distribution, distribution and drainage pipes placement, and most importantly in the water flow direction. VFs are commonly fed intermittently with large batches of wastewater (Vymazal 2016). The combination of HF and VF in the so-called hybrid systems is currently becoming more and more popular (Vymazal 2013). The main reason for using the hybrid system is to exploit the advantages of each filter in order to fulfill the requirements for the quality of the discharged water and especially to fulfill nitrogen concentration limits (Tunçsiper 2009). HF CWs are anaerobic and provide the necessary conditions to attain denitrification, while the intermittently fed VF CWs are aerobic, thus enabling nitrification (Vymazal and Kröpfelová 2015). The removal of total nitrogen in hybrid CWs may amount to 85% (Kantawanichkul et al. 2001). The hybrid CW Kotenčice, Czech Republic, was built in 2011 in Central Bohemia and consists of various HF beds with the total area of 911 m^2 and VF beds with the total area of 300 m^2 being alternately connected (Hudcová et al. 2013). This hybrid CW has been designed to test the optimum combination of the HF and VF beds. The results from this CW shows that nitrogen was reduced up to 79% in the summer period and up to 62% in the winter period.

18.1.2 CWs for Treatment of Agro-Industrial Wastewaters

Constructed wetlands are used, among others, for treating agricultural wastewaters, especially wastewaters from animal feedlots (Knight et al. 2000) or drainage waters (Xue et al. 1999; Poe et al. 2003). The problem of feedlot wastewater is that the organics content characterized by biochemical oxygen demand (BOD_5) is usually one order of magnitude higher in domestic waters (Cronk 1996). Healy et al. (2007) reported BOD_5 concentrations reaching up to 7130 mg L^{-1} (the average of seven observed CWs treating concentrated animal waste). Dairy, swine, and cattle wastewater respectively are also rich in total suspended solids (TSS). Knight et al. (2000) reports the TSS concentrations reaching up to 1111 mg L^{-1}, 128 mg L^{-1}, and 291 mg L^{-1}, respectively. Total nitrogen concentrations from pig farms, poultry farms, and dairies averaged 407 mg L^{-1}, 89 mg L^{-1}, and 103 mg L^{-1}, respectively. Agricultural wastewater also includes food processing. Such wastewaters can be characterized by high loads of organics. Especially chemical oxygen demand (COD) loads can reach up to 7406 mg L^{-1} during the wine making season; however, even during the rest of the year it can be as high as 1721 mg L^{-1} (Grismer et al. 2003). Some authors report even higher concentrations—up to 15,400 mg L^{-1} (Grismer et al. 2003) or 25,400 mg L^{-1} (Šereš et al. 2017)—in winemaking facilities. The results of the review done by Sultana et al. (2015) show that the use of hybrid systems is desirable for treating such waters because VF CWs are able to treat extremely high loads of pollution.

18.1.3 Emerging Pollutants and CWs

Due to the increased consumption of pharmaceutical products, there is a rising concern regarding the harmful effects of such substances on the environment (Reif et al. 2008). The concerns are also connected with the overuse of veterinary pharmaceuticals and other active substances belonging to the so-called pharmaceutical and personal care products (PPCPs). The persistence of pharmaceutical residues and their metabolites in the environment is one of the most significant problems that can lead to a cumulative effect on the nontarget organisms in the aquatic environment (Han et al. 2010). One of the most frequently used groups of drugs that can be often found in water environments are so-called nonsteroidal anti-inflammatory drugs (NSAIDs) (Wang et al. 2011). Some studies already examined the elimination capacity of constructed wetlands for most common NSAIDs—ibuprofen, diclofenac, naproxen, and ketoprofen (Hijosa-Valsero et al. 2010). In the groundwater analysis performed in the Czech Republic, these four substances were the most abundant pharmaceuticals in the collected samples (Helenkár et al. 2010; Kotowska et al. 2014). Marsik et al. (2017) confirm significant concentrations of the NSAIDs in the Elbe river and its tributaries. In this work, the efficiency of the hybrid constructed wetland Chrámce, Ústí region, in the removal of basic chemical parameters (COD, BOD_5, TSS, N, and P) and NSAIDs from mixed domestic and agro-industrial wastewater was evaluated.

18.2 Materials and Methods

18.2.1 Experimental Plant Description

This study was performed in a hybrid constructed wetland Chrámce, Ústí region, Czech Republic. This CW receives sewage water from a residential building (negligible part of the total flow); wastewaters from fruit, fruit juice, and wine processing (the total area of orchards and vineyards is 195 ha and 1.9 ha, respectively); and wastewater from a sheep farm (150 sheep in total). The total calculated capacity of the CW is 50 population equivalent (PE) and the average calculated flow through the system is 0.09 L s^{-1}, which represents 7.8 m^3 d^{-1}. The calculated theoretical retention time is 10.5 days and the tracers tests that were performed according to Kadlec and Wallace (2009) using a KBr indicator confirmed the mean retention time to be 10 days. The CW has been operating since 2012. The inflow is not continuous, but the CW is batch fed by vacuum trucks.

The system consists of eight treatment stages (see Fig. 18.1): a reservoir for wastewater (3 × 10 m^3), a three-chamber septic tank (14.1 m^3), a series of three wetland filters—horizontal filter 1 (HF1), vertical filter (VF), and horizontal filter 2 (HF2). Tertiary treatment of wastewater is performed in three small stabilization ponds with littoral zones connected with a shallow meandering stream (SP1–SP3). The treated water is discharged into the existing pond. More detailed characteristics are summarized in Table 18.1.

Filters are planted with reed canary grass (*Phalaris arundinacea*) and common reed (*Phragmites australis*). Meandering streams and littoral zones are planted with sweet manna grass (*Glyceria maxima*), purple loosestrife (*Lythrum salicaria*), yellow iris (*Iris pseudacorus*), Siberian iris (*Iris sibirica*), sweet flag (*Acorus calamus*), beaked sedge (*Carex rostrata*), and broadleaf cattail (*Typha latifolia*).

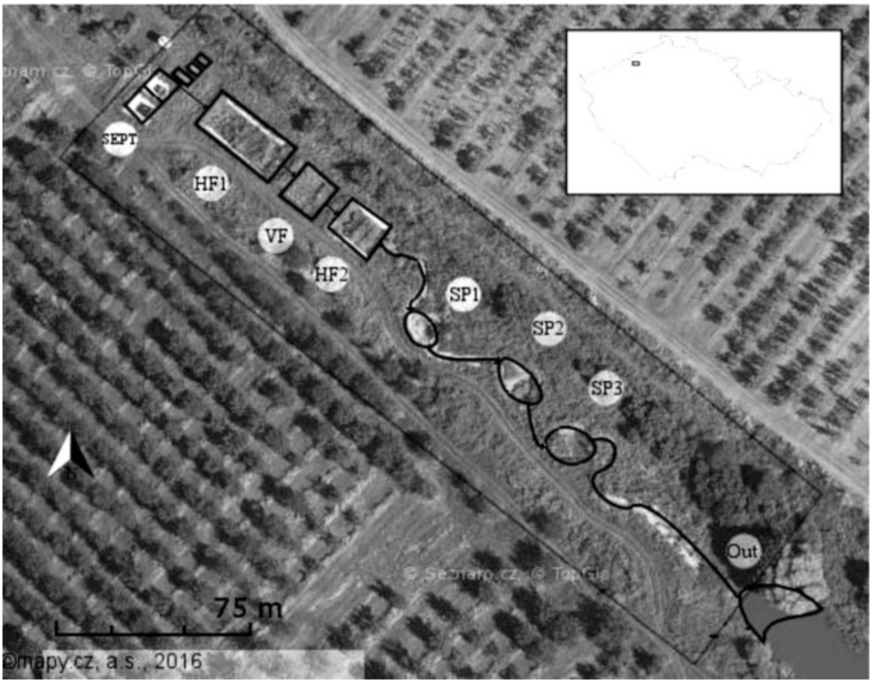

Fig. 18.1 Map and layout of the hybrid constructed wetland in Chrámce

Table 18.1 Major design parameters of constructed wetland

Parameter	Effective area (m^2)	Depth (m)	Filtration material*	Volume (m^3)	HLR (cm/d)
HF1	133	0.7	a, b, c	97.2	3.8
VF	49	1.3	a, b, c, d	50.4	10.2
HF2	70	0.9	a, b, c, e	48.6	7.1

Adapted from Šereš et al. (2017)

*(a) Washed gravel 2–4 mm (protective material); (b) washed gravel 4–8 mm, porosity: 0.45, conductivity 16 cm/s; (c) washed gravel 8–16 mm, porosity: 0.44, conductivity 94 cm/s; (d) washed gravel 32–64 mm, porosity: 0.46, conductivity 350 cm/s; (e) slag 8–16 mm, porosity: 0.51, conductivity 47 cm/s

18.2.2 *CW Performance Evaluation*

Sampling is performed at ten sampling points—two samples in septic tank (1st and 3rd chamber), two samples in HF1 (middle and outlet), other six samples are taken at the outflow from the outlets at each resting stage (VF, HF2, SP1–SP3, and final discharge point). For the treatment efficiency evaluation, the following parameters regulated by Czech laws were monitored: COD, BOD$_5$, TP, TN, NH$_4^+$, TSS. The samples were analyzed according to the standard methods (APHA 1998). Detailed sampling was performed on a weekly basis during the periods September–October 2013 (n = 10 samples) during the high production season. Sampling then continued during the period April–October 2014 (n = 17 samples) and February–October 2015 (n = 17 samples).

18.2.3 NSAIDs Removal Evaluation

For the evaluation of NSAIDs removal in the constructed wetland, an in vitro analytical method was developed and an analysis of these antiphlogistics in a real system was performed thereafter. Metabolic potential of cell cultures of *Melilotus officinalis* and *Rheum palmatum* in vitro was verified in *Phragmites australis* cells, tissues, and whole plants, and finally tested in the CW. Some measurements of NSAIDs concentrations in this CW were reported by Marsik et al. (2015).

The sampling and preparation procedure of the samples was done according to Marsik et al. (2015). The samples were collected in the period June–September 2014 and February–June 2015 (15 samples in total) from the same sampling points as mentioned above. Wastewater samples were analyzed using UPLC-MS/MS. For the analysis, the samples were first acidified with acetic acid at pH 2.5 and then they were filtered through a nylon membrane filter (0.45 μm). For quantitative analysis, system Q-Trap 4000 (AB Sciex, USA) was used. The sample (25 mL) was applied on solid phase extraction columns (Strata C8 (55 μm, 70 A), 500 mg/3 mL, Phenomenex, USA) preconditioned with 5 mL of methanol and 10 mL of deionized water. The columns were then washed with acidified water (acetic acid, pH 2) and eluted with 5 mL of methanol. The samples were then evaporated under stream of nitrogen to dryness and stored at −80 °C. Before the analysis, the samples were dissolved in 1 mL of methanol and then dissolved 100× for analysis.

18.3 Results and Discussion

18.3.1 Performance of the Hybrid Constructed Wetland

In 2013 the monitoring was focused mainly on the period of wine harvesting and processing. In Table 18.2, mean concentrations of COD, BOD_5, and TSS are summarized. The results show that the discharge limits (see in Table 18.3) were fulfilled at the outflow from the CW filters; however, the quality of the water decreased in the stabilization ponds when the COD increased from 40 mg L^{-1} at the outlet from HF2 to 181 mg L^{-1} at the outlet from SP3. As for the TSS, the C^{out} increased from average 3 mg L^{-1} at the outlet from HF2 to 1047 mg L^{-1}at the outlet from SP3. This increase was caused mainly by the growth of algae and decay of plants in the stabilization ponds (see Fig. 18.2). Shepherd et al. (2001) reported very

Table 18.2 CW performance after 1 year of operation (September–November 2013) in high production season

	CW filters[a]			Stabilization ponds[b]	
Parameter	C^{in} (mg/L)	C^{out} (mg/L)	RE (%)	C^{out} (mg/L)	RE^{c} (%)
COD	17,012 ± 11,862	40 ± 22	96.3 ± 5	181 ± 135	83.6 ± 22.3
BOD_5	1806 ± 1179	1 ± 0.5	99.7 ± 0.3	14 ± 15	98.5 ± 1.8
TSS	43,723 ± 30,797	3 ± 1	99.6 ± 0.5	1047 ± 1121	20.1 ± 100

[a]CW filters (HF1-VF-HF2); [b]Stabilization ponds (SP1–SP3); [c]Total value of RE after flowing through SP

Table 18.3 Discharge limits set for the constructed wetland Chrámce

Parameter	Mean limit (mg/L)	Max limit (mg/L)	Removal limit (%)
COD	110	170	70–75%
BOD_5	30	50	80–85%
TSS	40	60	90–95%

Fig. 18.2 Algae bloom observed stabilization ponds (2013)

high COD concentrations, rising up to 45,500 mg L^{-1}, in winery wastewaters and the CW treating such wastewaters still provided great removal efficiency (RE), up to 99%.

The quality of agro-industrial wastewaters varies during the year. Such variations can be observed even here if we compare the inlet concentrations of all the parameters in the high season of 2013 (Table 18.2) and average of the off-seasons in years

Table 18.4 CW performance after 2 and 3 years of operation (2014–2015)

	Root filters[a]			Stabilization ponds[b]	
Parameter	C^{in} (mg/L)	C^{out} (mg/L)	RE (%)	C^{out} (mg/L)	RE^{c} (%)
COD	477 ± 442	65 ± 101	73 ± 32	42 ± 77	77 ± 42
BOD_5	226 ± 228	14 ± 23	86 ± 19	3 ± 1	89 ± 21
TSS	240 ± 416	3 ± 1	76 ± 47	5 ± 6	76 ± 46
N_{total}	33 ± 21	4 ± 4	86 ± 10	3 ± 3	87 ± 15
P_{total}	10 ± 14	2 ± 7	75 ± 44	0.1 ± 0.1	97 ± 3
Ammonia	25 ± 17	0.1 ± 0.1	98 ± 4	0.1 ± 0.1	98 ± 4

[a] CW filters (HF1-VF-HF2); [b] Stabilization ponds (SP1–SP3); [c] Total value of RE after flowing through SP

2014 and 2015 (Table 18.4). We can observe for COD 35× higher C^{in}, for BOD_5 8× higher C^{in}, and for TSS even 200× higher C^{in}.

The observation shows that during the study period 2014 and 2015 the average elimination efficiency of the hybrid systems fulfilled the legislative requirements for most of the observed parameters. The results are also comparable with other hybrid constructed wetlands (Vymazal 2010, 2013) and previous studies done on this CW (Šereš et al. 2017). We observed relatively high inlet concentrations of COD and BOD_5; however, the outflow concentrations were very low already at the outflow from the CW filters (65 and 15 mg L^{-1} respectively) and decreased even more at the outflow from the stabilization ponds (42 and 3 mg L^{-1} respectively). In comparison with the year 2013, we can observe a decrease in the removal efficiency for COD, BOD_5, and TSS, yet we can observe lower inlet concentrations. As some authors explains, the percentage efficiency depends on the inflow concentration, and, thus, lower C^{in} can cause lower RE (Ghermandi et al. 2007). We can also observe that COD and BOD were reduced from 52 and 56% reduced in HF1 (see Fig. 18.3). The only parameter that in average outlet concentrations did not fulfill the legislative limit was the TSS removal efficiency. Mean C^{in} of TSS were reduced by only 76% in both stages (wetland filters and stabilization ponds). Nevertheless, the outflow concentrations were far below the discharge limits (3–5 mg L^{-1}). The higher outlet concentrations of the suspended solids might be caused by the presence of planktonic algae in the stabilization ponds (Hijosa-Valsero et al. 2012) or by filtration material decomposition (Ghermandi et al. 2007).

As for the nitrogen removal, we can observe that total nitrogen was eliminated by 87%. From Fig. 18.3, we can see that almost 65% of N_{total} that was formed mainly by ammonia N was eliminated in the first horizontal filter and almost 86% of N_{total} was eliminated after the flow through the whole hybrid system. The stabilization ponds made an insignificant contribution to the nitrogen removal. Very high nitrogen removal is common (Vymazal 2013) compared to single HF or VF CWs. Vymazal and Kröpfelová (2015) report similar overall RE of N_{total} (79.9%) in their three-stage CW (VF-VF-HF).

Ammonia N was removed from wastewater mostly in HF1 (61%) and the overall removal reached 98%. Although hybrid CWs are efficient in the removal of total

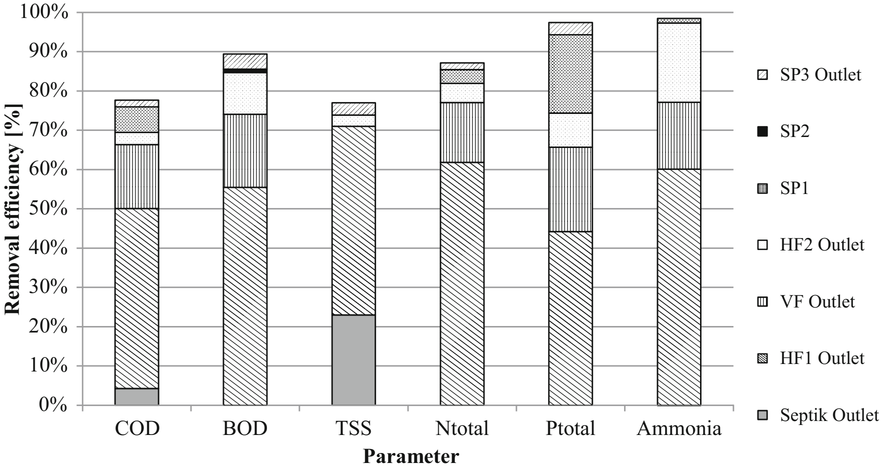

Fig. 18.3 Mean removal efficiencies for the monitored chemical parameters in each stage of the CW

nitrogen, the elimination of ammonia is usually promoted in aerobic conditions of vertical filters. Such high ammonia removal might be explained by higher exploitation of anammox processes in the CW filter environment (Tao et al. 2012). For removal of phosphorus in CWs, mostly physio-chemical processes are applied (Abe et al. 2010), but macrophytes could also be important in phosphorus storage in the short term (Wang et al. 2018). Phosphorus removal of instream CWs usually reaches up to 62% (Kasak et al. 2018); however, Zhu et al. (2012) report the P_{total} removal reaching up to 95% from the livestock wastewater containing 17.99 mg L^{-1}. In our study, we observed the P removal reaching 97% with the average inlet concentrations of 10 mg L^{-1}.

18.3.2 Elimination of NSAIDs in Hybrid CW Chrámce

In this study, the elimination of four selected NSAIDs in hybrid intermittently fed CW was evaluated. In Fig. 18.4, the concentrations observed in each treatment step of the CW are summarized for each NSAID. These results show that ibuprofen, diclofenac, ketoprofen, and naproxen are efficiently degraded and accumulated after flowing through the whole wetland system and all the analyzed pharmaceuticals at the outflow from the stabilization ponds were on average close to the detection limits. The average removal of diclofenac was 81% (see Fig. 18.4) that corresponds with the findings of Hijosa-Valsero et al. (2010) who report the average removal of diclofenac in wetland treatment systems 65–87% while conventional activated sludge wastewater treatment plant removes diclofenac only by 44% (Ávila and García 2015). The average inlet concentration of diclofenac in our study was 389.6 ng L^{-1} and the C^{out} was in average 21.3 ng L^{-1}. Vystavna et al. (2017)

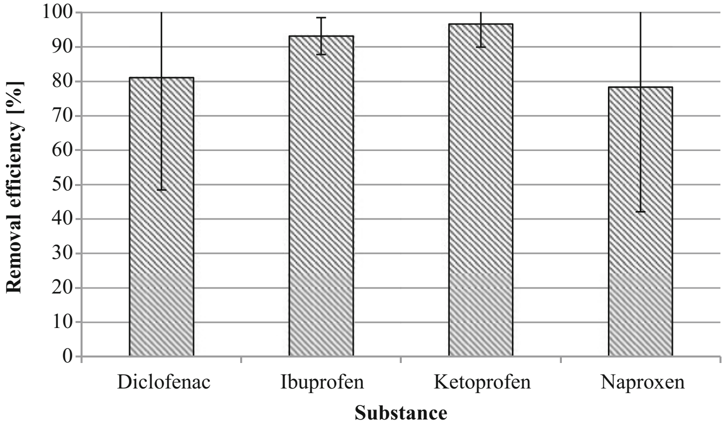

Fig. 18.4 Removal efficiency of four selected NSAIDs in hybrid CW

reported the inlet concentrations of diclofenac to the four-stage CW treating hospital wastewaters to be 1862 ng L^{-1}. Removal efficiency of this CW for diclofenac was higher than 50%. The mean inlet concentration 361.4 ng L^{-1} of ibuprofen was in our system removed by 93.2% from which 40% corresponds to the vertical filter. Ávila et al. (2015) report the removal of ibuprofen reaching 100% also mainly in the VF. Matamoros and Salvadó (2012) report the removal efficiency of ibuprofen, ketoprofen, and naproxen to be 93%, 98%, and 88%, respectively, which is similar to our study. In our study we also observed the highest RE for ketoprofen (97%) and the lowest for naproxen (78%). In the work of Vystavna et al. (2017), the highest removal efficiency was reached for naproxen (>80%) and only above 50% for other observed substances, which conflicted with our findings. However, in their work, the inlet concentration of naproxen was only 0.7 ng L^{-1}.

From Fig. 18.5, we can observe that concentrations of all four substances were reduced mainly after the VF, which is in line with the observations of Ávila and García (2015) who propose that aerobic pathways are more efficient in degradation of PPCPs. Nevertheless, the stabilization ponds played a major role in reaching the RE reported above. The removal of the selected pharmaceuticals was probably performed by combination of the processes. It is expected that some portion of pharmaceuticals might be assimilated by plants; however, Zhang et al. (2013) reported that the naproxen uptake by the wetland plants reached only 4% of the total substance mass; therefore, other processes such as biodegradation must be used here. Also Matamoros et al. (2008) suggest that ibuprofen is removed by degradation rather than by sorption. Biodegradability of all the observed substances is proven also by Yu et al. (2006). In the graph below, we can observe that the concentration of all the substances slightly increased in the first horizontal filter. As we can observe, the removal of NSAIDs did not occur in HF1; therefore, concentrating of substances in this filter might have taken place in the long term and the biodegradation occurred after the VF.

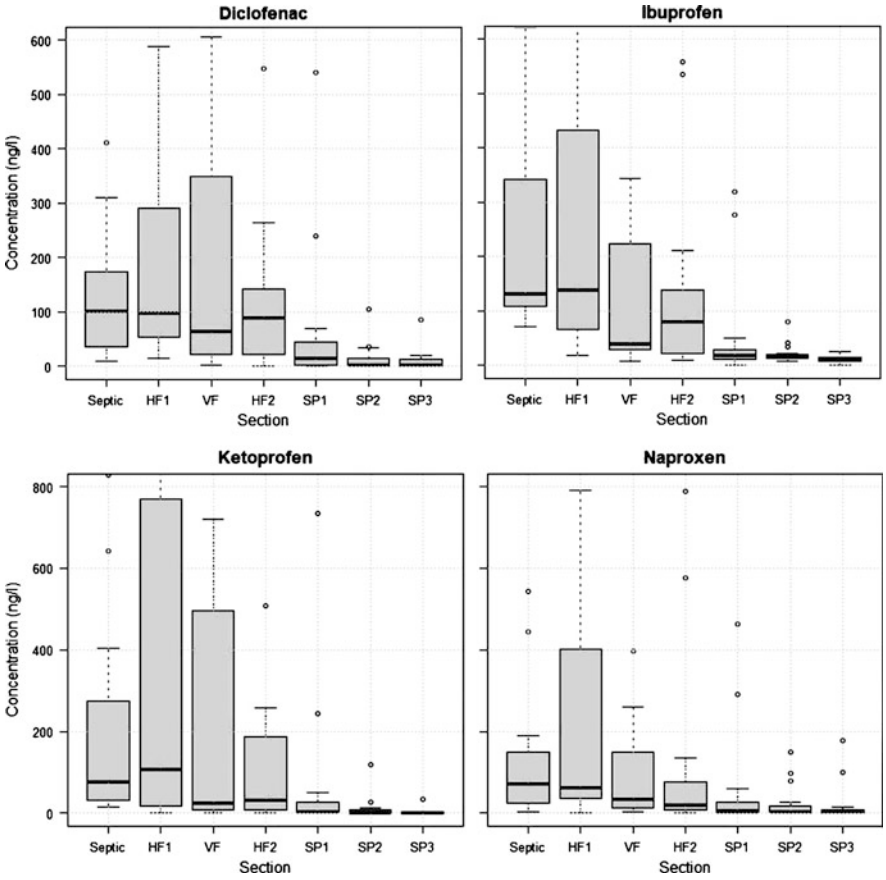

Fig. 18.5 Boxplot diagram of four NSAIDs (diclofenac, ibuprofen, ketoprofen, naproxen) in each CW section

18.4 Conclusions

The hybrid constructed wetland in Chrámce treating mixed household and agro-industrial wastewaters coming mainly from sheep farm and wine and juice production facility performed high treatment efficiencies. The main conclusions are:

- The removal efficiency for COD, BOD_5, and TSS in high wine processing season is 96.3%, 99.7%, and 99.6, respectively, in wetland filters but overall removal efficiency is only 83.6%, 98.5%, and 20.1%, respectively, due to algae growth and decay of plants in the stabilization ponds.
- The average RE of the system in seasons 2014–2015 of COD, $BOD_{5,}$ and TSS was 77%, 89%, and 76% and the mean outlet concentrations easily fulfilled the discharge limits.

- Nitrogen was effectively removed in the hybrid CW because of the combination of three subsurface wetlands with various feeding strategy. The removal of N_{total} reached 87%. The ammonia removal reached 97% and more work should be done in the future to discover if anammox might take place in the ammonia removal.
- Monitoring four widely used human pharmaceuticals from the group of nonsteroidal anti-inflammatory drugs showed very high removal efficiency of the hybrid CW. The average removal efficiency for diclofenac, ibuprofen, ketoprofen, and naproxen, was 81.1%, 93.2%, 96.7%, and 78.3%, respectively.

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