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Pilot-scale trials of stone wool substrate media filters as a potential technology for tertiary domestic wastewater treatment

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Abstract

The aim of the present study was to investigate the application of trickling filters with stone wool medium for the tertiary on-site wastewater treatment. The experiment with three filters (0.5 m in diameter and with 1.0 m, 1.5 m and 2.0 m medium height) feeding three different flow rates (0.25 m³/d, 0.5 m³/d and 0.75 m³/d) of treated wastewater was carried out over a period of 430 days and at influent wastewater temperature of 1.9–23.2 °C. Depending on a filter height and a flow rate, mean 7-day biochemical oxygen demand (BOD₇) and mean suspended solids (SS) removal efficiencies fluctuated from 60 to 85 % and from 50 to 70 % respectively. The removal efficiencies increased when increasing SS and BOD₇ influent concentrations, however no dependence on volumetric loading (up to 242 g SS/m³/d and 228 g BOD₇/m³/d) was observed. The main nitrogen transformation/removal processes were nitrification and the removal of organic nitrogen (N-org). Due to low temperature, full nitrification was first achieved on day 177 and from that point was dependent on volumetric ammonium loading, but did not depend on volumetric organic loading in the range of 5–228 g BOD₇/m³/d and wastewater temperature in the range of 5.6–23.2 °C. Mean N-org removal efficiency ranged from approximately 30 to 70 % and was positively dependent on N-org influent concentration. Total phosphorus removal was unstable in all the filters with its mean values of –18–13 % depending on a filter height and a flow rate.

Keywords: Trickling filter; tertiary treatment; filter medium; stone wool; wastewater.

1. Introduction

There is a large number of households in many countries around the world that are not connected to a centralized sewerage system. This problem is particularly relevant in developing countries [1], though in developed countries decentralized wastewater management also plays an important role. For example, in 2007 20% of households in the USA were served by septic systems [2].

Due to various reasons (such as considerable fluctuations of domestic wastewater composition, inadequate maintenance or insufficient instruction of the owners), the performance of many on-site wastewater treatment systems is not satisfactory [3], thus significantly contributing to both surface and groundwater contamination. For example, U.S. EPA states that failing septic systems are the third source of groundwater contamination in the USA [4]. 61% of nitrogen released to the environment with wastewater is discharged through decentralized wastewater treatment systems [5]. In Sweden only 15% of the population is connected to on-site wastewater treatment systems, though the total discharge of phosphorus from these systems exceeds the total discharge from all municipal treatment plants [6].

Tertiary filtration could be one of the solutions to improve decentralized effluent wastewater quality. Apart from traditional sand filters, some other tertiary filters such as recycled glass and date-palm fibres filters were also investigated [7–8].

A trickling filter with stone wool medium proved to be an effective wastewater treatment method for decentralized areas [9–10] as it successfully removed organic matter, suspended solids (SS), ammonium and organic nitrogen (N-org) from wastewater. The objective of the study reported herein was to investigate the application of such filters for the tertiary on-site wastewater treatment. The removal of organic matter, SS, nitrogen and phosphorus compounds was evaluated under various design, operational and water quality conditions.

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2. Materials and methods

2.1. Experimental set-up and procedure

The pilot-scale experimental set-up comprised of the influent wastewater feed tank, three filters run in parallel, three pumps and the effluent tank. The system was installed outside, in Wastewater Treatment Plant Laboratory of the Certification Centre of Building Products (Maišiagala, Lithuania), and operated at an influent wastewater temperature of 1.9–23.2 °C. The experiment was carried out over a period of 430 days. The schematic diagram of the experimental set-up is presented in Fig. 1a.

Influent wastewater from the feed tank flowed by gravity to three filters and was introduced at the top of each filter using manually rotated six arm stainless steel distributor.

Each filter had a cover, was cylindrical in shape with a diameter of 0.5 m and an overall height of 1.55 m (labelled as 1F), 2.05 m (labelled as 2F) and 2.55 m (labelled as 3F) (Fig. 1b). Stone wool substrate *Growcube* (Grodan, The Netherlands) for container plant cultivation supplied in 2×2×2 cm cubes was used as the filter medium. Detailed information on the medium used can be found in Kirjanova et al. (2011) [9]. The substrate was not submerged and therefore acted as a trickling filter, with the exception of not providing ventilation/aeration to the filters. Even though the aeration was not provided, aerobic conditions prevailed in the filters which was proved by occasional dissolved oxygen measurements.

The effluent was pumped into the three chamber effluent tank where effluent samples were taken.

The first 108 days of the experiment were considered as a start-up period for the biofilm formation. The maturation of the biofilm was accomplished naturally, by feeding influent wastewater to the filters. Such a long inoculation period was determined by low influent wastewater temperature during the set-up period, which was in the range of 3.9–8.1 °C.

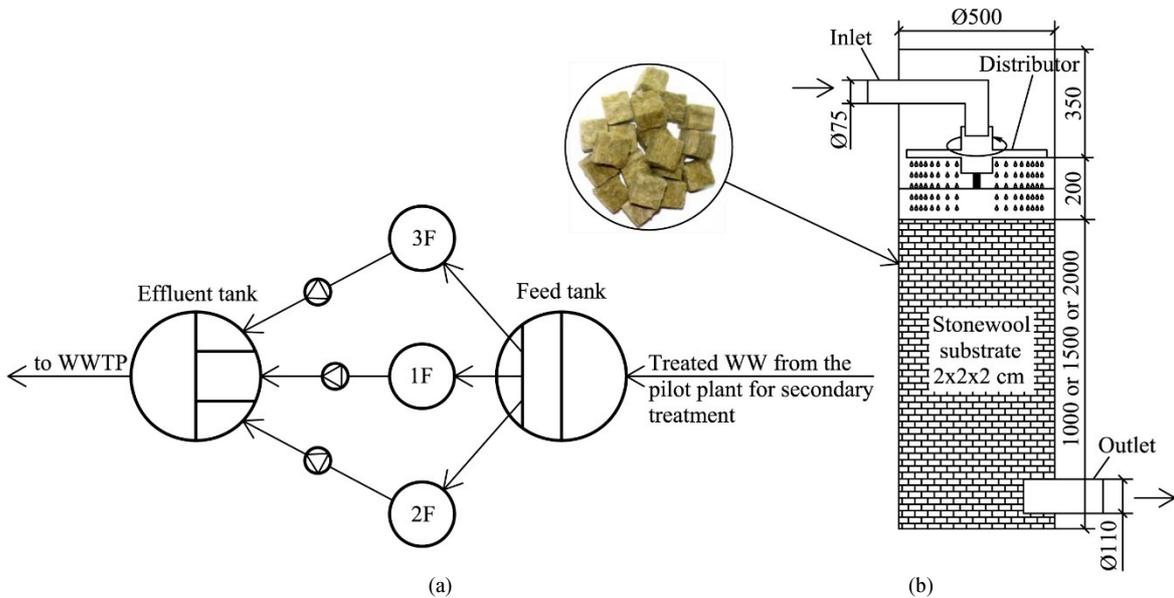


Fig. 1. The schematic diagram of the experimental set-up (a) and the filters (b)

In order to analyze the influence of a flow rate and thus hydraulic and volumetric loadings on the performance of the filters, the filters were fed with three different flow rates (Q) of 0.25 m³/d, 0.50 m³/d and 0.75 m³/d which represented the daily wastewater amount generated by 2, 4 and 6 persons and corresponded to 1.3, 2.6 and 3.8 m³/m²/d hydraulic loading respectively.

Wastewater to the experimental set-up was fed simulating the conditions of daily water use in a household, according to LST EN 12566-3+A1: 2009 [11]. The daily flow pattern is presented in Table 1.

Table 1. Daily flow pattern in a single household

Period, h	Percentage of daily volume, %
3	30
3	15
6	0
2	40
3	15
7	0

2.2. Feed wastewater

Wastewater treated in the pilot plant for secondary treatment described in Kirjanova *et al.* (2011) [9] was used as the influent to the experimental set-up. The composition of the treated wastewater fluctuated throughout the experimental period. The characteristics of the wastewater fed to the filters are presented in Table 2.

Table 2. Chemical composition of the wastewater fed to the filters

Parameter	Range mg/l	Mean	Median	St. dev.	<i>n</i> ^a
SS	3–128	22	12	24	41
BOD ₇	8–97	26	21	17	41
COD	31–246	87	88	37	42
NH ₄ -N	2–71	21	18	18	37
NO ₂ -N	0.7–7.4	2.3	1.7	1.8	33
NO ₃ -N	0–40	13	13	9	42
N-org	2.4–15	7.4	6.4	3.2	34
N-total	17–79	45	44	17	37
o-PO ₄ -P	2.2–8.5	4.7	4.5	1.6	42
P-org + p-PO ₄ -P	0.1–1.7	0.6	0.6	0.3	38
P-total	2.8–8.8	5.3	5.1	1.6	42
pH	6.90–8.64	7.67	7.58	0.44	42

Note: ^a*n* – number of samples.

2.3. Analytical methods

Starting from 109 day of the experiment, influent and effluent samples of the filters were taken once a week. They were either grab or 24-h composite. Grab samples were taken manually, whereas 24-h composite samples were taken with the portable water samplers *Buhler 1000* (Hach Lange, Germany). Influent samples were taken in the feed tank, while effluent samples – in the corresponding chamber of the effluent tank. The samples were analyzed for pH, SS, 7-day biochemical oxygen demand (BOD₇), chemical oxygen demand (COD), ammonium nitrogen (NH₄-N), nitrite nitrogen (NO₂-N), nitrate nitrogen (NO₃-N), total Kjeldahl nitrogen (TKN), orthophosphate phosphorus (o-PO₄-P) and total phosphorus (P-total) concentrations according to standard methods. N-org concentration was determined by subtracting NH₄-N concentration from TKN concentration. N-total concentration was determined by summing TKN, NO₂-N and NO₃-N concentrations. The sum of the concentrations of P-org and polyphosphate phosphorus (p-PO₄-P) was determined by subtracting o-PO₄-P concentration from P-total concentration. Wastewater temperature (T) was measured in the feed tank before the filters with *SevenGo pro SG6* dissolved oxygen meter (Mettler Toledo, Switzerland). pH was measured with *MultiLine P4/SET* measuring device (WTW, Germany).

2.4. Statistical analysis

Experimental results were statistically evaluated using *SPSS Statistics 20* package (IBM Corporation, USA). If the regression between influent concentrations, volumetric loadings and removal efficiencies was statistically significant (F-test, $p < 0.05$), the correlations between these variables were defined by either Pearson (*r*) or Spearman (*r_s*) correlation coefficients depending on the distribution and homoscedasticity of the residuals.

3. Results and discussion

3.1. Organic matter and SS removal

On day 109, when the first samples were taken, organic matter removal was high and it was assumed that steady state conditions were reached. As can be seen from Table 3, mean BOD₇ removal efficiency fluctuated from 60 to 85% throughout the experimental period, while mean COD removal efficiency was lower – in the range of 40–60%. Mean SS removal efficiency in all the filters ranged from approximately 50 to 70%. At a flow rate of 0.50 m³/d all the filters experienced a slight deterioration in their performance which likely could be caused by decreased influent concentrations at this flow rate. Dependence of BOD₇ and SS removal efficiency on the correspondent influent concentrations is presented in Figure 2. As can be seen, the higher the concentration, the better the removal. Moreover, the removal was also dependent on the filter height: the highest – 2 m height 3F filter – performed better than 1.5 m height 2F filter, while the latter operated better than the smallest – 1 m height 1F filter. On the other hand, no dependence of removal efficiency on volumetric

loading (up to 242 g SS/m³/d, 228 g BOD₇/m³/d and 586 g COD/m³/d) was observed. As can be seen from Table 3, comparing to 1.3 m³/m²/d hydraulic loading (0.25 m³/d flow rate), increased hydraulic loading (3.8 m³/m²/d or 0.75 m³/d flow rate) slightly improved SS removal, but reduced organic matter removal efficiency.

Table 3. Organic matter and SS removal as a function of design and operational conditions

Filter	Flow rate, m ³ /d	Hydraulic loading, m ³ /m ² /d	SS			BOD ₇			COD		
			C _{in} ^a , mg/l	C _{out} ^b , mg/l	E ^c , %	C _{in} , mg/l	C _{out} , mg/l	E, %	C _{in} , mg/l	C _{out} , mg/l	E, %
1F	0.25	1.3	25±34	6.9±9.5	55±22	23±20	3.8±2.4	80±14	87±46	37±15	54±16
	0.50	2.6	15±10	7.1±4.9	51±20	20±11	5.8±1.3	61±24	73±18	41±11	41±23
	0.75	3.8	24±16	9.8±10	56±28	36±15	8.5±4.8	75±12	109±29	53±15	50±11
2F	0.25	1.3	25±34	4.4±2.1	62±21	23±20	3.6±2.4	82±12	87±46	36±16	58±8
	0.50	2.6	15±10	8.6±11	50±28	20±11	5.3±1.3	64±25	73±18	40±9	42±17
	0.75	3.8	24±16	5.9±3.4	69±20	36±15	6.4±2.3	79±9	109±29	45±8	57±9
3F	0.25	1.3	25±34	3.9±1.4	64±22	23±20	3.2±2.2	84±11	87±46	33±12	59±11
	0.50	2.6	15±10	5.9±3.3	58±17	20±11	5.1±1.8	63±30	73±18	36±8	49±18
	0.75	3.8	24±16	5.9±4.1	69±21	36±15	4.9±1.9	84±8	109±29	42±11	60±11

Notes: ^aC_{in} – influent concentration;
^bC_{out} – effluent concentration;
^cE – removal efficiency.

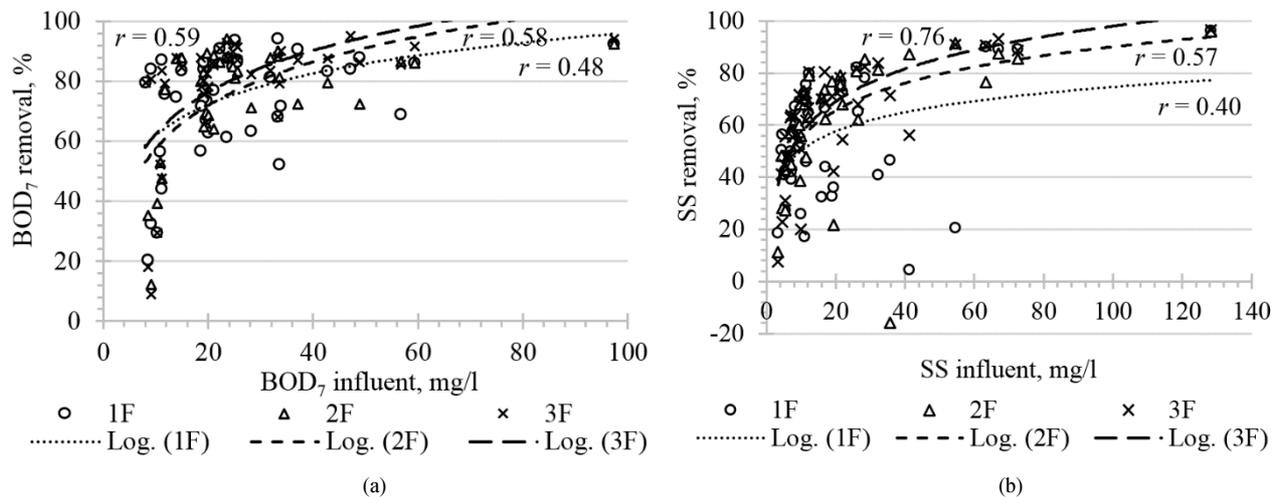


Fig. 2. BOD₇ (a) and SS (b) removal dependence on influent concentration

3.2. Nitrogen compounds removal

Contrary to organic matter removal, it took 177 days to reach steady ammonium removal. On the first day of taking samples, i.e. 109, no ammonium removal was observed. Later on NH₄-N concentrations in the filters effluent started to decrease, while NO₃-N concentrations started to increase indicating gradual inoculation of nitrifying bacteria (Fig. 3). Complete NH₄-N removal was first reached on day 177, when it was higher than 99% in all the filters. Such a long set-up period for nitrification could be related to the low influent wastewater temperature (which was only 5.6 °C on day 177) as nitrifying bacteria are known to be sensitive to low temperature [12].

NH₄-N, as well as N-org and N-total removal characteristics are presented in Table 4. NH₄-N and N-total are calculated excluding maturation period for nitrifying bacteria, i.e. first 176 days of the experiment. As can be seen from Table 4, NH₄-N removal efficiency decreased when increasing a flow rate and herewith NH₄-N influent concentration. Strong negative linear correlation between NH₄-N influent concentration and its removal efficiency is depicted in Fig. 4a. Moreover, the removal was also dependent on the filter height: the higher the filter, the better the removal. This can be explained by nitrification dependence on volumetric ammonium loading (Fig. 4b).

During the experiment, BOD₇ and COD concentrations in the wastewater fed to the filters almost always were higher than 12 and 30 mg/l respectively which are necessary for tertiary nitrification in trickling filters [13–14]. On the other hand, mean volumetric organic loading in the filters ranged from 58 g BOD₅/m³/d in 1F filter to 29 g BOD₅/m³/d in 3F filter. These values are lower than applied to trickling filters for combined organic matter removal and nitrification [15]. Therefore, the design of the tested filters was somewhat between trickling filters for combined organic matter and ammonium removal and trickling filters for tertiary nitrification. Based on multiple linear regression analysis, it was

concluded that NH₄-N removal was mostly dependent on volumetric ammonium loading, while volumetric organic loading in the range of 5–228 g BOD₇/m³/d did not have an impact on NH₄-N removal. Therefore, the tested filters acted as tertiary nitrification trickling filters, despite the fact that organic matter concentration in influent wastewater was higher than it supposed to be for this type of trickling filters. This can be explained by stone wool fibrous structure and its extremely high specific surface area compared to stone or plastic trickling filter packings. Contrary to conventional trickling filters where the top of the packing is populated with more competitive heterotrophic bacteria and autotrophic nitrifying bacteria occupy the lower part of the filter [16], here due to the high specific surface area heterotrophic and nitrifying bacteria can coexist in the whole volume of the filter.

Once inoculation of nitrifying bacteria was done, temperature in the range of 5.6–23.2 °C also did not influence NH₄-N removal which is inconsistent with the data presented in the literature [13].

All in all, in order to achieve 80% and 90% NH₄-N removal efficiency, volumetric ammonium loading should not exceed 55 g NH₄-N/m³/d and 24 g NH₄-N/m³/d respectively.

Prior to 240 day of the experiment, the amount of removed NH₄-N was similar to or lower than the amount of NO₂-N + NO₃-N produced. Increase in the production of NO₂-N and NO₃-N can be attributed to ammonification and hydrolysis processes during which N-org was converted to NH₄-N and subsequent nitrification of the latter. Starting from 240 day of the experiment, the amount of removed NH₄-N was higher than the amount of NO₂-N + NO₃-N produced which theoretically can be explained by either denitrification, volatilisation, anammox process, metabolic uptake by microorganisms in the biofilm or their combination. Denitrification though is not expected to take place in the filters tested as there was not enough organic matter for denitrifying bacteria. Volatilisation also did not seem to be an issue as pH shifts did not follow any pattern. On the other hand, Lydmark *et al.* (2006) [17] found a small number of presumably anaerobic anammox bacteria in the lower part of a full-scale well-aerated nitrifying trickling filter. Though not numerous and with an unknown activity, the detection of these bacteria exhibits the complexity of biofilm systems and stresses the need for their further detailed investigation.

As can be seen from Table 4, mean N-org removal efficiency in all the filters ranged from approximately 30 to 70 %. The removal of N-org in the filters were associated with ammonification and hydrolysis of both soluble N-org and particulate N-org that was mechanically retained in the filter media. N-org removal in the filters was dependent on its influent concentration: the higher the N-org concentration, the better the removal ($r_s = 0.48$). Furthermore, the removal was also dependent on the filter height, though no dependence on volumetric N-org loading (up to 59 g N-org/m³/d) was observed. Finally, comparing to 1.3 m³/m²/d hydraulic loading (0.25 m³/d flow rate), increased hydraulic loading (3.8 m³/m²/d or 0.75 m³/d flow rate) reduced N-org removal efficiency, even though N-org influent concentration was higher then.

N-total removal in the filters was not high with mean efficiency in the range of 10–30%. As aerobic conditions in the filters prevailed, the main nitrogen transformation/removal processes were nitrification and the removal of N-org. Other processes such as denitrification and volatilisation are not expected to take place in the filters due to inadequate conditions. Some anammox process could theoretically take place in anaerobic pockets, presumably in the lower part of the filters.

Table 4. Nitrogen compounds removal as a function of design and operational conditions

Filter	Flow rate, m ³ /d	Hydraulic loading, m ³ /m ² /d	NH ₄ -N ^a			N-org			N-total ^a		
			C _{in} , mg/l	C _{out} , mg/l	E, %	C _{in} , mg/l	C _{out} , mg/l	E, %	C _{in} , mg/l	C _{out} , mg/l	E, %
1F	0.25	1.3	8.9±5.6	0.1±0.2	99±1	7.1±3.1	2.4±0.8	61±17	37±13	31±12	15±15
	0.50	2.6	27±22	13±12	70±27	6.3±2.4	15±10	35±34	49±27	42±23	13±7
	0.75	3.8	38±22	21±17	52±20	8.7±3.9	23±15	39±38	51±22	44±16	11±13
2F	0.25	1.3	8.9±5.6	0.2±0.3	98±2	7.1±3.1	2.2±0.8	65±15	37±13	33±13	13±12
	0.50	2.6	27±22	9.8±9.4	77±22	6.3±2.4	4.5±3.4	31±39	49±27	43±24	13±8
	0.75	3.8	38±22	16±15	67±19	8.7±3.9	3.2±1.8	58±29	51±22	39±21	28±11
3F	0.25	1.3	8.9±5.6	0.3±0.9	98±5	7.1±3.1	2.1±0.7	66±14	37±13	31±13	19±21
	0.50	2.6	27±22	10±10	73±22	6.3±2.4	2.8±1.8	51±30	49±27	41±22	15±7
	0.75	3.8	38±22	12±14	79±21	8.7±3.9	3.0±1.3	60±20	51±22	38±19	27±8

Note: ^aNH₄-N and N-total values are calculated on the basis of the data obtained on 177–430 days of the experiment.

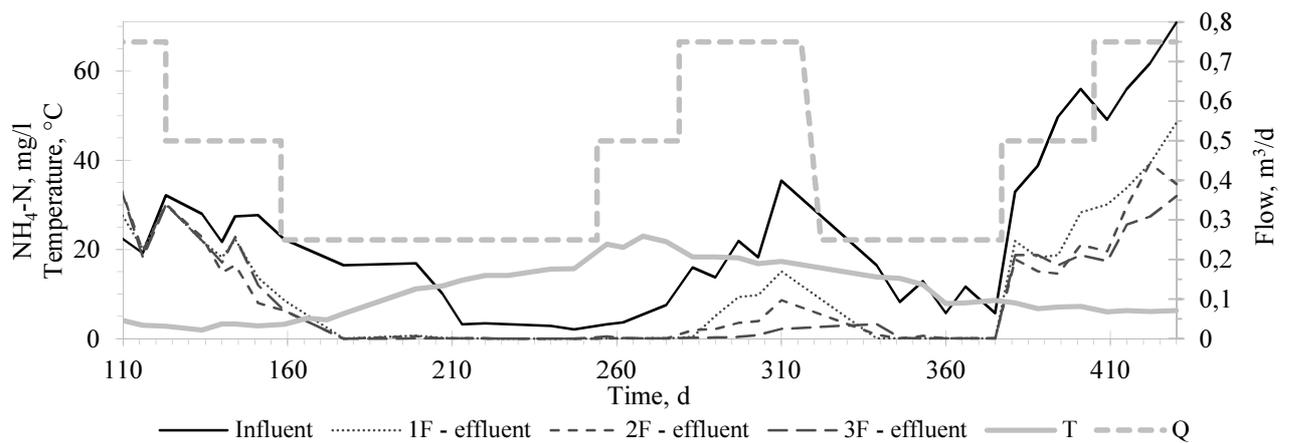


Fig. 3. NH₄-N concentrations in the influent and effluent of the filters

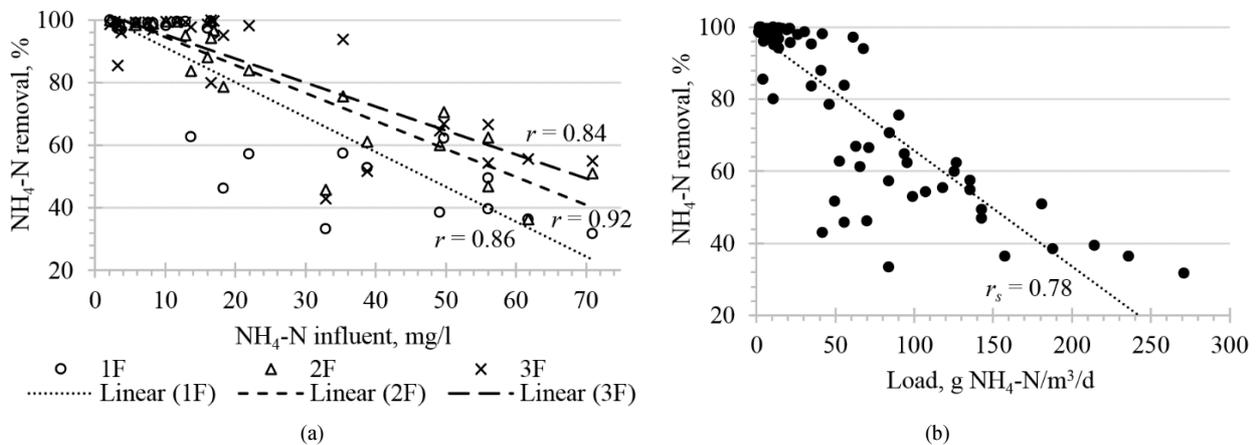


Fig. 4. NH₄-N removal dependence on influent concentration (a) and volumetric loading (b)

3.3. Phosphorus compounds removal

P-total removal was not high and its mean values fluctuated from -18 to 13% depending on the filter height and hydraulic loading (Table 5). P-org + p-PO₄-P concentration in the influent was very small with a mean value of 0.60±0.32 mg/l, therefore no dependencies of its removal on operational and design parameters were observed. Mean P-org and polyphosphates removal efficiency in all the filters ranged from approximately 30 to 65% and, as in case of N-org, was related to the physical retention, decomposition and hydrolysis of these phosphorus forms. PO₄-P amounted 89±7% of P-total in the influent with its mean concentration of 4.7±1.6 mg/l. PO₄-P removal did not depend on any operational and design parameters and was unstable in all the filters during the experimental period with considerable fluctuations of removal efficiencies from negative to positive values. This can be attributed to the combination of several processes: the conversion of P-org and polyphosphates to PO₄-P through decomposition and hydrolysis and metabolic uptake of PO₄-P by microorganisms in the biofilm.

Table 5. Phosphorus compounds removal as a function of design and operational conditions

Filter	Flow rate, m ³ /d	Hydraulic loading, m ³ /m ² /d	o-PO ₄ -P			P-org + p-PO ₄ -P			P-total		
			C _{in} , mg/l	C _{out} , mg/l	E, %	C _{in} , mg/l	C _{out} , mg/l	E, %	C _{in} , mg/l	C _{out} , mg/l	E, %
1F	0.25	1.3	3.8±0.9	3.8±0.9	-0.7±19	0.56±0.28	0.24±0.31	56±47	4.3±0.9	3.9±0.8	7.5±17
	0.50	2.6	5.3±1.4	5.4±1.2	-3.1±12	0.63±0.28	0.28±0.37	59±45	5.8±1.2	5.7±1.1	2.3±9.0
	0.75	3.8	5.4±2.1	5.5±2.1	-0.2±5.5	0.64±0.43	0.26±0.33	57±44	6.0±2.0	5.7±2.0	5.7±8.4
2F	0.25	1.3	3.8±0.9	3.8±0.9	-2.4±15	0.56±0.28	0.28±0.35	54±43	4.3±0.9	4.1±0.9	5.0±12
	0.50	2.6	5.3±1.4	5.3±1.3	-1.2±7.4	0.63±0.28	0.34±0.20	32±57	5.8±1.2	5.6±1.2	3.9±7.5
	0.75	3.8	5.4±2.1	5.5±1.8	-2.7±7.8	0.64±0.43	0.32±0.29	44±48	6.0±2.0	5.8±1.7	3.8±3.5
3F	0.25	1.3	3.8±0.9	3.5±1.0	4.1±25	0.56±0.28	0.19±0.21	61±48	4.3±0.9	3.7±1.0	13±19

0.50	2.6	5.3±1.4	4.8±1.5	7.9±20	0.63±0.28	0.21±0.26	63±37	5.8±1.2	5.0±1.4	13±16
0.75	3.8	5.4±2.1	6.6±2.8	-28±62	0.64±0.43	0.33±0.34	32±64	6.0±2.0	6.9±3.0	-18±54

4. Conclusions

A trickling filter with stone wool medium can potentially be used for the tertiary on-site wastewater treatment. SS, organic matter removal, nitrification, removal of N-org, P-org and polyphosphates were the main processes taking place in these filters. SS removal efficiency in the filters reached up to 69%, BOD₇ – up to 84%, COD – up to 60%, NH₄-N – up to 99% and N-org – up to 66% depending on filter design and operational conditions. SS and organic matter removal efficiencies increased when increasing SS and BOD₇ influent concentrations, however no dependence on volumetric loading (up to 242 g SS/m³/d and 228 g BOD₇/m³/d) was observed. Nitrification was dependent on volumetric ammonium loading, but did not depend on volumetric organic loading in the range of 5–228 g BOD₇/m³/d and wastewater temperature in the range of 5.6–23.2 °C. Nitrification efficiency of 90% can be achieved when volumetric ammonium loading does not exceed 24 g NH₄-N/m³/d. P-total removal in the filters was not stable and was mainly attributed to P-org and polyphosphates removal. The latter amounted only 11% of P-total, therefore P-total removal was not high and fluctuated from -18 to 13%.

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