Biological nitrate removal in a laboratory-scale slow sand filter

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Abstract

This research evaluated removal of nitrates from drinking waters in a slow sand filter (SSF). Batch experiments were performed to determine optimum carbon to NO₃-N (C/N) ratio for the filtration experiments. The filter column was filled with filter sand of an effective diameter of 0.5 mm and uniformity coefficient of 1.23. The filter was operated at filtration rates of between 0.02 to 0.120 m/h and 0.01 to 0.25 m/h with concentrations of 22.6 and 45.2 mg NO₃-N/ ℓ , respectively, and effluent samples of the SSF were taken at 6 depths of 10, 15, 20, 40, 60, 80 cm, and the bottom. Optimum C/N ratio was found to be 1.5 when using ethanol in batch tests when the removal efficiencies of NO₃-N and C were higher than 90%. Although increasing filtration rates decreased NO₃-N removal, effluent NO₃-N concentration at the effluent port of the SSF was lower than the limit value. Most of the NO₃-N removal was carried out at the upper layer of (10 cm) the filter bed. Concentrations of 22.6 mg NO₃-N/ ℓ . As expected, increasing influent NO₃-N concentration to 45.2 mg/ ℓ increased NO₃-N, NO₂-N, and C concentrations in the effluent water. The SSF process was unable to provide NO₃-N removal rate of more than 228 g N/m³-d (0.2 m/h flow rate, 217g N/m²-d of surface loading rate). The NO₃-N removal efficiency dropped slightly from 96 to 95% when the loading rate increased from 228 to 285 g/m³-d, but the effluent water contained higher concentrations of NO₂-N (8.4 mg/ ℓ) than the standard value. The results of the SSF experiment demonstrated that averaged nitrogen conversion to volatile solids was about 0.77 mg VS/mg NO₃-N.

Keywords: biodenitrification, slow sand filtration, drinking water

Introduction

Nitrate contamination in groundwater arises from agricultural practices and improper discharge of industrial and municipal wastes. Accumulation of various forms of nitrogen in water can lead to adverse effects including depletion of dissolved oxygen in receiving waters, ammonia toxicity to aquatic life, and public health problems related to the presence of nitrate in drinking water supplies (Elefsiniotis and Li, 2006).

Regulations for drinking water are required in order to limit human risks and environmental pollution for NO₃-N and NO₂-N in drinking water. While the United States Environmental Protection Agency (USEPA) has set maximum contaminant level goal (MCLG) of 10 mg NO₃-N and 1.0 mg NO₂-N/ ℓ , the World Health Organisation (WHO, 1984) and European Economic Community (EU, 1998) have set standards of 11.3 mg NO₃-N/ ℓ and 0.03mg NO₃-N/ ℓ .

It is necessary to reduce NO_3 -N from drinking water supplies for human consumption when the NO_3 -N concentration exceeds the drinking water standards. Among the various NO_3 -N removal methods such as ion exchange, biodenitrification, reverse osmosis, electrodialysis and distillation, biological processes have been shown to be more efficient and convenient.

The slow sand filter (SSF) has been known to be a simple to operate, low cost, efficient and reliable technique and has been used successfully to remove micro-organisms in drinking water since 1900. It has been well documented in the literature for many decades. In conventional water treatment works, SSF

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reservoir storage and rapid filtration, prior to disinfection (Ellis, 1985). However, SSF can also provide a single-stage treatment for raw waters within certain water quality limits of turbidity and algal content (Bowles et al., 1983). Simplicity, low capital cost, and operating costs are other principal advantages of SSF compared with more sophisticated methods of water treatment (Campos et al., 2002). Among the more important mechanisms for the removal of contaminants in water through SSF are: biological activity at the upper layer of the filter, adsorption, mechanical filtration, and surface catalysed degradation. These process features make SSF most attractive for advanced treatment of effluents (Adin, 2003). Recent attention has focused on the use of SSFs for tertiary wastewater treatment (Nakhla and Farooq, 2003). The SSF was operated in drinking and wastewater treatment for removal of carbon and nitrification (Rodgers et al., 2005), pathogenic bacteria (Bomo et al., 2003), protozoan parasite (Timms et al., 1995), and suspended solids (Christopherson et al., 2005; Aslan, 2005; Aslan and Turkman 2006; Rocca et al., 2005). The SSF was applied to remove nitrogen in wastewater using biological nitrification and denitrification (Nakhla and Farooq, 2003).

is generally the third stage of water treatment purification after

Although the SSF has been applied for the removal of pathogens, suspended solids and organics in wastewater, very few studies have been conducted on the treatment of NO₃-N removal in drinking water. The objective of this study is to present experimental data on the biological removal of NO₃-N through the SSF depths operating at various filtration rates under laboratory conditions.

Materials and methods

The SSF was inoculated with micro-organisms taken from the aeration basin at Cumhuriyet University Wastewater Treatment

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Plant in Sivas, Turkey, acclimatised to ethanol and NO_3 -N with medium solution prepared daily in distilled water. The inoculation lasted about one month for microbial growth with daily replenishment of NO_3 -N and ethanol in medium solution in a 500 m ℓ bottle.

Batch experiments

Batch experiments were carried out to determine the optimal carbon to nitrogen (C/N) ratio for microbial activity. Experiments were performed in 500 ml glass bottles containing medium solutions. C/N ratios varied from 1.1 to 3.0 for C using ethanol while the NO₃-N concentration was kept constant at 22.6 mg/l. Acclimated micro-organisms were added into the flasks and cultures were placed on a shaking incubator at 30°C at 100 r/min. NO₃-N, NO₂-N, and chemical oxygen demand (COD) analyses were performed in the feed solution and in the clear samples at the end of the reaction.

Experimental set-up of the biological SSF

The biological SSF experimental set-up consisted of a cylindrical stainless steel biological reactor, 10 cm inner diameter and 100 cm height, completely submerged and operating in down flow mode (Fig. 1). The filter was equipped with 6 water sampling and effluent ports. Screens were placed at the bottom and sampling ports of the filter to prevent clogging of the column outlet.

The synthetic medium solution was stored in 30 ℓ plastic containers of C and NO₃-N sources including trace elements at room temperature ($17 \pm 3^{\circ}$ C). The outer surfaces of the containers and tubes were wrapped with aluminium foil to prevent algal growth. NO₃-N and C concentration in the containers were measured periodically throughout the experimental study. A piston pump was used to transfer synthetic medium solutions to the SSF. Transfer tubes were washed with acidic solution weekly to prevent microbial growth.

The filter column was filled with filter sand of an effective diameter of 0.5 mm and uniformity coefficient of 1.23. The SSF had a liquid volume 2 ℓ after packing sand and 5 cm water depth on the top of the filter was maintained throughout the experiment. The sand was washed several times to remove impurities before packing the filter.

The filter was operated at filtration rates between 0.02 to 0.120 m/h and 0.01 to 0.25 m/h with concentrations of 22.6 and 45.2 mg NO₃-N/ ℓ , respectively, and effluent samples of the sand filter were taken at 6 depths of 10, 15, 20, 40, 60, 80 cm, and the bottom. The nitrogen loading rate (NLR) was varied by increasing the influent flow rate. The flow rate was adjusted by a valve at the bottom of the filter. The operational condition of the SSF system is summarised at Table 1. Manometers were installed at each sampling ports to measure head loss. The filter was operated primarily to assess the impact of filtration rates and filter depths on the NO₃-N removal performance.

Prior to operation, the SSF was filled with synthetic medium solution and 10 m ℓ inoculated micro-organisms in the batch unit was added to the upper layer of the filter. The inoculation of the SSF was carried out at the lowest filtration rate (0.01 m/h) and the filter effluent was withdrawn daily at the bottom of the filter to monitor the performance by measuring NO₃-N, NO₂-N, and C concentration throughout the acclimation period. After about 3 d, effluent NO₃-N concentration started to decrease and complete NO₃-N removal was achieved after 15 d of stable operation of the SSF.



Figure 1 Schematic representation of the biological SSF experimental set-up

TABLE 1 Operational condition of the SSF system			
	0.02	11.4	10.8
(22.6 mg/ℓ)	0.03	17.1	16.3
	0.055	31.4	29.8
	0.07	40	38
	0.1	57.1	54.2
	0.12	68.5	65.1
	0.01	11.4	10.8
(45.2 mg/ℓ)	0.02	22.8	21.7
	0.055	62.8	59.7
	0.088	100.5	95.5
	0.18	205.5	195.3
	0.2	228.3	217
	0.25	285.4	271.2

When the flow rate through the filter could not be maintained and the head loss reached 20 cm, the water layer above the sand bed was drained and 2 cm depth of the top layer of the filter scraped out to remove micro-organisms in the sand. Volatile solids (VS) measurement was carried out on the removed sand. After scraping and cleaning the sand, the upper layer of the filter was filled with clean sand, and the filter was operated for at least 3d to promote microbial growth at the top of the filter until achieving higher than 90% NO₃-N removal at the bottom of the filter.

SSF was operated at room temperature. After completing the first experiment at 22.6 mg NO₃-N/ ℓ , 10 cm depth of upper layer of the filter was scrapped out, the concentrations of VS onto the sand was measured and then clean sand was replaced onto the filter. NO₃-N concentration of the synthetic medium solution after mixing with C solution was increased to 45.2 mg/ ℓ while maintaining the C/N ratio at 1.5 during the second step.

Synthetic medium composition

The liquid medium used consisted of a mineral base supplemented with NO₂-N as sole electron acceptor and ethanol as electron donor. The medium constituents were KNO₂, ethanol, KH₂PO₄ (150 mg/ ℓ), and NaHCO₂ (325 mg/ ℓ). This medium was supplemented with 1% v/v of a solution containing FeSO₄.7H₂O (0.20 mg/ ℓ), titriplex (0.565 mg/ ℓ), and with 0.1% v/v of a trace nutrient solution containing ZnSO4.7H2O (0.1 g/l), MnCl2.4H2O (0.03 g/l), H₃BO₃ (0.3 g/l), CoCl₂.6H₂O (0.2 g/l), CuCl₂.2H₂O (0.01 g/l), NiCl₂.6H₂O (0.02 g/l), and NaMoO₄.2H₂O (0.03 g/l)(Aslan, 2005). The final pH of the medium was adjusted to 7.5 using NaOH solution.

Analytical methods

NO₂-N, NO₂-N, and COD concentrations of influent solutions were measured routinely. Samples were withdrawn daily from 6 effluent points and the bottom of the SSF and centrifuged at 6 000 r/min for 30 min to remove suspended solids from the effluent. NO₂-N, NO₂-N, and COD analyses were performed with clear samples and pH was measured. Biomass concentrations were determined as VS by scraping a 2 cm deep layer of sand off the SSF. The sand was then washed gently in distilled water and VS were measured in 50 ml eloquent and the sand was placed back onto the sand filter. COD concentrations of the influent and effluent samples and the VS were determined according to Standard Methods (1995) and C concentrations were calculated according to its stoichiometric relationship with COD for ethanol. NO₂-N and NO₂-N were analysed with the Merck photometer Nova 60 using analytical kits; NO₂-N (14776), and NO₂-N (14773).

Results and discussion

Determination of optimum C/N ratio in batch units

The temperature affects the removal rate of NO₂-N and the microbial growth rate. About complete NO₃-N removal was observed at temperature between 22 and 37°C (Aslan and Turkman, 2004). Because of this reason, batch experiments were performed at 30°C for 3 d after which at least 90% NO₂-N removal efficiency was observed to obtain optimal C/N ratio for the biological SSF system. The optimum C/N ratio was assumed to be the ratio which allowed achieving maximum removal of NO3-N with minimum excess C in the effluent.

100

90

70 (l/gm)

20

10

NO₃-N (50

effluen 30

It can be seen in Fig. 2 that the low C/Nratio resulted in low NO₃-N removal efficiency and high NO₃-N level at the end of the reaction. Optimum C/N ratio was found to be 1.5 when using ethanol in batch tests. At C/N ratios below the optimum ratio, the NO₃-N removal was found to be dependent upon the C concentrations, causing a noticeable fall of NO3-N removal in batch units. Although no significant improvement in NO₂-N removal was observed with the C/N ratio in excess of the optimum value, excess amount of C remained in the water. The C consumption reported here is in the range of the values obtained by Richard (1989) and is about the same as the values reported by Delanghe et al. (1994) and Aslan

(2005), but is 50% higher than the values given by the Dahab and Sirigina (1994).

Since the low C/N ratio (C/N<1.5) of the influent caused incomplete denitrification, the accumulation of NO₂-N occurred and the $\Delta C/\Delta N$ ratio was higher than 4.0. The highest $\Delta C/\Delta N$ ratio was observed for 1.2 of the C/N ratio.

SSF experimental results

The SSF was operated at low velocity at the start of the experiments to promote microbial growth through the filter bed and NO₂-N, NO₂-N and C concentrations in the effluent were measured during this stage. After 15 d of operation, NO₃-N was not detected in the filter effluent.

The filtration rate was brought to 0.02 and 0.01 m/h (about 0.16 and 0.08 ℓ/h) during the initial runs and was then progressively increased to 0.12 (0.94 ℓ/h) and 0.25 m/h (2 ℓ/h) for 22.6 and 45.2 mg NO₂-N/ℓ concentrations, respectively. The filter was operated at 45.2 mg/l influent NO₃-N concentration to achieve maximum NLR.

First step NO₃-N removal through the SSF

NO₂-N concentration was kept at 22.6 mg/ℓ during the first step of the study. Most of the NO₂-N removal was observed at the upper layer of the 10 cm filter bed. NO₂-N, NO₂-N, and C were not detected in the effluent port and the 60 cm depth of the SSF throughout the first step of the study. Although increasing filtration rate increased NO₂-N, NO₂-N, and C concentrations, NO₂-N concentration was still below the acceptable level for the drinking water at the 0.02, 0.03, 0.055, and 0.07 m/h filtration rates at 10 cm filter depth and the 0.1 and 0.12 m/h filtration rates at 15 cm filter depth (Fig. 3).



various loading rates (influent concentration 22.6 mg NO₃-N/ℓ) ♦0.02 □ 0.03 ▲ 0.055 X 0.07 № 0.1 ∘ 0.12 m/h

The filtration rates varied from 0.02 and 0.03 m/h, corresponding to an overall average NO₃-N removal efficiency of 98% at 10 cm filter depth. Increasing filtration rates to 0.055, 0.07, 0.1, and 0.12 m/h, the removal efficiencies of NO₃-N decreased to 65, 56, 46, and 31%, respectively at the first sampling port.

Lower NO₃-N concentration was observed for the filtration rates between 0.02 to 0.07 m/h than the standard limit of 10 mg NO₃-N/ ℓ . Effluent NO₃-N concentrations ranged from 12.2 to 15.6 mg/ ℓ at the filtration rates of 0.1 and 0.12 m/h at the 10 cm filter depth and decreased to 0.5 and 2.8 mg NO₃-N/ ℓ , respectively at the 15 cm depth. Accordingly, about 22 and 7 mg NO₃-N/ ℓ were removed at the filter depth of 10 cm at filtration rates between 0.02 and 1.2 m/h, respectively, and the remaining were removed at higher depths.

The intermediate product NO₂-N of denitrification in the effluent water at the 40 cm filter depth did not exceed the maximum limit value of 0.03 mg NO₂- N/ℓ at the filtration rate of 0.07 m/h. NO₂-N distributions through the SSF are given in Fig. 4. The effluent NO₂-N concentrations were 0.26, 0.18, 0.05 mg/ ℓ , and lower than the detection limit at the 10, 15, 20, and 40 cm filter depths at 0.02 m/h filtration rate. Increasing filtration rate from 0.02 to 0.12 m/h increased the effluent NO2-N concentration at the effluent point. Although, effluent samples at the 40 cm depth contained more than 0.03 mg NO₂-N/ ℓ at the filtration rates of between 0.07 and 1.2 m/h, NO₂-N was not detected beyond the 40 cm depth.

The C content of the filtrate was high at the top layer and decreased gradually through the SSF (Fig. 5). The C outlet concentration was decreased through the filter depth and it was not detected beyond the 40 cm filter depth. The effluent C concentrations were 7.2, 5.9, 5.3, and 2.1 mg/ ℓ at 10, 15, 20, and 40 cm filter depths at 0.02 m/h filtration rate. The effluent C concentrations varied between about 47 and 9 mg/ ℓ at the top of the filter and 40 cm filter depth at 0.12 m/h filtration rate, respectively.

Second step NO₃-N removal through the SSF

As expected, increasing the NO₃-N concentration in the medium solution increased the NO₃-N, NO₂-N, and C concentrations in the effluent. The NO₃-N removal efficiency was 87% at the filtration rate of 0.01 m/h at the 10 cm depth and increased to 99% at the 20 cm and NO₃-N was not detected beyond the 20 cm depth. NO₃-N concentrations were higher than the standard limit of 10 mg NO₃-N/ ℓ for the filtration rates higher than 0.02 m/h at the 10 cm depth of and complete NO₃-N removal was observed at filtration rates lower than 0.2 m/h at the 60 cm depth (Fig. 6).

Increasing filtration rates above 0.055 m/h had little effect on the effluent concentration of NO_3 -N at the 10 cm filter depth. At the highest filtration rate, NO_3 -N removal efficiencies were between 39 and 95% through filter depths. When the SSF was operated at high filtration rate, the short contact time caused low NO_3 -N removal. Beyond the filter depth of 60 cm, NO_3 -N removal was not observed for the filtration rates of 0.2 and 0.25 m/h; however, NO_3 -N concentration was lower than the standard value.

Although relatively high concentrations of NO_2 -N were observed at the 10 cm filter depth, NO_2 -N was decreased through the filter, except at the highest filtration rate. However, at this rate a concentration of 8.4 mg NO₂-N was detected in the





Available on website http://www.wrc.org.za ISSN 0378-4738 = Water SA Vol. 34 No. 1 January 2008 ISSN 1816-7950 = Water SA (on-line) effluent, which was higher than the limit value (Fig. 7).

The C source was entirely consumed in the SSF when the filtration rate was low, but more than 5 mg/ ℓ C remained in the effluent at the filtration rate of 0.2 and 0.25 m/h. C concentrations were between 54 and 125 mg/ ℓ at the 10 cm filter depth at the filtrations rates applied during the study (Fig. 8).

In this study, more than 95% NO₂-N removal efficiency was achieved at the filtration rate of 0.25 m/h at the depth of 60 cm; by comparison, Nakhla and Faroog (2003) achieved about 80% denitrification efficiency in raw wastewater using the SSF at a depth of 80 cm, while 95% NO₂-N removal was obtained at the filter depth of 40 cm at a filtration rate of 0.18 m/h. The SSF was performed as a post-treatment after the biological process and it was assumed that the slowly biodegradable soluble COD in the wastewater might slow down the denitrification process. Aslan (2005), Aslan and Turkman (2005 and 2006) results indicated that about 20% of the NO₂-N remaining from the biological denitrification reactor was removed in the following sand filter at a depth of 30 cm. Because the SSF was operated at a high flow rate (0.125 m/h) and low C content of the influent water, NO,-N removal was lower than in this study.

The SSF showed good NO₃-N removal performance at the 10 cm filter depth with daily removal of 28.2 and 50 g NO₃-N/m³ at a filtration rate 0.02 and 0.01 m/h when influent concentrations were 22.6 and 45.2 mg/ ℓ , respectively. Daily removal decreased, with increasing filtration rates and depths, because most of the NO₃-N was removed at the upper layer of the SSF (Figs. 9 and 10).

In this study, the occurrence of the highest microbial activities in the top layer (0-40 cm filter depth) of the SSF was observed and beyond this depth significant microbial activity did not occur.

Complete NO₃-N removal was obtained with influent concentration of 22.6 mg NO₃-N/ ℓ . The maximum NO₃-N removal rate during this period was 68.5 g/m³·d and about complete NO₃-N removal was obtained at the 40 cm filter depth. Increasing the NLR from 68.5 to 285 g/m³·d (surface loading rate 171.2 g/m²·d) by increasing the influent flow rate and influent NO₃-N concentrations appeared to result in an incomplete denitrification.

It was evident that the process was unable to provide NO_3 -N removal rate of more than 228 g NO_3 -N/m³·d (0.2 m/h flow rate). Although the NO_3 -N removal efficiency dropped slightly from 96 to 95% when the NLR increased from 228 to 285 g NO_3/m^3 ·d, the effluent water contained a higher con-









(Influent concentration 45.2 mg NO3-N /l)



centration of NO₂-N (8.4 mg/ ℓ) than the standard value.

Alkalinity is produced during the conversion of NO_3 -N to nitrogen gas resulting in an increase in effluent pH. Throughout the experimental study, because of the denitrification process the final pH at the effluent was slightly higher than initial pH and in the range of 7.8-8.1.

When the flow rate through the filter could not be maintained, VS was measured by scraping the upper layer of the filter. The biomass yield was calculated for the top layer of the SSF by considering the nitrogen consumption: calculated by using the following equation:

Nitrogen consumption (mg) = treated water volume (ℓ) x total nitrogen (influent – effluent from the top layer) (mg/ ℓ).

Stoichiometric relationship including cell synthesis of heterotrophic denitrification with ethanol as a C source has been suggested by Mateju et al. (1992) as:

$$\begin{array}{l} 0.613 \text{ C}_2\text{H}_5\text{OH} + \text{NO}_3 \rightarrow 0.10 \text{ C}_5\text{H}_7\text{NO}_2 + 0.7124 \text{ CO}_2 \\ + 0.286 \text{ OH} + 0.98 \text{ H}_3\text{O} + 0.449 \text{ N}_3 \end{array} \tag{1}$$

where $C_5H_7NO_2$ represents biological cell formula. Based on Eq. (1), the reduction of 1 g NO₃-N theoretically produces 0.807 g new cells.

The results of the SSF experiment demonstrated that the average nitrogen conversion to VS was about 0.769 mg VS/mg NO_3 -N (Fig. 11). It was apparent that consumed NO_3 -N (mg) correlated well with conversion of nitrogen to micro-organisms which is proposed in the stoichiometric equation by Mateju et al. (1992).

The nitrogen balance throughout the SSF, considering inlet and outlet differences of NO₃-N and NO₂-N vs. total daily removed nitrogen showed high correlation $R^{2>}$ 0.89 at various depth of sampling ports. The average NO₃-N removal efficiency was 99.9 and 95% (Std. dev. = 3.3 ± 1 , n= 4 for each NLR) at NLR of 11.4 and 285.4 kg NO₃-N/m³·d, respectively.

Conclusions

Based on experimental results of this study, it can be concluded that SSF can be used effectively for NO₃-N removal in drinking water. Most of the NO₃-N was removed in the top layer of the filter. In this investigation, NO₃-N concentrations were reduced from the initial concentration of 22.6 mg/ ℓ to below the detection limit of NO₃-N at all filtration rates, and from the initial concentration of 45.2 to 2.3 mg/ ℓ at the highest filtration rate. No significant NO₂-N accumulation occurred in the denitrified water at the 22.6 mg NO₃-N/ ℓ influent concentration. Increasing filtration rates from 0.02 to 0.12 had no adverse effect on the filter effluent. However, 8.4 mg NO₂-N/ ℓ remained in the effluent water at the highest filtration rate when influent NO₃-N was 45.2 mg/ ℓ . The SSF was unable to provide NO₃-N removal rate of more than 228 g/m³·d. The average nitrogen conversion to VS was about 0.769 mg VS/mg NO₃-N in the SSF.

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