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Removal of ammonia from contaminated air in a biotrickling filter – Denitrifying bioreactor combination system

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ABSTRACT

The removal of gaseous ammonia in a system consisting of a biotrickling filter, a denitrification reactor and a polishing bioreactor for the trickling liquid was investigated. The system allowed sustained treatment of ammonia while preventing biological inhibition by accumulating nitrate and nitrite and avoiding generation of contaminated water. All bioreactors were packed with cattle bone composite ceramics, a porous support with a large interfacial area. Excellent removal of ammonia gas was obtained. The critical loading ranged from 60 to 120 $g\,m^{-3}\,h^{-1}$ depending on the conditions, and loadings below 56 $g\,m^{-3}\,h^{-1}$ resulted in essentially complete removal of ammonia. In addition, concentrations of ammonia, nitrite, nitrate and COD in the recycle liquid of the inlet and outlet of each reactor were measured to determine the fate of nitrogen in the reactor, close nitrogen balances and calculate nitrogen to COD ratios. Ammonia absorption and nitrification occurred in the biotrickling filter; nitrate and nitrite were biologically removed in the denitrification reactor and excess dissolved COD and ammonia were treated in the polishing bioreactor. Overall, ammonia gas was very successfully removed in the bioreactor system and steady state operation with respect to nitrogen species was achieved.

1. Introduction

Ammonia emissions are very common in operations such as composting, fertilizer manufacturing, and intensive swine, poultry or cattle production (NRC 2003; Arogo et al., 2003). Ammonia has a moderate odor threshold (5–20 ppm_v) and emissions are regulated both because of odor nuisances and air pollution concerns. Ammonia can be easily scrubbed chemically, although the costs of chemicals for scrubbing can be very significant and scrubbing results in large amounts of an acidic ammonium solution that needs to be disposed of.

Biological treatment of odorous air is an interesting alternative to conventional treatment which has been shown to be efficient and cost effective in a number of cases (Devinny et al., 1999; Gabriel and Deshusses, 2003). With respect to ammonia gas treatment, the complexity of the biological nitrogen cycle offers several possibilities for biotransformation, although many have so far not been fully exploited for gas treatment. For example, ammonia gas can be absorbed and then nitrified to nitrite and nitrate, and subsequently denitrified (autotrophically or heterotrophically) to nitrogen gas. There is a vast body of literature on nitrogen removal in wastewater treatment plants (see e.g., Metcalf and Eddy, 2003) on which researchers can draw to develop bioreactors for air pollution control.

Various studies have focused on the treatment of ammonia gas in biofilters and biotrickling filters. Unfortunately, in many

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cases, the ultimate fate of nitrogen is not clearly identified. This is because of the difficulty of closing the nitrogen balance in systems such as compost beds which already contain significant concentrations of ammonium and nitrate, and which are often operated in a pseudo-steady state with slow accumulation of substrates and metabolites (e.g., ammonium, nitrite, nitrate) in the packing. Thus, in most cases, treatment relied mostly on the absorption of ammonia, which was followed by partial nitrification to nitrite and nitrate, and accumulation of these species in the packing and sometimes partial purging from the system by leaching (Hartikainen et al., 1996; Smet et al., 2000; Chou and Wang, 2007; Taghipour et al., 2008). However, both free ammonia and free nitrous acid are known inhibitors of nitrification (Weckhuysen et al., 1994; Baeza et al., 1999; Baquerizo et al., 2005), and when considering the importance of pH effects on both ammonia absorption and on nitrification, it is not surprising to see reactors fail after some time due to the accumulation of metabolites (Hartikainen et al., 1996; Sorial et al., 2001; Smet et al., 2000). Thus, unless a flushing schedule and relatively tight control of pH are implemented, biofilters treating ammonia will be much more susceptible to failure than biotrickling filters. This is illustrated by Liang et al. (2000) studies which wrongfully concluded that ammonia inlet concentration over 200 ppm_v should be avoided because of the toxic effect of ammonia to the nitrifiers. A deeper insight into the fate of nitrogen, ammonia concentration and pH effects was provided by Baquerizo et al. (2007) who modeled the various parallel processes involved during the treatment of ammonia. The model simulations illustrated the importance of moisture and free water in the treatment of ammonia.

When high concentrations of ammonia need to be treated and water consumption should be minimized, nitrification to nitrite or nitrate, followed by denitrification to nitrogen gas is probably the most desirable route, since it will prevent generation of a stream of water contaminated with nitrate and/or nitrite. However, this requires several biotransformation carried out by different microorganisms. Typically, achieving successful nitrification and denitrification requires a careful control of pH, substrate and chemical oxygen demand (COD) concentrations, dissolved oxygen, etc. and preventing toxic or inhibitory metabolites such as free ammonia and free nitrous acid to accumulate. Hence, the purpose of this research was to determine technical feasibility and the performance of a biological treatment system comprising of a biotrickling filter and a separate denitrification reactor to treat high concentrations (300–500 ppm_v) of ammonia in air with conversion of ammonia to nitrogen gas.

2. Materials and methods

2.1. *Biotrickling filter and packing material*

Three reactors (biotrickling filter, denitrification, and later a post-treatment) were used in this study. All the reactors were constructed from clear polyvinyl chloride (PVC) piping and were 1.2 m in length and 10 cm in internal diameter. Fig. 1 provides a schematic of the setup. Synthetic waste air for the experiments was prepared passing compressed air through a humidifier and adding pure ammonia gas to reach the desired

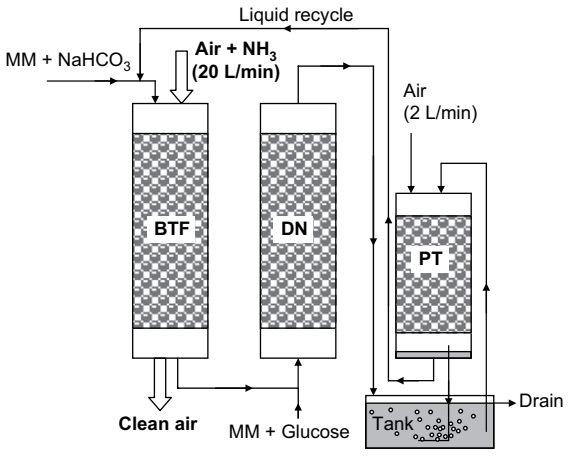

Fig. 1 – Schematic diagram of the experimental system (not to scale). BTF = biotrickling filter, DN = denitrification reactor, PT = post-treatment bioreactor, MM = mineral medium.

concentration. The synthetic contaminated air stream was supplied to the top of the biotrickling filter (downflow mode). Only the biotrickling filter and denitrification bioreactors were operated for the first 15 days after acclimation, after which the post-treatment bioreactor was added. The purpose of the latter (an aerated trickling filter) was to avoid feeding back residual organic substrate to the biotrickling filter. This will be discussed later. The effluent of the denitrification was directed to a holding tank (15 L) aerated (2 L min^{-1}) with the air from the post-treatment. The liquid recycle for the biotrickling filter was taken from this tank (see Fig. 1). The biotrickling filter and denitrification reactors were packed to a bed depth of 60 cm, while the post-treatment reactor had a bed depth of 40 cm. Cattle bone composite ceramic (CBP) beads (4 mm diameter, Aisin Takaoka Co, Ltd., Toyota, Japan) were used as a packing in this study. CBP had been shown to outperform other packings for toluene vapor removal in biofilters (Sakuma et al., 2006). It is made with 80% volume of the raw material used in the making of standard porous ceramics and 20% volume of cattle bone powder. During the making of the ceramic beads, part of the cattle bone powder burns leaving pore space and ashes, while the remainder of the cattle bone is believed to act as a slow release nutrient source for microorganisms.

2.2. *Startup of the bioreactors and operating conditions*

The biotrickling filter and denitrification reactors were inoculated with activated sludge from a local wastewater treatment plant. Mineral medium (2 L, see composition below), activated sludge (0.3 L) and 3 g $(NH_4)_2SO_4$ were circulated through the biotrickling filter and the denitrification beds for 24 h. The suspended solids were not monitored during this phase. The biotrickling filter was then incubated for a period of 4 months prior to the experiments to establish a dense culture of nitrifying organisms. The duration of the initial incubation period was not optimized. For the first 3 months, the biotrickling filter was operated as nitrification reactor fed with dissolved ammonium salt instead of ammonia in air.

Fresh liquid (8 mL min^{-1}) was continuously trickled (one pass) through the bioreactor. The liquid contained: 2.7 g L^{-1} $(NH_4)_2SO_4$, a quarter of the concentration of mineral medium listed at the end of this section and variable concentrations (1.25–5 g L^{-1}) of $NaHCO_3$. Nitrification performance was better with the addition of $NaHCO_3$ as was reported earlier by Martin et al. (1996). $NaHCO_3$ was used both as a carbon source for the nitrifiers and as pH buffer. For the next 35 days, 12 mL min^{-1} of ammonium-free liquid was continuously fed into the reactor from the top while about 400 ppm_v of ammonia gas in air was fed into the system at a flow rate of 20 L min^{-1}, corresponding to an empty bed residence time (EBRT) of 13.5 s. The trickling liquid contained 5 g L^{-1} $NaHCO_3$ and half the concentration of mineral medium as listed below.

During this period, the effects of the direction of airflow were compared. The synthetic foul air was applied from the bottom (upflow) for 22 days and from the top (downflow) for the next 13 days. Over 95% of ammonia gas removal was observed for both modes, however, markedly different nitrification performance between the two modes was determined by measuring inlet and outlet of ammonia, nitrite, and nitrate concentrations in the trickling liquid. The downflow mode exhibited a better nitrification performance. This is because most of the ammonia absorption occurred close to the gas inlet port and the absorbed ammonia had much more time to contact the biofilm in the packed bed when operated cocurrently. Therefore, the biotrickling filter was operated in a downflow mode for all further experiments.

The denitrification reactor was incubated for a period of 7 months prior to the experiments. This long incubation time was not optimized either; it ensured that a stable denitrifying culture was established. 8 mL min^{-1} of fresh liquid was continuously fed to the reactor from the bottom (one pass). The liquid for denitrification reactor contained: 4.125 g L^{-1} KNO_3, 1.53 g L^{-1} glucose, and a quarter of the concentration of mineral medium as listed below. After 1 month, the average nitrate removal performance reached 80%. The system had clogging problems because of excess biomass growth on day 75. At this time, the packing was removed from the reactor and washed with mineral medium. After restart, 70–100% of nitrate removal was observed until day 210.

The mineral medium contained: 1.443 g L^{-1} KH_2PO_4, 1.443 g L^{-1} K_2HPO_4, 1.0 g L^{-1} NaCl, 0.262 g L^{-1} $MgSO_4$, 0.0252 g L^{-1} $CaCl_2$, and 1 ml L^{-1} of a trace elements solution. The trace element solution contained: 12.2 g L^{-1} $FeCl_2 \cdot 4H_2O$, 0.16 g L^{-1} H_3BO_3, 4.09 g L^{-1} $MnCl_2 \cdot 4H_2O$, 0.927 g L^{-1} $CoCl_2 \cdot 6H_2O$, 2.37 g L^{-1} $ZnCl_2$, 0.067 g L^{-1} $NiCl_2 \cdot 6H_2O$, 0.616 g L^{-1} $CuCl_2 \cdot 2H_2O$, 0.579 g L^{-1} $NaMoO_4 \cdot 2H_2O$, 0.148 g L^{-1} KI, and 6.5 g L^{-1} EDTA $Na_4 \cdot 4(H_2O)$.

2.3. Standard operating conditions

The airflow rate was 20 L min^{-1}, corresponding to an empty bed residence time of 13.5 s. The ammonia concentration in the influent gas stream ranged from 270 to 700 ppm_v resulting in loadings to the biotrickling filter of 55–124 g m^{-3} h^{-1}. All experiments were carried out at a room temperature (22–25 °C). 14 mL min^{-1} of recycled liquid and 7 ml min^{-1} fresh liquid were continuously trickled through the biotrickling filter. The fresh liquid contained half the concentration of mineral medium as listed above and 5 g L^{-1} of $NaHCO_3$. The trickling liquid effluent was directly fed to the bottom of the denitrification reactor together with 7 mL min^{-1} of a 2.55 g L^{-1} solution of glucose. Day zero in the graphs corresponds to the day the biotrickling filter and denitrification bioreactors were connected together. On day 15, the post-treatment bioreactor was added to the system.

2.4. Analyses

Gaseous ammonia concentrations were measured by ammonia sensor from CITY Technology (Portsmouth, UK) with a detection limit of about 1–3 ppm_v. Liquid-phase concentrations of ammonia, nitrite, nitrate and COD were performed using quick test Vacu-vials kits from CHEMetrics (Calverton, VA). Pressure drop was measured using a U-shaped water gauge.

3. Results and discussion

The results of the continuous operation of the biotrickling filter with inlet concentrations of ammonia ranging from 270 to 700 ppm_v and an empty bed residence time of 13.5 s are shown in Fig. 2. During the initial 15 days of operation, the removal efficiency of ammonia was between 92 and 96%; however, a foul odor (rotten organic matter) was detected from the effluent air. Since ammonia treatment in the field is usually motivated by odor concerns, something had to be done to avoid foul odors. The odors were suspected to be from the buildup of anaerobic biodegradation by-products in the collection tank, which were then stripped when the liquid recycle was fed to the biotrickling filter. No further investigations were conducted to determine the nature of the odorous compounds, but in order to prevent their formation, a post-treatment reactor (see Fig. 1) consisting of an aerated trickling filter was installed on day 15. Liquid from the collection tank was trickled through the post-treatment reactor cocurrently with 2 L min^{-1} air stream, and the effluent liquid was used directly as trickling liquid in the biotrickling filter. The foul odor disappeared immediately.

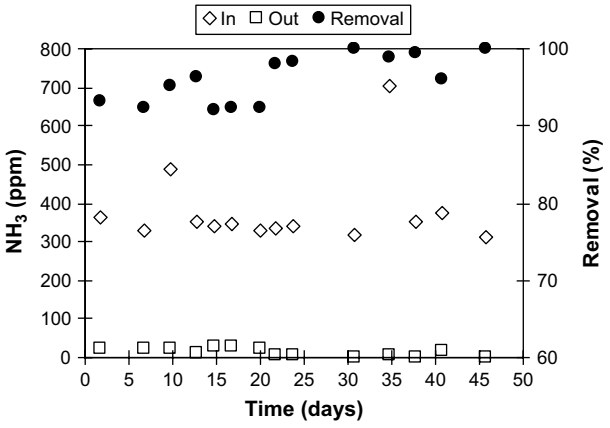

Fig. 2 – Inlet and outlet concentrations and removal of ammonia gas in the biotrickling filter over time. EBRT was 13.5 s.

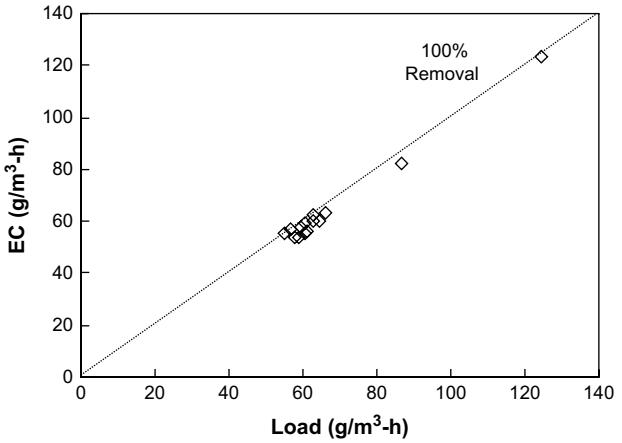

Fig. 3 – Ammonia elimination capacity (EC = (inlet–outlet concentration) × flow/bed volume) vs. (load = inlet concentration × flow/bed volume). The dashed diagonal line represents 100% removal.

During that time, the ammonia removal efficiency remained between 92 and 96%, possibly due to nutrient limitation. Therefore, the mineral medium concentration was doubled to provide ample supply of mineral nutrients, while the concentration of $NaHCO_3$ was halved on day 20 as carbonate was already supplied in excess. After day 21, the removal efficiency of the biotrickling filter never fell below 96%. In addition, 100% removal was observed on days 31 and 46 (Fig. 2). A plot of ammonia gas elimination capacity vs. loading is shown in Fig. 3. A complete removal of loadings up to 56 $g\,m^{-3}\,h^{-1}$ was observed and a critical load (defined as the maximum load at which 95% removal occurs) of 66 $g\,m^{-3}\,h^{-1}$ was obtained. The bioreactor system was also operated for 3 days at a much higher loading (125 $g\,m^{-3}\,h^{-1}$) which resulted in elimination capacities exceeding 120 $g\,m^{-3}\,h^{-1}$. During this time, the biotrickling filter effluent was analyzed for nitrate, nitrite and ammonium and closure of the nitrogen balance could be accomplished. It was not determined whether the removal rate of ammonia at the highest loading could be sustained over a long period of time. Overall, the ammonia removal rates that were observed are high compared to the average elimination capacity reported in other biofiltration studies. Most authors report values between 5 and 40 $NH_3\,g\,m^{-3}\,h^{-1}$ for the critical loading (Chung et al., 1997; Kim et al., 2000; Smet et al., 2000; Sorial et al., 2001; Yani et al., 1998; Taghipour et al., 2008).

Pressure drop through the biotrickling filter remained low during the first 20 days (Fig. 4). However, it rose sharply thereafter due to biomass growth which was clearly visible and resulted in the partial plugging of the packed bed. Therefore, the packing material was removed from the reactor and washed on day 36. The rapid biomass growth was unexpected since nitrifying organisms are known to be slow growers with low biomass yields. The probable reason for excessive biomass growth was that during the first 20 days (i.e., prior to starting the liquid post-treatment), excess COD from the denitrification reactor was fed to the biotrickling filter via the trickling liquid, leading to significant growth of heterotrophic organisms in the biotrickling filter. Heterotrophic growth was even more pronounced when a higher concentration of mineral nutrients was fed to the reactor (after day 20), resulting in faster growth and rapid increase of pressure drop. After washing the packing day 36, a low-pressure drop was re-established (<1 cm w.g.) and the conditions in the reactor were such that the pressure drop remained low for the rest of the experiment.

In Fig. 5, the effects of continuous operation of the biotrickling filter on liquid concentrations of key nitrogen species are shown. Over time, fluctuations of the various nitrogen species were observed. As will be discussed below, some of major changes could be directly attributed to the operation of the bioreactor system. Some fluctuations were also probably due to the sensitivity of the system to small changes in operating conditions, in particular pH, which affects the different acid–base equilibria and the concentrations of various biological inhibitors, as was modeled by

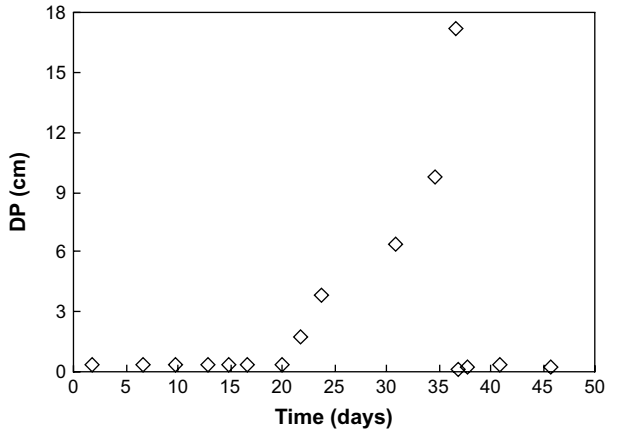

Fig. 4 – Pressure drop (in cm water gauge) over time in the biotrickling filter (EBRT = 13.5 s).

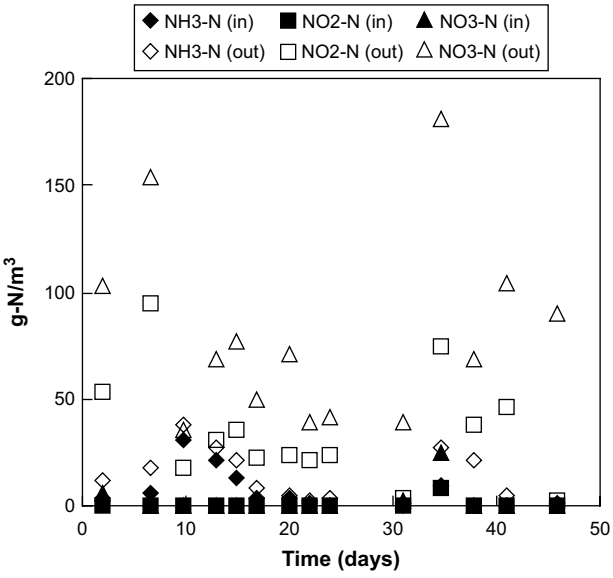

Fig. 5 – Nitrogen species in the influent (closed symbols) and effluent (open symbols) liquid streams of the biotrickling filter.

Baquerizo et al. (2007). Between day 10 and 20, 10–30 g N NH_3 m^{-3} were recycled back to the influent of the biotrickling filter indicating that incomplete nitrification occurred in the system. Thereafter, influent ammonium concentration remained low, probably because of the added nitrification capacity brought by the post-treatment and the aeration of the sump tank. Also, effluent ammonium concentration remained low indicating that a vast majority of the treated ammonia was nitrified in the biotrickling filter. Consequently, effluent nitrate and nitrite concentrations were high, usually ranging from 25 to 100 g N m^{-3}. The effluent pH decreased (not shown) as expected. In several occasions, nitrite concentrations rose to fairly high levels (~100 mg L^{-1}), raising concerns of possible inhibition of the nitratification (i.e., the second step in the nitrification process). Careful attention to the nitrite concentration is warranted since nitrite is toxic and its accumulation can potentially shut down all biological transformations in the process.

The nitrogen balance over the biotrickling filter was usually closed within 70–120%, i.e., all ammonia gas removed in the biotrickling filter could be accounted for in the different nitrogen species contained in the liquid effluent (there is no accumulation in the biotrickling filter). Altogether, the results in Figs. 5 and 6 indicate that ammonia was removed and biologically converted mostly by nitrification to nitrate and nitrite in the biotrickling filter.

In Figs. 7 and 8, the continuous operation of the denitrification reactor is shown. Nitrate and nitrite concentrations in the recycling liquid were usually close or below the detection limit after the denitrification reactor (Fig. 7). This is a requirement for the long term operation of the system, since accumulation of various N species such as HNO_2 is known to inhibit nitrification and have impacted earlier biofiltration investigations (Baquerizo et al., 2005; Buday et al., 1999; Demeestere et al., 2002; Dragt et al., 1987; Joshi et al., 2000). COD also decreased correspondingly during denitrification (Fig. 8) as expected, although there was some residual COD in the liquid. In general, denitrification is a relatively robust bioprocess, and does not pose too many challenges (Metcalf and Eddy, 2003). Here, no major issue was observed with the denitrification, besides the secondary problems caused by the residual COD when fed to the biotrickling filter resulting in odor and plugging, as mentioned earlier. These problems were resolved when post-treatment of the liquid was initiated prior to feeding the denitrified effluent to the biotrickling filter.

In Fig. 9, the continuous operation of the post-treatment reactor is reported. Ammonia and COD in the recycling liquid all decreased while nitrite and nitrate concentrations in the recycling liquid rarely increased. This indicates that the post-treatment reactor served its purpose. Because of the conditions in that reactor, it is likely that a combination of processes occurred. These include heterotrophic biodegradation of the residual COD (the average COD removal was about 60%, see Fig. 9 inset), possibly some denitrification in anaerobic pockets or deep in biofilms where oxygen limitation occurred, and nitrification of ammonia. Most importantly, the post-treatment reactor successfully removed the excess COD and possible traces of dissolved volatile odorous compounds which resolved

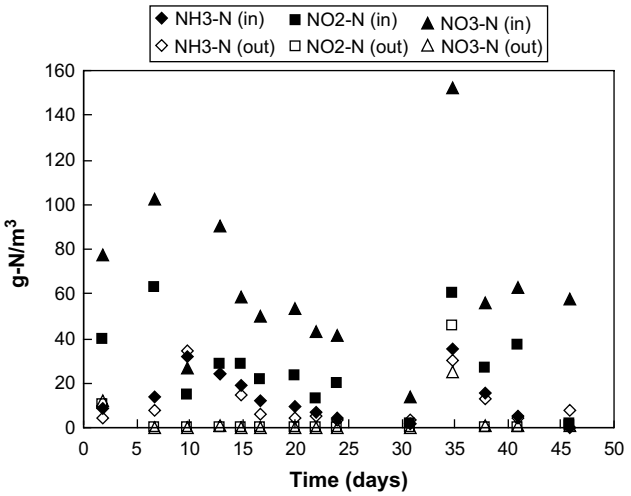

Fig. 7 – Influent and effluent nitrogen species in the denitrification reactor.

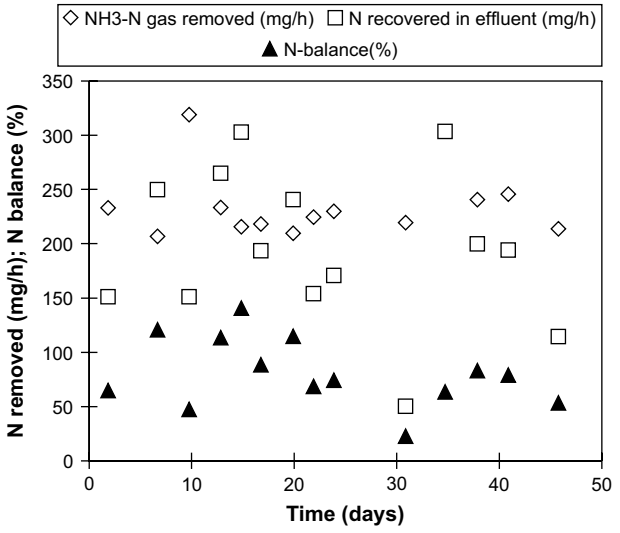

Fig. 6 – Nitrogen balance in the biotrickling filter.

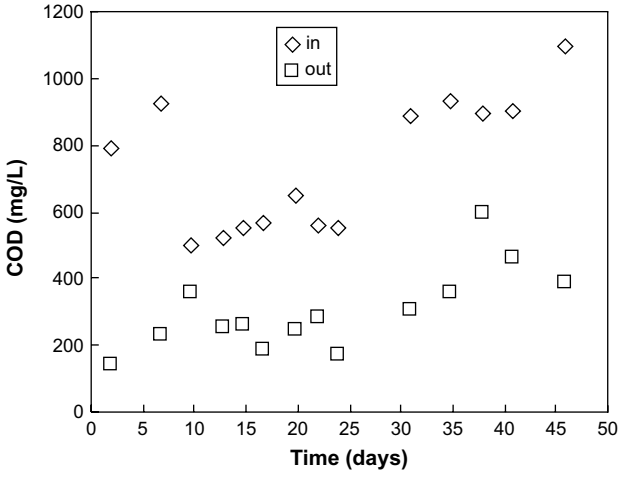

Fig. 8 – COD in the denitrification reactor.

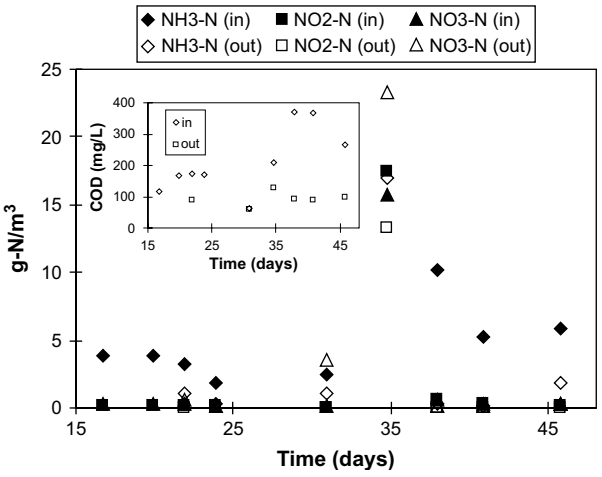

Fig. 9 – Influent and effluent N species for the post-treatment reactor. Ammonium inlet on day 35 (45 g N m^{-3}) is not shown on the graph. The inset shows COD influent and effluent concentrations.

subsequent odor and plugging problems in the biotrickling filter.

4. Conclusions

Overall, the results of this study demonstrate that ammonia vapors can be efficiently removed and converted to nitrogen gas in a biotreatment system comprising of a biotrickling filter, a denitrification reactor and a polishing bioreactor for the trickling liquid. Effective ammonia treatment was obtained in the biotrickling filter at a gas retention time of only 13.5 s. The critical load ranged from 60 to 120 g m^{-3} h^{-1} depending on the conditions and ammonia elimination capacities of about 60 g m^{-3} h^{-1} could be sustained over time. During short periods of high loadings the elimination capacity reached up to 120 g m^{-3} h^{-1}. These are all high specific performances compared to other biofiltration studies, which often only considered ammonia oxidation. In the present case, ammonia gas was sequentially nitrified and denitrified to nitrogen gas, thereby minimizing nitrate and nitrite contamination of any liquid effluent stream. This is certainly an advantage at a time where conservation of water is important. Obviously further optimization of the different bioreactor volumes and larger scale demonstration are needed before full-scale implementation of this treatment system is possible. Of particular relevance is optimizing the size of the liquid treatment system, understanding the limits for stable operation, and minimizing treatment costs, possibly by changing the COD source.

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