Nitrogen removal from high organic loading wastewater in modified Ludzack–Ettinger configuration MBBR system

Mojtaba Torkaman, Seyed Mehdi Borghei, Sepehr Tahmasebian and Mohammad Reza Andalibi

ABSTRACT

A moving bed biofilm reactor with pre-denitrification configuration was fed with a synthetic wastewater containing high chemical oxygen demand (COD) and ammonia. By changing different variables including ammonium and COD loading, nitrification rate in the aerobic reactor and denitrification rate in the anoxic reactor were monitored. Changing the influent loading was achieved via adjusting the inlet COD (956–2,096 mg/L), inlet ammonium (183–438 mg/L), and hydraulic retention time of the aerobic reactor (8, 12, and 18 hours). The overall organic loading rate was in the range of 3.60–17.37 $\frac{\text{g COD}}{\text{m}^2 \cdot \text{day}}$, of which 18.5–91% was removed in the anoxic reactor depending on the operational conditions. Considering the complementary role of the aerobic reactor, the overall COD removal was in the range 87.3–98.8%. In addition, nitrification rate increased with influent ammonium loading, the maximum rate reaching 3.05 $\frac{\text{g NH}_4}{\text{m}^2 \cdot \text{day}}$. One of the most important factors affecting nitrification rate was influent C:N entering the aerobic reactor, by increasing which nitrification rate decreased asymptotically. Nitrate removal efficiency in the anoxic reactor was also controlled by the inlet nitrate level entering the anoxic reactor. Furthermore, by increasing the nitrate loading rate from 0.91 to 3.49 $\frac{\text{g NO}_3}{\text{m}^2 \cdot \text{day}}$, denitrification rate increased from 0.496 to 2.47 $\frac{\text{g NO}_3}{\text{m}^2 \cdot \text{day}}$.

Key words | ammonium loading rate, denitrification rate, MBBR, nitrate loading rate, nitrification rate, organic loading rate

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INTRODUCTION

The presence of nutrients, including nitrogen, is one of the most detrimental factors deteriorating the water quality. Consequently, discharging industrial wastewater effluents high in chemical oxygen demand (COD) and nitrogen content into the surface and groundwater bodies may cause several issues such as eutrophication, toxicity, and oxygen depletion (Smith 2003; Luostarinen *et al.* 2006). Therefore, removal of total nitrogen (TN) is an important and necessary task in municipal and industrial wastewater treatment plants.

Combination of biological nitrification and denitrification is one of the most commonly used approaches for TN removal. The former is more cost-effective than physical and chemical methods and is more environmentally friendly (Ruiz *et al.* 2003; Behera *et al.* 2007; Fakhru'l-Razi *et al.* 2009). During nitrification process, the inlet ammonium entering the aerobic reactor oxidizes to nitrite and nitrate in the presence of oxygen (Wang *et al.* 2006). These compounds convert into nitric oxide (NO) and nitrous oxide (N_2O), respectively, and finally into nitrogen gas (N_2), in an anoxic reactor in the absence of oxygen. All these compounds are gaseous and may be released into atmosphere (Sedlak 1991; Tchobanoglous *et al.* 2003). Therefore, regarding the different operational conditions in the aerobic and anoxic reactors, it is preferred to carry out two processes in separate reactors (Wang *et al.* 2006).

Biological processes based on suspended growth biomass, such as activated sludge plants, have been favorably used for nutrient removal, in the past decades. However, large reactor tanks, settling of flocs, sludge returning requirements, and other factors have limited their use (Pastorelli *et al.* 1997a, b, 1999). Biofilm processes are preferred especially in cases where the presence of organisms

doi: 10.2166/wst.2015.343

with limited growth (e.g., nitrifiers) is necessary in the system. Nonetheless, disadvantages such as channeling and clogging may worsen the system performance (Broch-Due *et al.* 1997).

Hence, moving bed biofilm reactors (MBBRs) were developed in the late 1990s in Norway in order to take advantage of the positive features from attached growth systems (compactness, simplicity of operation, and stable removal efficiency) without their limitations (channeling, need of periodic backwashing, clogging, pressure drop, and distribution of the load on the whole carrier surface) (Hem *et al.* 1994; Ødegaard 1999; Rusten *et al.* 2006).

MBBR systems are continuously mixed reactors, which contain small plastic elements – the so-called carriers – that provide a large surface for biofilm growth (Martín-Pascual *et al.* 2013). The carriers provide a large surface area for biofilm growth with no need for biomass recycling. The current investigations indicate unique features of MBBR systems, including high biomass levels, superior oxygen transfer, higher organic loading rate, larger surface area facilitating the mass transfer, strong tolerance to loading impact, smaller volume, shorter hydraulic residence time (HRT), and eliminating the sludge bulking phenomenon. These benefits may be employed in order to improve the performance of activated sludge systems with overloading without adding a new tank (Ødegaard 1999; Tchobanoglous *et al.* 2003; Jing *et al.* 2009).

Industrial wastewaters include high loadings of organic and nutrient materials and hence their treatment methods are different from municipal wastewaters. One of the main factors that have to be considered in nitrogen removal processes is the influent condition, the organic and nutrient loadings of which may affect the competition between heterotrophic bacteria and nitrifiers (Hanaki *et al.* 1990; Cheng 1994). In this respect, the inlet C:N ratio in the nitrification process changes the population distribution of heterotrophic bacteria and nitrifiers in the biofilm layer as well as their oxygen and substrate consumption (Nogueira *et al.* 2002). On the other hand, in order to reduce the oxidized nitrogen compounds (e.g., nitrate and nitrite) inside the anoxic reactor, heterotrophic denitrifier bacteria need a suitable organic carbon compound to act as electron donor as well as energy source (Chudoba *et al.* 1998). As a result, biological nitrification-denitrification processes with pre-denitrification configuration are more suitable for nitrogen removal from wastewaters with high organic and nutrient loading.

Considering the promising features of MBBR systems in COD removal from industrial wastewaters and also the importance of treating wastewaters with high COD and ammonia level, the current study aims at investigating the simultaneous nitrogen and organic carbon removal from various high COD and ammonia wastewaters in a pre-denitrification MBBR system. The simultaneous effect of ammonium and organic loading on organic removal, the nitrification process, the denitrification process and also the complementary role of these processes inside the two aerobic and anoxic reactors were studied. More specifically, we investigated concurrent COD and nitrogen removal from high organic and ammonia wastewaters in an MBBR system consisting of an anoxic and an aerobic reactor with pre-denitrification configuration.

METHODS

Experimental setup

In the current work, a laboratory-scale aerobic reactor (14.4 L) which follows an anoxic reactor (7.2 L) devised in the form of modified Ludzack–Ettinger process with pre-denitrification configuration has been utilized.

A peristaltic pump was used to introduce the synthetic feed into the anoxic reactor (followed by the aerobic reactor). Roughly twice the influent flow rate was refluxed from the aerobic reactor outlet back to the anoxic reactor inlet using a pump. A sedimentation tank was also devised downstream of the MBBR reactors in order to separate the effluent from the sludge and also for sampling from the treated wastewater. Considering the flow rate range studied here, HRT and hydraulic loading rate varied in the ranges 1.5–3.375 hours and 6–13.5 $\frac{m^3}{m^2 \cdot day}$, respectively.

In the current investigation, biofilm carriers were made of polyethylene (density 0.96 g/cm^3), in the form of small cylinders (diameter = 1.2 cm; length = 1.4 cm) with cross fins on the internal surface, and longitudinal fins on the external surface. The specific surface area of these carriers was 480 m^2 per cubic meter bulk volume. Considering the ~50% filling fraction ($V_{carriers}/V_{reactor}$) in both reactors, the real specific surface area of the carriers would be 240 m^2/m^3 reactor.

Biofilm carrier movement inside the aerobic reactor was induced by porous diffusers placed at the bottom of the reactor. This type of diffuser prevents stagnant volume formation inside the reactor while providing good oxygen transfer into the liquid phase. In the anoxic reactor, a mechanical

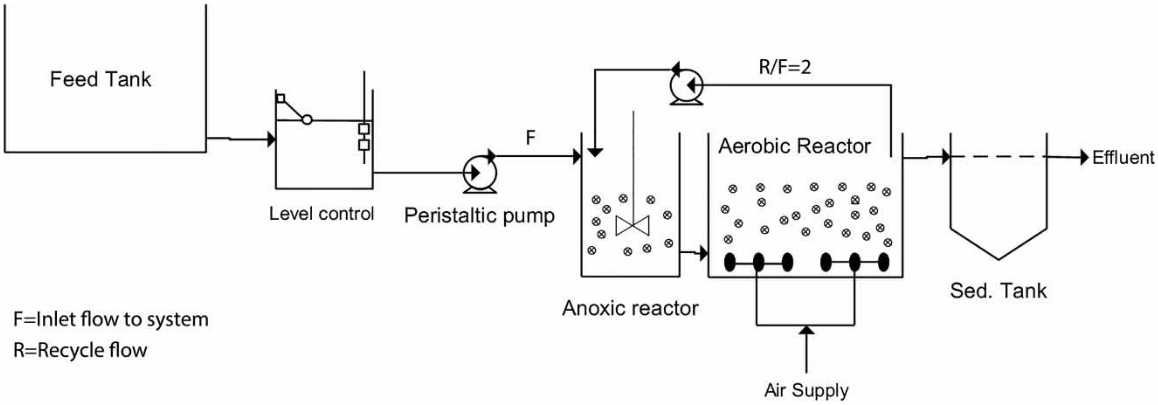

Figure 1 | Schematic diagram of the bench-scale MBBR system.

impeller with a speed of 50–60 rpm was employed to circulate the carriers. The laboratory-scale moving bed biofilm reactors are schematically shown in Figure 1.

Wastewater characteristics

In order to facilitate investigating the system efficiency at different inlet COD and ammonium concentrations, the influent wastewater was synthesized artificially. In this respect, molasses was used as the carbon source (COD provider) while urea, NH_4Cl, and $(NH_4)_2SO_4$ were the ammonium providers, and KH_2PO_4 and K_2HPO_4 were phosphate sources. As the pH value affects both nitrification and denitrification processes, in order to adjust the pH value $NaHCO_3$ was also used in addition to KH_2PO_4. Other micronutrients including Fe, Mg, Na, Ca, S, and Cl were also supplemented using $MgSO_4$, NaCl, $CaCl_2$, $MnCl_2$, and $FeCl_3$. Table 1 summarizes the detailed properties of the influent feed in the operational range investigated here.

Table 1 | Properties of the influent feed

Variable	Value
COD (mg/L)	956–2,096
NH_4-N (mg/L)	183–438
NO_3 (mg/L)	0–7
$MgSO_4$ (mg/L)	2–3
$FeCl_3$ (mg/L)	0.4–0.6
NaCl (mg/L)	1
$CaCl_2$ (mg/L)	0.5

Experimental procedure

The operation of MBBR reactors was started by inoculating with activated sludge derived from the aeration tank of Ekbatan Wastewater Treatment Plant (Tehran, Iran). A 50-day period for the startup and biofilm growth on the external surface of carriers was allowed. After the startup period, the continuous step was conducted in the aerobic reactor at three different HRTs of 18, 12, and 8 hours in a reducing stepwise manner. To provide the desired HRT value, the peristaltic pump was set to feed the synthetic wastewater at different rates of 19.2, 28.8, and 43.2 L/day, respectively (see Table 2 for details). For each value of hydraulic retention time, the inlet COD and ammonium

Table 2 | The time necessary for conducting various tests at each HRT value

Hydraulic loading rate (L/day)	Aerobic reactor HRT (hours)	Reflux flow rate from aerobic to anoxic reactor (L/day)	Anoxic reactor HRT (hours)	Number of tests carried out at each HRT value	Time allowed for reaching steady state conditions (days)	Total time period for each HRT (days)
19.2	18	38.4	3	12	12	144
28.8	12	57.6	2	12	11	132
42.2	8	86.4	1.33	12	10	120

level were changed from 956 mg/L to 2,096 mg/L and 183 mg/L to 430 mg/L (12 different permutations at each HRT value), respectively, and upon each change several days were allowed for the system to reach steady state conditions. In this way, the effects of three variables, i.e., HRT, inlet organic matter concentration, and inlet ammonium concentration were assessed by organic removal efficiency, as well as nitrification and denitrification rates. Table 2 summarizes the experimentation time in order to conduct the set of experiments at various hydraulic loading rates and corresponding retention times in each reactor.

During the continuous experiments, the dissolved oxygen (DO) concentration inside the aerobic reactor was ~5–6 mg/L and that in the anoxic reactor was 0.2–0.4 mg/L. Moreover, the pH in the aerobic reactor was kept at ~7.5 while it was kept between 7.2 and 7.6 inside the anoxic reactor. Temperature was controlled using a heater maintaining the system temperature at a value of 26 °C.

Analytical methods

In order to evaluate the effect of alterations, samples were derived from the inlet and outlet of both reactors and were analyzed immediately after filtering through 0.45 μm filter paper. Soluble COD, ammonium (NH_4^+-N), nitrate (NO_3^--N) and nitrite (NO_2^--N) concentrations were measured according to standard methods (Clesceri *et al.* 1998). During and throughout the experiments, the operational variables such as temperature, DO, and pH were kept constant. DO and pH were measured in each bioreactor using a 'YSI 55' DO meter (YSI Company, Inc., USA) and a pH electrode (Metrohm, Swaziland), respectively.

RESULTS AND DISCUSSION

After the startup period, the system operation was assessed under different influent conditions, in order to evaluate the extent of organic matter removal as well as nitrification and denitrification rates. In these experiments the inlet COD, inlet ammonium, and HRT were assumed as variables. In the following sections, the overall COD removal efficiency, aerobic nitrification rate and finally anoxic denitrification rate, along with parameters affecting them, were investigated.

Organic removal

The inlet COD concentration was in the range of 956–2,096 mg/L and the influent flow rate was between 19.2 and 43.2 L/day. The former gave rise to organic loading rates in the range of 3.6–17.37 $\frac{\text{g COD}}{\text{m}^2 \cdot \text{day}}$ (0.8–4.2 $\frac{\text{kg COD}}{\text{m}^3 \cdot \text{day}}$; based on total reactor volume). In this system, the influent feed first entered the anoxic reactor, and, depending on the inlet nitrate level and the operational conditions of the anoxic reactor, 18.5–91% of inlet COD was removed. Hence, the effluent COD, which is in turn an influent to the aerobic reactor, was lower than the system's primary inlet COD, and the residual COD was removed in the aerobic reactor. Figure 2 demonstrates the COD removal rate versus inlet organic loading under different operational conditions. According to this figure, the COD removal efficiency with respect to organic loading was very high, in the range of 87.3–98.8%. In the current reactor configuration, we behold that the complementary role of the aerobic reactor damps down the effect of loading variations on COD removal rate. In addition, there is roughly a linear relationship between the loading rate and COD removal rate. Previously, Pastorelli *et al.* (1997a) showed an analogous linear relation between the inlet organic loading rate and COD removal rate in MBBR systems with inlet loading less than 8 $\frac{\text{g COD}}{\text{m}^2 \cdot \text{day}}$. One reason behind the high COD removal might be the high feed degradability, beside the high concentration and activity of biomass in the MBBR reactor, the latter being a result of using constantly circulating, high-surface-area carriers (Andreottola *et al.* 2002; Ødegaard *et al.* 2004; Rusten *et al.* 2006). In this

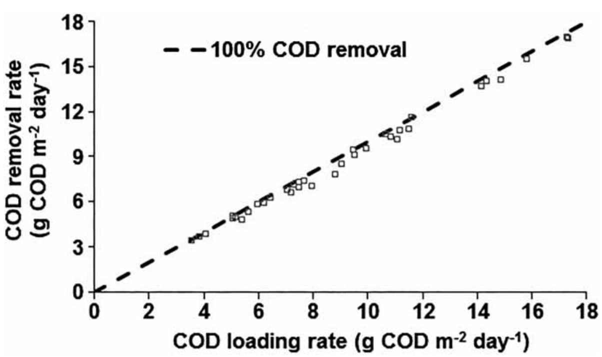

Figure 2 | COD removal rate versus inlet COD loading under different operational conditions.

regard, increasing the organic loading enhanced the attached biomass growth, which in turn led to an increased organic consumption. Corroborating previous studies on COD removal by MBBR systems imply that the former technology could tolerate high organic loadings as a result of possessing high specific surface area and also constant packing circulation (Aygun *et al.* 2008; Chen *et al.* 2008).

Nitrification

Nitrification rate is mainly a function of three factors, namely organic loading, ammonium loading, and DO. To assess the effect of these factors on nitrification rate in the aerobic reactor, the inlet COD concentration was varied between 251 and 1,800 mg/L, ammonium concentration between 183 and 438 mg/L, and HRT between 8 and 18 hours. Considering the constant reactor volume, hydraulic retention time was controlled by changing the inlet flow rate. Nitrification rates were calculated based on the inlet and outlet ammonium concentrations, inlet flow rate, and biofilm surface area in the aerobic reactor.

Effect of ammonium loading rate on nitrification rate

To evaluate the effect of inlet ammonium concentration and HRT on nitrification rate, the simultaneous effects of these two parameters were studied. By increasing either the inlet ammonium concentration (concentration loading) or the inlet flow rate (hydraulic loading), ammonium loading rate would increase. Figure 3 illustrates nitrification rate as a function of ammonium loading rate at constant inlet COD loading rates. As is obvious, at all the inlet COD loading rates, nitrification rate increases with inlet ammonium loading rate. By increasing the inlet ammonia concentration, its removal efficiency in the aerobic reactor was only slightly lessened, which is due to the nitrification rate being at its maximum kinetic capacity. In addition, by reducing the HRT (via increasing the inlet flow rate), ammonia removal would be less in the aerobic reactor as a result of limited time for the nitrifying bacterial activity. Hence, by either increasing the inlet ammonia or reducing the HRT, ammonia removal declines. Nonetheless, since nitrification rate is proportional to the product of ammonia concentration and inlet flow rate, increasing ammonia loading rate entering the aerobic reactor would promote the nitrification rate. The results from the article by Gupta & Gupta (2001) reporting nitrogen removal from wastewater in an aerobic rotating biological contactor (RBC) system are completely in line with our observations.

The effect of COD:N ratio on nitrification rate

Organic substrate concentration is one of the most essential factors affecting the nitrification process (Hanaki *et al.* 1990; Cheng 1994). In fact, the distribution of heterotrophic and nitrifying bacteria inside the biofilm layer, as well as oxygen and substrate diffusion into the layer, controls the nitrification rate (Rusten *et al.* 1995).

Figure 4 depicts nitrification rate in the aerobic reactor versus the influent COD:N ratio. The beheld reduction in nitrification rate may be analyzed from two different aspects. First of all, based on the previous studies, the growth rate is different in heterotrophic bacteria than in nitrifiers. Grady *et al.* (2012) state that the maximum growth rate of heterotrophic bacteria is roughly five times that of nitrifiers. Besides, at high inlet COD loading rates, heterotrophic bacteria grow faster than nitrifiers and a higher fraction of biofilm is occupied by heterotrophs (Harremoës 1982; Rusten *et al.* 1995). Satoh *et al.* (2000) investigated the relation between organic loading and the extent of ammonium oxidation and deduced that by raising the organic loading, as a result of reduced nitrifier population on the external surface of biofilms, the nitrification rate decreases. Likewise, by increasing the organic loading entering the aerobic reactor, nitrifying bacteria tend to accumulate at the bottom layer of biofilms, which limits the oxygen availability for these bacteria (Jing *et al.* 2009).

According to our results and documented studies, we find that by increasing the inlet organic loading entering the aerobic reactor, the space and dissolved oxygen available for the nitrifying bacteria would be less, which hinders ammonia oxidation and nitrification. In this regard, the maximum nitrification rate in the aerobic reactor at $COD{:}N = 1$ was $3.05\ \frac{g\ NH_4}{m^2 \cdot day}$.

In addition, according to our findings, the inverse relation between nitrification rate and inlet COD:N ratio is asymptotically decreasing and at higher COD:N ratios, nitrification rate does not change appreciably. DO concentration in the biofilm layer could be the reason behind such an asymptotic behavior. Investigations by Zhang *et al.* (1994) support this idea. In their studies, in order to examine the competitive behavior between the heterotrophic and nitrifying bacteria in using the space and substrate, they applied a microelectrode technique which

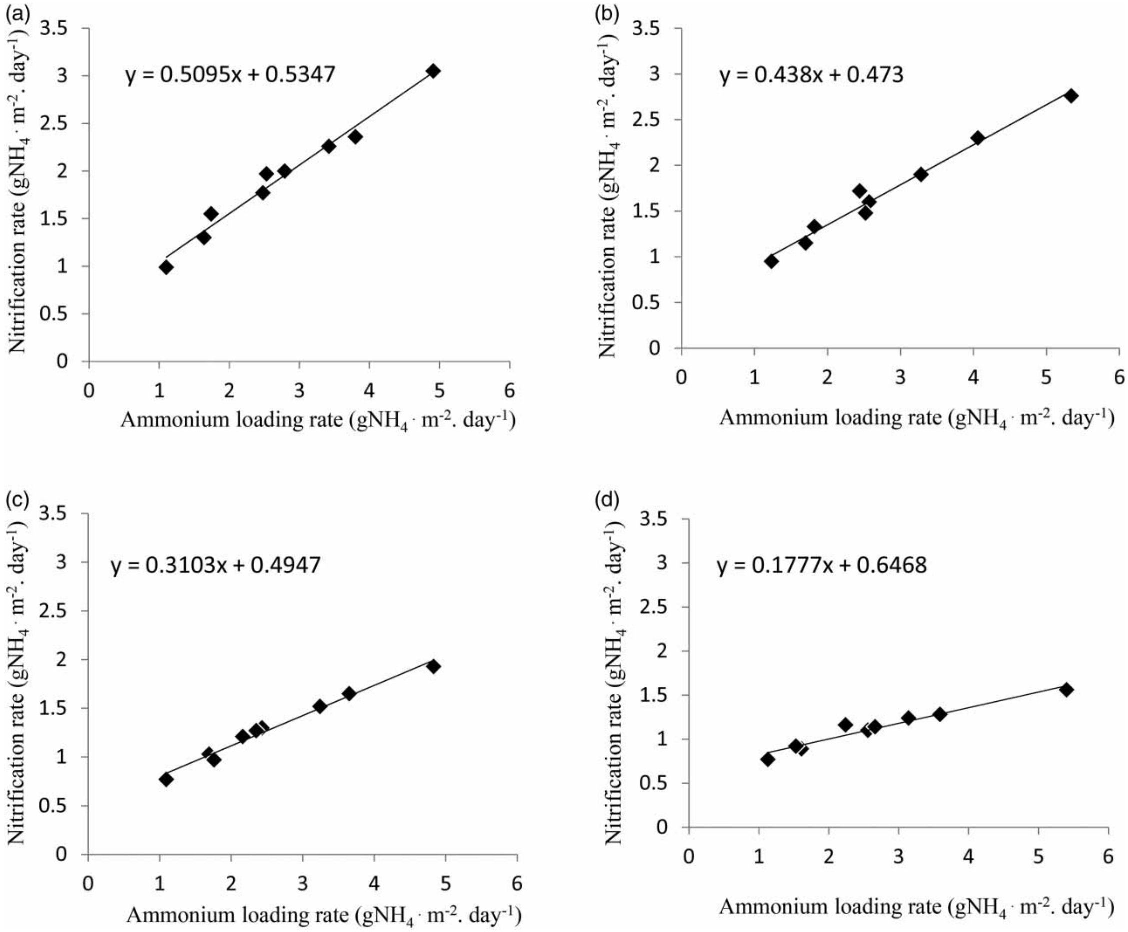

Figure 3 | Nitrification rate versus ammonium loading rate in aerobic reactor for (a) COD loading rate = 4.9 g COD m^{-2} day^{-1} ; (b) COD loading rate = 6.68 g COD m^{-2} day^{-1}; (c) COD loading rate = 10.43 g COD m^{-2} day^{-1}; (d) COD loading rate = 12.94 g COD m^{-2} day^{-1}.

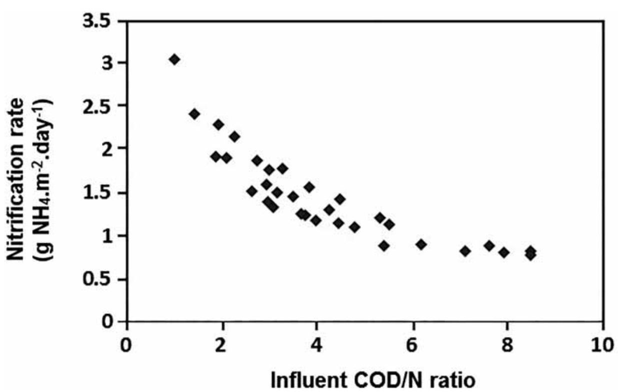

Figure 4 | Nitrification rate versus influent $\frac{COD}{N}$ ratio in aerobic reactor.

showed that by increasing the inlet organic loading, oxygen consumption by heterotrophic bacteria residing on the biofilm surface increases while surface DO concentration diminishes. Furthermore, when the organic loading exceeds a specific value, the DO profile remains unchanged inside the biofilm layer (Zhang *et al.* 1994). In addition to these studies, Ling and Chen examined nitrification in industrial wastewaters using three different biofilters and showed that by increasing the inlet COD:N ratio entering the aerobic reactor, nitrification rate drops asymptotically (Ling & Chen 2005). In another work, Carrera *et al.* (2004) used an activated sludge system for treating industrial wastewater and demonstrated the same descending asymptotic behavior.

Denitrification

The biological denitrification process refers to the reduction of nitrate and nitrite by heterotrophic bacteria in an anoxic reactor. The denitrification rate could be controlled by changing the nitrate loading, quality and quantity of the inlet organic loading, and oxygen concentration (or, instead, the presence of oxygen) in the anoxic reactor (Aspegren *et al.* 1998; Chudoba *et al.* 1998; Ødegaard 1999). In this study, based on the inlet flow rate and the reflux ratio from the aerobic reactor to the anoxic reactor, the performance of the anoxic reactor was evaluated at three different HRT values of 1.33, 2, and 3 hours.

In biological nitrification/denitrification systems, nitrate and nitrite produced inside the aerobic reactor enter the anoxic reactor, in which the NO_X loading rate depends on nitrification rate inside the aerobic reactor. In this respect, the inlet nitrate loading to the anoxic reactor increases with nitrate produced inside the aerobic reactor, which in turn increases with aerobic nitrification rate. Considering the high DO concentration inside the aerobic reactor, the amount of nitrite produced in the current study was pretty low. Figure 5 shows anoxic denitrification rate (in $\frac{g\ NO_3}{m^2 \cdot day}$) as a function of inlet nitrate loading rate ($\frac{g\ NO_3}{m^2 \cdot day}$) at various HRTs. Nitrate loading rate and denitrification rate were calculated using the inlet and outlet nitrate concentrations, influent flow rate, and biofilm surface area inside the anoxic reactor. Besides, nitrate loading rate was controlled by changing the inlet nitrate concentration (concentration loading) or inlet flow rate (hydraulic loading). As can be seen, in all the HRT values, denitrification rate increases linearly with nitrate loading rate. In fact, by increasing the inlet nitrate loading rate, heterotrophic bacteria become more active, which in turn increases denitrification rate. Moreover, at the same loading rates, by lowering the HRT (especially at HRT = 1.33 hours), due to hydraulic limitation, the activity of denitrifying bacteria lessens, and consequently the denitrification rate drops. As is shown there, the minimum denitrification rate was 0.496 $\frac{g\ NO_3}{m^2 \cdot day}$ (0.119 $\frac{kg\ NO_3}{m^3 \cdot day}$) at a loading rate of 0.91 $\frac{g\ NO_3}{m^2 \cdot day}$ (0.218 $\frac{kg\ NO_3}{m^3 \cdot day}$), while the maximum denitrification rate was 2.47 $\frac{g\ NO_3}{m^2 \cdot day}$ (0.593 $\frac{kg\ NO_3}{m^3 \cdot day}$) at a loading rate of 3.94 $\frac{g\ NO_3}{m^2 \cdot day}$ (0.946 $\frac{kg\ NO_3}{m^3 \cdot day}$). Earlier studies on denitrification also indicated the same direct linear relation between denitrification rate and nitrate loading rate in anoxic reactors (Chudoba *et al.* 1998; Kermani *et al.* 2008).

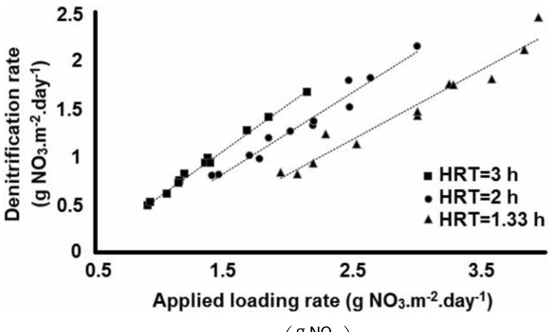

Figure 5 | Anoxic denitrification rate $\left(\frac{g\ NO_3}{m^2.day}\right)$ as a function of inlet nitrate loading rate $\left(\frac{g\ NO_3}{m^2.day}\right)$ at various retention times.

CONCLUSIONS

The current study revolves around the application of a continuous MBBR system with pre-denitrification configuration. This was used for simultaneous treatment of nitrogen and COD from a synthetic wastewater with high COD and ammonium concentration. The effects of COD concentration, ammonium concentration, and HRT on nitrification and denitrification rates were appraised. Considering the organic loading applied to the overall system (3.60–17.37 g$\frac{COD}{m^2 \cdot day}$), COD removal was in the range of 87.3–98.8%. In addition to high biodegradability of the inlet feed, maintaining the active biomass on the high specific surface area of the packings present inside the MBBR system led to significant COD removal, in a sense that increasing the influent organic loading would enhance attached biomass growth, ensuing faster organic consumption. The inlet C:N ratio entering the aerobic reactor was among the important factors affecting both ammonium removal and nitrification rate. By increasing this ratio, heterotrophic bacteria occupied a higher fraction of the biofilm surface and hence, nitrifiers had less access to DO and substrate. Thus, the nitrification rate dropped asymptotically.

Increasing the nitrate loading rate entering the anoxic reactor promoted the activity of heterotrophic denitrifying bacteria. This gives rise to an almost linear, direct relationship between nitrate loading rate (entering the anoxic reactor) and denitrification rate. Quantitatively, increasing the nitrate loading rate from 0.91 to 3.49 $\frac{g\ NO_3}{m^2 \cdot day}$ increases the denitrification rate from 0.496 to 2.47 $\frac{g\ NO_3}{m^2 \cdot day}$.

In summary, inlet COD was among the most critical factors affecting nitrogen removal from the wastewater studied here. Concerning the former, consuming a significant portion of COD in the anoxic reactor lowers the C:N ratio in the influent entering the aerobic reactor, which in turn enhances nitrification rate in the latter reactor. Enhanced nitrification boosts the influent nitrate entering the anoxic reactor, which brings about a faster denitrification.

ACKNOWLEDGEMENT

We are grateful to the Ekbatan Wastewater Treatment Plant (Tehran, Iran) for providing the initial activated sludge.

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First received 4 March 2015; accepted in revised form 22 June 2015. Available online 7 July 2015