Inorganic nitrogen removal of toilet wastewater with an airlift external circulation membrane bioreactor

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Abstract

Removal of inorganic nitrogen (inorganic-N) from toilet wastewater, using a pilot-scale airlift external circulation membrane bioreactor (AEC-MBR) was studied. The results showed that the use of AEC-MBR with limited addition of alkaline reagents and volumetric loading rates of inorganic-N of 0.19–0.40 kg inorganic-N/(m³·d) helped achieve the desired nitrification and denitrification. Furthermore, the effects of pH and dissolved oxygen (DO) on inorganic-N removal were examined. Under the condition of MLSS at 1.56–2.35 g/L, BOD₅/ammonia nitrogen (NH₄⁺-N) at 1.0, pH at 7.0–7.5, and DO at 1.0–2.0 mg/L, the removal efficiencies of NH₄⁺-N and inorganic-N were 91.5% and 70.0%, respectively, in the AEC-MBR. The cost of addition of alkaline reagent was approximately 0.5–1.5 RMB yuan/m³, and the energy consumption was approximately 0.72 kWh/m³ at the flux of 8 L/(m²·h).

Key words: inorganic nitrogen removal; treatment of wastewater from toilet; airlift external circulation membrane bioreactor (AEC-MBR); membrane bioreactor (MBR)

Introduction

Wastewaters from the toilets, when released into water bodies, are extremely harmful to the aquatic environment because they have high concentrations of suspended solids, uncountable number of microorganisms (including fecal bacteria, pathogenic bacteria, and even viruses) and large quantities of ammonia and organic pollutants (Tananka, 1997; Zeeman and Lettinga, 1999). Once wastewater from toilets is discharged directly into surface waters, serious water pollution occurs and increases the incidence of epidemic diseases. For this reason, the disposal and treatment of wastewater from toilets has always been an important issue of concern in many countries for the prevention of water pollution and protection of water bodies.

To eliminate the pollution caused by wastewater from toilets, various treatment processes, including biodegradation and membrane processes, have been developed to treat the wastewater from toilets. From 1960 to 1970, the main method that was used to treat the concentrated toilet wastewater was to treat the liquid of that diluted approximately 20 times with a process of an anaerobic or an aerobic digestion unit followed by an activated sludge process unit. The two-stage biological denitrification process that involved an approximately 10-time dilution of the wastewater was widely used in the later half of the 1970s. At the same time, many advanced technologies were applied to treat toilet wastewater, such as combined

processes involved coagulation sedimentation, ozone oxidation, sand filtration, and activated carbon absorption. In the early 1980s, the first high-load UF membrane separation facility for the treatment of toilet wastewater was put into operation in Japan. From then on, the facilities with membrane and membrane bioreactors (MBRs) were studied and novel facilities were rapidly developed to treat the toilet wastewater (Magara and Itoh, 1991; Magara *et al.*, 1992; Misaki and Matsui, 1996). MBRs were much simple processes that replaced the complex treatment processes incorporated with denitrification, coagulation, filtration, and activated carbon treatment (Magara and Itoh, 1991; Manem, 1996). In recent years, some reports showed that MBRs were used to treat toilet wastewater and gray water on cruise ships, such as the Copa MBR Technology[®] and the ZeeWeed[®] MBR of ZENON's system (Bentley and Ballard, 2000; OOZE, 2001).

Ammonia nitrogen (NH₄⁺-N) is an important pollutant in the treatment of toilet wastewater. Although many MBR systems could efficiently eliminate nitrogen, the processes of elimination of nitrogen was always time consuming and complex. MBRs for nitrogen removal from toilet wastewater should have higher nitrogen loading rates, higher nitrogen removal efficiency and lower energy consumption. Since 2000, Xu and Fan (2003) studied the treatment and reuse of toilet wastewater using a novel and simple process named airlift external circulation membrane bioreactor (AEC-MBR). Although a significant reduction in the chemical oxygen demand (COD) <47 mg/L and biochemical oxygen demand (BOD₅) <8.5 mg/L in the effluent was obtained using the AEC-MBR, the

ammonia in the effluent, <20 mg/L, was higher than water reuse standard and the removal efficiency of ammonia, 60%–81.9%, was also lower than required. With the establishment of a stricter standard, higher removal efficiency of $\text{NH}_4^+\text{-N}$ is required and more studies will be necessary to find optimum operation parameters and conditions for obtaining higher removal efficiency of $\text{NH}_4^+\text{-N}$ from toilet wastewater. However, few studies have been carried out with this objective.

In this study, the removal of inorganic nitrogen (inorganic-N) from toilet wastewater was investigated using an AEC-MBR on a scale of 10 m³/d. The objectives of this study were to investigate the performance of AEC-MBR, determine characteristics of nitrification and denitrification in the AEC-MBR, to examine the effects of operation parameters, such as pH and dissolved oxygen (DO), on the removal of inorganic-N, to determine the optimum values of the operation parameters, and to analyze energy consumption and cost of the AEC-MBR for inorganic-N removal. In addition, the characteristics of microorganisms in the AEC-MBR were also discussed.

1 Materials and methods

1.1 System description

Fig.1 shows the pilot-scale AEC-MBR system. The system with a capacity of 10 m³/d was installed at a public toilet in Beijing Zoo. The whole system of AEC-MBR mainly comprised the adjustment tank, acidification tank, AEC-MBR tank, and effluent tank, with working volumes of 6.7, 7.3, 10.2, and 6.8 m³, respectively, in which the aeration tank and membrane tank composed the AEC-MBR.

A septic tank was positioned ahead of the AEC-MBR system. From the septic tank, the raw wastewater from the toilet flowed through the acidification tank with filling materials and then into the adjustment tank. A level sensor fixed in the membrane tank was used to switch on the feed pump in the adjustment tank to maintain the water level of the AEC-MBR. In the AEC-MBR, a down-circulation pipe was fixed between the aeration tank and membrane

tank. The mixed liquids flowed over the weir between the membrane tank and the aeration tank and then flowed back to the membrane tank through the circulation pipe. The circulation flow of mixed liquids was driven by the density difference between the membrane tank and aeration tank resulting from the air blown from the aeration pipes under the membrane modules. In addition to maintaining circulation, the airflow in the membrane tank created a shear force to scour membrane fibers to control the membrane fouling. The effluent was drawn from the membrane modules and then sent to the effluent tank by a suction pump. A vacuum meter was mounted to monitor the transmembrane pressure (TMP). The total filtration area of hollow fiber membrane modules was 50 m². Table 1 shows the specifications of the AEC-MBR.

1.2 Toilet wastewater

Table 2 shows the characteristics of wastewater from the toilet, which was fed to the AEC-MBR system.

1.3 Experimental procedure

In order to enhance the removal efficiency of $\text{NH}_4^+\text{-N}$ as well as to investigate the effects of pH and DO on inorganic-N removal, the experiments were divided into 3 stages for nearly 6 months from Dec. 2003 to Jun. 2004. Table 3 summarized the operational parameters

Fig. 1 Schematic of AEC-MBR system for treatment of toilet wastewater. (1) septic tank; (2) acidification tank; (3) adjustment tank; (4) feed pump; (5) aeration pipe; (6) aeration tank; (7) circulation pipe; (8) overflow weir; (9) air compressor; (10) membrane module; (11) membrane tank; (12) suction pump; (13) effluent tank; (14) closestool; (15) tap water; (16) overfalling hole.

Table 1 Specifications of the AEC-MBR

Membrane		Module		Operation conditions	
Material	Polyvinylidene fluoride (PVDF)	Surface area (m ²)	50	HRT of the AEC-MBR (h)	20
Structure of membrane	Hollow fiber	Length (m)	1.5	SRT (d)	30–60
Pore size (μm)	0.2	Flux (L/(m ² ·h))	8.0	MLSS (g/L)	1.01–2.35
		TMP (MPa)	≤0.04	Load (kg $\text{NH}_4^+\text{-N}$ /(m ³ ·d))	0.18–0.39
				Load (kg Inorganic-N/(m ³ ·d))	0.19–0.40
				Load (g COD/(g MLSS·d))	0.27–0.87

Table 2 Characteristics of wastewater from the toilet

		COD (mg/L)	BOD ₅ (mg/L)	$\text{NH}_4^+\text{-N}$ (mg/L)	$\text{NO}_2^-\text{-N}$ (mg/L)	Inorganic-N (mg/L)	Alkalinity (CaCO ₃ mg/L)
Influent	Max.	665.2	323.3	317.6	18.3	335.9	1495.2
	Min.	211.0	149.7	149.7	1.3	151	1052.6
	Average	466.2	275.9	241.5	9.0	250.5	1283.4
Effluent	Average	229.5	8.81	8.31	70.26	78.57	228.5

Table 3 Operation parameters at the three experimental stages

Experimental stage	I	II	III
Time (d)	1–56	57–74	75–126
Hydraulic retention time (h)	20	20	20
Load (kg NH ₄ ⁺ -N/(m ³ ·d))	0.18–0.35	0.24–0.39	0.29–0.36
Load (kg inorganic-N/(m ³ ·d))	0.19–0.36	0.23–0.40	0.29–0.37
pH	5.58–6.02	7.0–7.5	7.0–7.5
DO (mg/L)	5.0–8.1	5.0–8.1	1.0–2.0
Sludge concentration (g SS/L)	1.01–1.79	1.37–1.95	1.56–2.35
BOD ₅ /NH ₄ ⁺ -N	0.54–1.09	1.5–1.85	1.01–1.45

at three experimental stages. In the whole experiment, the volumetric loads of NH₄⁺-N and inorganic-N were maintained in a range of 0.18–0.39 kg/(m³·d) and 0.19–0.40 kg/(m³·d), respectively. The pH of mixed liquid was adjusted by adding alkalinity to the influent of the system and the DO in mixed liquid was controlled by the airflow blew to the aeration tank and the membrane tank.

1.4 Analysis methods

The performance of the AEC-MBR system was monitored by the study of the following parameters: COD, BOD₅, inorganic-N (NH₄⁺-N, NO₃⁻-N and NO₂⁻-N), DO, pH, MLSS, and alkalinity. The samples were taken from influent, mixed liquid, and effluent of the AEC-MBR. The methods and instruments used in the experimental analyses are shown in Table 4.

Table 4 Methods and instruments used for experimental analysis

Parameter	Methods and instruments
COD	CTL-12 COD meter (Huatong Company, China)
BOD ₅	BODTrak™ (Hach Company, USA)
NH ₄ ⁺ -N	Nessler's reagent colorimetric method
NO ₃ ⁻ -N	Spectrophotometric method with phenol disulfonic acid
NO ₂ ⁻ -N	Spectrophotometric method
DO	Models 810Aplus dissolved oxygen meter (USA)
pH	pH meter (pHB-4, China)
MLSS	Weight method
Alkalinity	Acid-alkaline indicator titrimetric method

2 Results and discussion

2.1 Effect of pH on inorganic-N removal

As shown in Fig.2, the nitrogen compounds in the effluent appeared mostly as ammonium nitrogen and nitrate nitrogen. It indicated that the nitrification was not complete during the experimental stage I (1–56 d). In this stage, the average concentration of NH₄⁺-N and inorganic-N in the influent were 241.5 and 250.53 mg/L, respectively. The concentration of NH₄⁺-N in the effluent was high and ranged from 57.06 to 79.02 mg/L, with a low nitrification efficiency of 67.5%, and the concentration of inorganic-N in the effluent ranged from 88.54 to 157.32 mg/L, with the average denitrification efficiency of 45.9%. The average concentration of alkalinity (as calcium carbonate (CaCO₃)) in mixed liquids was only approximately 283.39

Fig. 2 Variation in NH₄⁺-N (a), NO₃⁻-N (b), and inorganic-N (c) concentration during the experiment.

mg/L. At the same time, pH was in the range of 5.73–5.96. However, according to previous studies (Painter and Loveless, 1983; Toerien *et al.*, 1990), the optimal pH for nitrification was 7.0–8.0 based on the calculation in theory, and the concentration of alkalinity was more than 2328.46 mg/L in the influent. Because of insufficient alkalinity in the influent and the demand of alkalinity for complete nitrification in mixed liquids, it is necessary to increase the alkalinity of the influent to the AEC-MBR. However, the quantity of alkaline reagent added in this type of AEC-MBR needs to be investigated and determined through experiments.

During experimental stage II (57–74 d), the effect of pH on nitrification was investigated. With pH of mixed liquids in the aeration tank maintained at 7.0–7.5 and with the addition of alkaline reagent, the nitrification was increased obviously, compared with that in experimental stage I. The concentration of NH₄⁺-N in the effluent was decreased rapidly from 80–90 mg/L to 45.25 mg/L, with an average nitrification efficiency of 77.6%, and the average concentration of inorganic-N in the effluent was 93.79 mg/L, with a removal efficiency of 56.3% (Fig.2). Furthermore, the concentration of alkalinity (as CaCO₃) was up to 400–600 mg/L in mixed liquids. These results showed that pH was a very important factor in the nitrification of the toilet wastewater, with higher ammonia concentration and a low

ratio of carbon to nitrogen (C/N).

2.2 Addition of alkaline reagent

The above results show that alkalinity is an important factor directly affecting the efficiency of nitrification, so the consumption of alkalinity must be determined. To investigate the correlative variation in alkalinity, pH, and $\text{NH}_4^+\text{-N}$ of the mixed liquids, the beaker test was carried out under the condition simulating experimental stage I. Table 5 shows the correlative variation in alkalinity, pH, and $\text{NH}_4^+\text{-N}$ of the mixed liquids that was obtained using the beaker test; it shows that the final values of alkalinity, pH, and $\text{NH}_4^+\text{-N}$ are also similar to that obtained during experimental stage I. The variation in $\text{NH}_4^+\text{-N}$ and alkalinity implies that alkalinity is a key factor for the removal of $\text{NH}_4^+\text{-N}$ in the AEC-MBR system for treatment of toilet wastewater.

The relationship between pH and logarithm of alkalinity was obtained through linear regression (Fig.3). The linear equation is as shown in Equation (1), and it can be used as an empirical equation to determine the alkalinity concentration of mixed liquids in the AEC-MBR.

$$\text{pH} = 4.1377\lg(\text{alkalinity}) - 3.935 \quad (1)$$

On the basis of Equation (1), when pH of mixed liquids was 7.0–7.5, the average concentration of alkalinity (as CaCO_3) was 400–580 mg/L.

The characteristics of five reagents used to optimize the alkalinity are shown in Table 6. Commonly, both CaCO_3 and sodium hydroxide (NaOH) were used as alkaline reagents in the conventional activated sludge process (Painter and Loveless, 1983; Toerien *et al.*, 1990). However, in the MBRs, although the use of CaCO_3 as alkaline reagent is very economical and effective to accommodate the buffering of pH for nitrification, the accumulation of calcium salts may be a potential hazard to membrane fouling. To avoid membrane fouling resulting from the addition of the alkaline reagent, it is recommended that effective carbonates, sodium carbonate (Na_2CO_3) or NaOH, be added to rapidly increase pH without the increase in Ca^{2+} in the AEC-MBR.

In the AEC-MBR, nitrification-denitrification accompanied the consumption or release of alkalinity. According to the stoichiometry of these biochemical reactions, 1 g $\text{NH}_4^+\text{-N}$ consumes 7.14 g (CaCO_3) of alkalinity, and 1 g $\text{NO}_3^-\text{-N}$ releases 3.57 g (CaCO_3) of alkalinity, which is half of $\text{NH}_4^+\text{-N}$ consumption (Toerien *et al.*, 1990; Daigger *et al.*, 1988). In the AEC-MBR, the total alkalinity mainly came from three approaches, such as the influent, addition of alkaline reagents and release of alkalinity during denitrification process, in which the third is a very important aspect to save alkaline reagents for nitrification.

Fig. 3 Linear correlativity of alkalinity with pH.

Fig.4 shows the ratios of alkalinity (CaCO_3) to $\text{NH}_4^+\text{-N}$ during the experimental stages II and III. The ratio of alkalinity (CaCO_3) to $\text{NH}_4^+\text{-N}$ in the influent was approximately 5.20 (ratio_{in}). However, based on the conception of total alkalinity, the ratio of $\text{NH}_4^+\text{-N}$ should be at 7.32 (CaCO_3) for complete nitrification (ratio_{co}). Thus, the ratio of the addition of alkaline reagent was approximately 2.12 (CaCO_3) (ratio_{ad}), where the ratio_{ad} is the difference between ratio_{co} and ratio_{in} . The amount of alkaline reagent added in the AEC-MBR system can be calculated by ratio_{ad} and concentration of $\text{NH}_4^+\text{-N}$ in the influent. From the experimental results, if the concentrations of $\text{NH}_4^+\text{-N}$ in the influent were at 150–300 mg/L, the consumption of alkalinity would be approximately 318–636 mg (CaCO_3)/L.

2.3 Effect of DO on inorganic-N removal

The nitrification and denitrification are two main processes involved in inorganic-N removal. Nitrification is an aerobic reaction, the nitrifying cells oxidize ammonia to nitrite or nitrate, demanding oxygen, whereas denitrification is an anaerobic reaction, denitrifying cells reduce nitrite or nitrate to nitrogen under anoxic and anaerobic conditions. In the AEC-MBR, because DO was controlled among 1.0–2.0 mg/L in the aeration tank and 7.6–9.4 mg/L in the membrane tank, the recycling of mixed liquids by airlift formed appropriate DO grads inside gel particles

Fig. 4 Ratio of alkalinity to $\text{NH}_4^+\text{-N}$ during the experimental stages II and III.

Table 5 Variation in alkalinity, pH, and $\text{NH}_4^+\text{-N}$ observed using the beaker test

HRT (h)	0	4	8	10	12	14	16	18	20
pH	8.52	7.89	7.31	6.74	5.89	5.71	5.68	5.62	5.6
Alkalinity (mg/L)	1064.61	717.72	550.25	263.16	263.16	239.24	215.32	215.32	215.32
$\text{NH}_4^+\text{-N}$ (mg/L)	194.62	117.96	110.2	79.62	76.1	75.66	73.01	73.01	66.84

Table 6 Comparisons of several medicaments as alkalinity

Medicament	pH	CO ₂	HCO ₃ ⁻	Ca ²⁺
CaCO ₃	+	-	+	+
NaHCO ₃	/	/	+	/
NaOH	+	-	+	/
Na ₂ CO ₃	+	-	+	/
Ca(OH) ₂	+	-	+	+

+: increase; -: decrease; /: invariable.

that was one of keys for efficiency of a single-stage biodenitrification process (Cao *et al.*, 2002).

As shown in Fig.2, the removal efficiency of NH₄⁺-N was complete despite the decrease in DO at experimental stage III (75–126 d). Although the concentration of DO reached 1.0 mg/L in the aeration tank, the concentrations of NH₄⁺-N in the effluent were below 10 mg/L, with an average removal efficiency of more than 91%, and the nitrogen compounds in the effluent appeared mostly as nitrate nitrogen. It indicates that nitrification was almost complete during experimental stage III. The concentrations of inorganic-N in the effluent also decreased from 93.79 to 77.21 mg/L, with an average removal efficiency of 70.0% after decrease in DO in the aeration tank.

At the same time, when DO was 1.0–2.0 mg/L, the biodegradation of organic contaminants was not effected negatively. The removal efficiencies of BOD₅ were maintained above 95.7% during experimental stage III, as shown in Fig.5.

2.4 Sludge characteristics

The specific nitrification rate (SNR), specific denitrification rate (SDNR), and different categories of microorganisms were used to analyze sludge characteristics in mixed liquids. Fig.6 shows the SNR and SDNR based on the concentration of inorganic-N in the experiment. It can be seen that the nitrogen removal rate was limited by lower SNR at experiment stage I. Till experimental stage II, the SNR increased sharply from 0.12 to 0.33 g NH₄⁺-N/(g MLSS·d) and the SDNR increased from 0.08 to 0.28 g inorganic-N/(g MLSS·d) when the pH was maintained at 7.0–7.5 with the addition of alkaline reagent. However, at experimental stage III and at same pH as that in experimental stage II, the SNR tended toward a drop from 0.33 to 0.13 g NH₄⁺-N/(g MLSS·d) and the SDNR from 0.28 to 0.08 g inorganic-N/(g MLSS·d), the main reason for which was the increase in MLSS from 1.56 to

Fig. 5 Variation in BOD₅ concentration during experimental stages II and III.

Fig. 6 Velocities of nitrification and denitrification during the experiment.

2.35 g/L.

Some reports indicated that it was difficult to find protozoans and metazoans in the MBR systems with external circulation (Zhang and Yamamoto, 1996; Xing *et al.*, 2001). However, in the AEC-MBR, the microorganisms were saved and microbial floc was well maintained because there was no circulation pump in the process. Abundant microorganisms especially subminiature animals were found in the mixed liquids, such as *Aspidisca* sp., *Vorticella* sp., *Suctorina* sp., and *Rotifer* sp. The microbial communities and their activities were similar to those in the conventional activated sludge processes. Moreover, *Aeoloosma hemprichii* was also detected, which always survived in the anaerobic condition.

2.5 Energy consumption and cost analysis

According to the circulation mode of the AEC-MBR, the energy consumption mainly resulted from air compressor, raw wastewater pump, and suction pump, of which air compressor consumed the largest part of the energy. The energy consumption for air compressor was calculated as shown in Equation (2):

$$N = \frac{G \times P}{3600 \times \eta} \quad (2)$$

where N is the shaft power of the air compressors, kW; G is the airflow rate, m³/h; P is the wind pressure, kPa; and η is the available power efficiency of blower, 0.5.

On the basis of the statistic, 85%–94% of the energy consumption was derived from the aeration. Therefore, the average energy consumption was at 0.72 kWh/m³ at the aeration rate of 18 m³/h at the flux of 8 L/(m²·h) in this experiment. Generally, the energy consumption was 2–10 kWh/m³ in the normal external circulation MBRs and 0.2–0.4 kWh/m³ in the normal submerged MBRs (Zhang and Yamamoto, 1996; Stephenson *et al.*, 2000; Churchouse and Wildgoose, 2002). The energy consumption of AEC-MBR was much lower than that of the normal external circulation MBRs and was close to that of the normal submerged MBRs.

When the concentrations of NH₄⁺-N in influent was 100–300 mg/L, with the ratio of alkalinity (CaCO₃)/NH₄⁺-N at 2.12, the consumption of alkalinity will be approximately 212–636 (CaCO₃) mg/L. The cost of addition of alkaline reagent was approximately 0.5–1.5 RMB yuan/m³ if the price of the alkalinity reagent is 2.5 RMB yuan/kg.

3 Conclusions

For inorganic-N removal from toilet wastewater, with an AEC-MBR, the SNR, SDNR, and the loads of nitrification and denitrification, the effect of pH, addition of alkaline reagent, and DO on inorganic-N removal, the sludge characteristics, energy consumption, and the cost for treatment of toilet wastewater were investigated in this study. The results can be summarized as follows:

(1) In the AEC-MBR, pH was an important factor affecting nitrification and denitrification. The SNR ranged from 0.13 to 0.33 g $\text{NH}_4^+\text{-N}/(\text{g MLSS}\cdot\text{d})$; the SDNR, 0.08 to 0.28 g inorganic-N/(g MLSS·d); pH 7–7.5; MLSS, 1.56–2.35 g/L; and DO, 1.0–2.0 mg/L. Under the same conditions as those mentioned above, the removal efficiency of $\text{NH}_4^+\text{-N}$, total inorganic-N, and BOD_5 were 91.5%, 70.0%, and 95.7%, respectively, in the novel type of MBR.

(2) Absence of circulation pump in the circulation model prevented the microorganisms and sludge flocs from being crushed, so that abundant microorganisms especially subminiature animals were found in the mixed liquids, such as *Aspidisca* sp., *Vorticella* sp., *Suctorina* sp., *Rotifer* sp., and *Aeolosma hemprichii*, which played an important role in increasing the removal efficiencies of nitrogen and organic matter.

(3) In AEC-MBR, for the complete removal of $\text{NH}_4^+\text{-N}$, the ratio for addition of alkaline reagent was approximately 2.12 alkalinity (CaCO_3)/ $\text{NH}_4^+\text{-N}$. The cost of addition of alkaline reagent was approximately 0.5–1.5 RMB yuan/ m^3 if the price of alkalinity reagent was 2.5 RMB yuan/kg and $\text{NH}_4^+\text{-N}$ in the influent was approximately 100–300 mg/L. The energy consumption was approximately 0.72 kWh/ m^3 at the aeration rate of 18 m^3/h at the flux of 8 L/($\text{m}^2\cdot\text{h}$).

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