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Enhanced nitrogen removal from sludge reject water by methanol addition using sequencing batch biofilm reactor

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ABSTRACT

Reject water from sludge contributes to the total nitrogen load at municipal wastewater treatment plants (WWTPs) and the development of new processes to treat it is crucial. A laboratory-scale sequencing batch biofilm reactor was investigated to treat reject water for the improved nitrogen removal. Due to the low dissolved organics (120–200 mg L^{-1}) and alkalinity (1,400 mg $CaCO_3$ L^{-1}) in reject water, an additional supply of alkalinity and external carbon for nitrification and denitrification was necessary. For the most optimal improved nitrogen removal, three conditions were tested. When both alkalinity and external carbon source (methanol) were supplied (Case I), nitrification and total nitrogen removal efficiencies were 97 and 91%, respectively. When only alkalinity was supplied (Case II), nitrification efficiency reached 97%, but with low denitrification. When only methanol was provided (Case III), nitrification and total nitrogen removal efficiencies were 89 and 80%, respectively. Nitrogen loss occurred when methanol was added (Cases I, 38.2% and Case III, 30.7%), whereas only nitrification occurred without methanol addition (Case II, 0.7%). The nitrogen loss in Case II, during the 10-min static fill step, may be associated to the occurrence of simultaneous nitrification and denitrification, resulted from the presence of nitrifiers and denitrifiers in the biofilm microenvironments. This process can be successfully applied to the removal of nitrogen from reject water as a separate treatment, saving cost from the alkalinity compensation for nitrification from denitrification as well as saving space in WWTP.

Keywords: Denitrification; Nitrification; Nitrogen removal; Organic carbon; Reject water; Sequencing batch biofilm reactor

1. Introduction

The wastewater treatment process can be divided into the water treatment line and the sludge treatment line. Reject water is generated at the sludge treatment line from thickening, anaerobic digestion, and dewatering processes. It has high ammonium and low organic carbon concentrations and is frequently mixed with the influent, contributing to the increase of total nitrogen load for the plant. Nitrogen removal is generally achieved at the wastewater treatment plant (WWTP) by the incorporation of additional volumes of

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aeration tanks, which represents a substantial investment [1]. In comparison, a separated treatment for reject water might significantly save cost as well as space. A variety of physical–chemical methods including struvite precipitation, ammonia stripping, and membrane separation have been developed to treat reject water [2–4]. The biological treatment process, however, has been known to be the most stable and economical one [5]. Biological wastewater treatments use either suspended or immobilized culture, and there have been considerable developments in fixed bed biofilm processes with such advantages as reduced operation and energy costs, smaller reactor volume, minimized need for settling capacity, operational simplicity, and reduced sludge production [6–8].

A continuous flow reactor has been used for most processes developed so far to treat reject water, such as ANAMMOX and SHARON [9,10]. However, the waste sludge disposal from anaerobic digester and dewatering equipment is not under 24-h continuous operation. Therefore, it is crucial to consider the batch process to treat reject water. The sequencing batch biofilm reactor (SBBR) is a fill-and-draw reactor where the biomass is fixed on a support medium. In this system, wastewater is added to a single batch reactor and treated to remove undesirable components before discharged. Compared to most activated sludge sequencing batch reactor (SBR) systems, which require the settle period to separate activated sludge, the SBBR system typically does not need the settle period for solid separation as well as the sludge recycle devices, still maintaining high microbial concentrations inside the reactor [11,12]. While the removal of nitrogen from reject water has been studied in pilot-scales using SBR and SBBR systems [13,14], the reactors were operated to achieve nitrification only and the denitrification was carried out in the anoxic zone of the mainstream-activated sludge plant.

In this study, the nitrogen removal from reject water containing low concentration ratios of alkalinity to ammonium and organics was evaluated as a separate treatment employing SBBR. The nitrogen removal rates were compared, depending on the presence/addition of alkalinity as well as methanol as an external carbon source during the anoxic react step. Furthermore, nitrification and nitrogen loss from the reject water were also investigated during the static fill step.

2. Materials and methods

2.1. Characteristics of reject water

Samples were obtained from the filtrate generated from the dewatering process using the belt press filter for the anaerobic digested sludge at "J" regional municipal WWTP in Seoul, Korea. The pH of this filtrate was 7.6 ± 0.1 and the concentration of soluble chemical oxygen demand (SCOD) was $160 \pm 56\ \mathrm{mg\ L^{-1}}$. The influent nitrogen was mostly present in the form of ammonium at $390 \pm 103\ \mathrm{mg\ N\ L^{-1}}$. The average concentrations of nitrite, nitrate, and alkalinity were 1.0 ± 0.4, and $6.0 \pm 0.5\ \mathrm{mg\ N\ L^{-1}}$ and $1{,}400 \pm 46.2\ \mathrm{mg\ CaCO_3\ L^{-1}}$, respectively. Measurements were performed in triplicates. The average ratio of alkalinity to ammonium for the filtrate was approximately 3.6, lower than the theoretically required amount of 7.1 for nitrification [15]. Therefore, the additional supplementation of alkalinity was necessary to complete nitrification. According to Peng et al. [16], the denitrification potential of wastewater is related to the available organic carbon, which is commonly expressed by the COD/TN ratio. For the satisfactory or complete denitrification to occur, the range of COD/TN might be between 4 and 15. In the current study, the ratio was not sufficient (0.4) and the addition of methanol as an external carbon source was necessary to increase the denitrification potential.

2.2. Experimental apparatus

A schematic diagram of the SBBR used for the removal of nitrogen from reject water is shown in Fig. 1. The reactor was made of acrylic cylinder i.d., 5 cm; height, 120 cm). In order to support the media inside, the plates perforated with 3-mm diameter holes were installed at both top and bottom portions of the filled media. Sampling ports were installed from the bottom of the filled layer, at 15 cm intervals along the column. The reactor was filled with blasted clay granules (particle size, 5–8 mm) up to the level of 75 cm, with the void fraction of 0.33. The reactor was operated upflow with the intermittent aeration, using a compressor, from its bottom at the flow rate of $1{,}000\ \mathrm{cm^3\ min^{-1}}$, following the sequence red/ox condition (0.5–4.5 h in Case I and 5–6.5 h in Cases II and III). The dissolved oxygen (DO) concentration was maintained at $4–8\ \mathrm{mg\ L^{-1}}$ and below $0.1\ \mathrm{mg\ L^{-1}}$ in aerobic and anaerobic/anoxic tanks, respectively, and the pH ranged from 7.0 to 8.0 for both processes.

The feed contained reject water mixed with alkalinity ($NaHCO_3$, 20 L at $2{,}400\ \mathrm{mg\ L^{-1}}$) and entered the reactor through the bottom. During the aerobic react step, ammonium was oxidized to nitrite and nitrate. The aeration stopped during the anoxic react step, and nitrate was reduced to nitrogen gas by denitrifiers with the additional supply of methanol (from the methanol tank, 100 mL for Case II and 50 mL for

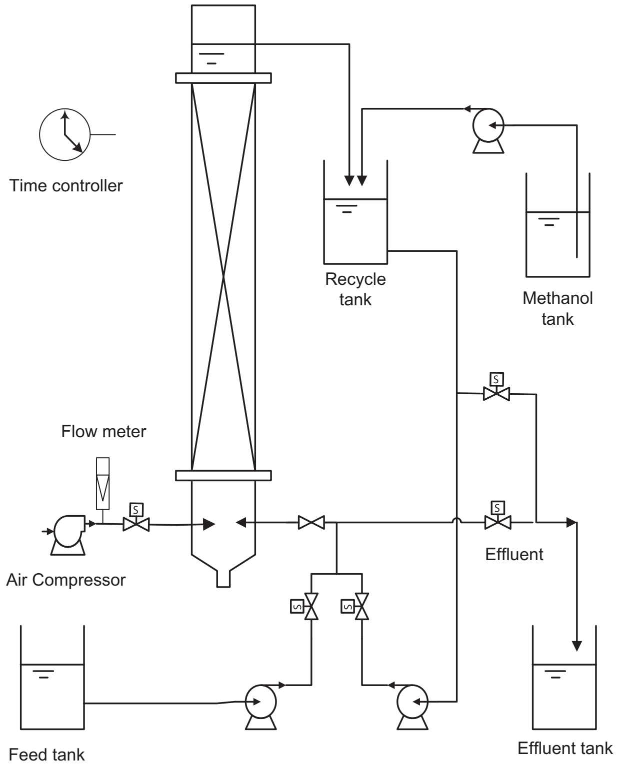


Fig. 1. A schematic diagram of the SBBR for the SND.

Case III). The reactor was seeded with the activated sludge from WWTP and the biofilm attached gradually on supporting media. Both effluent and reject water were recirculated, and the reactor was acclimated/operated in a batch mode for about 2 weeks before the simultaneous nitrification and denitrification (SND) experiments started.

The superficial water velocity ($m^3\ m^{-2}\ h^{-1}$) varied from 0.4 to 6.0 and the total reactor cycle time started from 4 to 12 h, depending on whether each run for nitrification and denitrification, respectively. For nitrification, the reactor was initially operated with 7.1 h of EBCT (empty bed contact time) and the analysis started after 18 d. After 38 d, $NaHCO_3$ was added to the influent and the ratio of alkalinity to ammonia-N was maintained at 5–7. For denitrification, the reactor was initially operated with 3.8 h of EBCT, 2.3 h after 42 d, and 2.6 h after 90 d, while the reactor temperature was 15–35℃. The loading rate of total oxidized N (NO_x-N) for influent was 0.2–0.4 kg $m^{-3}\ d^{-1}$.

2.3. Reactor operation

The SBBR was initially seeded with the activated sludge obtained from the municipal WWTP mentioned above. Experiments were performed under three different cases. In Case I, $NaHCO_3$ (as alkalinity) was added (20 L) to the influent reject water and methanol (as external carbon source) was added (100 mL) under the anoxic condition. In Case II, only $NaHCO_3$ was added (20 L), and in Case III, methanol (50 mL) was added but without $NaHCO_3$ in order to utilize alkalinity generated in the denitrification process. The operational schemes for each case are detailed in Table 1. In this study, two different sequences of redox conditions were applied, which were Anoxic–Aerobic–Anoxic (Case I) and Anoxic–Aerobic–Anoxic–Aerobic–Anoxic (Cases II and III), with the same total cycle time to further investigate the effect of redox sequences on nitrogen removal efficiency. During the react step, which consisted of aerobic and anoxic conditions, biological reactions occurred and mixing was provided by recirculation at the superficial water velocity of 5 $m^3\ m^{-2}\ h^{-1}$, from the recycle tank to the bottom of the reactor. During the aerobic react step, aeration was provided using compressor to achieve nitrification, whereas during the anoxic react step, air was not provided and methanol was added to achieve denitrification. During the draw step, 100% of the supernatant was withdrawn. The idle step occurred between the draw and fill steps, while maintaining microorganisms attached onto media, before the new cycle started.

2.4. Analytical methods

The overall water quality data were obtained by analyzing samples from the effluent tank after one day of treatment. The time-dependent data for each experimental case were obtained by analyzing 5-mL sample aliquots withdrawn from the recycle tank. The concentrations of SCOD, NH_4^+-N, NO_2^--N, NO_3^--N, and alkalinity were analyzed according to the Standard Methods [17]. The pH was measured using a pH meter (Orion 510A, Thermo Electron Orion, USA), and temperature and DO were checked using a DO meter (YSI 58, Yellow Springs Incorporated, USA). The DO was measured from the top portion of the filled layer in the reactor.

3. Results and discussion

3.1. Overall performance and nitrogen removal characteristics in SBBR

The reject water generally shows low levels of alkalinity and organic carbon, necessary for nitrification and denitrification processes, respectively. Fig. 2 shows the concentration variations for total inorganic nitrogen (TIN) of the influent and for ammonium,

Table 1
Operational conditions of bench-scale SBBR for the SND

		Time (min)		
Phase	Reactor condition	Case I	Case II	Case III
Fill	Reactor filled to max. volume	10	10	10
React	Anoxic: recirculation on	20	50	50
	Aerobic: aeration on	240	90	90
	Anoxic: aeration off	200	150	150
	Aerobic: aeration on	–	90	90
	Anoxic: aeration off	–	80	80
Idle/draw	Idle: recirculation off	10	10	10
	Draw: 100% of filled volume drained			
Total cycle time		480	480	480
Alkalinity addition to reject water[a]		$NaHCO_3$	$NaHCO_3$	–
Carbon source addition during anoxic step		Methanol[b]	–	Methanol[c]

[a] $NaHCO_3$ was added to the feed tank (20 L at 2,400 mg L^{-1}).

[b] 100 mL of methanol was added to the recycle tank 10 min after the anoxic react step started.

[c] 50 mL of methanol was fed at the beginning of each 10 min anoxic react step.

nitrite, and nitrate of the effluent during the operational period. In Case I, the TIN concentration started to decrease around 2 weeks after the start of operation, and then the effluent concentration of inorganic nitrogen was maintained at ≤10 mg N L^{-1}, resulting in the average of 97% nitrification and 91% nitrogen removal efficiencies. When $NaHCO_3$ was added without external carbon source (Case II), the effluent concentration of ammonium was maintained low. However, since the denitrification process was not carried out due to the lack of external carbon source, the concentrations of nitrite and nitrate were maintained at 70–160 and 240–320 mg N L^{-1}, respectively, resulting in the average of 97% nitrification but only 5% nitrogen removal efficiency, which implies that almost no denitrification occurred. When only methanol was added (Case III), the average effluent concentration of ammonium was approximately 47 mg N L^{-1}. Based on the influent ratio of alkalinity to ammonium, 50% nitrification was expected to occur, but 89% nitrification occurred instead, due to the alkalinity generated from the denitrification process, resulting in 80% nitrogen removal efficiency. Fig. 3 shows the details of cycles after the reactor reached the stable condition in Cases I–III.

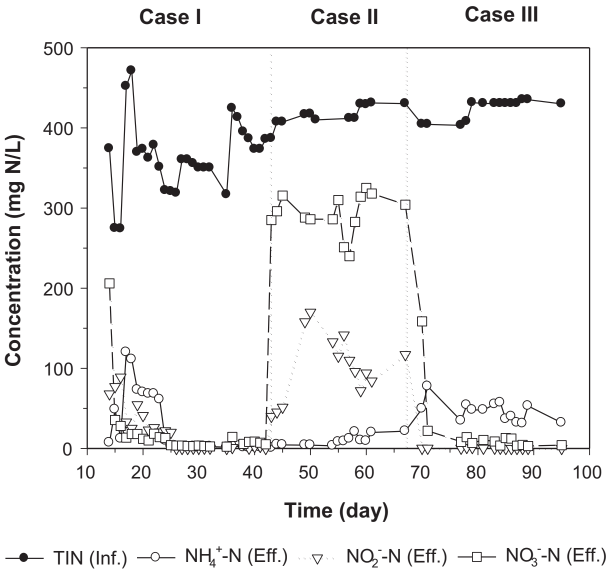
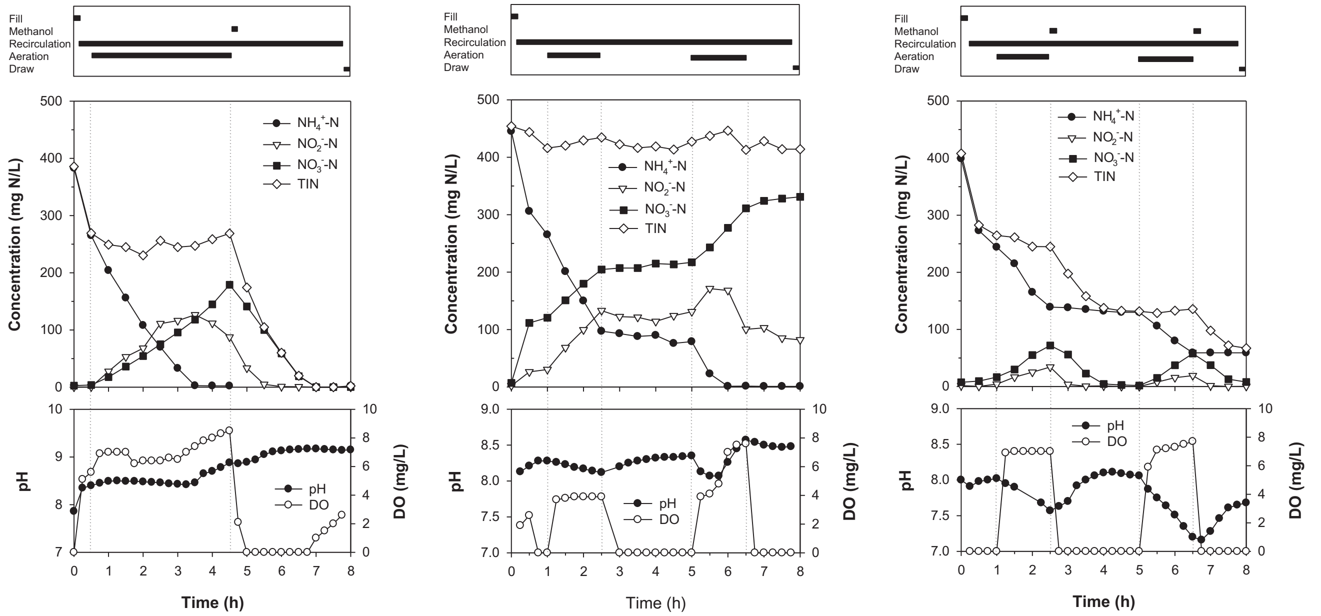


Fig. 2. Concentration profiles of influent and effluent for each experimental case (Case I, both $NaHCO_3$ and methanol added; Case II, only $NaHCO_3$ added; Case III, only methanol added).

3.1.1. Case I (complete removal of ammonium by addition of both alkalinity and methanol)

As shown in Fig. 3(a), the ammonium concentration drastically decreased even without nitrite or nitrate generation during the first 30 min, resulting in about 30% nitrogen loss from the influent, and was completely nitrified within 3 h after the fill step. The nitrite concentration increased until about 3 h during the aeration step and then decreased gradually afterward. The nitrate–nitrogen concentration increased greatly until the end of the aerobic react step. After the addition of methanol during the anoxic react step, the concentrations of nitrite and nitrate rapidly decreased due to denitrification, resulting in the



(a) (b) (c)

Fig. 3. Concentration profiles of ammonium, nitrite, nitrate, and TIN during the Case I (a), II (b), and III (c) SBBR cycles.

effluent TIN concentration of ≤10 mg N L^{-1}. The pH, on the other hand, was maintained at around 8.2 during the aerobic react step due to the addition of alkalinity to the influent. However, during the anoxic react step, high pH values were observed mainly as a result of the increase of alkalinity produced by denitrification. For this reason, the addition of alkalinity was not necessary and additional costs could be saved via the utilization of SBBR to treat reject water.

3.1.2. *Case II (incomplete removal of ammonium by addition of alkalinity only)*

The ammonium concentration decreased to approximately 300 mg N L^{-1} followed by the increase of nitrate–nitrogen concentration to 100 mg N L^{-1}, after the fill period, implying the occurrence of nitrification even without the provision of air during the fill period, as shown in Fig. 3(b). About 35% nitrification was achieved as a result of 50 min recirculation during the anoxic react step, but without any significant change in terms of the TIN concentration. Following the subsequent 1.5 h aerobic react step, the ammonium concentration decreased to about 100 mg N L^{-1} due to nitrification and the nitrite concentration increased to about 140 mg N L^{-1}. Then, during the anoxic react step without the addition of methanol, there were almost no changes in the concentrations of ammonium, nitrite, and nitrate. Finally, following the subsequent aerobic react step, the ammonium was completely nitrified and converted to nitrate. The low COD/TN concentration ratio resulted in a poor performance of denitrifiers due to the unavailable organic carbon in reject water and consequently, the low TIN loss was observed.

3.1.3. *Case III (complete removal of ammonium by addition of methanol only)*

About 30% TIN loss occurred after the static fill step, as shown in Fig. 3(c). The ammonium concentration decreased to 150 mg N L^{-1} and the concentrations of nitrite– and nitrate–nitrogen increased to 45 and 80 mg N L^{-1}, respectively, after the first aerobic react step due to nitrification, and the pH decreased to 7.5. Then, after the addition of methanol during the anoxic react step, the pH increased to 8.2 and both nitrite and nitrate were completely removed after about 1.5 h, due to denitrification, but the ammonium concentration showed no change. During the second aerobic react step, however, the ammonium concentration declined to 60 mg N L^{-1} and the pH started to decrease again down to 7.2, as a result of nitrification. Finally, during the subsequent anoxic react step, the denitrification of nitrite and nitrate resumed, resulting again in the increase of pH due to the alkalinity production, which might compensate the alkalinity loss during nitrification to some extent [18]. The alkalinity supplement may not be necessary for the current SBBR system, which could further reduce the operational cost.

3.2. Relationship between alkalinity and nitrogen removal

Since the average concentration ratio of alkalinity to ammonium for the reject water in this study was 3.6, only about 50% nitrification was theoretically expected to occur without any additional supply of alkalinity. However, 89% nitrification was achieved due to the alkalinity generated from the denitrification process. In addition, approximately 3.2 mg of alkalinity was consumed for 1 mg of ammonium nitrified without the alkalinity addition (Case III). Considering the alkalinity of 6.7 mg $CaCO_3$ $mg^{-1}NH_4^+$-N actually consumed in the biological aerated filter reactor previously studied [6], the alkalinity generated from the denitrification process would be considered 3.5 mg $CaCO_3$ per NO_x-N. In general, approximately 7.1 mg of alkalinity (as $CaCO_3$) is required for 1 mg of ammonium during the nitrification process, whereas 2.8–3.6 mg of alkalinity is generated while 1 mg of nitrate–nitrogen is denitrified [12,19].

3.3. Nitrification and nitrogen loss during static fill period

As shown in Fig. 3(a) through 3(c), when no external carbon source was added (Case II), the ammonium concentration drastically decreased, along with the increase of nitrate–nitrogen, resulting in about 30% nitrification, whereas approximately 30% nitrogen loss occurred when the external carbon source was added (Cases I and III). This phenomenon took place throughout the operational period for the SBBR, as shown in Table 2. When both alkalinity and methanol were added (Case I), about 30% loss of ammonium occurred 42 d after the start, whereas the loss of ammonium was 34% but only 11% loss of TIN occurred just 1 d after in Case II. Considering the conversion of ammonium to nitrite or nitrate afterward, the ammonium seemed to be removed by nitrification. When only methanol was added (Case III), about 30% TIN loss appeared again, probably due to the dilution effect resulted from the liquid remaining inside the reactor and the feeding tube. For the 30% removal of ammonium to occur by dilution only, approximately 0.43 L water is necessary for the 1 L added, but the amount remained inside the reactor and the feeding tube was almost negligible (data not shown).

Fig. 4 shows the changes in nitrogen concentrations during the fill-and-draw periods for Case II. As the feeding frequency progressed, the ammonium concentration decreased, along with the increases in nitrite and nitrate concentrations, and the TIN concentration remained similar, implying the occurrence of nitrification. After four cycles, approximately 45% nitrification occurred. Fig. 5 shows the nitrogen concentrations measured for the 100-mL effluent samples, after filling up the reactor with the influent during Case III experiments. In case of ammonium, the initial effluent concentration decreased to 150 mg N L^{-1}, but gradually reached almost the same as the influent when 1,000 mL passed through the reactor. In comparison, however, the concentrations of nitrite and nitrate were very low, resulting in about 30% TIN loss while 1,000 mL of influent passed through the reactor. Even though these results may suggest that the ammonium might have been adsorbed on biofilm and/or media, the removal of ammonium was not considered by adsorption, based on the Case II results where the conversion of ammonium to nitrite and nitrate was carried out. Instead, it might have been due to the probable occurrence of SND, commonly found in activated sludge systems. This phenomenon, especially the aerobic denitrification, has been described for various SBR systems. It was reported in a bench-scale combined anaerobic–aerobic SBR for the treatment of piggery wastewater [20] and an intermittent aeration process for the domestic wastewater treatment [21,22]. The SND can be divided into two categories, physical and biological. For the physical, a conventional explanation is that it occurs as a consequence of the DO concentration gradient within microbial floc or biofilm, due to the diffusional limitations, creating anoxic and/or anaerobic zones. The nitrifiers, therefore, exist in regions with high DO concentrations, whereas the denitrifiers would preferably be active in zones with very low DO concentrations. In comparison, the biological explanation for SND is somewhat contradictory to the traditional engineering concept of nitrification and denitrification. Microbiologists have reported the existence of aerobic denitrifiers as well as heterotrophic nitrifiers. Moreover, nitrification under the fully anaerobic conditions has also been shown possible [16].

Results showed that the nitrogen loss occurred when methanol was added as the external carbon source (Cases I and III), whereas only nitrification took place when methanol was not added (Case II). Guisasola et al. [23] showed the experimental evidence of carbon source limitation effect on nitrogen removal and indicated that the limitation started at values lower than 3.0 mmol L^{-1}. The SND, therefore, was considered to occur by ordinary nitrifiers and denitrifiers, rather than by aerobic denitrifiers or heterotrophic nitrifiers, possibly due to the DO concentration

Table 2
Nitrification and nitrogen loss after fill periods

Case	Time (d)	Influent (mg N L^{-1}) NH$_4^+$-N	NO$_2^-$-N	NO$_3^-$-N	TIN	After feeding (mg N L^{-1}) NH$_4^+$-N	NO$_2^-$-N	NO$_3^-$-N	TIN	NH$_4^+$-N loss (%)	TIN loss (%)	Sampling time (h)
I	22	369	0.2	4.5	374	165	40.0	26.8	231	55.3	38.2	1.0
	35	345	0.0	5.8	351	187	56.0	12.0	255	45.8	27.3	0.5
	42	383	0.1	2.6	386	265	0.5	3.7	269	30.8	30.1	0.5
II	43	378	0.6	5.5	384	249	3.4	88.4	341	34.1	11.3	0.5
	50	410	0.2	7.0	417	292	56.6	58.3	407	28.8	2.4	1.0
	53	394	7.7	4.4	406	246	42.7	115.0	403	37.7	0.7	0.5
III	78	399	1.6	7.4	408	273	0.2	9.4	283	31.6	30.7	0.5
	95	418	1.2	6.5	425	283	0.4	7.1	291	32.3	31.6	0.5

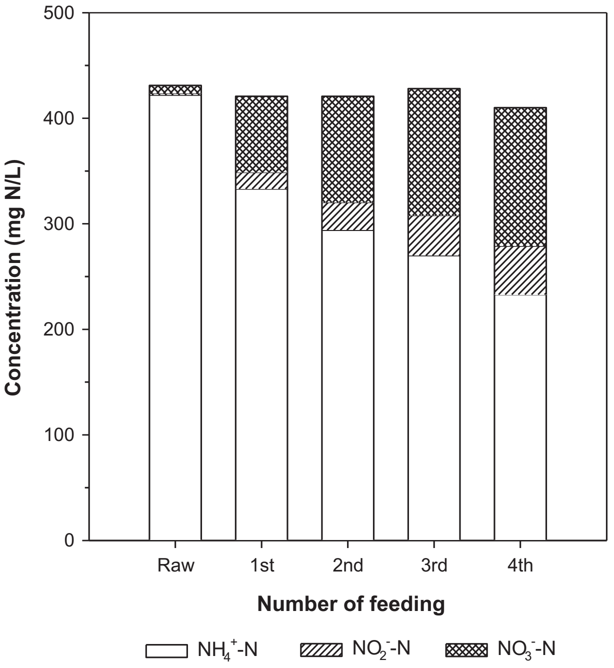


Fig. 4. Concentration profiles of ammonium, nitrite, and nitrate during the fill-and-draw periods for Case II.

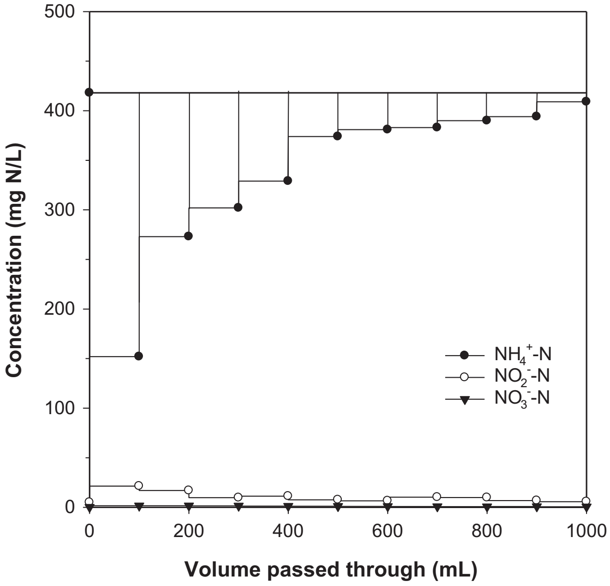


Fig. 5. Concentration profiles of ammonium, nitrite, and nitrate, depending on the influent volume passing through, during the static fill step for Case III.

difference within the biofilm microenvironments. According to Tay et al. [24], the N/COD ratio imparts a significant impact on the microbial population, where high substrate N/COD ratios enhanced nitrifying and denitrifying activity and consequently, the aerobic/heterotrophic populations were found to decrease in such circumstances.

During the idle step, the biofilm was exposed to the air, resulting in further nitrification of the influent added, due to the contact of nitrifiers with outside oxygen as the electron acceptor, and denitrification also occurred by denitrifiers residing inside the reactor, utilizing organic carbon sources present in the influent, while nitrification progressed simultaneously.

4. Conclusions

Results from nitrification and denitrification of reject water in the SBBR showed that the nitrogen removal efficiency was maintained ≥90% at the removal rate of 0.7 kg N m^{-3} d^{-1}, after methanol was added as the external carbon source during the 4-h cycle of fill and aerobic react steps and the 4-h cycle of anaerobic react and draw steps, with the 8-h total cycle time. When only alkalinity was added to the reject water, 97% nitrification was achieved but no denitrification was induced. When only methanol was added under the anoxic condition, 89% nitrification and 80% nitrogen removal efficiency were achieved due to the alkalinity generated from the denitrification process. Therefore, by applying this SBBR process to the wastewater especially containing low concentrations of alkalinity and organics, the high nitrogen removal would still be possible with only the addition of external carbon source but without the additional supply of alkalinity. On the other hand, during the static fill step, about 30% nitrification and 30% nitrogen loss occurred with and without the addition of methanol, respectively, and the nitrogen loss was considered due to the occurrence of SND, possibly resulted from the presence of nitrifiers outside and denitrifiers inside of the biofilm. This process can be successfully applied to the removal of nitrogen from reject water as a separate treatment, saving cost due to the alkalinity compensation for nitrification from denitrification as well as saving space in WWTP as a result of no anoxic tank requirement.

Acknowledgments

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