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Characteristic of nitrous oxide production in partial denitrification process with high nitrite accumulation



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HIGHLIGHTS

• N₂O production in partial denitrification with high NO₂⁻-N accumulated was studied.

• N₂O production could be ignored during NO₂⁻-N accumulation before NO₃⁻-N depletion.

• Accumulation of N₂O was strongly related to the NO₂⁻-N reduction process.

• Low pH condition significantly enhanced N₂O production.

• N₂O production could be alleviated by controlling pH turning point.

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ABSTRACT

Nitrous oxide (N₂O) production during the partial denitrification process with nitrate (NO₃⁻-N) to nitrite (NO₂⁻-N) transformation ratio of 80% was investigated in this study. Results showed that N₂O was seldom observed before complete depletion of NO₃⁻-N, but it was closely related to the reduction of NO₂⁻-N rather than NO₃⁻-N. High COD/NO₃⁻-N was in favor of N₂O production in partial denitrification with high NO₂⁻-N accumulation. It was seriously enhanced at constant acidic pH due to the free nitrous acid (FNA) inhibition. However, the N₂O production was much lower at initial pH of 5.5 and 6.5 due to the pH increase during denitrification process. Significantly, the pH turning point could be chosen as a controlled parameter to denote the end of NO₃⁻-N reduction, which could not only achieve high NO₂⁻-N accumulation but also decrease the N₂O production significantly for practical application.

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1. Introduction

The anammox process using NO₂⁻N as electron acceptor for NH₄⁺-N oxidization to nitrogen gas (N₂) is deemed as the most economic and effective nitrogen removal process for wastewater treatment. It hold great advantages of low energy consumption, low sludge production and high nitrogen removal rate (Kartal et al., 2010; Tang et al., 2011). But one of the bottlenecks for wide application of anammox process is the stable NO₂⁻-N production in the aerobic short-cut nitration process, especially for the low nitrogen containing wastewater (Jin et al., 2013). On the other hand, NO₂⁻-N accumulation in denitrification has drawn much attention in recently years as it could serve as another way to supply electron donor for anammox (Waki et al., 2013; Gong et al., 2012a; Kalyuzhnyia et al., 2006). Gong et al. (2012a) had reported that

the NO_2^--N accumulation ratio achieved 60% under batch-flow mode in acetate feast-famine condition.

Recent study showed that high NO₂⁻-N accumulation could be obtained in denitrification process, with as high as 80% of NO₃⁻-N -to- NO₂⁻-N transformation ratio maintained stably in a long-term operation, indicated that partial denitrification (NO₃⁻-N \rightarrow NO₂⁻-N) could be realized (Cao et al., 2013a). Thus, as to the NO₃⁻-N contained wastewater, partial denitrification combined with anammox process shows advantaged prospect compared with the conventionally complete denitrification, such as lower carbon consumption, less excess sludge production.

 N_2O emission from wastewater treatment plants (WWTPs) has been investigated substantially, and the accumulation of N_2O in denitrification has been deemed to the unbalanced rates of nitric oxide (NO) and N_2O reduction. Many factors have been reported to affect N_2O accumulation during denitrification, such as pH (Pan et al., 2012; Hanaki et al., 1992), FNA (Zhou et al., 2008), ratios of chemical oxygen demand to nitrate (COD/NO₃⁻-N)



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(Itokawa et al., 2001; Kishida et al., 2004), dissolved oxygen (DO) concentrations (Tallec et al., 2008; Gong et al., 2012b) and the types of carbon sources (Adouani et al., 2010). Among these, NO₂-N had a significant influence on N₂O accumulation (Alinsafi et al., 2008; Schulthess et al., 1995). Alinsafi et al. (2008) suggested that the NO₂-N concentration had a direct effect on the N₂O emission in the denitrification process. High NO₂-N concentration during denitrification was found to result in a lower denitrification rate and accumulation of NO and N₂O (Schulthess et al., 1995). Thus, it is imperative to investigate the nitrous oxide production in the denitrification process with high nitrite accumulation.

Although the factors mentioned above have been investigated for the effect on N₂O emission during denitrification, these studies mainly focused on the complete denitrification process. The characteristic and mechanism of N₂O production during the partial denitrification process with a high NO₂⁻-N accumulation was still unclear. In this study, a serial batch experiments were designed to establish a relationship between the N2O production and NO₃-N reduction as well as NO₂-N accumulation. The laboratorial sludge was taken from an anoxic denitrification sequencing batch reactor (SBR) with ~80% conversion ratio of NO₃⁻-N to NO₂⁻-N (Cao et al., 2013a). The N₂O production properties were investigated systematically with different parameters including COD/NO₃-N, constant acid pH and different initial pH. The operation strategies were also discussed for alleviating N₂O production in practical application of partial denitrification process for the first time

2. Methods

2.1. SBR operation for partial denitrification

A laboratory-scale SBR with a working volume of 5 L was performed to achieve partial denitrification. The seeding sludge was taken from an anaerobic fermentation coupling anoxic denitrification reactor as described in previous study (Cao et al., 2013a). The SBR was operated at room temperature (18.5.0–27.9 °C) with two cycles a day. Each cycle was consisted of 12 h operation, including 10 min feeding with NO₃⁻-N contained wastewater, 1 min feeding carbon source, 20-40 min anoxic reaction, 30 min settling, 9 min discharging and then the idle phase. The reactor was mixed using a mechanical stirrer at 100 rpm. Firstly, 3.2 L of synthetic wastewater was pumped into the reactor in the 10 min feed period, and then pumped the carbon source to the reactor at the end of feeding phase with a initial COD/NO₃⁻N of 3.0. At last, 3.2 L supernatant was discharged after settling. It should be noted that the SBR reactor was operated without sludge discharge during the overall operation period. The mixed liquor suspended solids (MLSS) concentration was 3600 ± 225 mg/L, and the mixed liquor volatile suspended solids (MLVSS) concentration was 2080 ± 120 mg/L.

The composition of the wastewater was: 182.1 mg/L NaNO₃ (NO₃⁻-N of 30 mg/L), 11.1 mg/L KH₂PO₄, 6 mg/L MgSO₄·7H₂O,

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The detail	conditions	for	batch	experiments.

Table 1

3 mg/L CaCl₂·2H₂O and 1 mL trace-element, which contained 1500 mg/L FeCl₃·6H₂O, 30 mg/L CuSO₄·5H₂O, 120 mg/L MnCl₂·4H₂O, 60 mg/L Na₂MoO₄·2H₂O, 120 mg/L ZnSO₄·7H₂O, 150 mg/L CoCl₂· 6H₂O, 180 mg/L KI, 150 mg/L H₃BO₃ and 10 g/L ethylenediamine tetraacetic acid (EDTA) according to Cao et al. (2013a). The sodium acetate solution (5 g COD/L) was used as a carbon source to supply the electron donor for NO₃⁻-N reduction.

2.2. Batch experiments

2.2.1. Reactor and sludge

All batch tests were carried out in conical flask reactors with working volume of 0.5 L. The reactors were completely sealed and performed in a laboratory with temperature varying between 26.0 and 28.0 °C. For each batch experiment, 150 mL mixed sludge was taken from the SBR at idle phase and washed three times with deionized water. The composition of the medium was added to the flasks as the same as the original SBR, expect the NO₃⁻-N and acetate amount. Then, N₂ was pumped into the reactors for 5~10 min to establish an anoxic environment. The batch reactors were mixed with a magnetic stirrer at speed of 100 rpm. Sterile injection syringe were used to take samples.

In order to minimize pH deviation, buffer solution with KH_2PO_4 and Na_2HPO_4 was used as described by Cao et al. (2013b), as well as 0.5 M HCl or 0.5 M NaOH solution in the Test 1 to Test 4. In Test 1–4, 50 mL pH buffer solutions were added, following by adjusted with 0.5 M HCl during the experiment. In Test 5, pH was adjusted with 0.5 M HCl or 0.5 M NaOH at the beginning of experiments without pH buffer solutions involved.

2.2.2. Effect of COD/NO $_3^-$ -N on N₂O production in partial denitrification

The batch experimental matrix was described in Table 1. Test 1 and 2 were conducted to investigate the effect of COD/NO_3^-N on N₂O production. The NO_3^--N stock solution was added to achieve an initial NO_3^--N concentration of 20 mg/L (Test 1) and 80 mg/L (Test 2) at the beginning of the test. Then different amount of sodium acetate solution was injected into the batch reactor to achieve varied initial COD/NO_3^--N ratios (Table 1). Each experiment lasted for 120 min and the pH was controlled at 8.5 ± 0.1.

2.2.3. Effect of different pH on N₂O production in partial denitrification

The N₂O production at constant pH 5.5 (with NO₃⁻-N of 20 mg/L and 40 mg/L) and constant pH 6.5 (with NO₃⁻-N of 20 mg/L, 40 mg/L and 80 mg/L) was investigated in Test 3 and Test 4, respectively. Furthermore, the effect of initial pH (5.5, 6.5 and 7.5) on N₂O production was conducted in Test 5 with NO₃⁻-N concentration of 40 mg/L and COD/NO₃⁻-N at 3.2.

2.3. Sampling and analytical methods

The influent and effluent samples during long-term operation of partial denitrification SBR were collected on daily basis and

Test	Experiment	Remarks	MLVSS (mg/L)
1	Culture (150 mL) + 6 levels of $COD/NO_3^{-}N$	$NO_{3}^{-}-N = 20 \text{ mg/L}$	1050 ± 75
	(0.8, 1.6, 2.4, 3.2, 4.0 and 8.0)	$pH = 8.5 \pm 0.1$	
2	Culture (150 mL) + 5 levels of COD/NO ₃ -N	$NO_{3}^{-}-N = 80 \text{ mg/L}$	1120 ± 60
	(1.6, 2.4, 3.2, 4.0 and 8.0)	$pH = 8.5 \pm 0.1$	
3	Culture (150 mL) + 2 levels of NO_3^-N	$COD/NO_3 - N = 3.2$	1200 ± 70
	(20 and 40 mg/L)	$pH = 5.5 \pm 0.1$	
4	Culture (150 mL) + 3 levels of NO_3^-N	$COD/NO_{3}^{-}-N = 3.2$	1350 ± 30
	(20, 40 and 80 mg/L)	$pH = 6.5 \pm 0.1$	
5	Culture $(150 \text{ mL}) + 3$ levels of initial pH	$COD/NO_3 - N = 3.2$	1270 ± 50
	(5.5, 6.5 and 7.5)	$NO_{3}^{-}-N = 40 \text{ mg/L}$	

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