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# 新始章 (HUANJING KEXUE)

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## C/N 比对反硝化过程中亚硝酸盐积累的影响分析

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摘要:在 SBR 反应器中利用乙酸钠为底物,研究 C/N (COD/NO<sub>3</sub>-N) 比对反硝化过程中亚硝酸盐积累的影响。在 SBR 连续运行过程中(HRT 为6 h),C/N 比为3 时,亚硝酸盐积累率可达 45%。批式处理研究表明,C/N 比为 2.5 和 3.0 时 亚硝酸盐的积 累率较高,分别为 47.50% ±1.005% 和 45.28% ±5.469%。C/N 比为 2.5 时获得的亚硝酸盐比积累速率为 (30.17 ± 1.70)  $\text{mg}\cdot(\text{g}\cdot\text{h})^{-1}$ ,C/N 比为 3 时获得的亚硝酸盐比积累速率为 (29.92 ± 1.90)  $\text{mg}\cdot(\text{g}\cdot\text{h})^{-1}$ 。C/N 比在 2.5 ~ 4 范围内时,C/N 比对硝酸盐的还原速率基本无影响,但对亚硝酸盐的积累速率影响显著,C/N 比为 2.5 和 3.0 时有利于亚硝酸盐的积累,C/N 比 3.5 时,亚硝酸盐积累率下降显著。

关键词: 半反硝化; 亚硝酸盐积累; SBR 反应器

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### Effect of C/N Ratio on Nitrite Accumulation During Denitrification Process

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**Abstract**: Effect of C/N (COD/NO $_3^-$ -N) ratio on nitrite accumulation during denitrification process was investigated in a sequencing batch reactor (SBR) with acetic sodium as the electron donor. The nitrite accumulation ratio was 45% at a C/N ratio of 3 during operation in SBR with HRT at 6 h. According to the results of batch experiments, Nitrite accumulation ratios were higher at C/N ratios of 2.5 and 3.0, and the values were 47.50%  $\pm 1.005\%$  and 45.28%  $\pm 5.469\%$ , respectively. The nitrite specific accumulation rate was (30.17  $\pm 1.70$ ) mg·(g·h) $^{-1}$  at a C/N ratio of 2.5, and was (29.92  $\pm 1.90$ ) mg·(g·h) $^{-1}$  at a C/N ratio of 3.0. C/N ratios of 2.5-4.0 did not affect the nitrate reduction rate but obviously affected the nitrite accumulation rate. C/N ratios of 2.5 and 3.0 were favorable for nitrite accumulation, and a C/N ratio $\geqslant$ 3.5 would lead to decrease in nitrite accumulation (SBR).

Key words: partial denitrification; nitrite accumulation; sequencing batch reactor(SBR)

近几年兴起的厌氧氨氧化技术能在无需碳源的前提下,实现废水的生物脱氮. 其原理是在厌氧条件下,以亚硝态氮为电子受体,氨氮为电子供体,生成氮气. 第一座生产规模的 ANAMMOX 反应器也在荷兰鹿特丹的 Dokhaven 市政污水处理厂运行成功<sup>[1]</sup>. 目前该技术的最高氮负荷已达 76kg·(m³·d) -1<sup>[2]</sup>,显示出很好的应用前景. 由于该技术以亚硝态氮为电子受体<sup>[3]</sup>,工程师们多采用半亚硝化-ANAMMOX 联合工艺处理低 C/N 比的含氨废水,如城市垃圾填埋场的垃圾渗滤液<sup>[4,5]</sup>、污泥上清液<sup>[6,7]</sup>、味精废水<sup>[8]</sup>、焦化废水<sup>[9]</sup>、养猪场废水<sup>[10]</sup>. 但对于同时含有氨氮和硝态氮的低碳废水,如电镀废水,关键是需先将废水中的硝态氮转化为亚硝态氮,然后利用氨氮作电子供体,通过厌氧氨氧化途径脱氮.

通常不易发现反硝化过程中的亚硝态氮累积现象. 而反硝化过程是分步进行的:  $NO_3^- \longrightarrow NO_2^- \longrightarrow NO \longrightarrow N_2O \longrightarrow N_2$ , 在某些条件下, 可以观察到亚硝态氮得以累积的现象. 在已有的研究中表

明,影响反硝化过程中NO<sub>2</sub>-N积累的因子有微生物种类<sup>[11]</sup>、pH 值<sup>[12,13]</sup>、碳源种类<sup>[14~18]</sup>和 C/N比<sup>[18,19]</sup>等. 因此可以把反硝化控制在半反硝化(亚硝酸盐积累)阶段,与厌氧氨氧化工艺联合处理同时含氨和硝酸盐的废水,能实现高效脱氮,且不需曝气.能源消耗少,是低碳的途径.

Sun 等<sup>[14]</sup>认为碳源对半反硝化有重要影响,乙酸钠为碳源时,反硝化过程中获得的亚硝酸盐积累浓度最大,乙醇和甲醇次之;另外 C/N 比小于 3.75 时,生物脱氮的反硝化过程中有明显的亚硝态氮积累.曹相生等<sup>[18]</sup>则以甲醇为碳源,在 C/N 为 0~4 的条件下研究亚硝酸盐的积累,发现 C/N≥4 时难以获得亚硝酸盐的积累;当进水 C/N 为 2.4~3.2 之间时,能获得的亚硝态氮累积率大约为 25%; C/N < 2.4 时,亚

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硝酸盐的积累率降低. 因此 C/N 比是影响反硝化进程的另一个重要影响因素. 本研究以乙酸钠为碳源时,实现高效稳定的  $NO_2^-$  累积率的最佳 C/N 比,获得半反硝化工艺控制条件,以期为同时含氨氮和硝酸盐废水的工程治理提供技术支持.

#### 1 材料与方法

#### 1.1 试验仪器及设备

半反硝化污泥的培养采用 3.2 L 的圆柱形厌氧 SBR 反应器(图 1). 反应器的运行模式为:充水比 1/2;每天运行 8 个周期;每个运行周期 t=3 h(也即 HRT = 6 h),其中进水 15 min,反应 45 min,沉淀 90 min,出水 15 min,排泥 15 min. 反应器置于 30℃ 的恒温水浴中,搅拌速度维持在 52 r·min<sup>-1</sup>.

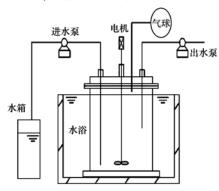


图 1 半反硝化试验装置

Fig. 1 Experimental setup for partial denitrification

### 1.2 批式试验

硝态氮起始浓度为  $60~\text{mg}\cdot\text{L}^{-1}$ , pH 值为 5.5~左 右,分别控制 C/N 比为 2.5:1、3.0:1、3.5:1 和 4.0 : 1,研究不同 C/N 比对反硝化过程中亚硝酸盐积累速率的影响. 将淘洗浓缩后的污泥分装到 18~支50~mL 比色管中,使污泥浓度 (MLSS) 为  $3400~\text{mg}\cdot\text{L}^{-1}$ . 将原水用氮气吹脱 5~min,然后分别加入 18~支比色管中,并定容至 50~mL,迅速将其密封,横着放入 30℃的恒温水浴振荡器中进行培养并定时取样.

#### 1.3 接种污泥及人工配水

SBR 反应器的接种污泥取自苏州新区第一污水厂,SBR 反应器的污泥浓度 MLSS 为3 200  $\text{mg} \cdot \text{L}^{-1}$ . 人工配水的成分为( $\text{mg} \cdot \text{L}^{-1}$ ):硝酸钾和乙酸钠按需投加;氯化钙 220,硫酸镁 82,磷酸二氢钾 5,碳酸氢钠 300. 微量元素 $^{[3]}$ 1.25  $\text{mL} \cdot \text{L}^{-1}$ .

Abeling 等发现[20], 当亚硝酸 $(HNO_2)$ 的浓度达到  $0.13 \text{ mg} \cdot \text{L}^{-1}$ 时, 便会抑制反硝化. 因而低的 pH 值能使反硝化过程产生的亚硝酸盐以亚硝酸的形式

存在而抑制反硝化过程, 出现亚硝酸盐的积累. Thomsen 等 $^{[12]}$ 也发现 pH 在 5.5 时, 反硝化过程中有较高的亚硝酸盐积累; 随着 pH 的升高, 亚硝酸盐的积累浓度逐步降低; 当 pH = 8.5 时, 硝态氮可直接转化为氮气, 无亚硝酸盐的积累. 因而本试验的原水用1  $\text{mol·L}^{-1}$ 的 HCl 调节 pH 为 5.5 ~ 6 之间, 以期获得亚硝酸盐的积累.

在试验控制的 pH 值范围内,溶液中的氨氮将以NH<sub>4</sub><sup>+</sup>-N的形式存在,不会对微生物产生抑制,并且其在有机缺氧环境下不会发生氧化反应. 因此在本试验中,未在原水中添加氨氮,未考察氨氮对反硝化过程中亚硝酸盐积累的影响.

### 1.4 试验采用的测量方法

各项指标测定方法均参照文献 [21], 其中 $NH_4^+$ -N采用纳氏试剂分光光度法;  $NO_2^-$ -N采用 N-(1-萘基)-乙二胺分光光度法;  $NO_3^-$ -N采用紫外分光光度法; COD 采用快速消解分光光度法; pH 采用玻璃电极法.

#### 1.5 计算公式

尽管反硝化副产物有 NO、N<sub>2</sub>O 等,但是 pH 会 使硝酸盐和亚硝酸盐的还原过程中的产物发生变 化,因此本文用简化的反硝化模式进行分析:

$$NO_3^- \longrightarrow NO_2^- \longrightarrow N_2$$

反硝化的两步反应的比转化速率可描述如下.

硝酸盐的比转化速率:

$$\frac{\gamma_{\text{NO}_{\bar{3}}}}{X} = -\frac{\Delta \text{ NO}_{\bar{3}}^{-}\text{-N}}{X\Delta t}$$
 (1)

亚硝酸盐的比转化速率(有硝酸盐时):

$$\frac{\gamma_{\text{NO}_{2}^{-}}}{X} = \frac{\Delta \text{ NO}_{2}^{-} - \text{N}}{X\Delta t}$$
 (2)

亚硝酸盐的比转化速率(无硝酸盐时):

$$\frac{\gamma_{\text{NO}_2^-}}{X} = -\frac{\Delta \text{ NO}_2^- - N}{X\Delta t}$$
 (3)

式中,  $\gamma_{NO_3^-}$  为硝酸盐(以 N 计)的转化速率 [ $mg\cdot(L\cdot h^{-1})$ ];  $\gamma_{NO_2^-}$  为亚硝酸盐(以 N 计)的转化速率 [ $mg\cdot(L\cdot h^{-1})$ ]; X 为污泥浓度,即 MLSS ( $g\cdot L^{-1}$ ).

#### 2 结果与讨论

#### 2.1 反应器运行试验结果与讨论

反应器运行分为两个阶段. 0~7 d是反应器启动阶段,提高污泥的反硝化活性. 第8~70 d为半反硝化的条件控制研究阶段,考察 C/N 比对半反硝化的

影响. 试验结果见图 2. 在反应器的整个运行期间, 氨氮的生成量都低于1 mg·L<sup>-1</sup>(试验结果未列出).

 $0 \sim 7$  d 为反应器启动阶段,反应器进水含 COD 和NO<sub>3</sub><sup>-</sup>-N分别为 105 mg·L<sup>-1</sup>和 30 mg·L<sup>-1</sup>, C/N 比为 3.5,进水 pH 值为 7 左右. 除前 3 d 出现不适应低碳源的现象外,反应器显示出较强的反硝化能力,没有出现NO<sub>5</sub><sup>-</sup>-N的积累,NO<sub>3</sub><sup>-</sup>-N全转化为 N<sub>3</sub>.

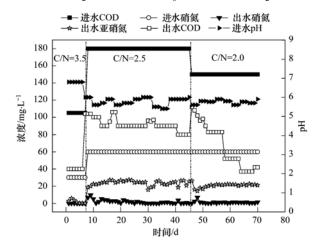


图 2 反应器进出水水质变化

Fig. 2 Variation of substrates in SBR reactor

第 8 ~ 45 d, 把进水 pH 值降为 5.5 左右, 同时进水NO $_3^-$ -N浓度提高至 60 mg·L $^{-1}$ , COD 浓度提高至 180 mg·L $^{-1}$ , C/N 比为 3. 试验期间, 硝酸盐在反硝化过程中几乎全被转化, 而亚硝酸盐开始有明显的积累, 最高达 27.2 mg·L $^{-1}$ (图 2), 积累率达 45%(图 3), 优于曹相生等 $^{[18]}$ 的 25% 积累水平. 反应器的出水 COD 由最初的 92 mg·L $^{-1}$ 左右, 逐渐稳定为83 mg·L $^{-1}$ 左右.

第 46 ~ 70 d,原水 $NO_3^-$ -N浓度仍为 60 mg· $L^{-1}$ , pH 值为 5.8 左右,把 COD 浓度降至 150 mg· $L^{-1}$ , C/N 比为 2.5,其它运行条件不变. 试验表明,COD浓度对反硝化过程中亚硝酸盐的积累有明

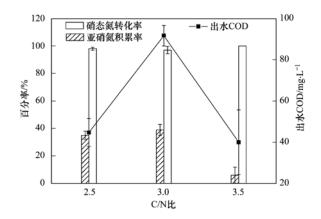


图 3 SBR 反应器中 C/N 比对反硝化过程的 亚硝酸盐积累率的影响

Fig. 3 Effect of C/N ratios on nitrite accumulation during denitrifying process in SBR reactor

显影响. 反硝化过程中硝酸盐几乎全被转化,而亚硝酸盐的积累率先有所下降,最低仅为 24%. 随着反应器中污泥对碳源浓度的适应,亚硝酸盐的积累率有所上升,最高达 37%,相应的 $NO_2^-$ -N出水浓度为 22. 4  $mg \cdot L^{-1}$ ,出水 COD 为 42  $mg \cdot L^{-1}$ . SBR 反应器中相应的污泥浓度(MLSS) 为3 400  $mg \cdot L^{-1}$ .

在整个反应期间,未对 SBR 反应器进行排泥,而反应器的运行也未出现出水带泥的情况,静置期的泥面未明显升高. 而 Mateju 等<sup>[22]</sup>的研究曾表明,若把微生物的分子式记为 C<sub>5</sub>H<sub>7</sub>NO<sub>2</sub>,用乙酸进行反硝化时的污泥产率系数为 0.07,而用典型有机物进行反硝化时的污泥产率系数为 0.11,用葡萄糖进行反硝化时的污泥产率系数为 0.18; McAdan 等<sup>[23]</sup>的研究则表明,利用乙醇进行反硝化时的污泥产率系数为 0.14(见表 1).可见,用乙酸或乙酸钠进行反硝化时的污泥产量较低,可以降低剩余污泥的处置费用. 而 ANAMMOX 细菌的世代期长,若该反应与ANAMMOX 工艺联用时,因反硝化细菌生长过快而将 ANAMMOX 菌淘汰的可能性降低.

表 1 反硝化的计量反应方程式

Table 1 Stoichiometries of denitrification reactions

碳源种类	计量反应方程式	文献
乙酸	$0.\ 82 \mathrm{CH_{3}C00H} + \mathrm{NO_{3}^{-}} \longrightarrow 0.\ 07 \mathrm{C_{5}H_{7}NO_{2}} + \mathrm{HCO_{3}^{-}} + 0.\ 30 \mathrm{CO_{2}} + 0.\ 90 \mathrm{H_{2}O} + 0.\ 47 \mathrm{N_{2}}$	[22]
典型有机物	$0.3C_{5}H_{3}NO + NO_{3}^{-} + H^{+} \longrightarrow 0.11C_{5}H_{7}NO_{2} + 0.95CO_{2} + 1.17H_{2}O + 0.5N_{2} + 0.19NH_{4}^{+}$	[ 22 ]
乙醇	$0.69C_2H_5OH + NO_3^- + H^+ \longrightarrow 0.14C_5H_7NO_2 + 0.67CO_2 + 2.07H_2O + 0.43N_2$	[23]
葡萄糖	$0.\ 36C_{6}H_{12}O_{6} + NO_{3}^{-} + 0.\ 18\ NH_{4}^{+} + 0.\ 82H^{+} \longrightarrow 0.\ 18\ C_{5}H_{7}NO_{2} + 1.\ 25CO_{2} + 2.\ 28H_{2}O + 0.\ 5N_{2}$	[ 22 ]

# 2.2 不同 C/N 比对反硝化过程中亚硝酸盐积累的 影响

在批式处理研究中发现,C/N 比为 2.5:1、3.0:1、3.5:1和 4.0:1时,硝酸盐的转化基本都在 45 min

内完成; 而亚硝酸盐都在 45 min 时达到最高值, 随后逐渐下降; pH 值逐步升高; COD 值逐渐下降(见图 4).

反硝化过程的半反应方程式如式(4)和式(5)

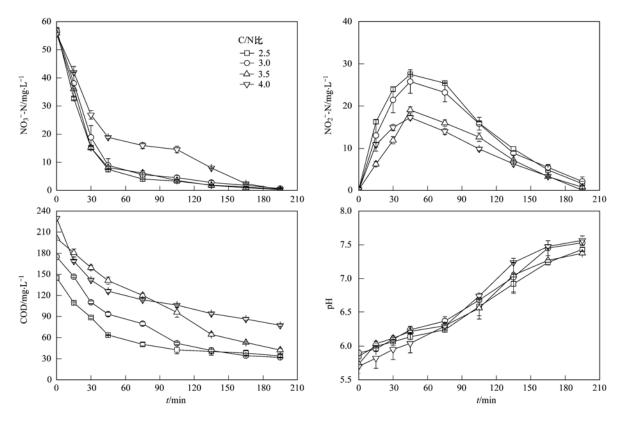


图 4 不同 C/N 时各底物的变化

Fig. 4 Variation of substrates during denitrification at different initial C/N ratios in batch experiment

所示[24]:

$$\frac{1}{2}NO_{3}^{-} + \frac{1}{2}H^{+} + e^{-} \longrightarrow \frac{1}{2}NO_{2}^{-} + \frac{1}{2}OH^{-},$$

$$\Delta G^{0'} = -41.45 \text{ kJ} \cdot \text{mol}^{-1} \qquad (4)$$

$$\frac{1}{3}NO_{2}^{-} + \frac{2}{3}H^{+} + e^{-} \longrightarrow \frac{1}{6}N_{2} + \frac{2}{3}OH^{-},$$

$$\Delta G^{0'} = -30.75 \text{ kJ} \cdot \text{mol}^{-1} \qquad (5)$$

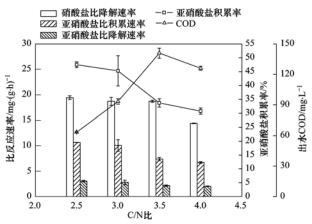
式中, $\Delta C^{0}$ 为标准状态和 pH = 7 时,反应释放的自由能.

由式(4)和式(5)可以看出,获得单位摩尔的电子当量,硝酸盐还原比亚硝酸盐还原释放更多的自由能.因此在相同条件下,硝酸盐的还原过程比亚硝酸盐的还原更易发生,适当的反应时间可导致亚硝酸盐的积累.

有类似研究表明,当 Paracocus denitrificans 和 Pseudomonas fluorescens 纯培养菌在进行硝酸盐和亚硝酸盐的还原时,若基质所提供的电子有限时,则存在对底物电子的竞争,硝酸盐比亚硝酸更容易作为电子受体<sup>[12,25]</sup>.

C/N 为 2. 5、3. 0 和 3. 5 时获得的硝酸盐比转 化速率相近,C/N 比为 4. 0 时的略低(见图 5),说明 C/N 为 2. 5~4. 0 时,对硝酸盐的转化基本无影响.

而研究报道,只有当硝酸盐的浓度低于 0.3 mg·L<sup>-1</sup>时,反硝化速率才会受到抑制<sup>[26]</sup>.本研究中, C/N为 2.5 以上,碳源充足,因此微生物的生长仅与电子受体硝酸盐的浓度有关.



COD 为亚硝酸盐积累浓度最大时的剩余浓度 图 5 不同 C/N 比对反硝化速率的影响

Fig. 5 Effect of C/N ratios on denitrification rates in batch experiment

C/N 比为 2. 5 和 3. 0 时的亚硝酸盐比积累速率相近,分别为(30. 17 ± 1. 70) mg·(g·h)  $^{-1}$ 和(29. 92 ± 1. 90) mg·(g·h)  $^{-1}$ ,相应亚硝酸盐的积累率分别为47. 50% ± 1. 005%和45. 28% ± 5. 469%.而 C/N

比为3.5和4.0时的亚硝酸盐比转化速率和亚硝酸盐的比积累速率下降明显,亚硝酸盐的积累率也随之分别下降至33.70%±1.496%和30.75%±1.107%.说明C/N比对亚硝酸盐的积累有较大影响,C/N比为2.5~3.0有利于亚硝酸盐的积累.

虽然 C/N 比 2.5 和 3.0 时的亚硝酸盐积累率相近,但是 C/N 比为 2.5 条件下,当亚硝酸盐积累浓度达到最高时的剩余 COD 浓度低于 60 mg·L<sup>-1</sup>,一方面可减少反硝化过程的外加碳源量而减少药剂费用的支出,另外也可减少对 COD 的后续处理费用,降低有机物对 ANAMMOX 过程的不利影响.

#### 3 结论

- (1)在 SBR 运行过程中, C/N 为 3 时, 获得的亚硝酸盐积累率可达 45%.
- (2) SBR 连续试验和批式试验都表明, MLSS 为  $3.4 \text{ g} \cdot \text{L}^{-1}$ 时, 反应 45 min 时可获得最大的亚硝酸盐积累率.
- (3) C/N 为 2.5 ~ 4.0 时,对硝酸盐的转化速率基本无影响,但对亚硝酸盐的积累速率影响显著, C/N 为 2.5 和 3.0 时有利于亚硝酸盐的积累, C/N  $\geqslant$  3.5 时,亚硝酸盐积累率下降显著. C/N 为 2.5 和 3 时,亚硝酸盐积累率分别为 47.50% ± 1.005% 和 45.28% ± 5.469%,亚硝酸盐比积累速率分别为 (30.17 ± 1.70) mg·(g·h)  $^{-1}$  和 (29.92 ± 1.90) mg·(g·h)  $^{-1}$ .

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