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硫自养填充床反应器降解水中高浓度高氯酸盐的特性 及菌群分析

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摘要:采用硫自养填充床反应器处理模拟高浓度高氯酸盐(ClO_4^-)污染水,考察不同进水 ClO_4^- 浓度及水力停留时间(HRT) 下的 ClO_4^- 降解特性. 结果表明,HRT 为 12 h 时,进水 ClO_4^- 浓度由 50 mg· L^{-1} 增加到 194 mg· L^{-1} 时, ClO_4^- 能被完全降解;进水 ClO_4^- 浓度为 194 mg· L^{-1} 时,HRT 由 12 h 减少至 4 h 时, ClO_4^- 法除率仅为 74%; SO_4^{2-} 的产量随着进水 ClO_4^- 浓度与 HRT 的增加而增加;进水 PH 和碱度(以 $CaCO_3$ 计)分别为 8.0 和 500 mg· L^{-1} ,出水 PH 和碱度(以 $CaCO_3$ 计)分别为 6.7 和 100 mg· L^{-1} ;反应器底部氧化还原电位(ORP)稳定在 $-380 \sim -330$ mV,反应器上部氧化还原电位(ORP)稳定在 $-300 \sim -250$ mV. 分子生物学分析表明,反应器内的菌群结构随高度的变化而变化,硫氧化菌 Sulfurovum 随反应器高度的增加而减少,由底部 57.78%减少到上部的 32.19%,同时硫化氢氧化菌 Hydrogenophilaceae 随反应器高度的增加而增加,由底部 4.35% 增加到上部的 22.24%.

关键词: 硫自养; 高氯酸盐; 水力停留时间(HRT); 氧化还原电位(ORP); 菌群分析

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Characteristics of Perchlorate Reduction and Analysis of Consortium Structure in a Sulfur-Based Reactor at a High Perchlorate Concentration

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Abstract: The effects of perchlorate concentration and hydraulic retention time (HRT) on perchlorate reduction characteristics were investigated in a sulfur-based perchlorate reduction reactor. The results showed that the perchlorate was completely removed at HRT of 12 h and the influent perchlorate concentration ranged from 50 mg·L⁻¹ to 194 mg·L⁻¹; The perchlorate removal efficiency was 74% at HRT of 4 h and the influent perchlorate concentration was 194 mg·L⁻¹; The yield of sulfate was increased by increasing the influent perchlorate concentration and HRT; The influent pH and alkalinity was approximately 8.0 and 500 mg·L⁻¹ CaCO₃, and the effluent pH and alkalinity was approximately 6.7 and 100 mg·L⁻¹ CaCO₃, respectively; The oxidation reduction potential (ORP) ranged from –380 mV to –330 mV at the bottom of the reactor, however, ORP ranged from –300 mV to –250 mV at the top of the reactor; The molecular biological analysis showed that the microbial consortium structure was different along the flow path in the reactor, Sulfurovum which is known to oxidize sulfur was decreased from 57.78% to 32.19% and Hydrogenophilaceae which is known to oxidize hydrogen sulfide was increased from 4.35% to 22.24%.

Key words: sulfur autotrophic; perchlorate; hydraulic retention time (HRT); oxidation reduction potential (ORP); consortium structure analysis

近些年,地表水和地下水中的高氯酸盐(ClO₄⁻)污染已成为十分严峻的问题^[1,2]. ClO₄⁻ 能抑制甲状腺对碘离子的吸收从而影响人体的健康^[3,4],因此,ClO₄⁻ 污染问题逐渐得到广泛关注. 由于 ClO₄⁻ 具有非挥发性、强稳定性、高溶解性等特点,一般的物理化学法很难将其彻底去除,并且耗资大^[5~7]. 生物法因其较彻底,成本低,成为最受关注的处理ClO₄⁻ 废水的方法之一^[8,9]. 生物法分为异养生物法

和自养生物法,其中异养生物法以有机物为电子供体,污泥产量高,易造成二次污染;而自养生物法以

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无机物作为电子供体,从而避免了这些问题[10,12].

自养还原 ClO_4 所需电子供体通常是氢气、还原态硫(S^{2-} 、 S^0 、 $S_2O_3^{2-}$)、零价铁等无机物 $^{[13-18]}$,因硫颗粒微溶于水,具有来源广泛、价格低廉等优点成为自养法降解 ClO_4 的研究热点 $^{[13,15]}$. 硫自养还原 ClO_4 的理论反应如式(1)所示 $^{[15]}$:

 $3ClO_4^- + 4S + 4H_2O \longrightarrow 3Cl^- + 4SO_4^- + 8H^+ (1)$ 微生物以硫颗粒作为电子供体,以 ClO_4^- 作为电子受体,最终将 ClO_4^- 完全转化为无毒无害的 Cl^- 同时产生副产物 $SO_4^{2-[19,20]}$.

2007 年 Ju 等 [10] 通过首次研究了硫颗粒作为电子供体自养还原 ClO_4^- 的可行性,同时分析了 SO_4^{2-} 实际产量高于理论产量的原因是由于硫歧化的发生.目前许多研究者都是利用硫自养反应器降解低浓度的 ClO_4^- 废水.如 2009 年 Gao 等 [21] 利用硫自养反应器将 ClO_4^- 浓度为 $4 \sim 8$ mg·L $^{-1}$ 的废水降至 0.05 mg·L $^{-1}$ 以下. 2011 年 Boles 等 [16] 利用硫自养反应器进行了中试规模的实验,将 ClO_4^- 浓度为 12.6 mg·L $^{-1}$ 的废水降至 15 μ g·L $^{-1}$ 以下.除此之外,利用硫自养反应器进行污染物如 ClO_4^- 、 NO_3^- 等的降解时,最大的问题就是出水 SO_4^{2-} 浓度过高 [21,22].

尽管近年来对于硫自养填充床反应器降解 ClO_4^- 的研究已逐渐成熟,但目前对于硫自养反应器 处理高浓度 ClO_4^- 废水,和如何控制硫自养反应器 出水 $SO_4^{2^-}$ 过高的问题还没有很好的解决方案. 基于以上研究背景,本文以硫自养填充床反应器降解 ClO_4^- 为体系,通过改变进水 ClO_4^- 浓度和 HRT,着 重考察硫自养反应器去除 ClO_4^- 的效率、 $SO_4^{2^-}$ 的产率及反应过程中 pH 及碱度的消耗量,同时对反应器内菌群结构进行分析,进一步考察其降解 ClO_4^- 的规律,以期为该技术的实际应用提供参考.

1 材料与方法

1.1 实验装置

本实验所用硫自养 ClO_4^- 还原反应器为升流式固定床反应器,图 1 为实验装置. 反应器内径为 10 cm,有效高度为 45 cm,材质为有机玻璃. 反应器内填充载体为硫单质和石英砂($V_{\text{fl}}: V_{\text{fl}}=2:1$),装填体积为 3.6 L. 硫颗粒和石英砂粒径均为 2~3 mm,反应器内孔隙率为 0.4,反应器有 4 个不同高度的出水口,分别为出水口 1(60 mm),出水口 2(180 mm),出水口 3(300 mm),出水口 4(390 mm),菌群

结构分析所取泥样分别来自取样口1(60 mm)和取样口3(300 mm).

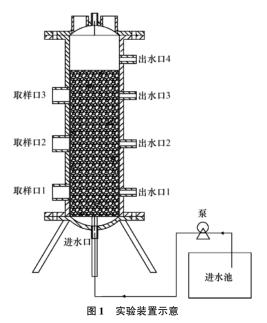


Fig. 1 Schematic of the experimental apparatus

1.2 实验材料

本实验采用的活性污泥取自石家庄市桥西污水 处理厂的二次沉淀池,将污泥沉降半小时,取沉降后 的污泥与按比例混匀的硫和石英砂混匀,直接填充 至反应器内,测得此时的污泥量(以 SS 计)为 8 $g \cdot L^{-1}$. 然后开始间歇式驯化挂膜 7 d(先进水后停 滞 12 h,然后连续运行 12 h,依次重复 7 d),开始连 续进水. 采用人工模拟高浓度 ClO4 地下水,反应器 进水均用自来水配制,其主要成分: K,HPO4·3H,O, $0.25 \text{ g} \cdot \text{L}^{-1}$; NaHCO₃ · H₂O, $1.50 \text{ g} \cdot \text{L}^{-1}$; NH₄Cl, 0.15 g·L⁻¹; NaClO₄, 0.14~0.27 g·L⁻¹(在不同阶 段 ClO₄ 的浓度由 50 mg·L⁻¹增加到 194 mg·L⁻¹); 微量元素为1 mL·L-1. 微量元素培养基的组成: EDTA, 0.50 mg·L⁻¹; MnSO₄·H₂O, 0.50 mg·L⁻¹; $FeSO_4 \cdot 7H_2O_7$, 0. 10 $mg \cdot L^{-1}$; $CaCl_2$, 0. 10 $mg \cdot L^{-1}$; $ZnSO_4 \cdot 7H_2O$, 0. 10 mg·L⁻¹; $CuSO_4 \cdot 5H_2O$, 0. 01 $\text{mg} \cdot \text{L}^{-1}$; $\text{Na}_2 \text{MoO}_4 \cdot 2\text{H}_2\text{O}$, 0.01 $\text{mg} \cdot \text{L}^{-1}$; $\text{Na}_2 \text{WO}_4 \cdot$ $2H_2O$, 0.01 mg·L⁻¹, NiCl₂·6H₂O, 0.02 mg·L⁻¹.

1.3 运行条件

实验运行调控参数如表 1,前 4 个阶段水力停留时间(HRT)为 12 h,ClO $_4$ 的浓度由 50 mg·L $^{-1}$ 依次增加到 100、150、194 mg·L $^{-1}$,然后 ClO $_4$ 浓度稳定在 194 mg·L $^{-1}$,减少 HRT 分别为 9 h 和 4 h,共分为 6 个阶段. 每个阶段内,定时监测进出水各离子浓度、pH 值和碱度等指标,待出水 ClO $_4$ 浓度连续

4 d 去除率在 96% 以上,则改变条件开展下一阶段 的实验.

表 1 运行调控参数

Table 1 Operation control para	ameters
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阶段	I	П	Ш	IV	V	VI
时间/d	1 ~ 25	26 ~ 37	38 ~ 51	52 ~61	62 ~ 89	90 ~ 116
$ClO_4^-/mg \cdot L^{-1}$	50	100	150	194	194	194
流量/mL·h ⁻¹	300	300	300	300	400	900
HRT/h	12	12	12	12	9	4
ClO ₄ 负荷/mg·(L·h) -1	4. 17	8. 33	12. 5	16. 17	21.56	48. 5

1.4 分析方法

ClO₄ 、SO₄ ² 、Cl ⁻ 、S² 都用离子色谱 (Dionex 1100)进行测定,所用分析柱为 AS20 (4 mm × 250 mm)和保护柱 AG20 (4 mm × 50 mm). 采用梯度淋洗的方法,淋洗液为 KOH, (0 ~ 10 min 为 15 mmol·L ⁻¹淋洗, 10 ~ 21 min 为 40 mmol·L ⁻¹淋洗),淋洗液流速 1.0 mL·min ⁻¹,柱温 30℃. 反应器不同位置的菌群结构基于 16S rDNA 基因的 V3-V4 区 DNA 序列 PCR 扩增与高通量测序技术进行分析,其中测试的序列数分别为 1361 (反应器上部菌群结构)和 1498 (反应器下部菌群结构) (上海生工生物工程有限公司);pH 使用 PHS225 C 型数字酸度计进行测定;碱度采用标准方法进行测定 [²³].

2 结果与讨论

2.1 硫自养反应器高氯酸盐的去除及硫酸盐的产生 反应器在运行期间 ClO_4^- 的去除效果及 SO_4^{2-} 的产生量如图 2 所示. 反应器在运行的前 4 个阶段 HRT 为 12 h,主要考察进水 ClO_4^- 的浓度分别为 50、100、150 和 194 $mg \cdot L^{-1}$ 时对反应器降解 ClO_4^- 及生成 SO_4^{2-} 的影响;反应器运行的第 $IV \times V \times II$ 阶段主要考察在进水 ClO_4^- 浓度为 194 $mg \cdot L^{-1}$ 时,HRT 分别为 12、9、4 h 时反应器降解 ClO_4^- 及生成 SO_4^{2-} 的影响.

反应器在运行的前 5 个阶段分别在第 23、33、48、58 和 85 d 时 ClO_4^- 的去除率达到了 100%; 第 VI 阶段,HRT 减少为 4 h,进水 ClO_4^- 浓度 194 mg·L⁻¹, ClO_4^- 的去除率最终稳定在 72% 左右. 以上结果表明,在维持较长的 HRT 条件下,进水 ClO_4^- 的浓度增加至 194 mg·L⁻¹时, ClO_4^- 的去除率并不会受太大影响,然而减少 HRT,降低了 ClO_4^- 传递至生物膜内的时间,导致 ClO_4^- 的去除率受到了较大影响,所以传质是限制 ClO_4^- 降解速率的主要原

因^[10]. Sahinkaya 等^[22]研究表明较高的上升流速会限制硫的溶解从而影响了硫自养的降解效率.

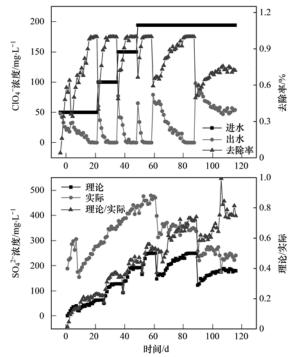


图 2 硫自养反应器运行过程中高氯酸盐及硫酸盐的变化

Fig. 2 Perchlorate and sulfate concentration variations during operation of the sulfur-based reactor

随着进水 ClO_4^- 浓度的增加, SO_4^{2-} 的产生量也在增加,进水 ClO_4^- 浓度为 194 mg·L⁻¹时,由反应方程式(1)得对应的理论 SO_4^{2-} 的量 250 mg·L⁻¹(国家饮用水标准),而实际产生的 SO_4^{2-} 的量高达 500 mg·L⁻¹. 万东锦等^[24]的研究表明在缺溶解氧及缺少电子受体的条件下,硫颗粒上的歧化菌即会发生歧化反应. Ju 等^[10]通过序批式实验观察到了硫自养 ClO_4^- 还原过程中存在着硫歧化反应. 当进水 ClO_4^- 的浓度为 194 mg·L⁻¹,HRT 由 12 h 减少到 4 h时,由图 2 可得,出水 SO_4^{2-} 的量由 500 mg·L⁻¹减少到 250 mg·L⁻¹. 其原因可能是进水流速增加抑制了硫歧化细菌的生长,并且在较快的水力冲刷速度下

减少了硫歧化的反应时间^[10]. 同时由图 2 可得理论产生 SO_4^{2-} 的量与实际产生 SO_4^{2-} 的量的比值随着进水 CIO_4^- 浓度的增加而增加,硫的利用率也在增加. 结果表明,增加进水 CIO_4^- 浓度及减少 HRT都会提高硫的利用率,但当上升流速过快时,由于 CIO_4^- 的去除率低,对 SO_4^{2-} 的产量影响较大.

2.2 pH 和碱度的变化

如图 3 所示, 反应器的进水 pH 稳定在 8.0 左 右,出水 pH 稳定在 6.5~7.0 之间,反应方程式(1) 表明硫自养降解 ClO₄ 是一个产酸的反应,1 mol 的 ClO_4 产生 2.66 mol 氢质子. 在前 4 个阶段,碱度消 耗随着进水 ClO4 浓度的增加而增加,进水 ClO4 浓 度由 50 mg·L⁻¹增加到 194 mg·L⁻¹时对应的 pH 变 化不大,并且稳定在 6.7 左右,同时碱度(以 CaCO, 计)消耗由 200 mg·L⁻¹增加到 450 mg·L⁻¹,在后 3 个阶段, HRT 对出水 pH 波动较大, 并且随着 HRT 的减少而增加,碱度消耗量随着 HRT 的减少而降 低. 进水 ClO₄ 浓度为 194 mg·L⁻¹, 当 HRT 由 12 h 减少到 4 h 时, 碱度(以 CaCO, 计)消耗由 450 mg·L⁻¹减少到 300 mg·L⁻¹. Sahinkaya 等^[22]研究表 明硫自养降解过程中会有硫歧化的发生,该反应是 一个产酸的反应. 在缺溶解氧、缺少电子受体的条 件下,会发生硫歧化反应,增加上升流速,抑制了硫

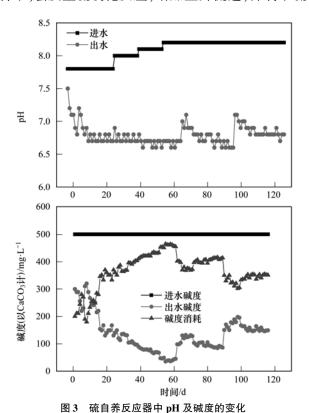


Fig. 3 Variations of pH and alkalinity in the sulfur-based reactor

歧化菌的增长,缩短了硫歧化的反应时间,即减少了硫歧化反应的发生,同时减少了酸的产生量,也减少了碱度消耗^[8,21]. 结果表明,碱度消耗量随进水ClO₄ 浓度的增加而增加,随 HRT的减少而减少.

2.3 硫自养反应器内 ORP 的变化

ORP 的变化与反应器体系内的反应相关,所以通过检测反应器内不同位置的 ORP 能够间接地反映反应器不同位置所发生的反应^[25].通过检测不同位置 ORP、出水 SO²⁻和 ClO⁻浓度的变化,来表征反应器内不同位置主反应的变化,同时对反应器的不同位置进行菌群结构分析.

如图 4 所示,反应器下部(距反应器底部 60 mm),在运行第 10 d 的时候基本稳定在 - 380 mV,并且随进水 ClO₄ 浓度的变化并没有太大变化,但当 HRT 为 9 h 和 4 h 时,ORP 分别为 - 360 mV 和 - 330 mV. 溶解氧的减少会导致 ORP 的降低^[26],减少 HRT 即增加进水流速,导致进水溶解氧的消耗减慢,从而使 ORP 增加. 反应器上部(距反应器底部 300 mm)的 ORP 随着驯化时间的增加,由反应器最开始的 - 370 mV 增加到 - 260 mV,由此可得,反应器上部发生的主体反应在发生变化,同时反应器下部和反应器上部的菌群结构在发生变化.

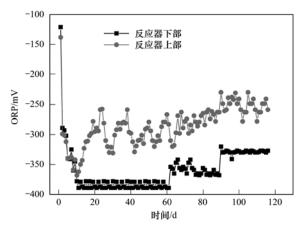


图 4 硫自养反应器中 ORP 的变化

Fig. 4 Variations of ORP in the sulfur-based reactor

如图 5 所示,在反应器的不同出水高度进行取样测定,在前 4 个阶段中,ClO₄ 在距反应器底部 300 mm 的高度时就已经降解完全,且在距反应器底部 390 mm 的高度中 SO₄ 的产生量并不会随着 ClO₄ 已经降解完全而减少,说明在反应器的上部发生的是其他的副反应如硫歧化反应^[16].为了进一步分析硫自养反应器内不同位置发生的不同反应,从反应器上部和下部中分别采样进行菌群结构分析,如图 6 所示,实验结果展示了属水平菌群分布随

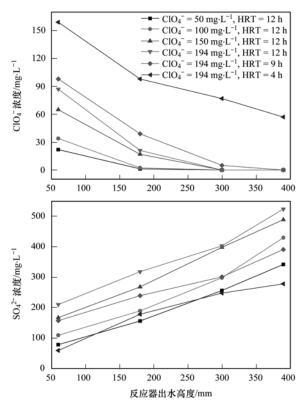
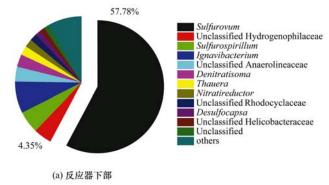


图 5 高氯酸盐及硫酸盐浓度随反应器出水高度的变化

Fig. 5 Variations of perchlorate and sulfate concentrations along the flow path in the reactor



反应器高度的变化,反应器下部与反应器上部的优势菌群均为 Sulfurovum,所占比例分别为 57.78% 和 32.19%,但反应器上部 Hydrogenophilaceae 的比例为 22.24%,而反应器下部 Hydrogenophilaceae 的比例仅为 4.35%. Gao 等 [21] 得出 Sulfurovum 为硫氧化菌可以将硫氧化成 SO_4^{2-} ,同时将 CIO_4^{-} 还原成无毒无害的 CI^{-} . Hydrogenophilaceae 为一种嗜氢菌,分析可能是由于反应器内存在硫歧化反应进而产生硫化氢,而反应器下部的比例要小于反应器上部的比例,所以反应器内下部主要是硫的氧化及 CIO_4^{-} 的降解,而反应器上部主要的反应体系为硫氧化及硫化氢的氧化.

3 结论

- (1)SO₄⁻ 理论产生的量与实际产生的量的比值,由第 I 阶段的 0.2 最终增加到第 VI 阶段的 0.8,说明硫自养降解 ClO₄⁻ 过程中硫的利用率会随着 ClO₄⁻ 的进水浓度增加及 HRT 的减少而增加.
- (2) 进水 pH 值与碱度(以 CaCO₃ 计)分别稳定在 8.0 左右和 500 mg·L⁻¹,而出水 pH 值与碱度(以 CaCO₃ 计)分别为 6.7 和 100 mg·L⁻¹,说明硫自养降解ClO₄ 的过程是一个产酸耗碱的过程,同时碱

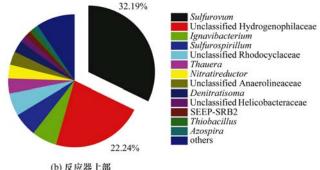


图 6 属水平菌群分布随反应器高度的变化

Fig. 6 Variation of microbial flora by genus along the flow path in the reactor

度消耗随进水 ClO₄ 浓度的增而增加,随 HRT 减少而减少.

(3)硫自养反应器内的菌群结构随高度的变化而变化,硫氧化菌 Sulfurovum 在反应器下部与上部的比例分别为 57. 78% 与 32. 19%, 硫化氢氧化菌 Hydrogenophilaceae 在反应器下部与上部的比例分别为 4. 35% 与 22. 24%.

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