Contents lists available at ScienceDirect

Ecological Engineering

journal homepage: www.elsevier.com/locate/ecoleng

The start-up of an anammox reactor as the second step for the treatment of ammonium rich refinery (IGCC) wastewater with high C_{org}/N ratio

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ARTICLE INFO

Article history: Received 6 September 2016 Received in revised form 27 April 2017 Accepted 27 April 2017 Available online 12 June 2017

Keywords: Anammox Nitrogen removal Industrial wastewater Integrated gasification combined cycle Organic carbon Petrochemical wastewater

ABSTRACT

In this study, the refinery wastewater produced by the integrated gasification combined cycle (IGCC) and pre-treated in a lab-scale partial nitritation chemostat was fed to a granular anammox reactor, in order to evaluate its feasibility as the final treatment step. The IGCC wastewater was characterized by high NH₄-N concentration ($540 \pm 82 \text{ mg L}^{-1}$), high organic carbon to nitrogen ratio (C_{org}/N), and by the presence of toxic substances. A conservative exponential law was adopted to progressively replace the synthetic influent with the pre-treated IGCC wastewater, in order to avoid any stressful conditions which could hinder the process. An increase in specific anammox activity (SAA) up to 0.104 gNO₂-N gVSS⁻¹ d⁻¹ was initially observed, suggesting that stimulation may occur if pre-treated IGCC wastewater dilution is sufficiently high. A system malfunction caused a worsening of process performance, which was partially restored: when only pre-treated IGCC wastewater was fed, the nitrogen removal efficiency and SAA were $71 \pm 3\%$ and 0.045 ± 0.002 gNO₂-N gVSS⁻¹ d⁻¹, respectively, and the removal of organic matter due to denitrification was negligible. As to physical/morphological properties of anammox granules, they did not change significantly during the whole experimental campaign. Results showed that the anammox process can be successfully applied to treat complex industrial wastewaters with high Corg/N ratio, if a conservative start-up strategy is adopted and the preliminary partial nitritation step guarantees an efficient removal of readily degradable organic matter.

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1. Introduction

The reduction of nitrogen discharge in water bodies (including freshwater, estuarine and coastal marine ecosystems) has become a priority, since high levels in water represent a threat for the environment, as well as for human health (Daverey et al., 2013; Camargo and Alonso, 2006). Anaerobic ammonium oxidation (anammox) has been recognised as a cost-effective alternative for biological nitrogen removal, compared with conventional systems. In this process, ammonium is oxidized to dinitrogen gas using nitrite as the electron acceptor, with the formation of NO₃-N as minor product: according to Strous et al. (1998), 1.32 mol of NO₂-N are required for the oxidation of 1 mol of NH₄-N(Eq. (1)). Recently, Lotti et al. (2014a) proposed a slightly different anammox stoichiome-

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http://dx.doi.org/10.1016/j.ecoleng.2017.04.068 0925-8574/© 2017 Elsevier B.V. All rights reserved. try, whereas 1.146 mol of NO₂-N are required per mole of NH₄-N (Eq. (2)).

$$\begin{split} \mathsf{N}H_4^+ + 1.32\mathsf{N}O_2^- + 0.066\mathsf{H}\mathsf{C}O_3^- + 0.13H^+ &\to 1.02\mathsf{N}_2 + 0.26\mathsf{N}O_3^- \\ + 0.066\mathsf{C}H_2\mathsf{O}_{0.5}\mathsf{N}_{0.15} + 2.03H_2\mathsf{O} \end{split} \tag{1}$$

$$NH_4^+ + 1.146NO_2^- + 0.071HCO_3^- + 0.057H^+ \to 0.986N_2 + 0.161NO_3^- + 0.071CH_{1.74}O_{0.31}N_{0.20} + 2.002H_2O$$
(2)

Since nitrite is not usually present in wastewater, the anammox process requires a pre-treatment step based on partial nitritation, where around 50% of influent ammonium is converted into nitrite by ammonium oxidizing bacteria (AOB), under aerobic conditions (Eq. (3)).

$$NH_4^+ + HCO_3^- + 0.75O_2 \rightarrow 0.5NH_4^+ + 0.5NO_2^- + CO_2 + 1.5H_2O$$
 (3)





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Nom	encl	ature

Glossary			
Anammox Anaerobic ammonium oxidation			
AOB	Ammonium oxidizing bacteria		
CSTR	Complete stirred-tank reactor		
Corg/N	Organic carbon to nitrogen ratio		
HBCR	Hybrid biofilm-carrier reactor		
HRT	Hydraulic retention time		
IGCC	Integrated gasification combined cycle		
MBR	Membrane bioreactor		
NitDR	Nitrite discharge rate		
NLR	Nitrogen loading rate		
NRE	Nitrogen removal efficiency		
NRR	Nitrogen removal rate		
SAA	Specific anammox activity		
SBR	Sequencing batch reactor		
SHARON	Single reactor for high activity ammonium removal		
	over nitrite		
SRT	Sludge retention time		
TN	Total nitrogen		
TOC	Total organic carbon		
TSS	Total suspended solids		
UABR	Upflow anaerobic bioreactor		
UASB	Upflow anaerobic sludge blanket		
VFA	Volatile fatty acids		
VSS	Volatile suspended solids		

Nowadays, the combination of a partial nitritation step followed by anammox represents a reliable, cost-effective and energetically efficient alternative for the treatment of ammonium-rich wastewaters with a low content of readily degradable organic carbon (i.e., low C_{org}/N ratio) like sludge digester supernatant, landfill leachate and livestock manure digester liquor (Jenni et al., 2014; Scaglione et al., 2012; Yamamoto et al., 2011). Compared to conventional nitrification-denitrification process, the combination of partial nitritation and anammox requires less energy for oxygen supply (-60%), there is no need for external carbon sources (the process is completely autotrophic), surplus sludge production is reduced significantly (-90%) due to the extremely low growth rate of anammox bacteria, and CO₂ emissions are reduced by 85% (Van Loosdrecht, 2008; Lackner et al., 2014). This widely accepted economical comparison makes the partial nitritation-anammox process an attractive solution even when the influent organic carbon is sufficient to sustain heterotrophic denitrification. On the other hand, if the ammonium-rich wastewater is characterized also by a high Corg/N ratio, the partial nitritation-anammox process may lack in terms of nitrogen and organic matter removal: Mosquera-Corral et al. (2005) observed a 10% inhibition of AOB activity when acetate was fed to a partial nitritation reactor (the resulting Corg/N ratio was 0.3 gC/gN). As to the subsequent treatment step (anammox), the presence of significant amounts of readily degradable organic substrate fosters the development of heterotrophic denitrifiers, since anammox bacteria are not able to compete for nitrite due to their lower growth rate (Molinuevo et al., 2009; Van Hulle et al., 2010; Tang et al., 2010). Moreover, the heterotrophic growth leads to a higher sludge production, with a corresponding decrease in the sludge retention time (SRT) due to the increasing sludge loss (Jenni et al., 2014), which is not favorable to anammox. Although the effects of readily degradable short chain fatty acids (VFAs) on anammox activity have been investigated, results are contradictory: acetate and formate were proved to inhibit anammox bacteria in continuously operated systems (Van de Graaf et al., 1996), while Kartal et al. (2007) showed that propionate, formate and acetate can be even co-metabolized by anammox biomass which use them as the electron donors, with nitrates as the electron acceptors. More specifically, Güven et al. (2005) stated that anammox bacteria have the organotrophic capability of oxidizing VFAs mostly to CO_2 , using nitrates as electron acceptors, while forming ammonium with nitrites as intermediates. Such capability was then used by Winkler et al. (2012) to explain the nitrate consumption and the low sludge production observed in two partial nitritation-anammox reactors treating VFAs containing wastewater.

Especially when dealing with complex wastewaters produced by industries like, for instance, coking, petrochemical, waste management, steel manufacturing and coal gasification factories, the presence of specific compounds may also hinder AOB or anammox activity (Ramos et al., 2015): complete inhibition of AOB was observed by Dyreborg and Arvin (1995) when phenol or cresol were present at low concentrations (3.7 and 1.3 mg L^{-1} , respectively); in batch experiments, initial concentrations of benzene, toluene and *m*-xylene above 10 mgC L^{-1} were shown to affect the ammonia oxidation pathway significantly, thus inducing a decrease in nitrification specific rates (Zepeda et al., 2006). Although anammox activity is hindered by toxic compounds like phenol, cyanides and thiocyanates at high concentrations, acclimation was shown to be achievable (Toh and Ashbolt, 2002). Ramos et al. (2015) observed that p-nitrophenol, guinolone, o-cresole and o-chlorophenol, which are commonly present in complex industrial wastewaters, caused a reduction of specific anammox activity even at low concentrations (up to 25 mg L^{-1}).

So far, only few studies have focused on the application of the partial nitritation-anammox process to the treatment of complex industrial wastewaters characterized by high ammonium concentration, high content of readily degradable organic matter (i.e., high C_{org}/N ratio), and by the presence of toxic/inhibiting substances: the feasibility of partial nitritation as the preliminary treatment of refinery wastewater produced by the integrated gasification combined cycle (IGCC) and characterized by high amounts of ammonium and readily degradable formate (Corg/N ratio up to 1.1 gC/gN), as well as by the presence of toxic cyanides and phenols, has been recently investigated (Milia et al., 2016a). Based on those results, a granular anammox sequencing batch reactor (SBR) was started-up and fed with the pre-treated IGCC wastewater, in order to evaluate its feasibility as the final treatment step in a double stage system based on partial nitritation and anammox. The replacement of the synthetic influent with the pre-treated IGCC wastewater followed a conservative exponential law, in order to implement an operating strategy which would avoid stressful conditions for anammox biomass and promote its acclimation; reactor performance was monitored in terms of nitrogen removal rate (NRR), nitrogen removal efficiency (NRE), nitrite discharge rate (NitDR), specific anammox activity (SAA), total organic carbon (TOC) removal efficiency, granules physical and morphological properties, overall effluent quality. A comprehensive set of information concerning operating strategies and process performance was gathered, which can be useful for the treatment of similar wastewaters characterized by a high Corg/N ratio, as well as by the presence of toxic substances.

2. Materials and methods

2.1. Granular anammox SBR

A glass SBR with a working volume of 2 L (inner diameter, 13 cm; working height, 15.1 cm) was used to carry out the experiments. The reactor was operated in fed-batch mode with a 8-h cycle (110–400 min mixed feeding, 70–360 min reaction, 5 min settling and 5 min effluent withdrawal). Mechanical mixing was provided

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