



# Removal of reverse-fluxed ammonium by anammox in a forward osmosis system using ammonium bicarbonate as a draw solute

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## ARTICLE INFO

### Article history:

Received 9 July 2015

Received in revised form

19 August 2015

Accepted 21 August 2015

Available online 22 August 2015

### Keywords:

Forward osmosis

Anammox

Ammonium bicarbonate

Reverse salt flux

Wastewater treatment

## ABSTRACT

Reverse flux of ammonium draw solute is a serious problem for applying forward osmosis (FO) in water/wastewater treatment. In this study, anaerobic ammonium oxidization (anammox) was synergistically linked to FO for removal of reverse-fluxed ammonium, thereby creating an osmotic anammox system. The feasibility of this system was demonstrated through both batch and continuous operation, and the anammox process was developed in two stages: sole anammox and nitrification–anammox. With addition of nitrite, the sole anammox process achieved an effluent ammonium concentration of  $9.9 \pm 9.5 \text{ mg N L}^{-1}$ . The nitrification–anammox maintained an ammonium concentration of  $3.1 \pm 4.2 \text{ mg N L}^{-1}$ , and increased the water flux to  $2.46 \pm 0.24 \text{ LMH (L m}^{-2} \text{ h}^{-1})$  compared with the sole anammox ( $1.90 \pm 0.14 \text{ LMH}$ ). The nitrification–anammox process exhibited advantages over anammox process in assisting the FO with respect to water flux improvement and chemical savings. The osmotic anammox system can be linked to previously developed microbial electrolysis cells that recover ammonium from high-strength wastes as a draw solute for FO operation. The results encourage further investigation of this system for effects of organic residues, decreasing nitrate accumulation, understanding biofilm on the FO membrane, and long-term performance with actual waste.

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## 1. Introduction

Wastewater reclamation and reuse represents a promising way to sustainable water resource management. To achieve a high-quality effluent, newly developed membrane technologies have been studied and applied extensively [1]. Among them, forward osmosis (FO) is of strong interest because of its reduced energy consumption and membrane fouling [2–4]. In an FO process, an osmotic pressure created by water potential difference drives water to permeate across a semipermeable membrane from a low concentration solution (feed solution) side to a concentrated solution (draw solution) side [5–7], resulting in a concentrated feed solution and a diluted draw solution. Draw solute, which should be highly soluble, easily separable and recoverable, non-toxic, non-reactive with membranes, and economically feasible, plays a key role in a successful FO system [8]. Recovery of draw solute accounts for most of the energy consumption in an FO process [5,8]. In addition, reverse salt flux causes the loss of draw solute, which results in increased operating cost due to draw solute replenishment, and creates potential environmental concerns as draw

solute is released into the feed solution.

In the past decade, much effort as well as significant progress has been made in the development of draw solutes for various potential applications that can generate a high osmotic pressure with low reverse salt permeation [9]. The thermolytic ammonium bicarbonate ( $\text{NH}_3\text{-CO}_2$ ) is considered as an effective draw solute because of its high solubility, osmotic efficiency, and circulation capacity [10]. More importantly, the diluted ammonium bicarbonate draw solution can be decomposed into ammonia and carbon dioxide at a temperature as low as  $60^\circ\text{C}$ , thereby achieving a low-energy recovery process for draw solute recycle [10]. The application of ammonium bicarbonate draw solute has been widely investigated at the lab scale [5,10–14], and also demonstrated in a pilot-scale desalination system for treating the produced water from natural gas extraction [15]. However, the loss of ammonium draw solute via reverse salt flux (RSF) can hardly be avoided, and the ammonium migrated into the feed solution may require further treatment because it is an important inorganic contaminant to water body.

Recently, we have demonstrated that ammonium recovered from a synthetic organic solution by a microbial electrolysis cell (MEC) can be applied as a draw solute in an FO system that extracted water from the MEC anode effluent [16]. It was observed that due to the reduced liquid volume and RSF, the ammonium concentration in the feed solution increased from  $0.24 \pm 0.03$  to

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$1.66 \pm 0.15 \text{ g L}^{-1}$ . Although this concentrated feed solution was returned to the MEC for further treatment, it reduced the treatment capacity of the MEC, which could receive only 50% of fresh medium in each batch due to the need of treating the returning solution. To avoid such a reduction in MEC treatment capacity, the reverse-fluxed ammonium should be removed or recovered in the FO.

Ammonium removal can be accomplished by biological reactions such as anaerobic ammonium oxidation (anammox), where anammox bacteria convert ammonium to  $\text{N}_2$  with nitrite as a terminal electron acceptor under an oxygen-limited condition [17]. Nitrification by ammonium oxidizing bacteria (AOB) is required to oxidize part of ammonium to nitrite [18]. Thus, the cooperation of AOB and anammox bacteria is essential to achieve completely autotrophic nitrogen removal. Typically, two methods can be applied to start up nitrification–anammox process, either by inoculating nitrifying sludge into anammox reactors [19,20] or by inoculation of anammox biomass into nitrification reactors [21,22]. The nitrification–anammox process represents an efficient and cost-effective approach for biological nitrogen removal with significantly reduced demand for oxygen and organic carbon.

Given the challenges of reverse-fluxed ammonium in the FO and the advantage of an anammox process in ammonia removal, in this study we have synergistically linked anammox to FO, and created an osmotic anammox system to remove the reverse-fluxed ammonium during the FO process. As a proof-of-concept study, the key objectives are to: (1) demonstrate the feasibility of the osmotic anammox system; and (2) develop a nitrification–anammox process associated with FO. Although the osmotic anammox is designed to be connected to a MEC that provides ammonium as draw solute, this study focused only on the osmotic anammox, and the MEC performance has been previously reported [16] and thus was not included here.

## 2. Materials and methods

### 2.1. Osmotic anammox setup

The schematic of a lab-scale osmotic anammox system is shown in Fig. 1. The system consisted of two units, an upflow anammox reactor and an FO unit, which were linked through the feed solution that was recirculated between the anammox reactor and the FO feed side at a recirculation flow rate of  $34 \text{ mL min}^{-1}$ , resulting in an upflow velocity of  $1.04 \text{ m h}^{-1}$ . The anammox reactor had a working volume of 1.5 L (height 0.75 m, diameter 0.05 m), and a plastic funnel was placed on its top to separate gas and liquid while reducing biomass loss. The pH inside the anammox reactor was maintained between 7 and 8 by adding HCl or

NaOH solution when necessary. The feed side of the FO unit had a working volume of 2 L. The cellulose triacetate (CTA) membrane with a surface area of  $0.01 \text{ m}^2$  (Hydration Technologies Inc., Albany, OR, USA) was installed with its active layer facing the feed solution [16]. A heating rod was used to maintain the temperature of  $30 \pm 1 \text{ }^\circ\text{C}$  in the feed side.

### 2.2. System operation

The anammox reactor was initially inoculated with a mixed biomass mainly containing anammox granules obtained from another anammox reactor (5 L) in our lab; fluorescent *in situ* hybridization (FISH) indicated that the anammox bacteria accounted for  $\sim 80\%$  enrichment. The sole source of ammonium into the anammox reactor was the reverse-fluxed ammonium ions from the draw side of the FO to its feed side. The extracted water from the anammox reactor was compensated by a mineral solution ( $480\text{--}580 \text{ mL d}^{-1}$ ) prepared according to a previous study [23]:  $\text{NaHCO}_3$   $0.42 \text{ g L}^{-1}$ ,  $\text{KH}_2\text{PO}_4$   $0.0272 \text{ g L}^{-1}$ ,  $\text{MgSO}_4$   $0.059 \text{ g L}^{-1}$ ,  $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$   $0.18 \text{ g L}^{-1}$  and  $1 \text{ mL L}^{-1}$  trace elements solution I and  $1 \text{ mL L}^{-1}$  trace elements solution II. The trace elements solution I contains ( $\text{g L}^{-1}$ ): EDTA, 5 and  $\text{FeSO}_4$ , 5. The trace elements solution II is composed of ( $\text{g L}^{-1}$ ): EDTA-2Na 15,  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$  0.25,  $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$  0.43,  $\text{NaMoO}_4 \cdot 2\text{H}_2\text{O}$  0.22,  $\text{MnCl}_2 \cdot 4\text{H}_2\text{O}$  0.99,  $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$  0.19,  $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$  0.24,  $\text{NaSeO}_4 \cdot 10\text{H}_2\text{O}$  0.21, and  $\text{H}_3\text{BO}_3$  0.014 [23]. The draw solution ( $0.5 \text{ M}$  ammonium bicarbonate,  $29.7 \pm 1.7 \text{ mS cm}^{-1}$ ) was fed to the draw side of the FO continuously at a rate of  $228 \text{ mL d}^{-1}$  and was recirculated at  $100 \text{ mL min}^{-1}$ . The experiment started in a batch mode and then was changed to a continuous mode. Each batch cycle used 200 mL of  $0.5 \text{ M}$   $\text{NH}_4\text{HCO}_3$  as the draw solution, and the synthetic solution contained 25 mM nitrite to support anammox bacteria growth; each cycle was operated for 1–2 days, depending on the changes of water flux and ammonium concentration in the feed side. The continuous operation had two stages (Table 1): in the stage I (39 days), only anammox process was examined and nitrite was externally added; and in the stage II (45 days), a nitrification–anammox process was developed by limiting aeration in the feed side while gradually decreasing nitrite supply. Aeration was controlled by a gas flow meter based on dissolved oxygen (DO) monitoring data and reactor performance. The hydraulic retention time (HRT) of the anammox reactor in the stages I and II was  $2.66 \pm 0.42 \text{ d}$  and  $3.12 \pm 0.06 \text{ d}$ , respectively.

### 2.3. Measurement and analysis

The concentration of ammonium ( $\text{NH}_4^+\text{-N}$ ) was measured by a standard ammonia ion selective electrode (Fisher Scientific, Pittsburgh, PA, USA). The concentrations of nitrite ( $\text{NO}_2^-\text{-N}$ ) and nitrate ( $\text{NO}_3^-\text{-N}$ ) were determined by a spectrophotometer (DR 890, Hach Company, Loveland, CO, USA). The pH was measured by a benchtop pH meter (Oakton Instruments, Vernon Hills, IL, USA). Conductivity was measured with a benchtop conductivity meter (Mettler-Toledo, Columbus, OH, USA). Water flux ( $\text{L m}^{-2} \text{ h}^{-1}$ , LMH) in the FO was calculated by the change of weight recorded

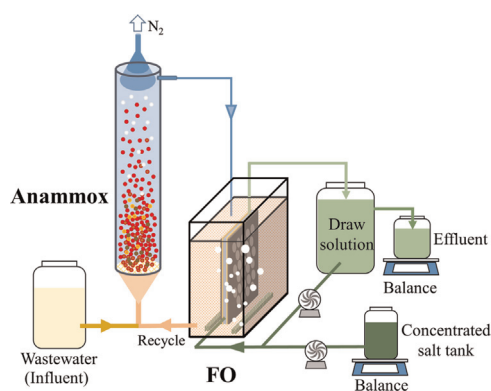


Fig. 1. The schematic of the osmotic anammox system.

Table 1  
Operation duration for Stage I and Stage II.

Stage	Period	Nitrite addition ( $\text{mg N L}^{-1}$ )	Operation duration (day)
I	A	280, 350, 420	1–16
	B	350	17–39
II	A (transitional)	280–140, 112, 70, 35–0	40–52
	B (stable)	0	53–84

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