



Recovery of ammonia and sulfate from waste streams and bioenergy production via bipolar bioelectrodialysis



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ABSTRACT

Ammonia and sulfate, which are prevalent pollutants in agricultural and industrial wastewaters, can cause serious inhibition in several biological treatment processes, such as anaerobic digestion. In this study, a novel bioelectrochemical approach termed bipolar bioelectrodialysis was developed to recover ammonia and sulfate from waste streams and thereby counteracting their toxicity during anaerobic digestion. Furthermore, hydrogen production and wastewater treatment were also accomplished. At an applied voltage of 1.2 V, nitrogen and sulfate fluxes of 5.1 g NH₄⁺-N/m²/d and 18.9 g SO₄²⁻/m²/d were obtained, resulting in a Coulombic and current efficiencies of 23.6% and 77.4%, respectively. Meanwhile, H₂ production of 0.29 L/L/d was achieved. Gas recirculation at the cathode increased the nitrogen and sulfate fluxes by 2.3 times. The applied voltage, initial (NH₄)₂SO₄ concentrations and coexistence of other ions were affecting the system performance. The energy balance revealed that net energy (≥ 16.8 kWh/kg-N recovered or ≥ 4.8 kWh/kg-H₂SO₄ recovered) was produced at all the applied voltages (0.8–1.4 V). Furthermore, the applicability of bipolar bioelectrodialysis was successfully demonstrated with cattle manure. The results provide new possibilities for development of cost-effective technologies, capable of waste resources recovery and renewable energy production.

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1. Introduction

Anaerobic digestion is an attractive technology widely used for organic waste treatment and production of energy in the form of biogas (Weiland, 2010). However, the stability of the processes is hard to maintain due to a wide variety of toxic substances, among which free ammonia (NH₃) and hydrogen sulphide (H₂S) produced from sulfate reduction are two most significant inhibitors (Angelidaki and Ahring, 1994; Siles et al., 2010). Ammonia (NH₃/NH₄⁺) and sulfate (SO₄²⁻) are often found at high levels (≥ 3 g NH₄⁺-N/L and up to 13 g SO₄²⁻-S/L, respectively) in the livestock manures and industry residues (Siles et al., 2010). As notorious environmental pollutants, these substances are often found in concentrations much higher than the inhibition levels. Inhibition can start at 1.5 g-NH₄⁺-N/L and/or 1.4 g-SO₄²⁻/L while 100% inhibition can be found at 6–13 g-NH₄⁺-N/L and/or 1.8 g-SO₄²⁻/L (El Hadj et al., 2009; Siles et al., 2010). Beside causing inhibition, H₂S derived from sulfate reduction can cause serious corrosion in compressors,

gas storage tanks and engines, which need to be removed from biogas through energy-extensive processes (Ryckebosch et al., 2011). Several biological, chemical and physical techniques such as bioaugmentation, air stripping and chemical precipitation have been developed to remove or recover excess ammonia and sulfate, and thereby preventing the inhibition (Angelidaki and Ahring, 1994; Angeles De la Rubia et al., 2010; Ippersiel et al., 2012; Westerholm et al., 2012). However, most of the methods are uneconomical, requiring high energy input, have low efficiency, or need of additional chemicals. Most importantly, none of these methods can recover both ammonia and sulfate.

Recently, microbial electrochemical systems (MES) such as Microbial fuel cell (MFC) and microbial electrolysis cell (MEC) have been demonstrated as promising alternatives to conventional air-stripping for ammonia recovery (Kelly and He, 2014; Kuntke et al., 2012; Wu and Modin, 2013). Though promising, there are still several challenges need to be addressed before field application. For example, anodic bacteria might be inhibited at high ammonia levels (>4 g NH₄⁺-N/L) (Kim et al., 2011; Nam et al., 2010). To meet this challenge, microbial desalination cell (MDC) has recently been proposed to recover ammonia from anaerobic reactors (Zhang and Angelidaki, 2014). The principle of MDC has also

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been successfully applied to upconcentrate nutrients from diluted urine (Tice and Kim, 2014). Nevertheless, the dramatic pH drop in the anode during recovery needs to be prevented before practical application (Qu et al., 2012). Sulfate is potential electron acceptor in MES (Sun et al., 2009; Zhao et al., 2008), but direct recovery of sulfate (e.g., as sulfuric acid) has never been reported. Therefore, it is of great importance to explore the feasibility of sulfate recovery using MES. Furthermore, an advanced system that can combine ammonia and sulphide recovery should be pursued. An MES namely microbial electrolysis desalination and chemical production cell (MEDCC) was recently developed for water desalination, acid and alkali production (Chen et al., 2012). With an applied voltage (~ 1.2 V), Cl^- and Na^+ in the desalination chamber are driven across an anion exchange membrane (AEM) and a cation exchange membrane (CEM) into acid-production and cathode chambers, where they are recovered as HCl and NaOH (Chen et al., 2012). It has been reported that NH_4^+ and SO_4^{2-} have higher ionic conductivity and diffusivity to transport through membranes than that of Na^+ and Cl^- (Tice and Kim, 2014). Thus, NH_4^+ and SO_4^{2-} could be captured in the form of NH_3 (from NH_4^+ and OH^-) and H_2SO_4 . In light of the above, the MEDCC could fulfill the requirements for ammonia and sulfate recovery, which has never been reported. Nevertheless, the extensive aeration in the cathode of MEDCC might be one of the key challenges for such new application.

In this study, we developed a bioelectrochemical system, called in the later as “bipolar bioelectrodialysis”, on the basis of MEDCC, for a brand new application with respect to simultaneous ammonia and sulfate recovery from waste streams and hydrogen production. Beside different application scopes, the main advantage of the bipolar bioelectrodialysis over the previous MEDCC is that the energy-intensive aeration at the cathode was replaced with cost-effective hydrogen production, which could offset the energy costs during recovery or be stored for further use. The objective of this study was to investigate the feasibility of the bipolar bioelectrodialysis system by varying operational parameters such as applied voltage, initial ammonia and sulfate concentration, and ionic species. Whereas the synthetic wastewater containing varied ammonia and sulfate concentrations was used for the investigation, also cattle manure was used as a proof of concept to demonstrate its applicability. This work demonstrates a new avenue to recover valuable resources from waste streams, produce bioenergy, and potentially prevent inhibitions during anaerobic digestion.

2. Materials and methods

2.1. Experimental setup

The bipolar bioelectrodialysis system composed of four chambers (inside dimensions 5 cm \times 5 cm \times 2 cm for each, Fig. 1) was made of nonconductive polycarbonate. A bipolar membrane (BMP, fumasep[®] FBM, FuMA-Tech GmbH, Germany), an AEM (AMI 7001, Membrane international, NJ) and a CEM (CMI 7000, Membrane international, NJ) were placed between the anode and cathode chambers. The acid-production chamber was close to the anode side for sulfate recovery. The working chamber was close to the cathode side for receiving waste streams. The liquid volume of each chamber except anode was 40 mL. There were several openings on each chamber connected with rubber tube for feeding and sampling. The anode electrode was made of a carbon fiber brush (5.0 cm diameter, 5.0 cm length, Mill-Rose, USA), which was pre-treated at 450 °C for 30 min prior to use (Alatraktchi et al., 2014). The pretreated anode was pre-acclimated with mature biofilm in a MFC operated with wastewater. The cathode was a stainless steel woven mesh (4.0 \times 4.0 cm, 0.24 mm wire diameter, 1.57 mm aperture, The Mesh Company, United Kingdom) coated with

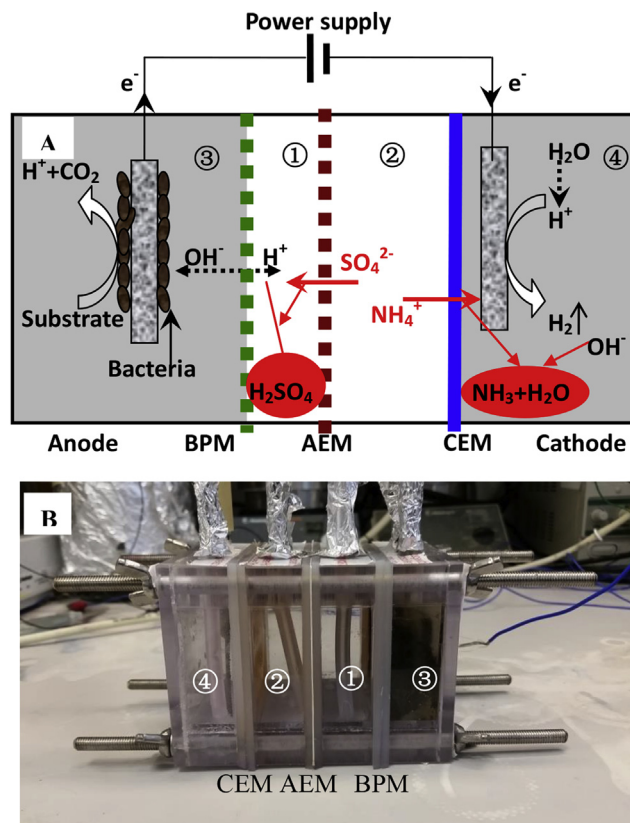


Fig. 1. Schematic illustration (A) and image (B) of the bipolar bioelectrodialysis from opposite view. ① Recovery chamber, ② Working chamber, ③ Anode, ④ Cathode.

0.5 mg/cm² Pt. All the electrical connection and electrode pre-treatment were performed according to previous study (Zhang and Angelidaki, 2012a).

2.2. Domestic wastewater, synthetic ammonia and sulfate-rich wastewater and cattle manure

Domestic wastewater was collected from primary clarifier (Lyngby Wastewater Treatment Plant, Copenhagen, Denmark). The characteristics of the wastewater were as following: chemical oxygen demand (COD) 296 mg/L, pH 7.8, conductivity 1.4 mS/cm, nitrogen 0.07 g NH_4^+ -N/L, phosphorus 0.01 g PO_4^{2-} -P/L, Na^+ 0.20 g/L, K^+ 0.07 g/L, Cl^- 0.19 g/L, and SO_4^{2-} 0.04 g/L. The synthetic wastewater was prepared with deionized water containing (pH 6.5): glucose 10 g/L, NaCl 0.10 g/L, $\text{MgCl}_6 \cdot \text{H}_2\text{O}$ 0.10 g/L, CaCl_2 0.05 g/L, NaHCO_3 0.50 g/L and trace mineral metals solution (≤ 0.01 g/L in total). NH_4^+ and SO_4^{2-} were added at different concentrations according to the tests. The cattle manure was collected from Vegger biogas plant, Denmark. The manure was sieved to remove coarse materials and stored at 4 °C before use. The characteristics of the manure were: pH 8.1, total solids 79.23 g/L, volatile solids 60.55 g/L, total Kjeldahl nitrogen 3.27 g/L, NH_4^+ -N 2.15 g/L, SO_4^{2-} 0.03 g/L, Cl^- 8.23 g/L, Ca^{2+} 0.82 g/L, K^+ 3.5 g/L, Na^+ 0.83 g/L. The manure was amended with Na_2SO_4 to mimic a mixture of ammonia and sulfate-rich waste, obtaining a final SO_4^{2-} concentration of 5 g/L.

2.3. Reactor operation

The domestic wastewater amended with acetate (2 g-COD/L in total) was recirculated from a feed reservoir (liquid volume of 500 mL) through anode at a recirculation rate of 50 mL/min using a

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