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Insights into enhanced current generation of an osmotic microbial fuel cell under membrane fouling condition



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ABSTRACT

Osmotic microbial fuel cells (OsMFC), which incorporates forward osmosis (FO) in MFC design, favors an improved electricity generation and effluent quality over conventional MFCs that use proton exchange membranes. However, fouling of the FO membrane during long-term operation is unavoidable, and it is unclear how this would affect the MFC performance. Here, we found that the current generation of an OsMFC increased upon membrane fouling despite of the loss of water flux, and the internal resistance decreased relative to the pristine membrane conditions. A further examination of the pH changes and ion flux by electrochemical test revealed that the diffusion of protons and other ions through the membrane was significantly promoted upon fouling. The underlying mechanisms of the fouling-induced diffusion enhancement were also investigated. The finding of this work broadens our understanding about the fouling properties of FO membrane, and implies that FO membrane could serve as a potential fouling-tolerant separator for MFCs and other bioelectrochemical applications.

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1. Introduction

Forward osmosis (FO) has recently emerged as an attractive water purification technology [1,2], with many advantages such as high contaminant removal, low-energy consumption, and less susceptible to membrane fouling over traditional pressure-driven membrane processes [3–5]. When being used as an electrolyte separator for microbial fuel cells (MFCs), FO membrane brings extra benefits of raising proton flux and anodic electrolyte conductivity over conventional proton exchange membrane (PEM), leading to higher power generation [6,7]. However, the long-term performance of such OsMFCs has not been investigated so far. Considering that membrane fouling would inevitably occur during long-term operation [8,9], it is necessary to understand how the OsMFC performance would be affected by membrane fouling.

A previous OsMFC study showed that membrane fouling led to water flux decline but the power generation was unaffected [6]. This phenomenon was contradictory to conventional MFC processes where the fouling of a PEM as the separator almost always led to electrochemical performance decline [10,11]. The fouling of FO membrane is known to significantly influence the permeation of ions

[12–14] and various trace organic contaminants [15–17]. However, the impacts of membrane fouling on ions flux in OsMFC are still unclear. Given the important role of cross-membrane diffusion of various ions for charge neutrality in electrochemical systems, it is of great interest to find out how the membrane properties and ion permeation behaviors of an FO membrane would change upon biofouling.

This study aims to fill in the above knowledge gaps. We investigated the current generation and internal resistance of an OsMFC, and surprisingly found a superior performance of the system when a fouled FO membrane was used against a pristine membrane. A further investigation of pH changes and ion flux by electrochemical test showed that ion flux (especially proton flux) through the FO membrane was significantly promoted upon fouling, which explains the decreased internal resistance of OsMFC with fouled membrane. These findings may have important implications for a long-term stable operation of FO membrane in MFCs and other bioelectrochemical systems (BESs).

2. Materials and methods

2.1. OsMFC setup and operation

A dual-chamber air-cathode OsMFC was constructed as

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described in our previous study [18]. A flat-sheet thin-film composite (TFC) FO membrane, purchased from Hydration Technology Innovations (HTI, Albany, USA), was used as the electrolyte separator. The membrane is made of a polyamide active layer embedded in a polysulfone support layer, and has good chemical stability under broad pH conditions (2–11). The pristine membrane was soaked in 18.2 Ω deionized (DI) water (Millipore Inc., USA) at 4 °C before use. For comparison, an identical FO membrane that had been pre-operated in another OsMFC for 2 months was used as the fouled membrane. The membranes (effective area 12.5 cm²) was installed in the MFC with active layer facing the feed solution.

A carbon felt (Beijing Sanye Carbon Co., China), with a projected area of 7.5 cm² and precultured in another single-chamber air-cathode MFC, was directly used as the bioanode. A carbon cloth (5 × 5 cm², GEFC Co., China) with 2 mg cm⁻² of Pt loading was used as the air-cathode. The electrodes were connected through an external circuit with a 100 Ω resistor. Both the feed and draw solutions were connected to external reservoirs and continuously recycled through peristaltic pumps at a constant recirculation flow rate of 20 mL/min. The draw solution reservoir was placed on an analytical balance (JA2100, Yueping Co., China) for weighing.

During the operation, the anode chambers were fed with synthetic wastewater (feed solution), which contained (per liter): CH₃COONa, 2 g; NH₄Cl, 0.15 g; NaCl, 0.5 g; CaCl₂, 0.02 g; MgSO₄, 0.015 g; NaHCO₃, 0.1 g; KH₂PO₄, 0.53 g; K₂HPO₄, 1.07 g, and trace elements of 1 mL [7]. The cathode chamber was filled with 1 M NaCl solution. The MFC was run for five consecutive cycles. Each operating cycle was ended when the current dropped to below 0.1 mA, and then the membrane and electrolyte solutions was replaced. To avoid interference by other factors, the pristine and fouled membrane were alternately tested in the same MFC. The experiment was conducted at ambient temperature (~25 °C).

2.2. Ion diffusion tests

To better decipher how the membrane fouling affects the solute diffusion and electrochemical performance, the diffusion fluxes of several representative ions in wastewater (including K⁺, Na⁺, NO₃⁻, Cl⁻, and H⁺) through FO membranes were evaluated in an abiotic electrochemical cell. Here, an H-shaped glass electrochemical cell with a 100 mL volume for each chamber was used (Fig. 1). A carbon paper was used as the anode and a carbon felt as the cathode. The projected surface area of both electrodes was 2 cm². A pristine or fouled FO membrane (effective area 3.8 cm²) was assembled between the two chambers of the electrochemical cell. KNO₃ solution (10 mM) was added as the anolyte and NaCl solution (1 M) as the catholyte. The chemicals, all purchased from Sinopharm Chemical Reagent Co., China, were of > 99.8% purity. The electrolyte solutions were prepared using DI water (Millipore Inc., USA). The solution in each chamber were mixed by magnetic stirring. During operation, a constant current of 0.05 mA/cm² was applied to enable water electrolysis and drive the ion transport, which resembles the conditions of MFC operation. All the tests were conducted in triplicates at ambient temperature of ~25 °C.

2.3. Analysis

2.3.1. Membrane characterization

The FO membranes were characterized by scanning electron microscopy (SEM), energy-dispersive x-ray spectroscopy (EDX), atomic force microscopy (AFM), confocal laser scanning microscopy (CLSM) and attenuated total reflectance-Fourier transform infrared (ATR-FTIR).

For surface morphology observation, a piece of membrane was first immersed in 2.5% glutaraldehyde, and then subjected to

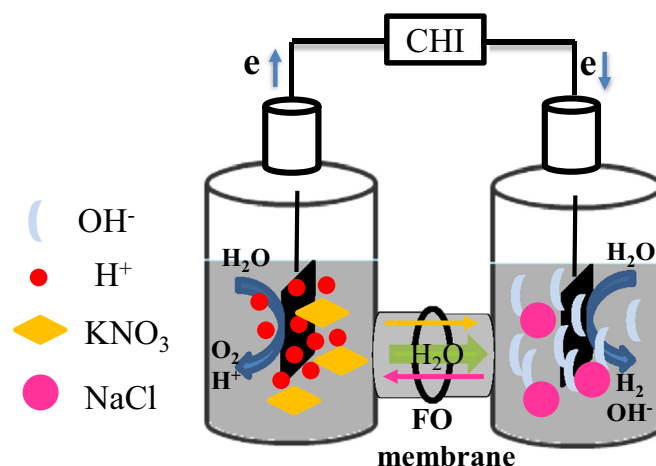


Fig. 1. Schematic diagram of the H-shaped electrochemical cell for ion diffusion test.

dehydration using ethanol series, dried completely at ambient temperature following the methods in literature [11,19]. Finally, the membrane samples were coated with a uniform layer of gold and analyzed using a Quanta-400FEG SEM (FEI, USA) coupled to an EDX.

The surface roughness and morphology of the FO membrane were also characterized by AFM imaging (Multimode, Veeco Inc., USA). The membrane surface was scanned at 10 $\mu\text{m} \times 10 \mu\text{m}$ area in tapping mode with an etched silicon probe. The probe frequency and scan rate were 233.4 Hz and 1.0 Hz, respectively. NanoScope 7.30 software was used to analyze AFM images.

The compositions and architecture of the FO membrane fouling layer were analyzed by CLSM (FluoView FV1000, Olympus Co., Japan). Fluorescent staining was conducted following a previously reported protocol [11,20]. Several fluorescence stains including Syto 63, fluorescing isothiocyanate (FITC), Calcofluor white, and Con A (Invitrogen Co., USA) were used to probe the cells, proteins, β -linked and α -linked polysaccharides, respectively.

ATR-FTIR spectroscopy was used to identify the functional groups of the active layer membrane surface. Prior to the measurement, the membrane samples were treated using a freeze-drier (HETO Power Dry PL3000, USA) at -50 °C for 12 h to remove the residual moisture. The measurements were conducted using an infrared spectrophotometer (Vector 22, Bruker Co., Germany) equipped with an ATR accessory. The recorded spectral range was 4000–600 cm⁻¹.

2.3.2. Electrochemical characterization of OsMFC

The voltage output (*V*) of the OsMFC was continuously recorded by a data acquisition system (34970A, Agilent Inc., USA) connected to a computer. The current (*I*) was calculated from Ohm's law ($I=V/R$). The anolyte conductivity was measured by a bench-top conductivity meter (DDSJ-308A, REX Instrument Co., China). The solution pH was measured using a pH meter (Mettler Toledo Inc., Switzerland).

When the voltage of the OsMFC became stable in each operating cycle, the polarization curves were measured using an electrochemical workstation (CHI 760, Chenhua Instrument, China), with the anode as the working electrode and the cathode as the counter electrode and reference electrode. Linear sweep voltammetry (LSV) were carried out at a potential scan rate of 1 mV/s from the open circuit voltage to 0 mV. The average values in two measurements were adopted for plotting the polarization curves. The total internal resistance was derived from the slopes of the polarization curves.

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