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Enhanced antifouling performance for modified carbon nanotubes filtration cathode by the electric field

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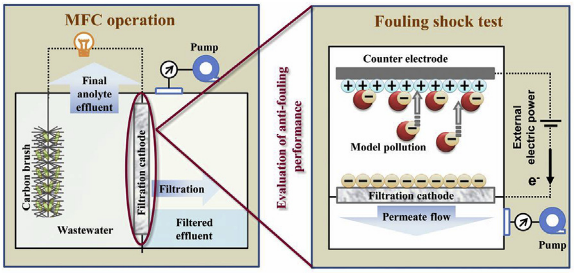
HIGHLIGHTS

- Carbon nanotubes as catalyst were added to active carbon filtration cathode.
- Power generation of the CNT filtration cathode was enhanced.
- COD removal was improved in the MFC with CNT filtration cathode.
- The electric field enhanced antifouling properties of the filtration cathode.

GRAPHICAL ABSTRACT



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ABSTRACT

A carbon nanotube filtration cathode as an air-cathode used in microbial fuel cells is fabricated following the phase inversion method, which couples membrane filtration with catalytic reduction. This novel activated carbon/carbon nanotube filtration cathode exhibits the superior electro-conductivity and antifouling performance. The filtration cathode with 10 wt% carbon nanotubes (CNT10) produces a maximum power density of 1469 ± 9.7 mW m^{-2}, which is 49% higher than that of the unmodified electrode (control). Cathode CNT10 exhibits the lowest charge transfer resistance of 17.9 ± 0.9 Ω, which is 64% lower than that of the control electrode. The minimum filtered total chemical oxygen demand of CNT10 is 23.1 ± 1.4 mg L^{-1}, which is 76% lower than that of the control (97.5 ± 3.6 mg L^{-1}). With an additional voltage of 1.5 V, CNT10 exhibits the highest bovine serum protein and humic acid rejection rates, indicating that the applied voltage improves both antifouling performance and electrochemical activity compared to those of the control cathode. These results demonstrate that the conductive filtration cathodes exhibit the better antifouling performance and electrochemical response.

1. Introduction

Microbial fuel cells (MFCs) are a potential wastewater treatment technology that can convert the chemical energy of organic compounds into electrical energy [1]. The electrons produced by the degradation of organics are transported to the cathode and react with oxygen to generate electricity [2]. Functional air-cathode MFCs, especially filtration MFCs exhibit promising development with better effluent water quality and a more favorable power density output than those of conventional air-cathode MFCs [1]. Although electrode electrocatalytic activity coupled with filtration could be applied in water treatment, fouling during filtration can clog the pores and form a cake layer. Hydrophobic organic and inorganic fouling, such as that from macromolecular organic pollutants and salts causes, the cathode to deactivate, which

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affects the oxygen reduction reaction rate and decrease power production [3,4]. Especially, the extracellular polymeric substances (EPS), which mainly contains protein, humic substances, etc., is considered as a key composition in the effluent of microbial fuel cell affecting both membrane fouling and bacterial adhesion on the filtration air-cathode [5,6]. It is, therefore, necessary to prepare an anti-fouling filtration electrode and improving the anti-fouling performance of the cathode is important for increasing power production and effluent water quality [7].

The phase inversion method is promising for the preparation of membrane [8] and has been used to prepare electrodes as anodes-supported in solid oxide fuel cells (SOFCs) [9] and cathodes in MFCs [10]. Compared to the rolling method, phase inversion could produce a thin filter layer on the cathode's surface [10], making it easy to fabricate a filtration cathode and modify it for anti-fouling.

Carbon-based materials, such as carbon black, graphene and carbon nanotubes (CNTs), have been adopted as air-cathode due to their excellent electrical conductivity, chemical stability and non-corrosiveness [11,12]. CNTs exhibit great promise as catalysts or modifiers for cathodes in fuel cells owing to their exceptional properties, such as nanometer size, high catalytic activity, good electronic conductivity, three-dimensional structure, and stability [13,14]. The three-dimensional structure could provide greater redox reaction site and high electricity production efficiency. However, the high specific surface area of CNTs increased the attachment of microorganism, and the microbial film could hinder oxygen transfer and limits the application of CNTs as catalysts of air-cathodes.

CNT-incorporated membranes have extraordinarily high transport rates to liquid water [15,16]. The flux of polyvinyl alcohol (PVA) membrane loaded with multi-walled carbon nanotubes (MWCNTs) was increased by the addition of MWCNTs for ethanol dehydration by pervaporation [17]. The permeation flux and separation of PVA membranes modified with chitosan-wrapped MWNCTs were improved for the pervaporation separation of benzene/cyclohexane mixtures, compared to that of unmodified PVA membranes [18]. Both the permeation flux and separation factor were increased after adding MWCNTs, and the flux and permeability were higher than those of the unmodified membrane [19]. These results indicated that the CNT had excellent performance for use in filtrated air-cathodes.

Electrochemical assistance could enhance the anti-fouling performance of electrodes by contaminant desorption because the charged substance could move in the electric fields [20]. Micropollutant molecules were removed via electrostatic repulsion from an electrode to achieve the regeneration of electrochemical activity [21]. The synergistic effects of electrostatic repulsion and electrochemical oxidation could improve the permeability, removal efficiency, and anti-fouling performance of electro-polarized membranes, especially cathodic polarized membranes [22].

In this paper, a filtration MFCs coupling physical entrapment with bioelectrochemical treatment were constructed with a CNT filtration cathode. The CNT filtration cathode was investigated as a modifier used to synthesize the filtration cathode following phase inversion method, which was designed and constructed to preserve the intrinsic electrochemical activity for power generation. The CNT filtration cathode was coupled membrane filtration to remove pollutant molecules and enable catalytic reduction for the air-cathode. The material properties and electrochemical activity were analyzed to evaluate the performance of the filtration cathode. The anti-fouling performance of the CNT filtration cathode was evaluated by a fouling shock test with an electric field. In the fouling shock test, the bovine serum protein (BSA) and humic acid (HA) as model humus and protein organic pollutants were used to accelerate the fouling of the CNT filtration cathode and investigate the cathode fouling process. These results indicated that the filtration cathode could efficiently maintain a higher anti-fouling performance, which could readily desorb organic molecules in the presence of an electric field and achieve the regeneration of electrochemical activity.

2. Materials and methods

2.1. Filtration cathode fabrication

Filtration cathodes were fabricated via phase inversion method [23]. A polymer solution was prepared with 12 wt% of a polyvinyl chloride binder (PVC, 1300 Da, Shenyang Chemical Co., Ltd., China), 6 wt% of a polyvinylpyrrolidone pore-forming agent (PVP, k30, Tianjin Guangfu Fine Chemical Research Institute, China) and N, N-dimethyl acetamide (DMAc, anhydrous, 99.8%, Tianjin Fuyu Fine Chemical Co., Ltd., China). The polymer solution was stirred for 24 h at an ambient temperature in the place to allow the PVC-co-PVP to completely dissolve. A settling period in a dark place was continued for 24 h to make the homogeneous solution to remove bubbles. The solution was left to settle under darkness for 24 h to homogenize the solution and remove bubbles.

To fabricate the filtration cathodes, 0.81 g of PVC-co-PVP, 0.58 g of activated carbon powder (AC, Carbosino Material Co., Ltd., China), 0.15 g of polyaniline (PANI, Hubei Yuanchengsaichuang Co., Ltd., China), 3 mL of 1-Methyl-2-pyrrolidinone (NMP, AR, Tianjin Zhiyuan Chemical Reagent Co., Ltd., China), and multiwalled carbon nanotubes (CNT, Suzhou Hengqiu Co., Ltd, Suzhou, China) were mixed. The CNT filtration cathodes were prepared with four different CNT mass ratios of AC = 0, 1:20, 1:10, and 1:6.6, which were denoted as the control, CNT5, CNT10, and CNT15, respectively. The mixture was coated on the two sides of the stainless-steel mesh (projected cross-sectional area of 7 cm^2) to form both the catalyst and diffusion layers after 30 min of ultrasonic agitation, which was further immersed into deionized (DI) for 20 min to prepare the electrode by phase inversion.

2.2. Filtration cathode characterization

The morphology of the filtration cathode was observed using a scanning electron microscope (SEM, ZEISS SUPRA 55) [24]. The roughness of the filtration cathode was characterized by atomic force microscopy (AFM, Veeco Instruments Inc, USA) and analyzed by using the Nanoscope III software [25].

The water contact angles of the filtration cathode were measured with the sessile drop technique using a surface contact angle measurement instrument (JYSP-360, USA, needlepoint 1.5 mm) [26]. The water droplets were measured after stabilization for 20 s. The surface energies (γ_{SV}, mN m^{-1}) were calculated using the following equations $\gamma_{SV} = \gamma_{SV}^{P} + \gamma_{SV}^{D}$ and $\gamma_{LV}(1+\cos\theta) = 2\sqrt{\gamma_{SV}^{D}\gamma_{LV}^{D}} + 2\sqrt{\gamma_{SV}^{P}\gamma_{LV}^{P}}$, and by using the contact angles of water ($\gamma_{LV} = 72.8\ \mathrm{mN\,m^{-1}}$, $\gamma_{LV}^{D} = 21.8\ \mathrm{mN\,m^{-1}}$, $\gamma_{LV}^{P} = 51.0\ \mathrm{mN\,m^{-1}}$) and methylene iodide ($\gamma_{LV} = 58.2\ \mathrm{mN\,m^{-1}}$, $\gamma_{LV}^{D} = 39.5\ \mathrm{mN\,m^{-1}}$, $\gamma_{LV}^{P} = 18.7\ \mathrm{mN\,m^{-1}}$), where θ is the contact angle (degree), and γ^{P} and γ^{D} are the polar and dispersion part of the free energy of the solid and liquid surface (mN m^{-1}), respectively [27].

Porosity was measured following the gravimetric method using the equation $\varepsilon = \Delta W/(V\rho_{water})$, where ΔW is the difference between wet and dry weight (g), V is the volume of membrane (cm^3), and ρ_{water} is the density of water (g cm^{-3}) [28]. The pure water flux (L_P, L m^{-2} s^{-1} MPa^{-1}) was calculated using $L_P = V/(PAT)$ where V (L) is the water volume, P (MPa) is the intensity of pressure, A (m^2) is the membrane area, and T (s) is the time [29]. Conductivity was tested using double testing digital four-probe tester (ST2263, Suzhou Jingge Electronic Co., Ltd, China).

2.3. Electrochemical characterization

All electrochemical measurements of the filtration cathode were investigated using an electrochemical workstation (Chenhua CH760E, China) with a three-electrode system using an electrolyte 50 mmol L^{-1} phosphate buffer solution (conductivity of 6.9 mS cm^{-1}) at ambient temperature (25 °C). The filtration cathode, a saturated calomel

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