



High-capacity carbon-coated titanium dioxide core–shell nanoparticles modified three dimensional anodes for improved energy output in microbial fuel cells



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HIGHLIGHTS

- A capacitive layer coated 3D anode was fabricated for targeting high-performance in MFCs.
- The capacitive layer was composed of TiO₂ and egg white protein-derived carbon.
- The capacitive 3D anode outperformed non capacitive anode.

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ABSTRACT

Three-dimensional (3D) electrodes have been intensively investigated as alternatives to conventional plate electrodes in the development of high-performance microbial fuel cells (MFCs). However, the energy output of the MFCs with the 3D anodes is still limited for practical applications. In this study, a 3D anode modified with a nano-structured capacitive layer is prepared to improve the performance of an microbial fuel cell (MFC). The capacitive layer composes of titanium dioxide (TiO₂) and egg white protein (EWP)-derived carbon assembled core–shell nanoparticles, which are integrated into loofah sponge carbon (LSC) to obtain a high-capacitive 3D electrode. The as-prepared 3D anode produces a power density of $2.59 \pm 0.12 \text{ W m}^{-2}$, which is 63% and 201% higher than that of the original LSC and graphite anodes, respectively. The increased energy output is contributed to the enhanced electrochemical capacitance of the 3D anodes as well as the synergetic effects between TiO₂ and EWP-derived carbon due to their unique properties, such as relatively high surface area, good biocompatibility, and favorable surface functionalization for interfacial microbial electron transfer. The results obtained in this study will benefit the optimized design of new 3D materials to achieve enhanced performance in MFCs.

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1. Introduction

Microbial fuel cells (MFCs) are a recently developed technology for the production of renewable energy that can efficiently convert organic matter into electricity using exoelectrogenic bacteria. Because MFCs are capable of utilizing organic matter from wastewater as fuel in an environmentally-friendly manner, they have attracted intensive research attention during the last decade [1–3]. However, real-world applications of MFCs are limited because of their low small-scale power output.

In MFCs, electron transfer is mediated by microbes that adhere to the anode surface. Hence, the development of novel anodic materials that can enhance bacterial adhesion and electron collection is vital to increasing the power production of MFCs [4,5]. In this regard, recent attempts have been made to design three-dimensional (3D) electrodes as alternatives to conventional plate electrodes. Examples include carbon nanotube-coated carbon foam or sponges, graphene-coated sponges, and polyaniline-hybridized graphene foams [6–9]. Despite significant efforts, the practical application of these 3D anodes has been hindered by a series of obstacles, including low biocompatibility, high cost, and complex fabrication procedures [6–9]. In this context, the direct carbonization of low-cost and naturally available materials, such as pomelo peel [10], kenaf [11], sustainable corrugated cardboard [12], and loofah sponges [13], provides a potential alternative for targeting high-performance 3D anodes. The improved anode

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performance was primarily attributed to the increased specific surface area of the 3D electrodes and the inspired extracellular electron transfer (EET) by the decorated materials.

In addition, MFCs integrated with external or internal capacitors have been proven to be an efficient strategy for harvesting more energy. For instance, Dewan et al. showed that the maximum power with an external capacitor was 111% higher than that without an external capacitor [14]. However, improved energy harvesting has only been achieved under the intermittent mode of operation. Furthermore, capacitive materials were recently integrated with anodes in an MFC system to function as internal capacitors for harvesting more energy [15,16]. These studies demonstrated that the capacitive anodes could improve the transient storage of electrical charge in the bioanodes, demonstrating the possibility of using the MFC simultaneously for the conversion and storage of bioenergy. It is well known that electrodes consisting of 3D interpenetrating structures can provide good electron storage capacities [17,18]. However, less attention has been given to the role of the capacitance properties of 3D anodes on the performance of MFCs.

In this study, a nano-structured capacitive layer was integrated with a loofah sponge carbon (LSC) electrode in order to increase the capacitance of the 3D anode for achieving higher energy output in an MFC. The capacitive layer composed of TiO_2 and egg white protein (EWP)-derived carbon, which were arrayed as a nano-structured hierarchical TiO_2 @C core-shell structure. On one hand, it has previously been reported that TiO_2 can increase the performance of the bioanodes in MFCs [19,20]. However, the mechanisms behind the enhanced performance of the reported TiO_2 -based electrode remain unclear. On the other hand, TiO_2 has been intensively investigated for use as a supercapacitor electrode material for supercapacitor due to its semiconducting properties and chemical stability [21]. In addition, it has been reported that the N-rich carbon derived from EWP is an ideal additive for high performance supercapacitor electrodes [22]. Hence, we used EWP as the precursor to produce TiO_2 @C to further enhance the capacitance of the TiO_2 , and a N-rich TiO_2 @C-coated LSC (LSC- TiO_2 @C) anode was achieved. Lab-scale MFCs equipped with this fabricated nano-structured 3D anode outperformed non-modified LSC anodes, and the enhanced performance was correlated with the integrated interior capacitors. With the high-capacitive anode it was possible to simultaneously convert and store renewable energy in MFCs.

2. Materials and methods

2.1. Electrode fabrication

The LSC electrode was prepared using a simple carbonization process as described previously [13]. The TiO_2 coated LSC electrode (LSC- TiO_2) was achieved in the following steps. First, 0.5 g of TiO_2 P25 (Evonik Degussa GmbH, Germany) was dispersed in a 100 mL aqueous solution by sonication. Then, a piece of loofah sponge (LS) was dipped into the TiO_2 dispersion then removed and dried at 60 °C. The dipping-and-drying process was repeated twice to increase the TiO_2 loading. Finally, the resulting LS/ TiO_2 composite was carbonized at 900 °C under nitrogen atmosphere to obtain the LSC- TiO_2 electrode. A similar procedure was employed to prepare N-rich carbon-coated TiO_2 modified LSC anodes (LSC- TiO_2 @C), with the only difference being that the LS was dipped into a mixed solution of EWP (derived from commercial chicken eggs) and TiO_2 . To prepare this mixture, EWP (30 mL) was first dispersed into 100 mL of an aqueous solution by sonication, and then TiO_2 nanopowder (0.5 g) was suspended and stirred in the EWP solution for 2 h. The loofah sponges were immersed in the EWP/ TiO_2 aqueous

dispersion. For comparison, the EWP coated LSs were also prepared through the same procedures. The obtained LSs were carbonized to produce the LSC- TiO_2 @C and LSC-C electrodes, respectively. A schematic of the electrode fabrication is shown in Fig. 1A.

2.2. MFC setup and operation

Air-cathode single-chamber MFCs with a liquid volume of 28 mL were fabricated as previously reported [13]. The cylindrical MFC chamber was made of plexiglass with a length of 4.0 cm and a diameter of 3.0 cm (Fig. S1A). An anode with a size of $2.0 \times 1.5 \times 0.5$ cm (length \times width \times thickness) connected with a titanium wire (Fig. S1B) was positioned in a concentric manner in the core of the cylindrical chamber. The cathode was prepared from 30% wet-proofed carbon cloth (type B) with four layers of PTFE coating. The other side of the cathode was coated with Pt C⁻¹ (0.5 mg cm^{-2} Pt loading) as an oxygen-reduction catalysis layer.

The MFC reactors were inoculated with 5.0 mL of pre-acclimated bacteria from another well-running MFC (originally inoculated with activated anaerobic sludge). The reactor was fed with a medium containing 1 g L^{-1} sodium acetate and culture medium solution. The culture medium solution contained the following components: $\text{Na}_2\text{HPO}_4 \cdot 12\text{H}_2\text{O}$ (11.40 g L^{-1}), $\text{NaH}_2\text{PO}_4 \cdot 2\text{H}_2\text{O}$ (2.77 g L^{-1}), KCl (0.13 g L^{-1}), NH_4Cl (0.31 g L^{-1}), a vitamin stock solution (12.5 mL L^{-1}) and a mineral stock solution (12.5 mL L^{-1}) [23]. Power density curves were obtained by varying the circuit resistor from 5000 to 50 Ω , with a single resistor used for a full batch cycle. All of the tests were conducted in batch mode in a 30 °C incubator. The power was normalized by the projected surface area of the anode (3.0 cm^2). All of the tests were conducted in triplicate, and the mean values are reported.

2.3. Electrochemical measurements

Cyclic voltammograms (CVs) of the anode biofilms were collected using a three electrode system with an Autolab (PGSTAT 302N). The 3D anode was used as the working electrode, a saturated calomel electrode (SCE) was used as the reference electrode, and the cathode was the counter electrode. For the electrochemical measurements of the biofilms in the absence of acetate, the electrolytes were not amended with nutrition and mineral solution to avoid interference. For data analyses of the CVs, the open-source software SOAS was used for baseline (capacitive current) correction for non-turnover conditions [24]. Chronoamperometric analyses of the electrode biofilm were run at a potential of +0.2 V vs. SCE. Before each chronoamperometric measurement, the electrochemical cell was switched off for 10 min of interruption followed by 30 min of closed circuit operation. Electrochemical impedance spectroscopy (EIS) was conducted over a frequency range of 1×10^5 to 0.1 Hz with a sinusoidal perturbation amplitude of 5 mV. Before starting each impedance measurement, the MFC was operated with a 1000 Ω external load discharging for over 1 h and then pre-polarized at +0.3 V for at least 15 min to attain the static state. The obtained Nyquist plots and circle fitting software were used to analyze the resistances of the anodes. The capacitance properties of the prepared electrodes ($2.0 \times 1.5 \times 0.5$ cm) were analyzed with CVs and galvanostatic charge/discharge. The CVs were tested in three-electrode systems in 0.5 M Na_2SO_4 solution at voltage scan rates of 5, 10, 30, 50, 70, 100 mV s^{-1} in the range from -0.4 to 0.1 V (vs. SCE) at room temperature. The galvanostatic charge/discharge (C/D) was evaluated between -0.4 V and a maximum potential of 0.1 V at different currents. The specific capacitances of the anodes were normalized by the projected surface area of the anode (3.0 cm^2).

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