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Application of surface-modified carbon powder in microbial fuel cells

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ABSTRACT

The catalytic activity of surface-modified carbon powder, Vulcan XC-72R (XC), for the oxygen reduction reaction (ORR) at an air cathode in a microbial fuel cell (MFC) has been investigated. The effects of treatment with different chemicals such as nitric acid and ammonia on the chemical characteristics of XC were studied. The catalysts were characterized by Fourier transform infrared spectroscopy (FTIR), Boehm titration, and X-ray photoelectron spectroscopy. FTIR analysis showed that the functional groups of the materials were changed by chemical treatment, with nitric acid causing the introduction of oxygen-containing groups, and ammonia leading to the introduction of nitrogen-containing groups. Electrochemical measurements of MFCs containing various modified carbon materials as ORR catalysts were performed, and the results showed that chemically modified carbon materials are promising catalysts in MFCs.

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1. Introduction

In recent years, microbial fuel cells (MFCs) have attracted much attention as devices that can convert the chemical energy stored in organic or inorganic substrates to electrical energy [1–3]. MFCs could potentially be used in biomass-based energy production. However, the high cost of the materials used in MFCs, particularly the cathode catalyst, has hindered their commercial application. To date, platinum (Pt)-based catalysts have been widely used as cathode catalysts in laboratory-scale MFCs because of their high catalytic activity in the oxygen reduction reaction (ORR). However, Pt-based catalysts are rare

and expensive [4]; therefore, development of alternative materials with high catalytic activity in the ORR and sufficient chemical stability is of high priority. Much effort has focused on the development of inexpensive, non-noble metal electrocatalysts to replace Pt-based catalysts. For example, phthalocyanines and porphyrins have been examined as alternatives to Pt in MFCs [5]. Subsequently, metal oxides such as MnO₂ [6] and PbO₂ [7] and heterocyclic complexes such as iron (Fe) and cobalt (Co) heterocycles [8,9] have been reported to possess good catalytic activity for the ORR. In addition, manganese phthalocyanine [10], carbon-supported cobalt hydroxide [11], manganese oxides and polypyrrole[12,13] composites prepared by

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electropolymerization have been investigated as potential ORR catalysts. However, these catalysts are unsuitable for practical applications because of the poor electrocatalytic stability of phthalocyanines and transition metal macrocycles, as well as the toxicity of metal dioxides.

Carbon powder has been widely used in fuel cells [14] and could be an inexpensive alternative to noble metal catalysts. The presence of different heteroatoms (O, H, S, and N) bonded at the active sites of carbon particles accounts for the surface activity of carbon powder [15]. Recently, Yuan et al. [16] used a polypyrrole/carbon black (Ppy/C) composite as a catalyst for the ORR in an air-cathode MFC. They assumed that the carbon atoms in the N-heterocyclic (pyrrole) ring provided active sites for chemical adsorption of oxygen, which decreased the oxygen activation energy. Their results demonstrated that although the power output with the Ppy/C cathode was lower than that with a commercial Pt cathode, the power per unit cost of the Ppy/C cathode was about 15 times greater than that of the Pt cathode.

Recent studies have shown that surface modification is a useful way to improve catalytic activity. Duteanu et al. [17] found that the electrocatalytic activity of carbon powder treated with nitric acid for the ORR was considerably higher than that of the untreated powder. Moreover, the current density was higher than that of an untreated carbon-supported Pt cathode. Cheng et al. [18] found that treatment of a carbon cloth anode with ammonia gas substantially increased its surface charge (from 0.38 to 3.99 meq/m²), and the combined effects of ammonia treatment of the anode and phosphate treatment of the solution enhanced the power production by 48% compared with their previous results using an air-cathode MFC. In addition, the start-up time was reduced by 50%. Wang and coworkers [19] investigated the electrocatalytic activity of ammonia-treated ordered mesoporous carbon in the ORR, finding that their modified catalyst exhibited high electrocatalytic performance.

In this study, to further improve the power output and reduce the cost of MFCs, carbon-based Vulcan XC-72R (XC) catalysts were modified by treatment with nitric acid and ammonia. Treatment introduced both oxygen- and nitrogen-containing groups into the XC catalysts. The obtained catalysts were characterized by Fourier transform infrared spectroscopy (FTIR), X-ray photoelectron spectroscopy (XPS), and Boehm titration. The electrocatalytic performance of the XC catalysts for the ORR in MFCs was also tested.

2. Experimental

2.1. Preparation of catalysts

XC (Cabot, USA) was heated under reflux in 30% nitric acid for 6 h at 98 °C. Then, the treated XC was filtered, washed with deionized water, and dried at 60 °C in a vacuum oven for 12 h. The obtained catalyst is denoted XC-N.

The XC-N catalyst was heated under reflux in 25% ammonia for 6 h at 60 °C. The catalyst was then filtered, washed, and dried. The obtained catalyst is denoted XC-NA. Untreated XC is denoted XC-U.

A 20%-XC-supported Pt catalyst (E-TEK, USA) was used for comparison purposes. It is denoted Pt/XC.

2.2. MFC configuration

An MFC with a single chamber was constructed with 75 cm³ (5 cm × 5 cm × 3 cm) polymethyl methacrylate, polyacrylonitrile-carbon (6 cm × 8 cm × 0.5 cm) and graphite felt as the anode catalyst. 20% Pt or an XC carbon powder (0.5 mg/cm²) was used as the cathode catalyst. The carbon base layer and cathode catalysts were coated onto the cathode carbon cloth by alternately spraying slurries (2.5 mg/mL, $V_{\text{ethanol}}:V_{\text{nafion}}:V_{\text{polytetrafluoroethylene}} = 8:1:1$) and drying at room temperature. Titanium wire was used as the cathode and anode leads. To reduce expense, the proton exchange membrane was replaced by an ion-exchange film purchased from Zhejiang Qianqiu Company, China, and was used to separate the anode and cathode. The acrylic plates, electrodes, and membrane were assembled with a silicon gasket to prevent leakage. A resistor (1000 Ω) was routinely used as the load resistor.

2.3. Enrichment and operation

The enrichment and adaptation of the electrochemically active bacteria in the MFC were performed in batches; activated sludge was obtained from the Yudai River, Panyu District, Guangzhou (Guangdong, China) for this purpose. Reactors were inoculated with activated sludge (12.5 mL) and glucose (1 g/L, 62.5 mL) culture media solution. The culture media solution contained KH₂PO₄ (13.6 g/L), NaOH (2.32 g/L), NH₄Cl (0.31 g/L), NaCl (1.0 g/L), and a mineral stock solution (12.5 mL/L) [20].

The single-chamber MFCs were operated at ambient temperature. Each experiment was performed in duplicate.

2.4. Analysis and calculation

The external circuit voltages (E) of the MFCs were measured by a data acquisition card (model ZP1001) from Guangzhou NXP Ltd, China. The power density (P) was measured by varying the external resistance in the MFC circuit from 30 to 80000 Ω. Parameters of the MFC were calculated using the formulae: $j = E/RA$, $P = E^2/RV$ and $E = U - Ir$, where U is the electromotive force (V), r is the battery internal resistance (Ω), R is the external resistance (Ω), A is the apparent area of the anode catalyst (cm²), V is the volume of the anode chamber, and I is the current (A). FTIR spectra of the carbon supports were collected on an infrared spectrometer (Tensor 27, Brooke, Germany). For XPS measurements, a high-resolution Kratos Axis Ultra (DLD) X-ray photoelectron spectrometer with an Al (K_{α}) radiation probe was used.

2.5. Electrochemical measurements

To investigate the ORR activity of the different catalysts, linear sweep voltammetry (LSV) was performed with a rotating disk electrode (RDE, Pine Instruments, USA). A Pt wire and

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