



Candle soot-derived carbon nanoparticles: An inexpensive and efficient electrode for microbial fuel cells

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ABSTRACT

Carbon nanoparticles (CNPs) derived from candle soot were used to prepare an efficient electrode of a microbial fuel cell (MFC). The candle soot were deposited on an ultrafine stainless steel (SS) wire disk, and the SS disk-supported CNPs were directly used as the electrodes. The physico-electro-chemical characterization tests showed the prepared electrode materials to be hierarchically porous, graphitic, and mechanically and electrochemically stable. The polarization studies using linear sweep voltammetry analysis revealed the maximum open circuit potential, power and current densities of the fabricated MFC to be 0.68 ± 0.03 V, 1650 ± 50 mW/m² and 7135 ± 110 mA/m² respectively. The method of preparation for the soot-derived CNP-based electrode material is simple, cost-effective, reproducible, and scalable, and the fabricated MFC has potential for producing high bioenergy.

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1. Introduction

Microbial fuel cells (MFCs) are an emerging and promising bio-electrochemical device which can convert chemical energy into electricity, using bio-catalysts (or microbes) and aqueous organic waste in a single step [1]. This is also considered to be a useful technique for the removal of toxic metals from wastewater [2–4]. It is, therefore, perceived that MFCs can meet the globally increasing demand for renewable energy as well as clean water, using industrial aqueous effluents [5,6]. A common configuration of MFCs includes an H-shaped cell containing an anode and a cathode separated by a cation exchange membrane (CEM) and connected via an external resistive load.

Significant efforts have been made in the last decade to develop efficient MFCs with low fabrication cost [1]. In this context, development of simple, efficient and cost-effective electrode materials are critical to producing this device economically and practically [1,7–10]. Whereas the anode material should be hierarchical porous (with micro- and mesopores) as well as biocompatible to promote the growth of biofilm on its surface [11], the cathode

material must serve as an active catalyst for oxygen reduction reaction (ORR) in MFCs [12,13].

Several types of carbon-based electrodes (anode as well as cathode) are used in MFCs. These include carbon cloths, carbon felt, granular activated carbon, reticulate carbon foam, and activated carbon fibers (ACFs) [14–18]. Such materials have also been functionalized with metals or metal oxides with a view to enhancing the metabolic reactions and ORR at anode and cathode, respectively [10,12]. In this context, composites of carbon and noble metals, primarily Pt and Ru, have been used as efficient electrodes for MFCs [12,19,20]. High cost of noble metal-based electrodes is, however, a concern. Therefore, transition metal/metal oxide (e.g., Fe₂O₃, MnO₂, and NiO)-dispersed carbon materials have been synthesized and indicated to be potentially alternative to noble metal-based electrode materials for MFCs [21–23]. In recent years, new types of carbonaceous materials including carbon nanotubes, carbon nanofibers, graphitic brush, graphene and graphene oxide have emerged as an efficient electrode material for MFCs [10,16,18,20,24]. Such materials are electro-conductive and show an improved catalytic activity towards ORR, with reduced charge transfer resistance (R_{ct}). The graphitic materials have also been doped with metal nanoparticles to increase the electro-conductivity of the electrodes [10,13,23]. Nitrogen doping has also

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been performed to facilitate electron transfer on the electrode surface [1,9,25]. Despite development of such numerous electrode materials, a number of drawbacks such as toxicity towards bio-catalysts, cost-ineffectiveness, difficulty in scaling, and complex methods of fabrication are hurdles in the successful commercialization of MFCs.

Carbon nanoparticles (CNPs) are commonly generated during the combustion processes, which contain a fractal-like interconnected porous structure [26]. Attributed to large surface area, hydrophobicity, mesoporosity, and good electrical conductivity, the candle soot-derived CNPs have recently been used for applications such as super capacitor, solar cell, coatings, and electrodes for lithium ion batteries [26–30]. In the present study, an inexpensive and facile technique is proposed for the fabrication of an efficient electrode material for MFC, based on the CNPs generated from candle soot. To the authors' knowledge, the low-cost candle soot-generated CNPs are introduced for the first time in this study, as an alternative to conventional graphitic materials used for the electrodes of a MFC.

2. Materials and methods

2.1. Materials

All chemicals used in preparing anolyte and catholyte, such as sodium acetate (CH_3COONa , purity > 98%), sucrose, potassium ferricyanide ($\text{K}_3\text{Fe}(\text{CN})_6$, purity > 99%), sodium chloride (NaCl), potassium chloride (KCl), potassium dihydrogen phosphate (KH_2PO_4) and anhydrous di-sodium hydrogen phosphate (Na_2HPO_4) were purchased from Merck, Germany. Nitrogen (purity > 99.999%) gas was purchased from Sigma Gases, India. The *Escherichia coli* (*E. coli*, K-12) culture was indigenously procured. Tryptone and yeast extract used for preparing Luria-Bertani (LB) medium were acquired from Thomas Baker Laboratory Reagent, India. Anolyte and catholyte used in this study were prepared in Milli-Q water. Ultrafine SS wire mesh (aperture size = ~0.35 mm and wire diameter = ~0.16 mm), the acrylic materials used for fabricating MFC, and candles were bought from the local market. The Nafion 119 CEM was purchased from Sinsil International, India. Activated carbon (AC) and carbon nanotube (CNT, type 4) were purchased from Merck, India and SRL, India respectively.

2.2. Synthesis of candle soot electrodes

The as-received SS wire mesh were cut into spherical shaped disks (thickness = 1.5 mm, diameter = 10 mm). The disks were successively pretreated in different reagents by sonication, namely, 1 N HCl, acetone, ethanol and Milli-Q water [26]. Next, candle (diameter = 20 mm) was lighted and the treated disk was held exactly over the tip of the flame. Soot were deposited from the tip of the flame on the disks for 30 min. The soot-deposited disks (area = 78 mm^2) were then compressed for 1 min at 120°C and 8 ton-pressure, using a hydraulic press. The compressed soot containing SS disks were directly used as electrodes (anode or cathode) of a double chamber MFC (DCMFC), without further treatment. The electrodes were prepared in duplicate to check the reproducibility. Also, new electrodes were used in all experiments. The steps involved in the fabrication of the electrodes are illustrated by Fig. 1.

2.3. Fabrication, operation and electrochemical analysis of DCMFC

In this study, an H-shaped MFC was used, which comprised of two chambers, each having an effective volume of ~50 mL. The chambers were fabricated from five acrylic plates ($L \times W = 75 \text{ mm} \times 75 \text{ mm}$) and were separated using a CEM (exposed

area = $\sim 0.001 \text{ m}^2$). The distance between the electrodes was kept at approximately 15 mm. A 0.2 mm-thick tungsten wire was used to externally connect the fabricated CNP-electrodes in the respective chamber, via a $5000\text{-}\Omega$ external resistance. An electronic multimeter (Model 2000, Keithley) was connected in parallel to the external resistance. The anolyte and catholyte were prepared using the previous protocol [10,13]. Briefly, 48 mL-volume of anolyte was prepared from an equal volume (12 mL each) of 10 mM PBS (pH = 7), 100 mM sucrose, *E. coli* LB broth ($10^8\text{--}10^9$ CFU/mL at pH = 7) and 12 mM CH_3COONa . The anolyte was purged with nitrogen gas for 30 min to ensure an anaerobic condition. Approximately 48 mL of 100 mM $\text{K}_3\text{Fe}(\text{CN})_6$ solution was prepared in 10 mM PBS and the mixture was used as the catholyte. Atmospheric air (~50 sccm) was continuously supplied to the cathode chamber using a peristaltic pump (speed = 50 rpm) throughout the experiment. The experiments were performed in batch mode at room temperature ($30 \pm 5^\circ\text{C}$).

Cyclic voltammetry (CV) tests were performed using a single channel electrochemical data acquisition workstation (AUTOLAB-PGSTAT302N, Metrohm, The Netherlands). A standard three-electrode configuration was used for the CV analysis, which had a working electrode (the prepared CNP-electrode), a counter electrode (Pt rod) and a reference electrode (Ag/AgCl electrode, 0.197 V vs. SHE, 3M KCl). Experiments were also performed using the SS disk as the working electrode, as per the same protocol. Kinetics of the electron transfer at the electrode surface was studied from the CV data fitted with the Randles-Sevcik equation as follows:

$$i_p = 0.4463nFAC \left(\frac{nFvD}{RT} \right)^{0.5} \quad (1)$$

where i_p is peak current (Ampere), n is the number of electrons transferred during redox reaction, $F = 96487$ (coulombs/mole), A is the area of the CNP working electrode, D is the diffusion coefficient of the electrolyte (cm^2/s), C is the initial molar concentration of reducible electrolyte, v is scan rate (V/s), R is the ideal gas constant and T is the operating temperature (K). Chronoamperometry tests were performed to determine the response time and investigate the kinetics of electrochemical reactions occurring at the surface of the working electrode. Further analysis of the data was carried out using the Cottrell equation as follows:

$$i = \frac{nFACD^{0.5}}{(\pi t)^{0.5}} \quad (2)$$

where i is current (ampere), n is the number of electrons transferred during redox reaction, $F = 96487$ (coulombs/mole), D is the diffusion coefficient of the electrolyte (cm^2/s), A is the area of working electrode (cm^2), C is the initial molar concentration of electrolyte, and t is time (s).

The ORR activity of the prepared cathode was determined using linear sweep voltammetry (LSV) at the scan rate of 10 mV/s in 10 mM oxygen-saturated PBS electrolyte at 600-rpm rotation speed. The three-electrode assembly was used for the measurements. The experiments were also performed using commercially available carbon materials, i.e., AC and CNT for the comparison purposes.

The LSV tests were performed using the same potentiostat as earlier described. The data acquisition workstation was connected to the cathode, whereas the anode was directly connected to the counter and reference electrodes of the workstation. Polarization curves were generated from 1 to 0 V at a scan rate of 1 mV/s, using NOVA 1.11 software. However, to determine the effect of scan rates on the prepared electrode, few experiments were performed at

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