



In situ nitrogen-doping of nickel nanoparticle-dispersed carbon nanofiber-based electrodes: Its positive effects on the performance of a microbial fuel cell



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ABSTRACT

Nitrogen (N) was doped *in situ* in the nickel (Ni) nanoparticle (NP)-dispersed carbon nanofibers (CNFs) that were grown on an activated carbon fiber (ACF) substrate, using catalytic chemical vapour deposition. Acetonitrile served as the single source of carbon and N for growing and doping CNFs, respectively. The prepared N-Ni-CNF/ACF was directly used as the electrodes in a microbial fuel cell (MFC) for bioelectricity production. The performance of MFCs considerably improved using the N-doped material, with the maximum power density measured to be $1850 \pm 20 \text{ mW/m}^2$, which was approximately two-fold higher than that of the Ni-CNF/ACF (without N-doping)-based MFCs. Furthermore, the charge transfer resistance of the N-Ni-CNF/ACF electrode-based MFC was significantly low. The N-doping provided a favourable growth condition for bacteria; enhanced the electrical conductivity of the electrode material, and improved the electro-catalytic activity towards oxygen reduction at the cathode. The method of N-doping the Ni-CNF/ACF electrode is simple and effective, and the prepared N-Ni-CNF/ACF is a promising electrode material for MFCs.

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1. Introduction

At present, the production of clean and renewable energy is one of the leading challenges of the scientific community. The focus is on the utilization of waste materials, which are inexpensive, abundant and easily accessible, as a renewable source of energy for the sustainable energy production. In this context, microbial fuel cell (MFC) is a novel technology that can serve these purposes together [1–3]. The fabrication of low cost and superior quality electrodes is a key challenge in developing high performing MFCs [1,4].

Carbon nanofibers (CNFs), carbon nanotubes (CNTs), and activated carbon have been used as efficient electrode materials for MFCs [5–11]. A fiber brush having graphitic structure has also been used as electrode materials and shown to improve the performance of MFCs to an appreciable extent [12]. A few recent studies have shown that the performance of MFCs was considerably enhanced using the nitrogen (N)-doped CNF/CNT-based electrodes, attributed to the N-induced charge delocalization in the electrode materials [13–16]. The N-doped carbon powders and

nanoparticles (NPs) have also been synthesized as electrode materials for MFCs [17–21]. From an overview of the aforementioned studies, it is clear that the electrode materials required processing after synthesis and before the end-application. Therefore, there is a need to prepare an efficient material via a simple route, to be directly used as electrodes in MFCs, i.e. without any post-synthesis step [22].

More recently, the nickel (Ni) NP-dispersed CNFs were grown over an activated carbon fiber (ACF) substrate, and the prepared Ni-CNF/ACF was directly used as efficient electrodes in a double chamber MFC for bio-energy generation [23]. A unique advantage of growing CNFs on an ACF substrate is that the method does not require a post-treatment step and the prepared multi-scale web of CNF/ACF can be directly used in an end-application. Also, the Brunauer-Emmet-Teller (BET) surface area (S_{BET}), mesoporous and graphitic contents of such materials are significantly high which provide a favourable condition for electrode applications.

In this work, a facile one-step fabrication of an N-doped Ni-CNF/ACF electrode is described and the resulting significantly enhanced performance of MFCs, fabricated using N-Ni-CNF/ACF, is experimentally demonstrated. In this method, the electrodes are prepared using chemical vapour deposition (CVD), with acetonitrile (CH_3CN) used as the source of carbon (C) for growing CNFs on

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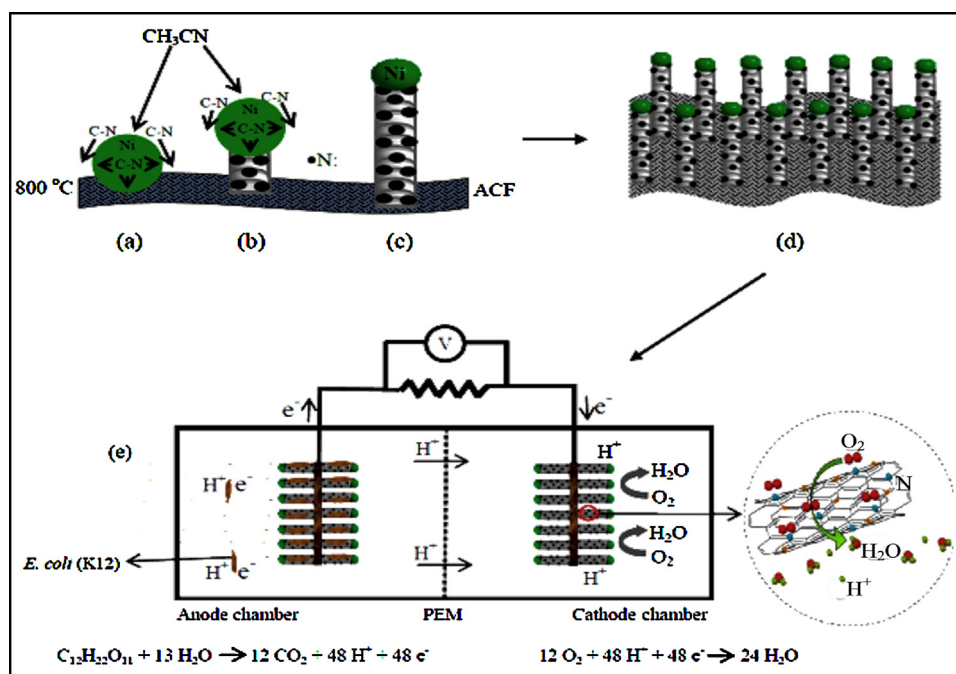


Fig. 1. Schematic illustration of (a–d) one-step CVD synthesis of N-Ni-CNF/ACF and (e) its application as the MFC electrodes.

the ACF substrate and that of N to simultaneously incorporate N-content in the CNFs. This way N-content was incorporated in the CNFs during CVD without any post-functionalization step. The prepared N-Ni-CNF/ACF was directly used as the electrodes, without any further treatment, in an H-shaped MFC.

2. Material and methods

2.1. Materials

The phenolic resin precursor-based ACFs were purchased from Gun Ei Chemical Industry Co. Ltd. (Japan). All high purity and analytical grade chemicals and reagents used in this study were procured from Merck (Germany). The high purity hydrogen (H_2) and nitrogen (N_2) gases were purchased from Sigma Gases (India). The *E. coli* (K12) culture was provided by Department of Biological Science and Bioengineering, Indian Institute of Technology Kanpur, India. Milli-Q water was used to prepare all aqueous solutions in this study. Proton exchange membrane (PEM, Nafion 117) and the standard Pt/C (0.5 mg/cm^2 Pt-loading) electrode were procured from Sinsil International (India).

2.2. Synthesis of N-Ni-CNF/ACF-based electrodes

The general procedure for the synthesis of N-Ni-CNF/ACF is the same as described in the previous study [23]. The salient difference between the previous and present study lies in using CH_3CN (source of C and N) at room temperature ($30 \pm 5^\circ\text{C}$), instead of benzene (source of C) at 8°C during CVD. Fig. 1 schematically describes the one-step preparation of N-Ni-CNF/ACF and its application as the electrode material in the MFC used for bioelectricity production. For the comparison purposes, a few non-N-doped samples (Ni-CNF/ACF) were also prepared.

2.3. MFC set-up, electrochemical measurement and analysis

The configuration of the H-shaped MFC, the composition of the electrolytes, and the protocols used for performing the

electrochemical measurements, using linear sweep voltammetry and electrochemical impedance spectroscopy (EIS), have been previously described [23]. Briefly, the power density and open circuit potential (OCP) were measured using the potentiostat (AUTOLAB-PGSTAT302N) and the linear sweep voltammetry (LSV) method. The scan rate was maintained at 1.0 mV s^{-1} from 1 V to 0 V. The polarization curves were drawn using Nova software. EIS measurements were performed for the complete cell, at the OCP of the respective electrodes, to calculate the internal resistances, namely, the solution/ohmic resistance (R_s), charge transfer resistance (R_{ct}) and Warburg mass transfer diffusion resistance (W), of the different prepared MFCs, using the FRA software and the potentiostat. For EIS measurements, the frequency was varied from 1,00,000 to 0.01 Hz at the potential amplitude of 0.01 V. The two electrodes-assembly was used for the electrochemical analysis, in which the counter and reference electrodes of the potentiostat were electrically shorted and connected to the cathode of the MFCs, whereas the working electrode was connected to the anode. Before electrochemical measurements, the MFCs were connected to a 5000Ω -load and left idle for 5 days to stabilize cell voltage. It was observed that the voltage slowly increased during the initial period of the tests and stabilized after ~ 1 day of initialization. The working area of the anode and cathode used in the present study was measured to be approximately 0.0006 m^2 . Three materials, namely, N-Ni-CNF/ACF, Ni-CNF/ACF and ACF were used at anode, keeping the commercially procured Pt/C fixed at the cathode of the MFCs. Further, tests were also performed using the other different combinations of the prepared materials including the ACF substrate, to determine the effects of Ni NPs, CNFs and N-doping on the performance of MFCs. The experiments were performed at

Table 1

Elemental (C–H–N) composition (% w/w) analysis of the prepared electrode materials.

Electrode materials	C (%)	H (%)	N (%)	Residue (%)
ACF	80.44	1.44	0.38	17.74
Ni-CNF/ACF	94.03	2.06	0.60	3.31
N-Ni-CNF/ACF	79.78	1.46	3.33	15.43

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