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A metal-free and non-precious multifunctional 3D carbon foam for high-energy density supercapacitors and enhanced power generation in microbial fuel cells

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ABSTRACT

This paper reports the synthesis of N-doped carbon foams (NCFs) using simple, environment-friendly, and self-developed freezing method. A robust structure of NCFs along with high surface area, hierarchical pore structure, and nitrogen/oxygen functionalities allows their application as free-standing electrode. Microbial fuel cell equipped with differently prepared NCFs as free-standing anode and metal-free cathode generates significantly higher power density, 35.74 $W m^{-3}$ than conventional electrodes. Furthermore, NCF with an optimized resorcinol-formaldehyde content shows significantly high-charge storage capacity, 799 $F g^{-1}$, at 0.25 $A g^{-1}$ and also delivers a high energy density, 111 $Wh kg^{-1}$, equivalent to that of a Li-ion battery.
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Introduction

The rapid increase in the world's population and industrial revolution has led to the wide extraction of fossil fuels and their rapid depletion. Under such conditions, the existing sources of fossil fuels are not expected to fulfill the world's energy needs by the end of the 21st century [1]. In addition, climate change, which is caused by environmental pollution from the burning of traditional energy sources, is widely acknowledged as the greatest environmental threat facing the world today [2]. To solve the energy problem with environmental issues, there is urgent need for clean and sustainable energy production and storage, which is also reflected by the major progress in theoretical and practical research and developments in energy production and storage [3–8]. Fuel cells and electrochemical supercapacitors/batteries are promising, effective, and upcoming technologies for the clean energy production and storage [9,10].

In both techniques, the electrodes play a very important role and make a major contribution to the cost and performance. The electrode characteristics required in both cases are low-cost, high surface area, and good electronic conductivity. Three dimensional (3D) electrodes offer a large surface area to volume ratio and are more suitable towards the practical applications of these techniques. In addition to these properties, the electrodes as an anode used in MFCs require a microorganism-favorable surface with sufficient pore openings [11], whereas as a cathode, they should have good catalytic activity for the oxygen reduction reaction (ORR) [12]. In contrast, the optimal pore size favors the electric double layer capacitance of the electrode with faster ion diffusion and charge kinetics [13].

3D carbon structures (cryogels, aerogels, and xerogels) obtained from resorcinol-formaldehyde (RF) sol–gels have attracted considerable attention as an electrode for MFCs and supercapacitors [14–16]. Although carbon aerogels and cryogels are interesting materials with high surface areas and large pore volumes, the supercritical and vacuum freeze-drying processes are extremely expensive, time consuming, difficult to scale-up, and require special instrumental facilities [17,18]. Although the solvent exchange method is available as an alternative for the vacuum freeze-drying, the excess use of toxic solvents is a major drawback that also contributes to the high cost [18–21]. Carbon xerogels obtained under ambient drying conditions are associated with

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large volume shrinkage (>90%) resulting in a comparatively low surface area and pore volume. The textural properties of aerogels are very important from the viewpoint of their applications in various fields. Therefore, there is urgent need to develop a low-cost, environmentally clean, and fast drying method for aerogels synthesis.

In the case of MFCs, carbon foam anode with an advantageous open pore structure and high external surface area can provide greater access and space for the growth of the microorganisms and contribute to higher energy production. On the other hand, nitrogen doping in the carbon texture offers exceptional ORR activity and stability, and can be replaced with traditionally used Pt catalysts to reduce the cost of MFCs without altering the overall performance [22]. In addition to the reports on development of 3D supercapacitors, very few studies have assessed the direct application of these materials as true 3D electrodes in supercapacitor. In most studies, the electrochemical properties of 3D materials were evaluated on an electrode fabricated by combining a few milligrams of powder with a binder/conducting agent [15,23,24] or sandwiched/pressed between a conducting support [25]. The charge storage capacity of the powder material is affected greatly by the material loading and coating thickness because of the higher diffusion and charge transfer resistance with a higher loading. For example, most electrode materials reported higher capacitance using their thin coating on a current collector and did not show the expected capacitance after the construction of a thick layer. Hu et al. [26] reported the dependence of the electrochemical performance on the mass and thickness of material. The increase in mass loading from 72 $\mu g\,cm^{-2}$ to 1.33 $mg\,cm^{-2}$, which was also an order of magnitude thinner than commercial ultracapacitor electrodes showed a 57% decrease in specific capacitance. The low energy density is a major disadvantage associated with supercapacitor applications to energy storage and many attempts have been made to achieve a comparable energy density to batteries [27].

The present paper focuses on the above mentioned problems in the development of carbon aerogels, MFCs, and supercapacitors, and reports a simple approach for the fabrication of N-doped carbon foam (NCF) with a newly developed drying technique. The prepared NCFs were also tested as an anode and cathode in MFCs to generate bioelectricity without the use of a Pt catalyst. An electrochemical study with a two and three electrode system was undertaken to evaluate the performance of NCFs as a metal-free 3D supercapacitor.

Experimental

Materials

A melamine sponge was purchased from Dae Han Co. Ltd., Korea. Resorcinol, formaldehyde solution, sodium phosphate monobasic dihydrate, sodium dihydrogen phosphate dodecahydrate, ammonium chloride, sodium carbonate, and potassium chloride were supplied by Duksan Pure Chemicals Co. Ltd., South Korea. The de-ionized (DI) water used in these experiments was obtained from a PUREROUP-30 water purification system. Carbon paper coated with a 0.5 $mg\,cm^{-2}$ Pt catalyst loading on one side (Fuel Cell Earth LLC, USA) was used as the cathode electrode.

Preparation of 3D N-doped carbon foams

Briefly, resorcinol (10 g) and a formaldehyde solution (37%; 14.7 g) were mixed in DI water (471 g) at a 1:2 molar ratio in a 1000 mL glass bottle. An aqueous sodium bicarbonate solution (0.2 wt.%; 24.1 g) was added as the base catalyst at a R/C ratio of 200. Pieces of melamine sponge ($3 \times 1 \times 7$ cm), which was used as a support framework and nitrogen source were immersed in the above solution, and a squeezing operation was then applied several times by compression and release followed by ultrasonication for 10 min to ensure complete wetting of the sponge. A glass jar was closed tightly and kept at 80 °C for 72 h. The obtained RF gel foam was frozen at −18 °C for >12 h (for electrochemical testing, the RF gel foam was cut into smaller pieces, $1 \times 1 \times 7$ cm in size). After the complete liquefaction of ice at room temperature, the obtained foams were dried at 80 °C using a hot air oven. Finally, carbonization of the foam was carried out at 900 °C for 1 h at a heating rate of 1 °C min^{-1} under flowing nitrogen. The resulting NCF was washed with DI water and dried at 80 °C in a hot air oven. The NCF was called NCF-5 based on the RF content (5%). A similar process was followed to synthesize the NCFs with different densities and nitrogen contents by altering the RF content and without varying the RF and RC ratio. The NCFs obtained with a RF content of 10 and 15% were labeled as NCF-10 and NCF-15, respectively.

Characterization of 3D N-doped carbon foams

Crystallographic phase identification of the sample was achieved by X-ray diffraction (XRD, PANalytical, X'pert PRO-MPD, Netherland) using Cu $K\alpha_1$ radiation ($\lambda = 0.15405$ nm). The different functional groups and chemical states were investigated by X-ray photoelectron spectroscopy (XPS, ESCALAB 250 XPS System, Thermo Fisher Scientific U.K.) using monochromatized Al Kα x-rays ($h\nu = 1486.6$ eV). The structural characteristics of the NCFs were observed by field emission scanning electron microscopy (FE-SEM, S-4200; Hitachi, Ltd.) at an accelerating voltage of 15 kV. Fourier Transform Infrared Spectroscopy (FTIR) and Raman spectroscopy were performed on a Frontier, PerkinElmer, USA and HR800 UV Raman microscope (Horiba Jobin-Yvon, France), respectively. The surface area measurements of the NCFs were carried out using a volumetric adsorption system (3Flex, Micromeritics Inc., USA) by obtaining N_2 adsorption/desorption isotherms at 77 K. The elemental composition of the samples was determined using a CHN analyzer (Perkin-Elmer, Optima 8300).

Microbial fuel cells assembly and operation

The cathodic and anodic performance of the as-prepared NCFs were analyzed in H-type MFCs, which were assembled according to previous work [16,28]. Briefly, two interconnected glass bottles (250 mL) separated by the proton exchange membrane to distinguish the cathode and anode chamber were filled separately with phosphate-buffered saline (pH 7) and LB media (250 mL) with *Shewanella oneidensis* MR-1 (5 mL), respectively. Pt-carbon paper was used as the cathode to assess the performance of NCFs as an anode. Nitrogen gas was bubbled for 10 min to remove the dissolved oxygen in the anode chamber and closed tightly to maintain an anaerobic environment for the development of EAB. On the other hand, continuous air bubbling was maintained in the cathode chamber using an aquarium air pump to provide sufficient oxygen for the ORR. All parts of the MFC were autoclaved prior to assembly to avoid bacterial contamination. The MFC was connected to a resistance box of 1000 Ω and all electrodes were hung by titanium wire. The voltage produced was monitored using a digital multimeter (Agilent 34405A, Agilent Technologies, Inc., USA) connected to a personal computer for precise data recording. After evaluating the anodic performance of the synthesized NCFs, the cathodic performance of the NCFs was also tested using NCF-10 as an anode. Data collected and stored by a data acquisition system connected to a computer was used to calculate the power density (P) by means of Eq. S1 and S2 (ESI†).

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