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Facile preparation of N-doped carbon nanofiber aerogels from bacterial cellulose as an efficient oxygen reduction reaction electrocatalyst

Fanlu Meng^{a,b,†}, Lin Li^{a,b,†}, Zhong Wu^{b,c}, Haixia Zhong^{b,c}, Jianchen Li^a, Junmin Yan^{a,*}^a Key Laboratory of Automobile Materials, Ministry of Education, and College of Materials Science and Engineering, Jilin University, Changchun 130012, Jilin, China^b State Key Laboratory of Rare Earth Resource Utilization, Changchun Institute of Applied Chemistry, Chinese Academy of Sciences, Changchun 130022, Jilin, China^c University of Chinese Academy of Sciences, Beijing 100049, China

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ABSTRACT

Carbon aerogels have attracted considerable attention over the past few decades as promising materials for catalyst supports, electrodes for supercapacitors and lithium-ion batteries, and adsorbents. However, expensive and toxic precursors as well as complicated synthetic methods dramatically limit their large-scale production and application. In this work, we developed a facile and effective route to prepare a N-doped carbon nanofiber aerogel (N-CNFA) with low mass density, continuous porosity, high specific surface area, and electrical conductivity from a bacterial cellulose precursor. Because of the highly porous and interconnected 3D structure, the obtained N-doped carbon aerogel was used directly as a catalyst for the oxygen reduction reaction (ORR), and it exhibited superior catalytic activity. This activity was much higher than that obtained without N-doping, and it can potentially be applied to high-performance fuel cells.

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1. Introduction

Carbon aerogels (CAs), as a special class of carbon foams, were first synthesized by Pekala [1] in 1989. Over the past few decades, CAs have attracted considerable attention because of their superior characteristics such as low mass density, controllable uniform micro- and meso-porosity, and large specific surface area [2–4]. Their superior properties derive from their unique three-dimensional interconnected network. Additionally, CAs are more chemically and thermally stable than other classes of porous materials such as porous silica, MOFs, and

zeolites [5]. Accordingly, CAs are promising materials for use as catalyst supports, in water purification, as electrode materials for supercapacitors and lithium-ion batteries, as adsorbents, and as gas sensors [6–14]. For example, Ahn and co-workers [15] prepared metal-doped CAs with a very high surface area of up to 3200 m² g^{−1}, and they exhibited excellent hydrogen storage ability. Chen et al. [16] demonstrated a high-performance supercapacitor using a MnO₂-modified carbon nanofiber aerogel (CNFA) as an electrode material. Owing to the low mass density and high specific surface area of the MnO₂-CNF electrode, a supercapacitor containing this electrode exhibited a

* Corresponding author. E-mail: junminyan@jlu.edu.cn

† These authors contributed equally to this work.

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high energy density of $32.91 \text{ W h kg}^{-1}$. Good cycling stability of 95.4% was retained with specific capacitance after 2000 cycles. Flexible and stretchable conductors prepared by the infiltration of flexible graphene foam with elastic polymers showed great potential [17]. Very recently, Wu et al. [18] reported the production of a CNFA from a template-directed hydrothermal carbonization process using glucose as the precursor. The as-prepared CNFA was used as an adsorbent to clean oil and chemical waste, and it demonstrated a very high sorption capacity and good recyclability.

Traditionally, CAs have been prepared via sol-gel transition, which involves the transformation of a wet gel precursor into a highly cross-linked aerogel. To preserve its tenuous network, freeze drying or supercritical drying are often used to remove the background liquid because of low surface tension. With Pekala's method, CAs are generally obtained from the carbonization of an organic precursor, and these are prepared by the sol-gel polycondensation of organic monomers such as resorcinol and formaldehyde [2–4,19]. Additionally, the porous structure can be modified by changing the synthesis conditions and synthesis routes to accommodate application requirements [5,20–29]. However, processing requires toxic and expensive precursors while alternative approaches to prepare CAs that are cheap, nontoxic, and environmentally friendly are desired.

Biomass materials such as bacterial cellulose (BC), a type of natural cellulose, have recently attracted much research interest. Typically, BCs are synthesized using certain bacteria (i.e. *acetobacter xylinum*) that are nontoxic, environmentally friendly, cheap, and easy to obtain [30]. BC pellicles are composed of highly interconnected cellulose nanofiber networks, and they are favorable precursors for the large-scale fabrication of CAs. Ultralight, flexible, and fire-resistant BC-derived CNFAs have been produced by a facile route and can potentially be used in pressure sensors and pollution adsorbents [31]. Very recently, a N-doped CNF derived from a polyaniline coated BC was used as an electrode material for the fabrication of a supercapacitor, and it had a high energy density and long cycling performance [32]. Many researchers have reported catalysts based on CAs, but little attention has been given to the catalytic activity of CNFAs derived from BCs.

Herein, we report a facile and effective way to produce a CNFA using BC as both a template and precursor. The catalytic activity of the as-prepared CNFA toward the oxygen reduction reaction (ORR) was evaluated. Owing to the highly porous and interconnected 3D structure, it was expected that carbon materials derived from BC should be good catalysts for the ORR. According to the literature, N-doping can result in more catalytically active sites and higher porosity, which can evidently enhance catalytic performance [33]. To further improve the catalytic activity, as illustrated in Fig. 1, we prepared a N-doped

CNFA (N-CNFA), and it showed superior catalytic performance. The N-doped CNFA also gave excellent electrical conductivity and had a low mass density, thus making it a promising candidate for application in future high-performance fuel cells.

2. Experimental

2.1. Material preparation

BC was purchased from the Hainan Yida Food Industry, China. The BC was washed using deionized water several times and then placed into liquid nitrogen to ensure a quick freeze followed by freeze drying overnight. This processing method can preserve the tenuous network owing to the low surface tension. The dehydrated BC was then heated in a tubular furnace with N_2 at $3 \text{ }^\circ\text{C min}^{-1}$ to $900 \text{ }^\circ\text{C}$ for 1 h to complete the carbonization. The resulting compound was then heated in a tubular furnace with ammonia gas at $3 \text{ }^\circ\text{C min}^{-1}$ to $900 \text{ }^\circ\text{C}$ for 1 h to complete the N-doping process.

2.2. Physical characterization

Scanning electron microscopy was performed using a HITACHI S-4800 field emission scanning electron microscope (FESEM). Transmission electron microscopy (TEM) and high-resolution transmission electron microscopy were undertaken on a FEI Tacnai G2 electron microscope operated at 200 kV. X-ray photoelectron spectroscopy (XPS) analysis was carried on a VG Scientific ESCALAB MKII X-ray photoelectron spectrometer.

2.3. Electrochemical measurements

All the electrochemical measurements were carried out using a VMP3 electrochemical workstation (Bio-logic Inc.) in a typical three-electrode cell at room temperature. Ag/AgCl was used as the reference electrode, and a platinum sheet was the counter electrode. A glassy carbon disk (5.0 mm diameter) served as a substrate for the working electrode to evaluate the ORR activity of various catalysts. The glassy carbon electrode was polished using aqueous alumina suspensions on felt polishing pads. The catalyst ink was prepared by blending the catalyst powder (5 mg) with a 50- μL Nafion solution (0.5 wt%) and 1 mL ethanol in an ultrasonic bath. Ten μL catalyst ink was then pipetted onto the GC surface. Linear sweep voltammograms in O_2 -saturated 0.1 mol L^{-1} KOH were measured at 1600 rpm with a sweep rate of 10 mV s^{-1} . To estimate the double-layer capacitance, the electrolyte was deaerated by bubbling with N_2 , and the voltammogram was re-evaluated in the deaerated electrolyte. The oxygen reduction current was taken as the difference between the currents measured in the deaerated and the oxygen-saturated electrolytes. Commercial 20 wt% Pt on Vulcan carbon black (Pt/C from BASF) was measured for comparison. The working electrode was prepared as follows: 5 mg Pt/C and 50 μL Nafion solution (0.5 wt%) was dispersed in 1 mL ethanol by sonication to obtain a well-dispersed ink. Ten μL catalyst ink was then pipetted onto

Fig. 1. Synthesis of a N-doped carbon nanofiber aerogel (CNFA) using bacterial cellulose (BC) as a template and precursor.

the GC surface.

The four-electron pathway in the ORR is favorable for fuel cells because it offers excellent ORR kinetics. The ORR occurred via a four-electron pathway on the Pt/C catalyst. The number of electrons transferred onto the N-CNF catalyst was calculated according to the Koutecky-Levich equation:

$$1/j_{\text{lim}} = 1/j_{\text{lev}} + 1/j_k = 1/(B\omega^{1/2}) + 1/j_k \quad (1)$$

$$B = 0.62nFC_0D_0^{2/3}\nu^{-1/6} \quad (2)$$

where j_{lim} (mA cm^{-2}) is the measured current density, which is related to the Levich current (j_{lev}) and the kinetic current (j_k), F is the Faraday constant (96485 C mol^{-1}), D_0 is the diffusion coefficient of oxygen in $0.1 \text{ mol L}^{-1} \text{ KOH}$ ($1.9 \times 10^{-5} \text{ cm}^2 \text{ s}^{-1}$), ν is the kinematic viscosity of water ($0.0107 \text{ cm}^2 \text{ s}^{-1}$), C_0 is the bulk concentration of oxygen in oxygen-saturated $0.1 \text{ mol L}^{-1} \text{ KOH}$ ($1.2 \times 10^{-2} \text{ mol cm}^{-3}$), ω is the rotating disc electrode (RDE) rotation rate, and n is the electron transfer number for the ORR. A linear plot of j_{lim}^{-1} versus $\omega^{-1/2}$ has a slope of $1/(0.62nFC_0D_0^{2/3}\nu^{-1/6})$. The constant 0.62 is used when the rotation speed is expressed in r/min .

3. Results and discussion

BC pellicles (Fig. 2(a)) were used as a precursor and template to fabricate the CNFA and N-CNFA after dehydration, carbonization, and N-doping. As shown in Fig. 2(b), (c), and (d), SEM images of the BC, the CNFA, and the N-CNFA show similar highly porous and interconnected morphology. This indicates a low mass density and a high specific surface area. These samples all form a filamentous network with little bundling. As shown in Fig. 2(b), the porosity length scale of the freeze-dried sample ranged from tens of nanometers up to 1 micrometer. However, a decrease in diameter was observed for the CNFA sample after the carbonization of BC with pore sizes ranging from tens to hundreds of nanometers, which is similar to the results reported in the literature [30,31]. Additionally, compared with the TEM image of the CNFA in Fig. 2(e), some cracks are present on the nanofibers of the N-CNFA sample (Fig. 2(f)), and this can be attributed to the etching of ammonia during the N-doping process. The inset in Fig. 2(f) clearly shows that the N-doping process resulted in the formation of a hierarchically porous structure. Micropores a few nanometers in size are uniformly distributed on the surface of the carbon nanofibers while mesopores and macropores are formed by the interconnected nanofibers. The N-doped CNFA samples thus have a larger specific surface area, higher porosity, and more catalytically active sites than the CNFA samples. Therefore, they are expected to have better catalytic performance than the CNFA samples.

XPS analysis indicates that N-CNFA is mainly composed of carbon, nitrogen, and oxygen, which mainly come from the BC pellicles and heat treatment under NH_3 . The XPS N 1s spectrum of N-CNFA reveals the presence of pyridinic N ($\sim 398.2 \text{ eV}$), nitrile N ($\sim 399.4 \text{ eV}$), pyrrolic N ($\sim 400.5 \text{ eV}$), and quaternary N ($\sim 401.6 \text{ eV}$) (Fig. 3). Because a small amount of adsorbed trace bacteria are present in the BC pellicles, CNFA might have acquired N from the BC pellicles, and N-CNFA mainly acquired N during pyrolysis under NH_3 , and it contained more nitrogen. At

Fig. 2. (a) Digital image of the BC; SEM images of the porous and interconnected structure of (b) BC after drying, (c) CNFA, and (d) N-CNFA; (e) and (f) are TEM images of CNFA and N-CNFA, respectively. The inset in (f) is a high-resolution image of a selected area.

a higher temperature, the amount of pyridinic N increased, and the N-CNFA treated at 900°C contained the most pyridinic N.

The electrocatalytic activity of the CNFA and N-CNFA materials for the ORR was examined by cyclic voltammetry (CV) in nitrogen and oxygen-saturated $0.1 \text{ mol L}^{-1} \text{ KOH}$ solutions at a scan rate of 50 mV s^{-1} . As shown in Fig. 4(a), a rectangular voltammogram without any obvious peak was observed for CNFA and N-CNFA in a nitrogen-saturated solution within a potential range from -1 to 0.2 V . A well-defined ORR peak centered at -0.14 V with a high reaction current was present in the CV plots when it was converted to oxygen, which means that high electrocatalytic activity is a characteristic of N-CNFA during oxygen reduction. The onset and peak potentials of N-CNFA during the ORR are positive, and the current density is much higher than that of the CNFA during the ORR. The excellent performance probably arises from the contribution of isolated N atoms (such as pyridine-like, pyrrole-like, and quaternary nitrogen atoms) upon ammonia treatment, which can act as active sites for the ORR.

For further insight into the ORR and reaction kinetics of CNFA and N-CNFA, RDE voltammetry (linear sweep voltammetry, LSV) was performed at a scan rate of 10 mV s^{-1} and at dif-

Fig. 3. XPS spectra: (a) survey scan of N-CNFA(900 °C); the N 1s of CNFA (b), N-CNFA(800 °C) (c), and N-CNFA(900 °C) (d).

ferent rotating speeds from 400 to 2025 rpm in an O_2 -saturated 0.1 mol L^{-1} KOH solution. As shown in Fig. 4(b), N-CNFA gave a well-defined single-step wide platform of diffusion-limiting currents below -0.3 V at all rotational speeds. This indicates an efficient surface electrocatalytic reaction with a direct four-electron ORR process. We further used the RDE to probe the effect of treatment temperature on the ORR catalytic activity of different catalysts. The LSV results for N-CNFA(800 °C) and N-CNFA(900 °C) are presented in Fig. 4(b). Notably, the onset potential of the N-CNFA(900 °C) catalyst was more positive than that of N-CNFA(800 °C). N-CNFA(900 °C) exhibited the most positive half-wave potential ($E_{1/2}$) and the highest kinetic current density. For comparison, RDE tests were also performed on commercial Pt/C. Notably, N-CNFA(900 °C) exhibited the highest onset potential, and this was a little lower than that of Pt/C, but the $E_{1/2}$ of N-CNFA(900 °C) shifted negatively by about 5.5 mV compared with Pt/C, which is very competitive. The diffusion-limiting polarization curves show a well-defined plateau, and the current density of N-CNFA(900 °C) was found to be slightly higher than that from the other catalysts and slightly lower than that from Pt/C. The data from the above-mentioned experiments convinced us that the novel N-CNFA gave the best ORR performance.

The LSV curves of Pt/C, CNFA, and N-CNFA at different rotation speeds and their Koutecky-Levich plots are shown in Fig. 4(c)–(f). The Koutecky-Levich plots show excellent linearity and parallelism for N-CNFA at various potentials compared with Pt/C, indicating first-order reaction kinetics for the ORR with respect to the concentration of dissolved oxygen. The

electron transfer number (n) was calculated at -0.5 V to be ~ 3.9 from the slopes of Koutecky-Levich plots (Eqs. (1) and (2)), emphasizing that N-CNFA(900 °C) mainly follows a four-electron ORR mechanism, similar to the ORR catalyzed by a high-quality commercial Pt/C catalyst when measured in the same 0.1 mol L^{-1} KOH electrolyte ($n = 4.0$ for Pt/C, Fig. 3(f)). The electron transfer number (n) of CNFA was calculated at -0.5 V to be ~ 2.7 , indicating a mainly two-electron ORR process. One reasonable interpretation of this N-CNFA result is that high porosity and nitrogen doping are a result of high-temperature treatment under ammonia. The doped and isolated N atoms (such as pyridine-like, pyrrole-like, and quaternary nitrogen atoms) are catalytically active sites during the ORR process. Thus, N-CNFA(900 °C) has a high diffusion-limiting current density, a high electron transfer number (~ 3.9), and a high positive half-wave potential, which is competitive with the commercial Pt/C electrocatalyst for the ORR in an alkaline electrolyte.

As an effective catalyst for the ORR, N-CNFA has the potential to replace commercially available Pt/C. We further evaluated its electrochemical stability and possible methanol crossover. The durability of N-CNFA and commercial Pt/C for the ORR was evaluated using a chronoamperometric method at -0.4 V in oxygen-saturated 0.1 mol L^{-1} KOH at a rotation rate of 1600 rpm. As shown in Fig. 5(a), the current density of N-CNFA showed a slight loss ($\sim 7\%$), indicating that the N-CNFA electrocatalyst is more stable than commercial Pt/C. The current-time (i - t) chronoamperometric response toward the ORR for Pt/C shows a sharp decrease in current upon the addition of

Fig. 4. (a) CV curves of CNFA and N-CNFA in nitrogen and oxygen-saturated 0.1 mol L⁻¹ KOH aqueous electrolyte solutions at a scan rate of 50 mV s⁻¹. (b) LSV curves of CNFA, N-CNFA, and Pt/C. LSV curves with various rotation rates for (c) CNFA, (d) N-CNFA, and (e) Pt/C. (f) Koutecky-Levich plots of CNFA, N-CNFA, and Pt/C.

2% (v/v) methanol (Fig. 5(b)). In contrast, the amperometric response from the N-CNFA electrode changed little even after the addition of methanol. However, the response does decrease with time. The stability can be attributed to its super-stable nanofiber structure, its high porosity, and its uniform nitrogen doping active sites.

4. Conclusions

We demonstrate that N-CNFA can be easily prepared as a highly efficient catalyst for the ORR. The as-prepared CNFA exhibited much lower catalytic activity than N-CNFA. This is mainly because of its higher specific surface area, hierarchical porous structure, and the higher number of catalytically active sites on N-CNFA. Compared with CNT-based CAs, our N-CNFA

had much higher catalytic activity, and it is comparable with commercial Pt/C. Additionally, the synthetic methods developed use BC as a template and a precursor in a facial manner and it is cost-effective for large-scale production. It is believed that the N-CNFA is a superior candidate for the fabrication of high-performance fuel cells. Furthermore, N-CNFA has low mass density and continuous porosity, which allows it to be used as an ideal electrode material for the production of high capacitance supercapacitors.

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Fig. 5. Chronoamperometric response toward the ORR for N-CNFA and Pt/C. (a) Durability evaluation of Pt/C and N-CNFA over 25000 s at -0.4 V vs Ag/AgCl and at a rotation rate of 1600 rpm. (b) Upon the addition of 2% (v/v) methanol after about 900 s.

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Graphical Abstract

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Facile preparation of N-doped carbon nanofiber aerogels from bacterial cellulose as an efficient oxygen reduction reaction electrocatalyst

Fanlu Meng, Lin Li, Zhong Wu, Haixia Zhong, Jianchen Li, Junmin Yan*
Jilin University;
Changchun Institute of Applied Chemistry, Chinese Academy of Sciences;
University of Chinese Academy of Sciences

N-doped carbon nanofiber aerogel (CNFA) derived from bacterial cellulose (BC) was used as an oxygen reduction reaction electrocatalyst, and it had comparable catalytic activity to commercial Pt/C.

以细菌纤维素为前驱体简便制备氮掺杂碳纤维气凝胶作为高效氧还原催化剂

孟凡陆^{a,b,†}, 李 霖^{a,b,†}, 吴 中^{b,c}, 钟海霞^{b,c}, 李建忱^a, 鄢俊敏^{a,*}

^a吉林大学材料科学与工程学院, 汽车材料教育部重点实验室, 吉林长春130012

^b中国科学院长春应用化学研究所, 稀土资源利用国家重点实验室, 吉林长春130022

^c中国科学院大学, 北京100049

摘要:数十年来, 碳气凝胶因其在催化剂载体、电容器和锂电池电极材料以及吸附剂等领域的潜在应用而备受关注。然而, 传统碳气凝胶的制备往往使用昂贵且有毒的前驱体, 其方法也较为复杂, 不利于大规模生产及应用。本文介绍了一种以细菌纤维素为前驱体制备氮掺杂碳纤维气凝胶的方法。该方法廉价高效, 简单易行且对环境无害。所制气凝胶具有密度低、孔隙度高、比表面积大以及导电性良好等优点。它继承了细菌纤维素生物质优异的三维交联多孔结构的特点, 可直接用作氧还原催化剂, 表现出优异的催化性能, 预示着其广泛的应用前景。这在该领域的应用报道尚属首次。

关键词: 碳纤维气凝胶; 氮掺杂碳; 氧还原反应; 细菌纤维素

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*通讯联系人. 电子信箱: junminyan@jlu.edu.cn

[†]同等贡献作者.

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