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One-Step Synthesis of N-Doped Carbon and Its Application as a Cost-Efficient Catalyst for the Oxygen Reduction Reaction in Microbial Fuel Cells

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Nitrogen-doped carbon with a high surface area was prepared by the one-step pyrolysis of cellulose paper under NH_3 gas, and has been developed as an alternative to Pt/C for the oxygen reduction reaction in microbial fuel cells (MFCs).The electrochemical tests showed that the catalytic performance of N-doped carbon in neutral media was significantly better than that of undoped carbon, with an onset of 0.21 V. The maximum power density of MFCs with the N-doped carbon catalyst (1041 ± 90 mW m^{-2}) was much higher than that of Pt/C-based MFC (584 ± 10 mW m^{-2}), while the cost was significantly lower. These results demonstrated that such N-doped carbon has the advantages of low cost and high efficiency, and can be a promising alternative to Pt/C in large-scale applications of MFCs.

Introduction

As a new green energy technology, microbial fuel cells (MFCs) have attracted tremendous attention in recent years.[1–3] MFCs can be used to generate electricity directly from wastewater, through microbial metabolism at the anode and reduction of electron acceptors at the cathode. Oxygen is considered as a promising electron acceptor due to its ready availability and low cost.[4] Similar to metal–air batteries and chemical fuel cells, the development of MFCs has been hindered by the poor kinetics of the oxygen reduction reaction (ORR), especially due to the neutral medium which is favorable for the microorganism.[5] The ORR can proceed via a 4 e^- or 2 e^- pathway, depending on catalyst. Platinum (Pt) is usually used as an efficient catalyst in MFCs to facilitate ORR through the 4 e^- pathway. However, the catalyst accounts for the majority of the MFC cost, and is the bottleneck for the large-scale application of MFCs.[6]

Extensive research has been conducted to find a cost-efficient alternative to Pt. Transition metal macrocyclic compounds, such as phthalocyanines and porphyrins, were developed as cathodic catalysts to replace Pt in MFCs,[7] and subsequently metal complexes and metal oxides were tested.[8] Some conducting polymers, such as polyaniline[9] and polypyrrole,[10] were also considered. But so far these catalysts have still not overcome face the challenges of limited performance and high cost due to high loading. Promising alternatives are carbon-based materials, including activated carbon (AC),[11] granules,[12,13] nanotubes,[14] biochar derived from sewage sludge,[15] due to the advantages of high electrocatalytic activity, environmental friendliness, and relatively low cost. Despite great progress in the development of carbon catalysts for the ORR, finding a better way to further improve the catalytic performance and satisfy the requirements for MFC application is still highly desirable.

An effective approach can be provided by heteroatom doping on carbon,[15–17] in particular nitrogen doping. But only a few studies about the application of N-doped carbon in MFCs have been reported so far. N-doped nanotubes, which were synthesized by chemical vapor deposition (CVD), obtained comparable power density to Pt/C in MFCs.[18] The researchers also applied N-doped graphene to MFC systems as a cathodic catalyst.[19] However, these catalysts were based on nanostructured carbon prepared by a complex procedure, and they are not practical for large-scale application. More attention has been paid to the choice of inexpensive activated carbon to improve the performance of cathodes, as it is among the most promising catalysts in the MFC field. For example, an MFC with an N-doped AC catalyst, which is synthesized by pyrolysis of AC in the presence of solid source of nitrogen in the presence of solid source of nitrogen, that is, cyanamide, outperformed the MFC with an Pt/C catalyst.[20] However, pretreatment of the AC with acidic and alkaline solutions was required before the pyrolysis, and the improved electrocatalytic activity was attributed predominantly to the pretreatment rather than the N-doping. The defects introduced by the pretreatment facilitate N-doping during the subsequent pyrolysis of the carbon material in the presence of nitrogen precur-

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sors. Meanwhile, the N-doped activated carbon (AC-N) prepared by pyrolysis of AC under NH_3 gas showed high ORR catalytic activity in MFCs.[21]

AC can be made from raw lignocellulosic biomass, such as coconut shells and wood chips. So it is quite possible to form N-doped carbon based on lignocellulosic biomass by a simplified process. And most importantly, the oxygen-rich functional groups in lignocellulosic biomass are considered to be favorable for N-doping, which is quite different from the N-doping of pure carbon.[22] Cellulose is one of the most abundant, sustainable, and oxygen-rich materials in nature. Herein, cellulose paper was heated with NH_3 gas to prepare N-doped carbon. The catalytic activity of N-doped carbon for the ORR in neutral medium was examined by electrochemical measurements. Finally, the performance of the N-doped carbon catalyst in MFCs was evaluated and compared with that of Pt/C in MFCs.

Results and Discussion

Characterization of the catalyst

The N-doped carbon catalyst was synthesized through the pyrolysis of cellulose paper in the presence of NH_3 gas. X-ray diffraction (XRD) analysis was used to investigate the effect of N-doping on the structure of the carbon catalyst. As shown in Figure 1 A, two diffraction peaks at $2\theta = 25^\circ$ and 43°, corresponding to the graphitic (002) and (101) planes, were observed in the XRD pattern. The shaper diffraction peaks at $2\theta = 25^\circ$ for the N-doped carbon indicate a higher graphitization degree than that in the undoped carbon. As shown in Figure 1 B, D, after the pyrolysis at 950 °C, the fiber-like morphology of the catalyst still remained, with the fiber diameter ranging from 2 to 20 μm. This was further validated in the transmission electron microscopy (TEM) images (Figure 1 C). Scanning TEM and element mapping images suggest that the nitrogen is uniformly distributed on the carbon, indicating the N atoms can be homogeneously doped into the carbon frameworks by this method (Figure 1 E–H). Furthermore, the energy-dispersive X-ray (EDX) spectra of the as-prepared sample indicate that the atomic ratios of N and O are about 2.31 % and 4.17 %, respectively.

XPS was performed to study the element content and its state of bonding with carbon framework. In synthesized N-doped carbon, the atomic ratios of N and O were 2.48 % and 4.23 %, respectively (Figure 2 A), which is agree with the EDX result. The XPS spectrum (Figure 2 B) has an apparent N 1s peak, which can be split into three peaks corresponding to pyridinic-N (398.3 eV), pyrrolic-N (400.1 eV), and graphitic-N (401.2 eV), strongly suggesting the successful incorporation of nitrogen atoms into carbon framework. Furthermore, the pyridinic-N and graphitic-N dominated in the N-doped carbon, both of which have positive effects on electrochemical performance of carbon catalyst.[25,26]

N_2 adsorption–desorption analysis was also performed to study the pore structure of the carbon catalyst (Figure 3 A). The BET surface area of the N-doped carbon is 1294.9 $m^2 g^{-1}$, which is much higher than that of undoped carbon (786.9 $m^2 g^{-1}$). Both doped and undoped carbon exhibit typical type I adsorption–desorption isotherms with a steep increase at low relative pressure ($P/P_0 < 0.1$), implying the characteristics of uniform microporosity. From the pore size distributions (PSD) plots, a sharp peak at about 2 nm was observed for N-doped carbon, while a broad peak over the mesopore range was observed for undoped carbon (Figure 3 B). After N-doping, the micropore area increased from 668.7 $m^2 g^{-1}$ to 1170.6 $m^2 g^{-1}$, accounting for major of the BET area. The increased micropores might due to the reaction between carbon atoms and NH_3 during the pyrolysis of cellulose paper. These abundant, dominant, and uniform micropores might provide additional active sites for ORR, thereby promoting the catalytic activity.[23] These results demonstrate that the N atoms doped in carbon changed the carbon surface properties slightly and the higher surface area plays a key role in improving the ORR catalytic activity.

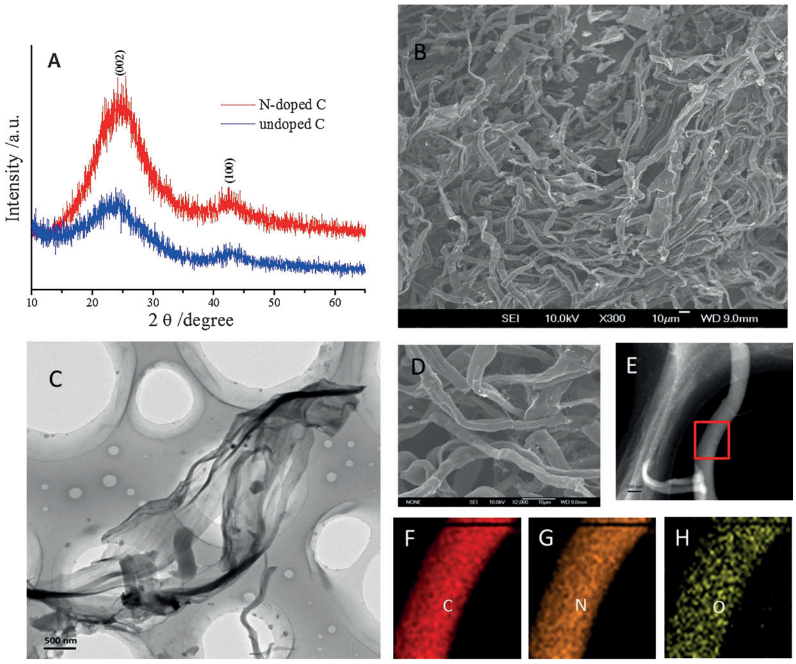

Figure 1. A) XRD patterns of N-doped and undoped carbon. SEM (B), TEM (C), high-resolution SEM (D), and STEM (E) images of the N-doped carbon. F–H) Elemental mappings of the rectangle area in (E).

Electrochemical Performance

The electrocatalytic activity of N-doped carbon was first evaluated by cyclic voltammetry (CV)

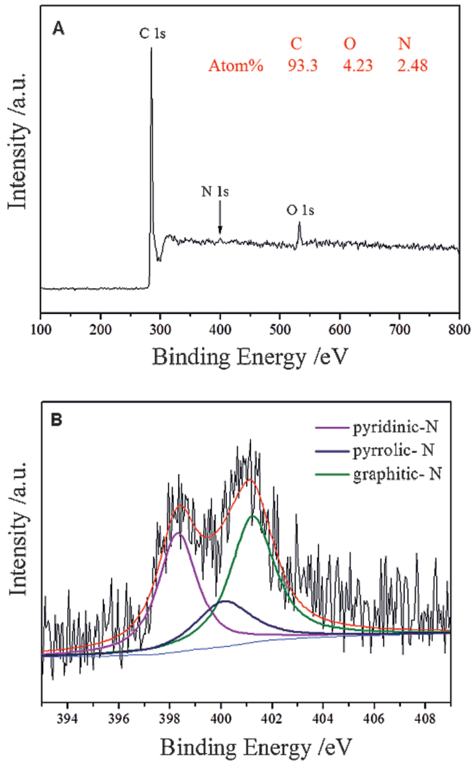

Figure 2. A) XPS survey and B) high-resolution N 1s spectrum of the N-doped carbon.

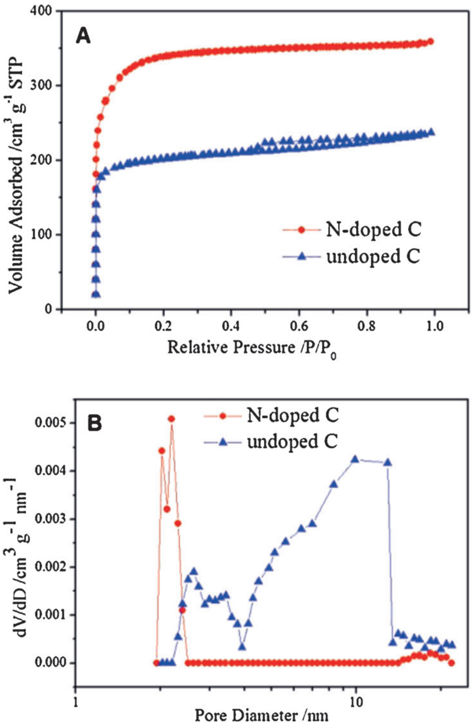

Figure 3. A) N_2 sorption isotherms and B) pore size distributions of N-doped and undoped carbon.

measurements in 50 mM phosphate-buffered saline (PBS), and compared to the performance of commercial Pt/C (20 wt %). As shown in Figure 4 A, N-doped carbon exhibited an obvious peak in O_2-saturated solution, whereas no peak appeared when it was measured in N_2-saturated solution, confirming the electrocatalytic activity of N-doped carbon for the ORR. The N-doped carbon displayed a peak potential of −0.11 V, which was more negative than that of commercial Pt/C (0.07 V). Subsequently, linear sweep voltammetry (LSV) measurements were performed to further investigate the electrocatalytic activities of the catalyst on a rotating disk electrode (RDE) at 1600 rpm in O_2-saturated solution. It can be observed from Figure 4 B that the onset potential of N-doped carbon was 0.21 V, which was not as excellent as that of Pt/C (0.32 V), but was greatly improved compared over that of the undoped carbon (−0.27 V). N-doping could break the electroneutrality of carbon and create favorable positive charged sites for O_2 adsorption, thus promoting ORR activity.[23]

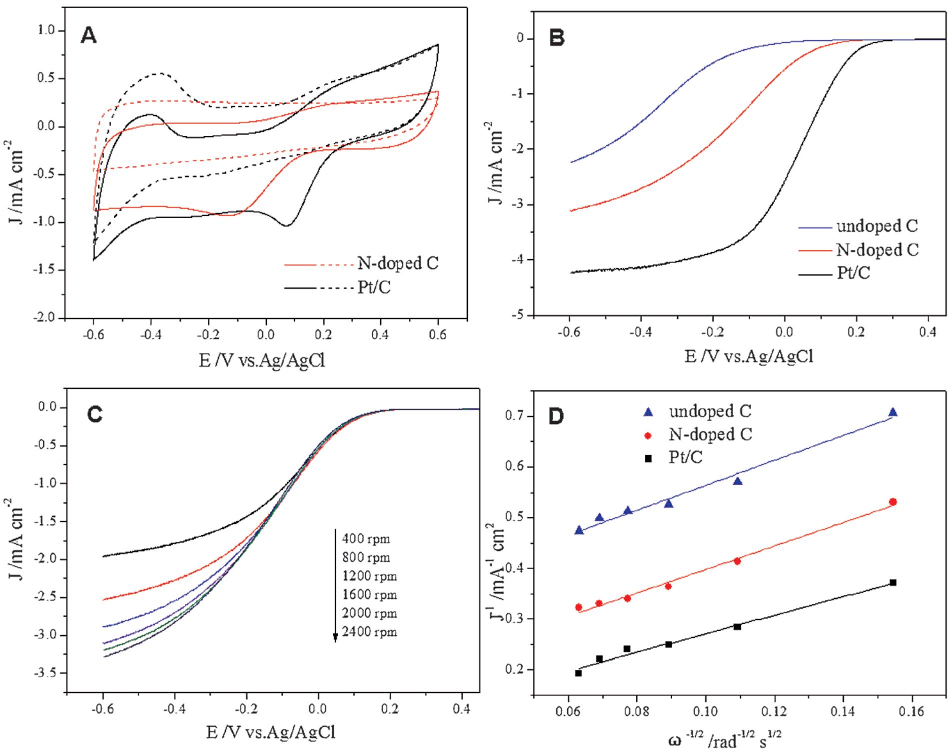

Figure 4. A) CV of N-doped and undoped carbon in O_2-saturated (solid line) and N_2-saturated (dotted line) 50 mM PBS. B) LSVs for different catalysts in O_2-saturated PBS at a rotation rate of 1600 rpm. C) LSVs for N-doped carbon at different rotation rates. D) K–L plots for different catalysts at −0.50 V.

The current density of N-doped carbon increased with the rotation rates (from 400 to 2400 rpm), as presented in Figure 4C. The Koutecky–Levich (K–L) plots at a potential of −0.5 V were obtained and displayed good linearity. The *n* value for N-doped carbon carbonate at −0.5 V was calculated to be 2.95, indicating a combined 2 e$^-$/4 e$^-$pathway for the N-doped catalyst. Whereas the *n* value for Pt/C was 3.74, which is higher than that for N-doped carbon. These results are in agreement with the CV results.

It is worth noting that the conditions of the RDE tests where diffusion limitation can be ignored due to aeration are quite different from the conditions in air-cathode MFCs. Besides, the loading of the N-doped carbon catalyst was almost 8 times higher than that of Pt/C (0.5 mg cm^{-2}) in the fabricated air-cathode, comparing to the constant loading in RDE test. Thus the catalytic performance of the catalyst reflected in RDE is likely not significant enough to predict the performance in a real air-cathode MFC.

Electricity Generation in MFCs

The voltage of MFCs at 1000 Ω became stable and repeatable after about one week. The cell voltage of about 530 mV achieved by N-doped carbon which was higher than that of Pt/C (490 mV), whereas the undoped carbon without NH_3 treatment only achieved a voltage of 420 mV. Moreover, no voltage decay occurred for N-doped carbon after more than 9 cycles, indicating very good durability—an important factor for the long-term operation of MFCs (Figure 5). Polarization and power density curves were examined to further evaluate the performances of different catalysts. As shown in Figure 6A, a highest open circuit voltage was achieved by Pt/C, but its polarization was more serious than N-doped carbon. And a maximum power density of 1041 ± 90 mW m^{-2} was generated by N-doped carbon, which was higher than that of undoped carbon (420 mW m^{-2}) and Pt/C (584 ± 10 mW m^{-2}) and agreed well with the cell voltage output results. According to the electrode potential curves, the cathodic catalyst was responsible for the improved power density of the MFC, since all anode potential curves were almost constant whereas the cathode potential varied with the catalyst (Figure 6B).These results demonstrated that N-doping could be an effective way to improve the ORR catalytic activity of carbon.

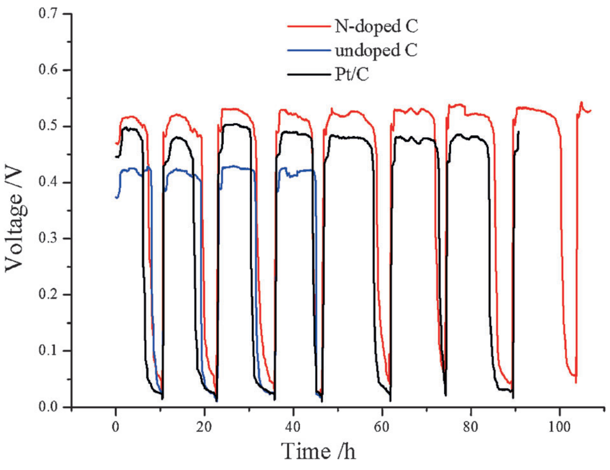

Figure 5. Voltage output curves of MFCs with different catalysts.

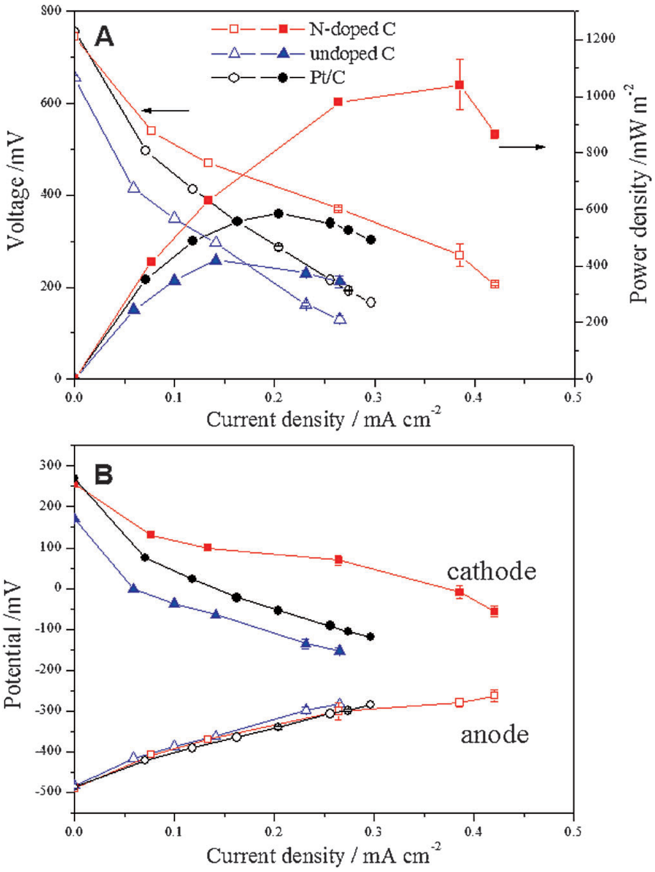

Figure 6. A) Polarization curves and power density curves, and B) electrode potentials curves of MFCs with different catalysts.

As mentioned previously, the performance of catalyst in real MFCs is quite different from that in RDE tests, due to the limiting diffusion of oxygen. Many factors, such as binder types, diffusion layers, and fabrication methods, affect the air-cathode performance in MFCs. In this study, the air-cathode with the N-doped carbon catalyst was fabricated by the rolling method with polytetrafluoroethylene (PTFE) binder, while the air-cathode with Pt/C was fabricated by the brushing method with Nafion binder considering the scarcity of Pt. The rolling method can be a promising method to produce low-cost, high-performance, and scalable air-cathodes.[27] In addition, the N-doped carbon catalyst was synthesized by heating cellulosic materials in NH_3 gas, with the advantage of low cost, renewability, and easy scale-up.

Conclusion

In this study, N-doped carbon was synthesized successfully by direct pyrolysis of cellulose paper under NH_3 and possesses a large surface area up to 1294.9 m^2 g^{-1}. The electrochemical tests revealed that the N-doped carbon exhibits high electrocatalytic activity toward the oxygen reduction reaction under neutral conditions. The application of N-doped carbon in air-cathode MFCs as a catalyst was examined. A maximum power density of 1041 ± 90 mW m^{-2} was achieved with N-doped carbon, higher than that achieved with commercial Pt/C (584 ± 10 mW m^{-2}). Both the nitrogen dopant and the large surface areas were considered to play a key role in the improved performance. The preparation of this N-doped carbon is cost-efficient and easily to be scaled-up, and therefore this catalyst is promising for application in MFCs.

Experimental Section

Materials

A 3 g sample of cellulose paper from tissue (Fuzhou, Fujian, China) was placed into a quartz tube furnace. The furnace was heated to 950 °C at a rate of 7.5 °C min^{-1} under NH_3 gas with a flow rate of 80 sccm, and maintained at the target temperature for 1 h.[21,22] After cooling down to room temperature, the black products were washed with abundant amounts of distilled water and dried in a vacuum drying oven overnight. For comparison, undoped carbon was also prepared by pyrolysis of cellulose paper under Ar gas under the same conditions.

Air-cathode fabrication

The air-cathode consisted of stainless-steel mesh (SSM) with a gas diffusion layer (GDL) on the air-facing side and a catalyst layer (CL) on the water-facing side.[23] The GDLs of all air-cathodes in this study were the same and made by rolling carbon black (ECP-600JD, Cuike Chemical Co. Ltd., Shanghai, China) and polytetrafluoroethylene (PTFE) emulsion (60 wt %, Dupont, USA) with the mass ratio of 3:7 into the SSM (mesh 40 × 40, type 304) to form a film (0.3 mm thickness), followed by sintering at 340 °C for 25 min. The carbon catalyst was ground evenly, and then mixed with PTFE binder with a mass ratio of 6:1. The mixture was roll-pressed to form a film of 0.2 mm thickness, and finally rolled onto the other side of the SSM to obtain the final air-cathode of 0.5 mm thickness. For comparison, the Pt-based air-cathode was prepared by brushing commercial Pt/C (20 wt %, Heson, China) onto the water-facing side of the SSM using 0.5 wt % Nafion solution as the binder, and the Pt loading was 0.5 mg cm^{-2}.

Characterization

The morphology of the N-doped carbon was observed by scanning electron microscopy (SEM, JEOL-6700F). Transmission electron microscope (TEM) images were obtained by a JEM-2010 instrument at an acceleration voltage of 200 kV. The elements on the surface of the N-doped carbon were identified by X-ray photoelectron spectroscopy (XPS, VG ESCALAB 250) with an Al Kα source at 1487 eV. XPSPEAK software was used for the peak fitting analysis. X-ray diffraction (XRD) was performed on a MiniFlex II diffractometer with Cu Kα radiation ($\lambda = 1.54$ Å).The specific surface areas were measured through N_2 adsorption at 77 K using ASAP 2010 and calculated by the Brunauer–Emmett–Teller (BET) equation. The PSD plots were acquired based on the density function theory model.

Electrochemical analysis

Electrochemical measurements were conducted at room temperature in a typical three-electrode system equipped with a platinum wire as the counter electrode and an Ag/Ag Cl electrode (saturated with KCl, 0.197 V vs. SHE) as the reference electrode. All potentials in this study were measured versus the Ag/AgCl reference electrode. To prepare the working electrode, 10 mg of each catalyst was first ultrasonically dispersed in 2 mL of 0.5 wt % Nafion solution. Then 4 μL of catalyst ink was pipetted onto the glassy carbon (GC) electrode (5 mm diameter), leading to a catalyst loading of 0.10 mg cm^{-2}. Finally the electrode was dried at room temperature before measurement.
In the electrochemical measurements a 50 mM phosphate-buffered solution (PBS, pH 7.0) solution containing 10.9233 g L^{-1} $Na_2HPO_4{\cdot}12H_2O$, 3.042 g L^{-1} $NaH_2PO_4{\cdot}2H_2O$, and 0.31 g L^{-1} NH_4Cl, 0.13 g L^{-1} KCl was used as the electrolyte. The cyclic voltammetry (CV) tests were run first in N_2-saturated solution, and then transferred to O_2-saturated solution which were then saturated with N_2, in the potential range from −0.6 V to 0.6 V at a scan rate of 0.05 V s^{-1}. The solutions were saturated with N_2 and O_2 by bubbling nitrogen and oxygen gas for 0.5 h, respectively. The liner sweep voltammetry (LSV) experiments using a rotating disk electrode (RDE) were performed with the O_2-saturated electrolyte at a scan rate of 0.005 V s^{-1}and at various rotation rates from 400 to 2400 rpm. O_2 was continuously streamed into the headspace of the electrolyte during the LSV test.

The electron transfer numbers (*n*) were calculated from the slopes of the Koutecky–Levich (K–L) plots using Equations (1) and (2).

$$J^{-1} = (J_L)^{-1} + (J_K)^{-1} = \left(B\omega^{1/2}\right)^{-1} + (J_K)^{-1} \quad (1)$$

$$B = 0.62nFC_0(D_0)^{2/3}v^{-1/6} \quad (2)$$

where *J* is the measured current; ω is the electrode rotation rate in rad s^{-1}; *F* is the Faraday constant (96 485 C mol^{-1}); C_0 is the concentration of O_2 (1.26×10^{-6} mol cm^{-3}); D_0 is the diffusion coefficient of O_2 (2.7×10^{-5} cm^2 s^{-1}), and ν is the kinematic viscosity of the electrolyte (0.01 cm^2 s^{-1}).

MFC construction and test

Single-chamber MFC reactors with total volume of 28 mL were constructed to measure the performances of the catalyst as previously described.[4] Three air-cathodes based on different catalysts including N-doped carbon, undoped carbon, commercial Pt were tested. Anodes were graphite fiber brushes (2.5 cm outer diameter and 2.5 cm long, Jilin Chemical Fiber Group Co., Ltd., China) which were soaked in acetone overnight and heated at 450 °C for 30 min before inoculation with microorganisms. MFC reactors were inoculated with wastewater from a local wastewater treatment plant and fed with a medium contained (per liter of 50 mM PBS): acetate (1 g), vitamin solution (5 mL), and trace element solution (12.5 mL). The medium was replaced when the voltage decreased below 50 mV. Unless otherwise noted, the external resistor was fixed at 1000 Ω. All MFCs were operated in duplicate at 30 °C.

Voltage output of MFC was monitored at 1 min intervals using a date acquisition system (LANHE, CT2001A, Wuhan, China). Polarization curves and power density curves were obtained by measuring the voltage generated at various external resistors from 1000 Ω to 30 Ω, with each resistor used for one complete cycle. The electrode potentials were measured by adding an Ag/AgCl reference electrode. Current density and power density were calculated as previously described and normalized to the projected area of the air-cathode (7 cm^2).[24]

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Keywords: cellulose · microbial fuel cells · N-doped carbon · oxygen reduction reaction

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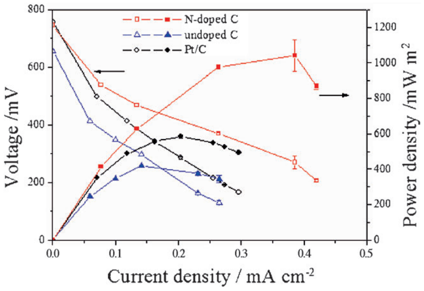
Heating cellulose under an ammonia atmosphere results in an N-doped carbon material with high surface area that exhibits high electrocatalytic activity for the oxygen reduction reaction in microbial fuel cells (MFCs). This N-doped carbon is an inexpensive, highly efficient catalyst, and can be a promising alternative to Pt/C in large-scale applications of MFCs.



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