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Biomass-derived N-doped carbon and its application in electrocatalysis

Xiao Zhao [a,b], Jianbing Zhu [a,b], Liang Liang [b], Chenyang Li [b], Changpeng Liu [b,**], Jianhui Liao [b], Wei Xing [a,*]

[a] State Key Laboratory of Electroanalytical Chemistry, Changchun Institute of Applied Chemistry, Chinese Academy of Sciences, Changchun, Jilin 130022, PR China

[b] Laboratory of Advanced Power Sources, Changchun Institute of Applied Chemistry, 5625 Renmin Street, Changchun 130022, PR China

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ABSTRACT

The economical, green and sustainable preparation of functional carbon materials is of interest for energy storage and conversion technology. Herein, a functional N-doped carbon material (NC) was obtained from green biomass and cheap carbon black. The as-prepared NC enables the formation of highly-dispersed Pt nanocatalyst. Interestingly, compared to a state-of-the-art commercial Pt/C catalyst, the as-prepared Pt/NC catalyst shows a superior electrocatalytic performance with a 1.4-fold improvement in mass activity and a 1.5-fold enhancement in stability for methanol electrooxidation reaction. Thus, the green and earth-abundant biomass materials exhibit the promise in designing and producing functional carbon materials for high-performance electrochemical devices.

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1. Introduction

Fuel cells are promising energy conversion devices. Until now, Pt-based metal have been the most effective electrocatalyst for fuel cell reactions. However, the scarcity and high cost of Pt metal limits its large-scale applications [1–7]. As a response, the functional carbon materials as electrocatalyst support display the potential in both boosting performance and reducing cost of electrocatalyst [3,5,8–17]. In particular, nitrogen-doped carbon (NC) has demonstrated the promise in facilitating the dispersion of metal nanoparticles (NPs) and generating the beneficial effects on electrochemical reactions [8,9,12]. However, the current synthesis procedures for NC are usually involved in toxic or expensive precursor such as pyridine [18], aniline [19] pyrrole [5] and porphyrins [8]. This impedes their large-scale and sustainable application to some extent.

To address this point, an intriguing direction is to use economical, green and sustainable biomass materials [20,21]. For instance, the carbon materials were obtained by the hydrothermal carbonization of biomass such as glucose, pine needles and sugar beets [20–22]. Chitosan (CS) as a natural biopolymer has many unique properties such as low-cost, biocompatibility and rich amines making it widely used in bio-fabrication technology [23,24]. However, few reports involve in using CS to synthesize NC although CS shows some attractive properties: (1) CS as biomass material has the economical, green and sustainable merits; (2) CS possesses much higher active nitrogen content than other biomass materials and should be an ideal nitrogen source [23]; (3) CS can uniformly cover substrates, which would promote the uniform incorporation of nitrogen into substrate.

In this report, NC was synthesized using green biomass CS and cheap carbon black. Firstly, the xerogel comprised of CS and carbon black was synthesized by a solvent-evaporation-induced gelation process. Secondly, the xerogel was then heat-treated at 900 °C to obtain NC. Interestingly, the Pt NPs supported onto the as-prepared NC showed a superior electrocatalytic performance for methanol electrooxidation reaction (MOR) compared to a commercial Pt/C and home-made Pt/C electrocatalysts. Thus, this report emphasizes not only the merit of chitosan as nitrogen precursor but also a superior electrocatalytic performance for Pt/NC catalyst.

2. Experimental

2.1. The synthesis of NC and Pt/NC electrocatalyst

Synthesis of NC: 1 g CS (the degree of deacetylation 80% and the molecular weight 50,000 g mol^{-1}) and 1 g Vulcan-XC 72 carbon

* Corresponding author at: 5625 Renmin Street, Changchun 130022, PR China. Tel.: +86 431 85262223; fax: +86 431 85685653.
** Corresponding author.
E-mail addresses: isliuchp@ciac.ac.cn (C. Liu), xingwei@ciac.jl.cn (W. Xing).

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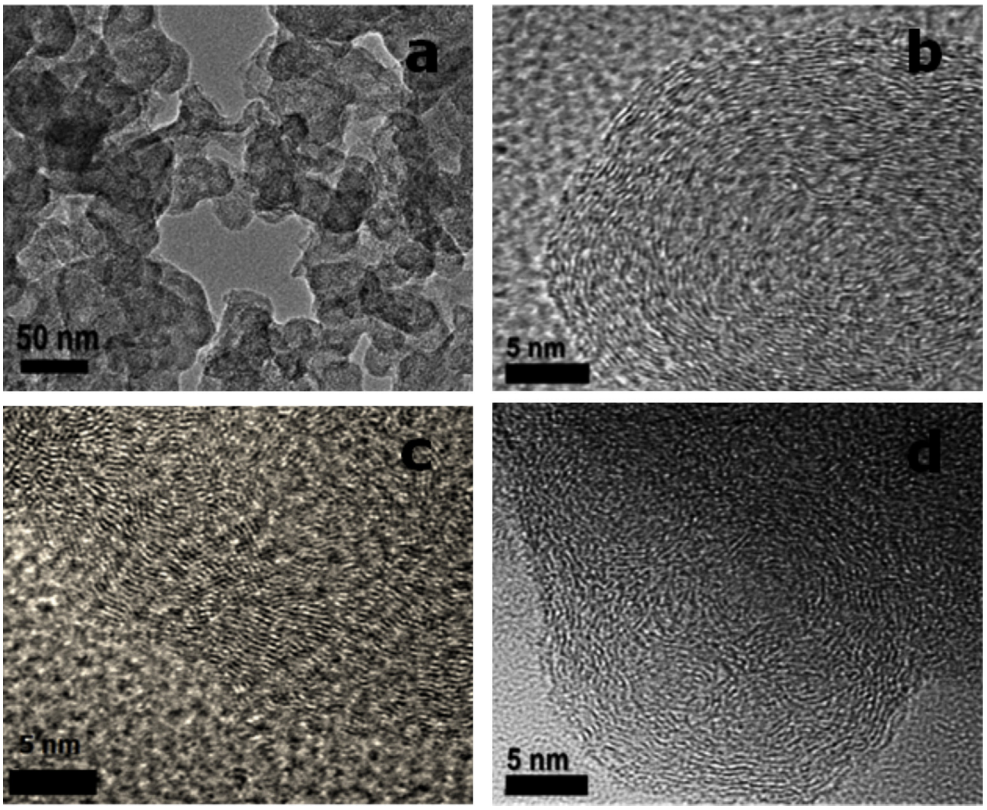

Fig. 1. The represntive TEM image of NC (a) and HRTEM images of NC paritcles (b–d).

black were added into 50 mL acetic acid. The resultant mixture was then extensively stirred and ultrasonically dispersed to form uniform ink. Then, a solvent evaporation-induced gelation process was conducted at 50 °C to obtain the xerogel. The as-prepared xerogel was then heat-treated at 900 °C for 1 h under N_2 atmosphere to obtain the NC.

Synthesis of Pt/NC and Pt/C electrocatalysts: the 20 wt% Pt electrocatalysts were synthesized by a microwave-assisted polyol process in ethylene glycol solution. Briefly, 20 mg NC were ultrasonically dispersed into 20 ml ethylene glycol to form a uniform ink. Then 357 μL H_2PtCl_6 solutions (14 mg mL^{-1}) were added and the pH value of the ink was adjusted to 11 using 0.1 M NaOH solution. Subsequently, the beaker was placed in the center of a microwave oven (800 W) with microwave heating for 90 s. The resulting solution was stirred for 8 h, filtered, washed, and dried in a vacuum oven to obtain Pt/NC. Pt/C was also synthesized using a similar procedure except Vulcan-XC 72 as catalyst supporter. A state-of-the-art commercial Pt/C-JM electrocatalyst (from Johnson Matthey Company, HiSPEC^{TM} 3000) was used as the benchmark electrocatalyst for comparison.

2.2. Physical characterizations

Transmission electron microscopy (TEM) and high resolution TEM (HRTEM) tests were conducted on a TECNAI G2 operating at 200 kV. X-ray photoelectron spectroscopy (XPS) measurements were carried out on a Kratos XSAM-800 spectrometer with an Mg Ka radiation source. X-ray diffraction (XRD) measurements were performed with a PW1700 diffractometer (Philips Co.) using a Cu Ka (λ = 1.5405 Å) radiation source. The obtained XRD patterns were treated with Jade 5.0 software.

2.3. Electrochemical characterizations

Electrochemical measurements were carried out with an EG&G mode 273 potentiostat/galvanostat and a conventional three electrode cell. The catalyst ink was prepared by ultrasonically dispersing the mixture containing 5 mg of catalyst, 950 μL of ethanol and 50 μL of a 5 wt% Nafion solution. Next, a 5 μL droplet of the well-dispersed ink was pipetted onto the pre-cleaned glassy carbon disk (diameter 3 mm) to produce a uniform thin film as working electrode. A Pt foil and a saturated calomel electrode (SCE) were used as the counter and the reference electrodes, respectively. All of the potentials are relative to the SCE electrode, unless otherwise noted. The working electrode was first electrochemically cleaned

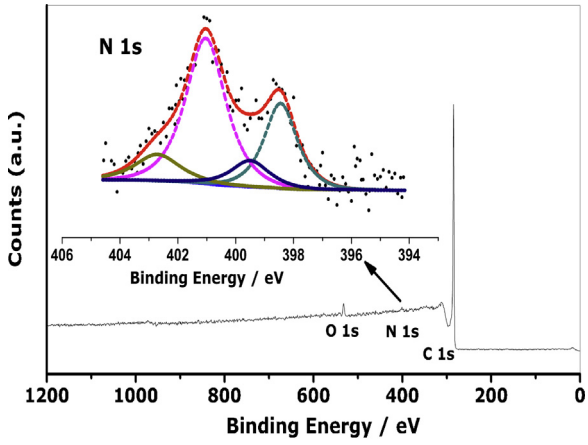

Fig. 2. XPS survey spectra of NC, the inset is high-resolution N 1s spectra.

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