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Preparation and characterization of nanoporous carbon-supported platinum as anode electrocatalyst for direct borohydride fuel cell

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ABSTRACT

The nanoporous carbon (NPC) is synthesized by carbonization of metal–organic framework-5 (MOF-5, $[\text{Zn}_4\text{O}(\text{bdc})_3]$, bdc = 1,4-benzenedicarboxylate) with furfuryl alcohol (FA) as carbon source and used as the carrier of the anode catalyst for the direct borohydride–hydrogen peroxide fuel cell (DBHFC). Then the NPC-supported Pt anode catalyst (Pt/NPC) is firstly prepared by a modified NaBH_4 reduction method. The obtained Pt/NPC catalyst is characterized by transmission electron microscopy (TEM), X-ray diffraction (XRD), energy dispersive spectrometry (EDS), cyclic voltammetry, chronopotentiometry, chronoamperometry and fuel cell test. The results show that the Pt/NPC is made up of the spherical Pt nanoparticles which disperse uniformly on the surface of the NPC with average size 2.38 nm, and exhibits 36.38% higher current density for directly borohydride oxidation than the Vulcan XC-72 carbon supported Pt (Pt/XC-72). Besides, the DBHFC using the Pt/NPC as anode electrocatalyst shows the maximum power density as high as 54.34 mW cm^{-2} at 25°C .

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1. Introduction

Fuel cells represent an environmentally friendly technology and are attracting considerable interest as a means of converting chemical energy into electrical energy with a high efficiency and low/zero-emission [1,2]. The direct borohydride fuel cell (DBFC) that uses borohydrides aqueous solution directly as an anode fuel is considered to be a promising candidate for portable power sources, because it has high theoretical open circuit voltage (OCV) of 1.64 V, high energy density of 9.3 Wh g^{-1} and fast anode kinetics at many metallic surfaces [3–7]. Additionally, borohydrides possess high

hydrogen contents, for example, NaBH_4 contains 10.6 wt.% hydrogen, which can eliminate hydrogen transportation and storage problems for DBFC [8,9]. Although DBFC has numerous advantages and is demanded from various industries, it has not been commercialized so far. The biggest obstacle is the high cost caused by the exclusive use of scarce precious metals (e.g., Pt, Au or Pd) as its primary catalysts [10,11]. Taking into account this situation, it is clear that increasing the utilization of catalysts through making the noble catalysts disperse as nanoparticles on electrically conducting carriers is one of the most effective pathways.

Up to 1990s carbon blacks are almost exclusively used as the catalyst carrier of fuel cell, but their properties are not

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completely satisfactory [12]. Recently, many works about the non-conventional carbon materials with high surface areas and good crystallinities to support the metallic catalysts have been reported, such as carbon nanotubes [13], graphitic carbon nanofibers [14], activated carbons [15] and mesostructured carbon materials [16]. Meanwhile, these studies have also revealed that the novel carbon carriers can not only maximize the availability surface area of the catalytic particles, but also facilitate electron transfer, leading to a better device performance [17]. Nonetheless, the corrosion of the carbon carrier is still problematic, which is recognized as the major cause for degrading the catalyst activity, and resulting in low durability of the fuel cell [18,19]. On this basis, many efforts have been made to develop the advanced materials with both special physical properties and good chemical stability as electrocatalyst carriers.

Nanostructured porous carbons are technologically important for various applications owing to their unique nature of nanoporous structures and high chemical/electrochemical durability [20–23]. In order to enhance these characteristics, lots of studies have been devoted mainly towards the synthesis of highly nanoporous carbons (NPCs). Currently, metal–organic frameworks (MOFs), a kind of emerging nanoporous materials consisting of transition-metal clusters as nodes and organic ligands as struts, have gained particular attention, because they can design the varied framework structures and diverse functions using their high degree of tunability of the both the organic and inorganic ligands [23–25]. The multitudinous structures, tunable pore sizes, high surface areas and large pore volumes of MOFs make them feasible as an alternative template for synthesis nanoporous carbon materials. Besides, considering their high carbon contents, the MOFs themselves could be used as excellent carbon precursors. Very recently, several MOFs, typically MOF-5 with a cubic three-dimensional extended porous structure, have demonstrated the promising precursors or templates to prepare NPCs through thermal conversion [26–28]. The resultant NPCs present exceptional porosity, high specific surface area ($3040 \text{ m}^2 \text{ g}^{-1}$), and show outstanding properties in wide applications, e.g. adsorbents, catalysis, sensing and electrode materials for supercapacitors [27–32]. As well known, these characteristics are essential for a good catalyst carrier in fuel cells. In addition, the NPCs possess lower manufacture cost and more simple technological process, which are in contrast with the currently hot carbon carriers, such as graphene, carbon nanotubes and carbon xerogel. To the best of our knowledge, the NPCs synthesized by MOFs templates as catalyst carriers are rarely reported in the studies of the fuel cells.

In our previous work, Vulcan XC-72 carbon supported Pt or Pt alloy catalysts as anode electrocatalysts for DBFC have been well studied [33,34]. Recently, we focused on the novel carbonaceous materials as electrocatalyst carriers in order to increase the utilization of the catalysts in DBFC [35,36]. In addition, our group has reported some work on the NPCs synthesized by MOF-5 precursor as electrode materials of supercapacitors [37]. Herein, the NPC was prepared using MOF-5 as template and FA as carbon source. In order to investigate the effect of the resultant NPC carrier on the anode performance of the Pt supported catalyst in DBFC, the Pt

nanoparticles were dispersed on the as-prepared NPC by a modified NaBH_4 reduction method at room temperature. The catalytic activity of borohydride oxidation on the Pt/NPC and the performance of single DBFC were investigated in detail.

2. Experimental

2.1. Synthesis of Pt/NPC electrocatalyst

The synthesis route of the Pt/NPC electrocatalyst is illustrated in Fig. 1. Briefly, the NPC was synthesized by carbonization of MOF-5 with furfuryl alcohol (FA) as carbon source [26,32]. The MOF-5 was prepared by solvothermal method with the optimal synthesis conditions according to the reported strategy [38]. The as-prepared MOF-5 was degassed at 200°C for 24 h to remove the solvent molecules. Then the FA was introduced into the pores of degassed MOF-5 via the incipient wetness technique. Subsequently, the FA/MOF-5 composite was transferred into a quartz boat and polymerized at 80°C for 24 h, then 150°C for 6 h in an Ar flow. Finally, the sample was carbonized at 900°C for 6 h. The resultant carbon material was denoted as NPC.

The Pt/NPC catalyst was prepared according to a modified NaBH_4 reduction method at room temperature. In brief, the required amounts of $\text{H}_2\text{PtCl}_6 \cdot 6\text{H}_2\text{O}$, NPC and polyvinyl pyrrolidone (PVP) were added to 100 mL deionized water under vigorous stirring for 30 min. Subsequently, 3 M NaOH solution was added to adjust the pH of the mixture to 10. To reduce Pt^{4+} , 1 mL of 1 M NaBH_4 was added dropwise. After an additional 24 h stirring, the resulting catalyst was filtered and washed with plenty of deionized water, then dried for 12 h at 80°C in vacuum. The Pt/XC-72 electrocatalyst was prepared by the same method for comparison. For each catalyst, the loading amount of metal is 20 wt.% of total catalyst weight.

2.2. Characterization of the Pt/NPC electrocatalyst

Elemental analysis of the as-prepared catalysts was performed by energy dispersive spectra (EDS) analysis, which was attached to the JEOL JSM-6360 scanning electron microscopy. TEM images were carried out using a JEOL JSM-2100F microscope operating at 200 kV. The TEM samples were prepared by placing several drops of dilute particle dispersion on carbon-coated copper TEM grids. Powder XRD analyses were performed on a D/Max-3C diffractometer with Cu $K\alpha$ radiation ($\lambda = 1.54056 \text{ \AA}$).

2.3. Electrochemical measurements

A conventional three-electrode system was used to perform the electrochemical tests. The system was composed of the Pt/NPC or Pt/XC-72 working electrode, an Ag/AgCl, KCl_{std} electrode and a Ni foam mesh with $3 \times 5 \text{ cm}^2$ as the reference and the counter electrode. Cyclic voltammetry, chronopotentiometry and chronoamperometry experiments were conducted with a VersaSTAT3 electrochemical workstation (Princeton, America).

The working electrode was prepared as follows: 10 mg Pt/NPC electrocatalyst was added to a mixture of 0.25 mL of

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