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Electrodeposition of palladium on carbon nanotubes modified nickel foam as an efficient electrocatalyst towards hydrogen peroxide reduction

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HIGHLIGHTS

- A novel Pd-CNT/Ni foam electrode is developed by a two-step preparation process.
- Reduction current is increased by 68.2% on Pd-CNT/Ni foam than that on Pd/Ni foam.
- The high-dispersed Pd nanoparticles are coated the surface of CNTs.

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ABSTRACT

In this article, a three-dimensional electrode (Pd-CNT/Ni foam) based on Pd nanoparticles and carbon nanotubes (CNTs) is successfully developed by a simple "dipping and drying" process and a potentiostatic deposition technology for H_2O_2 reduction in base medium. The composition and structure of Pd-CNT/Ni foam electrode are examined by X-ray diffractometer, scanning electron microscopy, energy-dispersive X-ray spectroscopy, transmission electron microscopy, respectively. The cyclic voltammetry (CV) and chronoamperometry (CA) techniques are applied to determine the electrochemical performance. The electrode exhibits a high catalytic activity for H_2O_2 electroreduction, and it outperforms Pd/Ni foam electrode without CNT coating. At the reduction potential of −0.8 V, the reduction currents on Pd-CNT/Ni foam electrode can reach 323 mA cm^{-2}, however, it is only 192 mA cm^{-2} on Pd/Ni foam electrode, which is increased by 68.2%. The impressive electrocatalytic performance is largely attributed to the superior open structure and high electronic conductivity, which allows the high utilization of Pd surfaces and makes the electrode have higher electrochemical activity. These findings may provide the opportunity on preparing binder-free carbon-supported electrode in the application of fuel cells.

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1. Introduction

Hydrogen peroxide as the high-efficiency oxidant is commonly used in the fuel cell engine replacing oxygen [1–12]. Currently, hydrogen peroxide has been investigated as a carbon-free energy carrier acted as the green fuel in direct peroxide–peroxide cell engine [13–18]. To obtain high performance of H_2O_2 reduction, the development of novel electrode with high catalytic activity and superior stability has received special attention for decades. Commonly, the electrode is consisted of catalyst and carrier. Literature has proved that noble metals exhibit high catalytic performance towards H_2O_2 reduction [6,10,11,18]. Pd is an exciting Pt-free alternative among various noble metal catalysts and many similar characteristics to Pt such as group of the periodic table, atomic size make it have very similar properties to Pt [19]. Besides, Pd is much cheaper and abundant than Pt (Pt is 1490 US$ per troy ounce and Pd is 626 US$ per troy ounce). Considering the expense of Pd and the improvement of catalytic activity, considerable effort has been concentrated on the structuring of Pd material on a nanometer scale. The group of Cao [15] prepared Pd nanothorns deposited on

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carbon fiber cloth as the efficient electrocatalyst towards H_2O_2 oxidation and reduction. The electrode displayed excellent catalytic performance for both H_2O_2 electrooxidation and electroreduction, and electrochemical results also reveal that the as-fabricated electrode is superior to commercial Pd/C powder made with the conventional binder-rich method. Wang et al. [20] have modified the glassy carbon electrode by Pd nanowires using Te nanowires as the template. Results have demonstrated that this ultra-thin Pd nanomaterial is an outstanding charge-transfer element for the ascorbic acid (AA) oxidation. Xia and co-workers [21] have also fabricated Pd concave nanocubes with high-index facets based on seeded growth method and proved its enhanced catalytic activities towards electrooxidation of formic acid.

Carbon nanotubes (CNTs) have drawn widely focus on account of the good conductivity, mechanical flexibility, and stable electrochemical behavior [22–26], which make them to be a very promising catalyst carrier for various catalytic reactions, especially for liquid-based reactions. The carbon nanotubes reported as the carrier for the first time can be traced back to 1994. Many metal catalysts including Pd, Pt, Ag, Fe or Ni supported on CNTs components can present desired catalytic performance [14,27–29]. Luo et al. [14] have manufactured Ni/CNT and Pd/CNT catalysts through an impregnation method. Among them, 5% Pd/CNT catalyst presents excellent performance for the hydrogenation of nitrocyclohexane to cyclohexanone oxime. Choi and Lee et al. [28] developed a by depositing $Pd_2(dba)_3$ on the surface of thiolated carbon nanotube to form a catalytic system for the hydrodehalogenation of aryl halides. The results have proved that CNTs have great influence on the catalytic properties of the CNT-supported metal catalysts towards hydrodehalogenation.

Based on the above considerations, CNT as the support for Pd loading could lead to high surface area and enhanced activity. The traditional preparation of electrode for fuel cells is commonly the carbon-supported and binder-enriched ones fabricated by the slurry-coating technique. The introduction of binders is generally dielectric, blocking the nanopores of catalysts, decreasing the mass transport of electrolyte, besides, the introduction of binders may also add the “dead volume” to the electrodes, increase the internal resistance, and thus weaken the catalytic performance. Moreover, the preparation processes of Pd/CNT composite materials are much too tedious and complicated. Therefore, the controllable preparation of CNT-based electrode with excellent performance and good mass transport is expected in the application of fuel cells.

Herein, a novel Pd-CNT/Ni foam electrode is developed by a two-step preparation process. Firstly, the surface of Ni foam substrate is covered with CNT through a simple “dipping and drying” process and Pd nanoparticles are loaded on the CNT by the following potentiostatic deposition technology. The whole preparation progress is simple and easy to operate, and also has no polymer binders to be introduced into. Ni foam has desirable 3D open-pore structure with high electronic conductivity, high surface area and strong absorbing ability, making it to be the more perfect support for catalyst loading. The obtained electrode is proved to have satisfactory activity and stability for H_2O_2 electroreduction in alkaline solution. We also compare the catalytic performance of Pd/Ni foam without CNT modification to that of Pd-CNT/Ni foam electrode.

2. Experimental

2.1. Reagents

Palladium chloride ($PdCl_2$) (>99.9%), nitric acid (HNO_3), potassium hydroxide (KOH) and hydrogen peroxide (H_2O_2) were obtained from Enterprise Group Chemicals Reagent Co., Ltd. China. Ni foam substrate (length * width * thickness = 10 * 10 * 1.1 mm, 110 PPI, 320 g m^{-2}) and multiwall carbon nanotubes (2 ~ 3 g) (MWNTs) were purchased from Changsha Lyrun Material Co., Ltd. China and Shenzhen Nanotech Port Co. Ltd., respectively. All chemicals are analytical grade and were used as-received without further purification. Ultrapure water (Millipore, 18 MΩ cm) was used throughout the study.

2.2. Preparation and characterization of Pd-CNT/Ni foam electrode

The Pd-CNT/Ni foam electrode was prepared by a two-step “dipping and drying” reaction and following electrodeposition process, which is also presented in Fig. 1. To prepare CNT/Ni foam substrate, nickel foam should be pretreated before use in the experiment according the literature [30]. Briefly, Ni foam substrate was degreased using acetone, etched using 6.0 mol L^{-1} HCl for 15 min, rinsed using water, soaked using 0.1 mmol L^{-1} $NiCl_2$ for 4 h, and then rinsed using water extensively. Multiwall carbon nanotubes (2 ~ 3 g) (MWNTs, Shenzhen Nanotech Port Co. Ltd.) were purified and shortened by ultrasonic treatment in concentrated H_2SO_4 (150 mL) and HNO_3 (50 mL). The solution was heated under stirring and the steam can be collected through condenser for 5 h. Then, the resulting solution was treated by vacuum filtering, washing, drying in a vacuum oven (100 °C, 4 h) and grinding. The cleaned Ni foam was immersed into a CNT solution, containing 40 mg multi-walled carbon nanotubes (MWNTs, Shenzhen Nanotech Port Co. Ltd.) and 50 mL ultrapure water (Milli-Q, 18 MΩcm). By sonicating the mixture at an ambient temperature, the CNT suspension can be obtained. After dipping for 30 s, Ni foam was gotten out from the CNT ink and dried at 373.15 K for 2 h in the vacuum oven. The dipping-drying cycle can be repeated several times to form the resulting CNT/Ni foam substrate. Then, Pd nanoparticles was electrodeposited on the CNT-coated Ni foam though the galvanostatic technique. In order to determine the optimal electrochemical parameters for the deposition of Pd on the surface of CNT/Ni foam by potentiostatic potential technique, the electrodeposition potential was kept at −0.2 V, the deposition time was set to be 7200 s, the solution concentration was varied

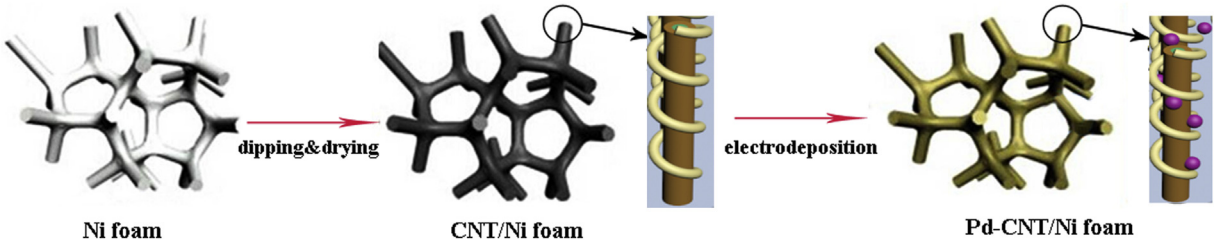

Fig. 1. Schematic diagram of the preparation process of Pd-CNT/Ni foam electrode.

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