



Combining Bimetallic-Alloy with Selenium Functionalized Carbon to Enhance Electrocatalytic Activity towards Glucose Oxidation



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ARTICLE INFO

Article history:

Received 8 April 2017

Received in revised form 13 May 2017

Accepted 13 May 2017

Available online 15 May 2017

Keywords:

Selenium functionalized carbon

Pd₃Sn alloy

Synergetic effect

Glucose oxidation reaction

ABSTRACT

To combine with the advantages of selenium functionalized carbon and bimetallic-alloy nanoparticles (NPs), a novel Se-C supported Pd₃Sn (Pd₃Sn/Se-C) electrocatalyst was successfully prepared and estimated towards glucose oxidation reaction (GOR) in alkaline medium. The results demonstrate that Pd₃Sn/Se-C electrocatalyst possesses superior catalytic performance towards GOR. Specifically, it presents larger electrochemically active surface area, higher electrocatalytic activity and better cycling stability compared to those of Pd₃Sn/C, Pd/Se-C and Pd/C. This satisfactory result mainly owes to the special properties of Se-functionalized carbon material, as well as the synergetic effect between Pd₃Sn NPs alloy and Se-C support. Additionally, the effects of the concentration of electrolyte and glucose as well as the temperature on the activity for GOR have been investigated. This work will shed new light on the investigation of excellent Se-functionalized-support based electrocatalysts in the future.

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1. Introduction

In the past several decades, a great number of attentions have gone into small organic molecules electrooxidation, such as methanol [1,2], ethanol [3,4], ethylene glycol [5,6], glycerol [7,8], formic acid [9,10], and so on, to develop high performance fuel cells. Noteworthy, among them, the glucose electrooxidation also is a promising orientation because of nontoxic, non-flammable, odourless and renewable properties [11,12], as well as its potential applied in direct glucose fuel cells (DGFCs) [11,13,14]. However, the kinetic of glucose electrooxidation is very sluggish on the state-of-art electrocatalysts. Therefore, for its practical applications, the most key point is the exploitation of efficient electrocatalysts with high activity and stability.

The selection of support is of importance to enhance the catalytic performance of electrocatalyst. Carbon black has been employed widely as an electrocatalyst support, because of its low cost, large surface areas and high electronic conductivity. However, carbon black is not an ideal support in the practical applications. Remarkably, functionalized carbon can tailor the surface electronic

structure by the introduction of heteroatoms into sp²-hybridized carbon frameworks. This is an effective way to immobilize NPs to produce efficient noble-metal electrocatalysts, as well as an encouraging material to improve catalytic performance [15,16]. In recent years, using N-doping [17,18], N, P-codoping [19], S-doping [20] and N, S-codoping [21] compounds to acquire functionalized carbon has been extensively investigated. While, as a chalcogenide group element in the periodic table, selenium (Se) is a semi-metal with six of valence electrons, and possesses similar chemical properties to S. For instance, there is a similar electronegativity between Se (2.55) and S (2.58) [22]. Except for the similarity with S, there are several special properties for Se element, including: i) Se possesses a superior conductivity on account of its stronger metallicity, in comparison with its counterparts in the same group, i.e., O and S, which promotes electron transport during the electrochemical reactions [23]; ii) Se displays a larger atomic size and higher polarizability than S; iii) Se shows comparable volumetric capacity, a high output voltage [24], as well as no shuttle effect [25]; iv) Se shows more metalline, when it bonded to metals, leading to superior chemical stability [26]. Nonetheless, there are still rare reports to employ Se-functionalized carbon as electrocatalyst support.

Base on the above, a novel Se-C support was introduced to electrocatalysts, expecting to promote catalytic performance for

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GOR. Simultaneously, among various nanoparticle candidates, the PdSn has been widely considered as a preeminent bimetallic electrocatalyst [5,27,31]. Thus, in this study, a Pd₃Sn/Se-C electrocatalyst was prepared by precipitation and heat treatment processes. As-prepared Se-C support is an effective material to fasten Pd₃Sn alloy NPs for producing satisfactory electrocatalysts, because it can tailor the surface electronic structure. Further, combining with the advantages of Se-C support and Pd₃Sn bimetallic-alloy NPs, the obtained Pd₃Sn/Se-C electrocatalyst would be favorable for GOR in alkaline medium.

2. Experimental

2.1. Preparation of electrocatalysts

2.1.1. Preparation of Se-C support

The Se-functionalized carbon support was prepared by following procedures: Firstly, triphenylphosphine (332.3 mg, PPh₃) was added to tetrahydrofuran (80 mL, C₄H₈O) and equably stirred until it was totally dissolved. Where triphenylphosphine was acted as a ligand to coordinate with Se in the reaction process, as well as can provide part of the carbon source in the process of calcination. Afterwards, Se powder (100.0 mg) was dropped into the above solution, and the obtained black slurry was placed for 0.5 h to acquire a clear solution. Secondly, to adsorb dissolved Se, a macroporous adsorption resin 101 (2.0 g, Shanghai Hualing Resin Co., Ltd.) was added in the above suspension and continuously stirred for 14 h. After that, the obtained ivory slurry was filtered, washed several times with ultrapure water, and then dried at 60 °C for 12 h. Thereafter, the resultant was heated at 800 °C in a tube furnace under N₂ atmosphere for 2 h. Finally, the Se-functionalized carbon support (Se-C, Se content: 6.08 wt.%) was obtained.

2.1.2. Preparation of Pd₃Sn/Se-C, Pd₃Sn/C, Pd/Se-C and Pd/C electrocatalysts

The Pd₃Sn/Se-C was prepared through a polyol reduction method. Briefly, palladium chloride (30.4 mg, PdCl₂) and stannic chloride pentahydrate (20.0 mg, SnCl₄·5H₂O) were dissolved into ethylene glycol (30 mL, EG) at sonication and stir conditions. Subsequently, sodium citrate (134.3 mg, C₆H₅Na₃O₇·2H₂O) was added and stirred for 1 h, and pH value of the system was adjusted to about ~8 by drop-wise addition of 5 wt.% KOH/EG solution. Then, the obtained Se-C support powder (100.0 mg) was dispersed into above mixture and stirred, sonicated for half an hour,

respectively. Thereafter, the obtained slurry was heated for 4 h at 180 °C in N₂ atmosphere. When cooling to room temperature, the sample was filtered, washed several times with ultrapure water, then dried in vacuum oven at 60 °C for 4 h. Finally, the Pd₃Sn/Se-C (14.58 wt.% Pd loading) electrocatalyst was obtained. For the sake of comparison, Pd₃Sn/C (14.58 wt.% Pd loading), Pd/Se-C (20.00 wt.% Pd loading) and Pd/C (20.00 wt.% Pd loading) electrocatalysts were prepared similarly.

2.2. Structural Characterizations

The morphology of as-prepared electrocatalysts was characterized by scanning electron microscopy (SEM, Carl Zeiss Ultra Plus, Germany), and transmission electron microscopy (TEM, FEI TECNAI G²TF20, America). X-ray diffraction (XRD) patterns were evaluated by diffractometer (Rigaku D/Max-2400, Japan) through a Cu K_α radiation source ($E = 40$ kV, $I = 150$ mA). X-ray photoelectron spectroscopy (XPS) was conducted on a spectrometer (Kratos Axis Ultra DLD, Japan) with the monochromatic Al K_α X-ray source ($h\nu = 1486.6$ eV). The degree of surface defects on Se-C and carbon was recorded on Raman spectroscopy (RFS 100/S, Germany).

2.3. Electrochemical Measurements

All electrochemical measurements were implemented by CHI 760E electrochemical workstation. A conventional three-electrode system is composed of working electrode (a modified glassy carbon electrode, 5 mm in diameter), reference electrode (Ag/AgCl electrode) and counter electrode (platinum wire electrode). The working electrode was prepared as following procedure: 5.0 mg electrocatalyst was ultrasonically dispersed in 1 mL Nafion/ethanol (0.25% Nafion) to acquire suspension ink. Subsequently, 8 μL ink was drop-cast on the surface of the disk glassy carbon electrode and dried. Before each measurement, all solutions were first deaerated with high purity Ar gas for about 25 min to guarantee gas saturated. All tests in this work were performed at room temperature, and all potentials were measured against Ag/AgCl.

3. Results and discussion

3.1. Physico-chemical characterization

Raman spectroscopy was used to investigate the structure and graphitic degree of carbon materials, as well as the doping

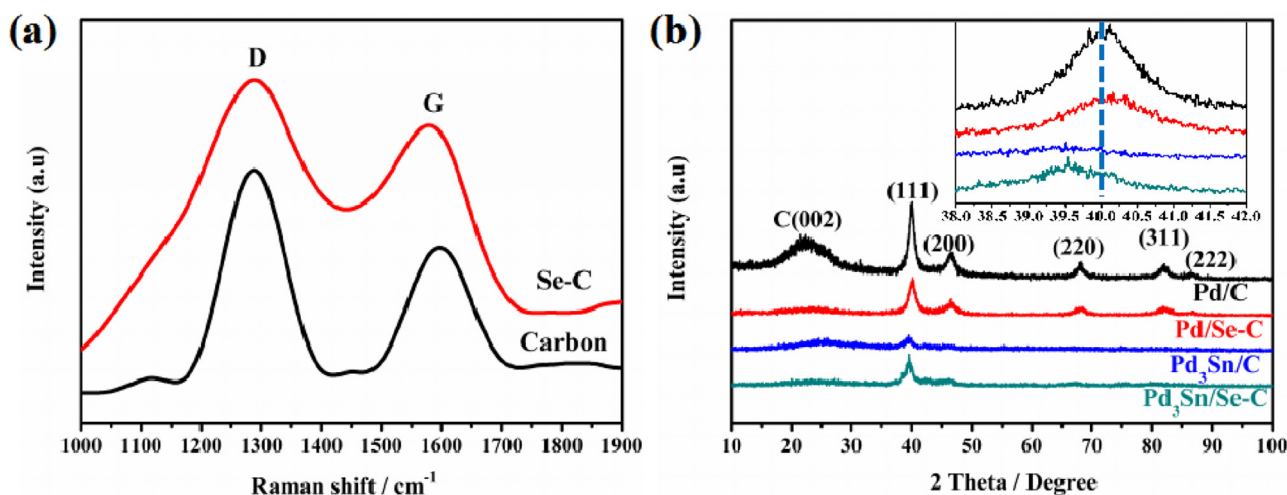


Fig. 1. (a) Raman spectra of Se-C and carbon; (b) XRD spectra of as-prepared Pd₃Sn/Se-C, Pd₃Sn/C, Pd/Se-C and Pd/C electrocatalysts; Inserted: Amplified Pd (111) reflection peaks of the corresponding electrocatalysts.

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