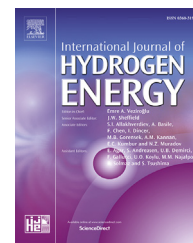


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Nickel phosphide decorated Pt nanocatalyst with enhanced electrocatalytic properties toward common small organic molecule oxidation and hydrogen evolution reaction: A strengthened composite supporting effect

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ABSTRACT

In this paper, well-dispersed Ni₂P-NiP₂-Pt/CNTs catalyst promoted by nickel-phosphorus compounds was readily synthesized by a two-step hydrothermal process. The as-synthesized Ni₂P-NiP₂-Pt/CNTs displayed improved electrocatalytic properties towards electro-oxidation of common small organic fuels such as methanol, ethanol and formic acid in contrast with Pt/CNTs and Pt/CNPs in acidic electrolytes. Meanwhile, the Ni₂P-NiP₂-Pt/CNTs catalyst also exhibited the excellent performance toward hydrogen evolution reaction with a more negative onset potential (−15 mV) and a smaller Tafel slope (29.8 mV dec^{−1}) when compared with Pt/CNTs (−29 mV, 30.6 mV dec^{−1}) and Pt/CNPs (−32 mV, 31.3 mV dec^{−1}) in 1.0 M H₂SO₄ solution. The catalytic activity enhancement possibly derives from the induced large specific surface area of carbon nanotubes as well as the strengthened synergistic effect between multiple supporting interactions.

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Introduction

In the 21st century, environmental pollution and energy crisis are both serious challenges to human society due to the rapidly increasing demand for energy consumption and depletion of large-scale fossil fuels [1]. Therefore, a large

number of researchers have shown great interest in searching for convenient, sustainable and environmentally friendly energy sources as well as their devices [2–9]. Among varieties of energy systems, proton exchange membrane fuel cells (PEMFCs) are considered to be a promising future power source and attracted tremendous attention in virtue of their high energy density, low operating temperature and portable

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electronic equipment [10–14]. Up to now, platinum (Pt) is the most extensively used catalyst for electro-oxidation of small organic molecule fuels such as methanol, ethanol and formic acid in PEMFCs. However, common pure Pt catalysts suffer from a lethal drawback of gradual deactivation in the electro-oxidation process due to being prone to poisoning by surface-adsorbed carbonaceous species such as CO and CHO [15–19]. In addition, the poor electrochemical activity and durability of the active carbon supported Pt catalyst is also reflected by a fast and significant loss of platinum electrochemical active surface area (EASA) over time during fuel cell operation [10,20]. On one hand, it is an effective approach for the introduction of one or more earth-abundant and non-precious constituents to improve the catalytic activity and durability as well as to abate the cost of Pt-based catalysts [18,21–23]. For example, in the initial research stage, people put forward the alloying of Pt with the other second metal to form Pt-M ($M = \text{Ru, Cu, Co, Ni, Ag, etc}$) alloy catalyst. These incorporated second-grade metals are capable of providing oxygen-containing species at low potentials (so-called bi-functional mechanism), triggering an obvious enhanced catalytic activities and anti-poisoning tolerance through accelerating the recycling of dissociate adsorption of reactant molecules together with oxidative removal of poisonous intermediates. On the other hand, researchers have found that supporting material with appropriate configuration and ideal physico-chemical properties is also a key factor to construct high-performance electrocatalyst [24,25]. During the past decades, different carbon-type supporting materials (i.e., active carbon, multi-walled carbon nanotubes (MWCNTs), graphite carbon nanofibers (GCNFs), graphene nanosheets, etc.) have been investigated frequently [17–19,26]. Among them, in virtue of the typical one-dimensional configuration, high-accessible surface area and good electric conductivity, carbon nanotubes (CNTs) are generally considered as attractive supporting material, which is beneficial to further improve the utilization of precious metals and trigger enhanced performance [20,21,27]. Except for the family of non-catalytic carbon materials, some co-catalytic materials were also implanted as supporting promoter to further increase the catalytic activity, such as metallic carbides (WC [28], Mo_2C [29], etc) and metallic oxides (CeO_2 [30,31], SnO_2 [32], etc). Recently, many attempts have already been taken to develop highly active composite catalysts by virtue of incorporation with earth-abundant transition metal sulfides (TMS, i.e., MoS_2 [33], WS_2 [34], etc) and phosphides (TMP, i.e., Ni_2P [13,35], Co_2P [36], Cu_3P [10], etc). It should also be mentioned that, relative to other metal compounds, transition metal phosphide (TMP) possesses a greater number of unsaturated surface atoms, which may substantially facilitate their higher catalytic activities. Meanwhile, noble Pt and earth-abundant TMP are both efficient electrocatalyst toward the half-side hydrogen evolution reaction (HER) in water electro-splitting process.

In the present paper, we explored the co-synergistic supporting effect of nickel-phosphorus compounds as well as different carbon-based support materials with Pt nanocatalysts. The catalytic performance of as-prepared nanocomposite catalyst was studied by using electro-catalytic oxidation reaction and hydrogen evolution reaction (HER) as two model systems. To the best of our knowledge, up to now,

there are few reports shedding light on the strengthened composite supporting effect in both electrocatalyst optimal design and electrocatalytic process. The hybrid use of promising carbon materials (MWCNTs) and transition metal compounds (Ni_2P , NiP_2) provides a promising way to construct high-performance catalyst composite supporting materials.

Experimental

Chemical reagents

Multi-walled carbon nanotubes (MWCNTs) and carbon nanoparticles (CNPs) were purchased from Shenzhen Nano Port Co. Ltd, China. The activation of both carbon support materials were carried out at 60 °C for 1 h in a concentrated HNO_3 (40%) prior to use, then washed repeatedly with ultra-purified water and evaporated to dryness. Potassium hexachloroplatinate (IV) (K_2PtCl_6) and nickel chloride hexahydrate ($\geq 98.0\%$, $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$) were purchased from Beijing Sinopharm Chemical Reagent Co., Ltd. Sodium hypophosphite monohydrate ($\geq 99.0\%$, $\text{NaH}_2\text{PO}_2 \cdot \text{H}_2\text{O}$) was purchased from Tianjin Kay Tong Chemical Reagent Co., Ltd. In all experiments, the ultra-pure water ($\sim 18.25 \text{ M}\Omega \text{ cm}$) produced through an UPT-II ultrapure water system was used to prepare the solutions. All of the chemicals used were of analytical grade and used as received without further purification.

Synthesis of catalysts

The synthesis of the catalyst in this paper is similar to that described in the previous paper [35]. In brief, the supported Ni_2P - NiP_2 -Pt/CNTs catalysts were prepared as follows: K_2PtCl_6 and $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ were selected as metal sources; $\text{NaH}_2\text{PO}_2 \cdot \text{H}_2\text{O}$ was selected as the source of phosphorus. First of all, the required stoichiometric amounts of Pt source salts were dissolved in ethylene glycol. Afterward, an appropriate amount of as-treated active carbon nanotubes was added in the mixture and ultrasonically stirred at room temperature ($\sim 25^\circ\text{C}$) for 1 h. Meanwhile, the pH of the suspension was adjusted to approx. 13 with 1.0 M NaOH solution. Then, the resulting suspension was placed in a reactor with a polytetrafluoroethylene liner for hydrothermal reaction. The autoclave was heated at 150 °C for 24 h in a hydrothermal autoclave, and then cooled to room temperature naturally. The suspension was finally filtered, washed and dried overnight at 60 °C in a vacuum oven to obtain the Pt/CNTs catalyst (ca. 25 wt% Pt). For the nickel phosphide promoter decoration, the initial as-synthesized Pt/CNTs sample with a mass of 20 mg, and the required stoichiometric nickel source and phosphorous salts were simultaneously dissolved in ultrapure water. The mixture was subjected to ultrasonic stirring until a mixed solution was obtained. The homogeneous resulting mixture was heated in a hydrothermal autoclave at 250 °C for 24 h, then cooled to room temperature naturally. The suspension was finally filtered, washed and dried overnight at 60 °C in a vacuum oven to obtain the Ni_2P - NiP_2 -Pt/CNTs catalyst (ca. 20 wt% Pt/10 wt% Ni-P). Using the similar hydrothermal strategy, the single Pt catalyst supported on carbon nanoparticles (denoted as Pt/CNPs (ca. 20 wt% Pt)) and the Pt-

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