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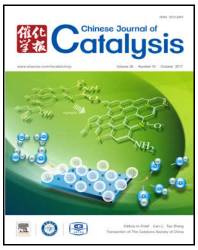

Article

Catalytic performance and synthesis of a Pt/graphene-TiO_2 catalyst using an environmentally friendly microwave-assisted solvothermal method

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ABSTRACT

A Pt/graphene-TiO_2 catalyst was prepared by a microwave-assisted solvothermal method and was characterized by X-ray diffraction, scanning electron microscopy, transmission electron microscopy, cyclic voltammetry, and linear sweep voltammetry. The cubic TiO_2 particles were approximately 60 nm in size and were distributed on the graphene sheets. The Pt nanoparticles were uniformly distributed between the TiO_2 particles and the graphene sheet. The catalyst exhibited a significant improvement in activity and stability towards the oxygen reduction reaction compared with Pt/C, which resulted from the high electronic conductivity of graphene and strong metal-support interactions.

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1. Introduction

A proton exchange membrane fuel cell (PEMFC) can directly convert hydrogen energy into electricity and is considered as a promising power source for portable electronic devices and electric vehicles owing to its high efficiency, high energy density, and low emission [1,2]. However, durability and high cost are the critical issues that have hindered its commercialization and widespread application.

The catalyst support plays a significant role in the activity and durability of the catalyst in a PEMFC. Therefore, the exploration of advanced catalyst supports has been a promising strategy for improving the performance of electrocatalysts [3–6]. Carbon black is a traditional support material for fuel-cell catalysts because of its high electrical and thermal conductivity, high surface area, and porosity [7,8]. However, carbon black is prone to corrosion under the operating conditions of fuel cells, which results in rapid degradation of the cell performance and durability. Therefore, extensive efforts have been made in the last ten years to develop new carbon-based materials and other alternative support materials for fuel-cell catalysts [9–15]. Transition metal oxides such as TiO_2 [16,17], SnO_2 [18], WO_3 [19], and CeO_2 [20,21] have been employed as alternatives to carbon supports to enhance the stability and durability of electrocatalysts. Among them, TiO_2 is more attractive owing to its inherent stability in an electrochemical environment and strong interactions with other metals [22–25]. However, its low electrical conductivity and small surface area have limited its widespread application in fuel cells. It is very necessary to design new catalyst support materials that can further enhance the electrical conductivity and surface area of TiO_2 when it is used as a catalyst support for fuel cells. Mean-

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while, graphene, a single layer of sp^2-bonded carbon atoms, has remarkable electrical conductivity and a high surface area [26]. It has been a potential material for fuel cells and other energy conversion and storage devices [27–29]. The high tendency of pristine graphene sheets to agglomerate is inevitable owing to the intense van der Waals forces [30], which lead to the aggregation of the fuel-cell catalysts and reduced performance.

Fortunately, the intercalation of TiO_2 with carbon materials has proven to be a good way to improve the electrochemical performance because of the synergistic effect between the materials [31–38]. For examples, Zhu *et al.* [31] successfully fabricated the nitrogen-doped graphitized carbon/TiO_2 supports by heat-treatment of polypyrrole/TiO_2 composites and the addition of nitrate as a graphitization catalyst, and then loading Pt nanoparticles by using a microwave-assisted polyol method in an ethylene glycol solution. The Pt/nitrogen-doped graphitized carbon/TiO_2 catalyst had high activity for the methanol oxidation reaction compared with the commercial Johnson Matthey catalyst. El-Deen *et al.* [35] prepared an rGO/TiO_2 composite with different TiO_2 loadings by using the alkaline hydrothermal method. Although these catalysts exhibited higher electrocatalytic performance than Pt/C, the preparation of these catalysts through the above-mentioned methods consumed a lot of time, which is not beneficial for mass production. Recently, some new synthesis methods have been developed. Jiang *et al.* [33] synthesized Pt/TiO_2-C catalysts with different particle sizes and TiO_2 content by a microwave-assisted polyol process. Zhao *et al.* [34] synthesized Pt/graphene-TiO_2 hybrid catalysts through a facile one-pot solvothermal method. Ye *et al.* [38] prepared a Pt/TiO_2/graphene composite under microwave irradiation. The catalysts showed higher catalytic activity and better tolerance to CO poisoning.

In this work, a more convenient, facile, and rapid method for preparing the catalyst Pt/graphene-TiO_2 is proposed. A graphene-TiO_2 composite support material and Pt catalyst were successfully prepared. X-ray diffraction (XRD), scanning electron microscopy (SEM), and transmission electron microscopy (TEM) were carried out to determine the crystalline structure and composition. Cyclic voltammetry (CV) and linear sweep voltammetry (LSV) techniques were also used to examine the electrochemical performance of the Pt/graphene-TiO_2 catalysts.

2. Experimental

2.1. Materials

All chemicals including graphite powder (100 μm, Qingdao Henglide Graphite Co., Ltd.), $KMnO_4$, KNO_3, H_2SO_4 ((95–97) wt%), H_2O_2 30 wt% (*m*/*m*), $K_2S_2O_8$, P_2O_5, tetrabutyl titanate (TBT), deionized (DI) water, acetic acid (HAc), 1-butyl-3-methylimidazolium tetrafluoroborate ([bmin]BF_4), and $H_2PtCl_6\cdot 6H_2O$ were purchased from Sinopharm, China. The Nafion solution (5 wt%) was purchased from Dupont. All chemicals were of analytical grade and used as received.

2.2. Synthesis of graphene oxide (GO)

The graphene oxide (GO) was synthesized using a modified Hummer's method [39]. Briefly, 5 g of graphite powder was pre-oxidized in a mixture of 4.2 g $K_2S_2O_8$, 4.2 g P_2O_5, and 60 mL H_2SO_4 at 80 °C for 4.5 h. The pre-oxidized sample was added into 120 mL H_2SO_4 at 0 °C, and then 2.5 g KNO_3 and 16 g $KMnO_4$ were slowly added. After the mixture was stirred for 2 h at 35 °C, 250 mL DI water was added, followed by the addition of 30 mL 30 wt% H_2O_2. The resultant mixture was washed with DI water, and then filtrated with HCl solution (1 mol/L) until the filtrate was almost neutral. Finally, the filter cake was dried at 60 °C in air.

2.3. Synthesis of cubic TiO_2

Anatase titanium dioxide [40] was prepared using a microwave-assisted ionothermal method. Specifically, 1 mL [bmin]BF_4, 2.5 mL H_2O, and 40 mL HAc were mixed well and then 1 mL TBT was added. The solution appeared light white and turned colorless and transparent after ultrasonic treatment for 30 min. The solution was then transferred into a microwave reactor (Explorer48, CEM) with a volume capacity of 50 mL and heated at 180 °C for 25 min. When it was cooled to room temperature, the white precipitate was isolated by centrifugation, rinsed several times with DI water and ethanol, and dried in a vacuum oven at 100 °C for 6 h.

2.4. Synthesis of Pt/graphene-TiO_2 catalyst

Pt/graphene-TiO_2 catalysts with different TiO_2 content were synthesized by using a microwave-assisted hydrothermal method. Typically, a calculated amount of GO and TiO_2 were dispersed into a mixture of ethylene glycol (EG) under ultrasonic treatment for 30 min, and kept stirring for 20 min. Then, the H_2PtCl_6-EG solution was added into the solution with agitation for 30 min. The pH of the solution was then adjusted to approximately 13 and the solution was transferred into a microwave reactor with a volume capacity of 50 mL and heated at 140 °C for 3 min. After cooling to room temperature, the pH of the solution was adjusted to 4. The mixture was washed repeatedly with ultrapure water. The obtained Pt/graphene-TiO_2 catalysts were dried at 80 °C in a vacuum oven for 5 h and subsequently treated in a reductive atmosphere at 150 °C for 2 h.

The TiO_2 content in the catalysts was relative to the total mass of the mixed support. For the sake of convenience, Pt/graphene-TiO_2 catalysts with 20 wt%, 40 wt%, and 60 wt% TiO_2 were denoted as Pt/graphene-TiO_2-20%, Pt/graphene-TiO_2-40%, and Pt/graphene-TiO_2-60%, respectively. The Pt metal loading of the hybrid catalysts was approximately 20 wt%. The metal loading of the samples was determined by burning off the carbon support and verifying the metal content by XRD. Samples of 50–100 mg were prepared to ensure good reproducibility and to minimize any errors.

2.5. Physical characterization

The XRD patterns of the as-prepared catalysts were obtained with a D/max-RB diffractometer (Japan) using a Cu K_α

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