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Comparison of two different catalyst preparation methods for graphene nanoplatelets supported platinum catalysts

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ABSTRACT

In this study, we compared two facile and effective approaches which are supercritical carbon dioxide (scCO₂) deposition and microwave irradiation methods to synthesize Pt nanoparticles uniformly dispersed on graphene nanoplatelets (G) having 750 m²/g surface area as the support material. The prepared catalysts were characterized by using XRD, TGA, TEM, CV and PEM fuel cell performance tests. Microwave irradiation (Pt/G1) and scCO₂ deposition (Pt/G2) methods resulted in the particle sizes of 3.1–3.4 nm and 1.5–1.6 nm, respectively. Higher electrochemically active surface area and PEM fuel cell performance were obtained for the Pt/G2 catalyst. An accelerated aging test, carbon corrosion, is also applied to the catalysts and Tafel slope losses were determined. Pt/G1 catalyst resulted in 50 mV Tafel slope loss whereas no significant loss was observed for Pt/G2 catalyst after carbon corrosion test.

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Introduction

Polymer electrolyte membrane fuel cells (PEMFCs) are considered as one of the most environmentally friendly energy converters and promising candidate for next generation power sources because of their low operation temperature, high energy efficiency, high power density and fast start up [1–7].

Carbon black (CB) is the most common commercial supporting material for PEMFC electrocatalysts which has some

advantages such as large surface area, high electrical conductivity and low cost. But long term stability of carbon black is considered as the major critical problem to be solved to improve the long term durability of PEMFC electrocatalysts [8]. Therefore, alternative carbon based supports (active carbon, carbon nanofiber, carbon nanotubes (CNT), etc.) and non-carbon based supports (conducting polymers, titania, indium oxide, alumina, silica, tungsten oxide nanostructures) are recommended in the literature [8–14].

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Graphene, a two-dimensional monolayer of carbon atoms, has seized great attention due to its unique structure and exceptional physical properties including excellent electronic conductivity, high specific surface area, high stability and large surface to volume ratio. These attracting properties make graphene a suitable alternative as a catalyst support for Pt nanoparticles [15–21] and bimetallic nanoparticles [20–25].

Support materials and synthesis methods are very important for better dispersion and narrow size distributions of Pt nanoparticles which significantly affect the electrocatalytic activity of the catalysts [26]. In recent years, various methods have been proposed to synthesize supported nanoparticles but choosing the most suitable method has a great importance in terms of the efficiency and structure of the catalysts.

Supercritical fluid deposition is a promising way to deposit metal nanoparticles onto the support materials. In this method, a metallic precursor is firstly dissolved in a supercritical fluid and then the dissolved precursor is exposed to porous support material. After adsorption of the precursor on the support, the metallic precursor is converted to its metal form by chemical or thermal reduction [27,28]. Previously, Vulcan supported Pt nanoparticles as the electrocatalysts for PEMFC were successfully developed by supercritical fluid deposition [26–28].

Microwave irradiation is also a heating method that provides rapid and uniform heating and it is gaining increasing attention at recent years among other catalyst preparation methods in terms of energy and synthesis time saving [29]. It has been demonstrated to be an effective way to prepare metallic nanoparticles, such as Pt, Ag, Au and Pd on support materials [3,30,31]. The required reaction temperature is reached by using microwave energy. By applying microwave energy to synthesis environment and the temperature of the reaction is high enough homogeneous reaction points are formed on the interface between the microwave-sensitive carbon support material and the precursor resulted in the formation of a homogeneous crystal nucleus [30]. The precursor can be converted its metallic form with the help of the microwave energy and also the reducing agent existing in the reaction environment. Previously, Sharma et al. reported the synthesis of Pt nanoparticle on graphene oxide (GO) using the microwave assisted polyol method that allowed partial reduction of GO and growth of Pt nanoparticles onto reduced GO. The TEM studies of Pt/RGO revealed the controlled size and distribution of Pt nanoparticles [32]. In another study, Shao et al. deposited Pt nanoparticles onto the PDDA-coated graphene nanoplatelets and CNT via microwave assisted polyol process and authors showed that the durability of Pt/GNP is significantly enhanced (2–3 times that of Pt/CNT and ETEK Pt/C) according to the oxygen reduction reaction (ORR) experiment [33]. Kundu et al. reported a rapid method to synthesize graphene supported Pt nanoparticles by the coreduction of GO and (H₂PtCl₆) using ethylene glycol as reducing agent under microwave irradiation [34]. Authors achieved 2–3 nm size of Pt nanoparticles onto the reduced graphene oxide, however fuel cell performance of resultant catalysts is not presented in the study [34].

In this study, graphene nanoplatelets is used as the catalyst support for the Pt nanoparticles. The synthesis of

supported Pt nanoparticles were achieved by using supercritical carbon dioxide deposition and microwave irradiation methods. The prepared catalysts were physicochemically and electrochemically characterized. Moreover, the resultant catalysts were evaluated in fuel cell conditions for their performances.

Experimental methods

Catalyst preparation and characterization

Graphene nanoplatelets (G), purchased from XG Sciences (xGnP® Grade C, surface area of 750 m²/g), was used as the catalyst support material. This product, described as aggregates of submicron platelets, has particle diameter less than 2 microns, particle thickness of a few nanometers, and a density of 0.2–0.4 g/mL [35]. Microwave irradiation method was employed to deposit highly active Pt nanoparticles on G support material. For this purpose, definite amount of support and aqueous solution of H₂PtCl₆ (Aldrich) were added in 50 mL ethylene glycol and then stirred for 30 min. The resulting mixture was put in an 800 W microwave oven. After microwave treatment for 1 min, the mixture was cooled down with cold water, then filtered, washed with acetone and deionized water and finally dried at 373 K for 12 h [36]. The resulting catalyst was called as Pt/G1.

scCO₂ deposition method was also employed to deposit highly active Pt nanoparticles on graphene nanoplatelets support material because of scCO₂ is emerging as an excellent medium to deposit metal nanoparticles into porous supports. 1,5-dimethyl platinum cyclooctadiene (Me₂PtCOD) was used as the Pt organometallic precursor. This precursor was dissolved in scCO₂ (at 333 K and 12 MPa) and then adsorbed on the graphene nanoplatelets support material. The precursor was decomposed to its metallic form with heat treatment at 673 K for 4 h in nitrogen atmosphere [28]. The resulting catalyst was called as Pt/G2.

The synthesized Pt/G catalysts were characterized by XRD, TGA, TEM in the as-synthesized condition with no subsequent cleaning or purification steps. XRD data were obtained by a Rigaku Miniflex X-ray diffractometer. The diffractometer with a CuK α ($\lambda = 1.5406 \text{ \AA}$) radiation source was operated in continuous scan mode at a scan rate of 0.6° min⁻¹ in the range of 10–90° (2 θ). Thermogravimetric analysis of Pt/G catalysts were studied in a Netzsch Thermal analyzer in air atmosphere from 298 to 1273 K in order to determine the Pt loading over graphene nanoplatelets. The structural changes and homogeneity of the Pt nanoparticles over the support material were characterized by transmission electron microscope (TEM) (JEOL JEM 2100F STEM).

Electrochemical characterization

Ex-situ electrochemical characterization of these catalysts was obtained by using cyclic voltammetry (CV) and PEM fuel cell performance test. CV measurements were carried out at 298 K in a standard three-electrode electrochemical cell system. A glassy carbon (GC) electrode was used as the working electrode, a platinum wire was used as the counter

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