



Research paper

Silver nanoparticles anchored reduced graphene oxide for enhanced electrocatalytic activity towards methanol oxidation

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ARTICLE INFO

Article history:

Received 13 October 2017

In final form 2 January 2018

Available online 3 January 2018

Keywords:

Reduced graphene oxide

Silver nanoparticles

Electrocatalytic activity

Methanol oxidation

ABSTRACT

In this report, silver nanoparticles (Ag NPs) anchored reduced graphene oxide (rGO) sheets (rGO/Ag) nanohybrid has been explored as anode material in direct methanol fuel cells (DMFCs). The synthesized rGO/Ag nanohybrid is characterized by XRD, XPS, FTIR spectroscopy and HRTEM techniques. Cyclic voltammograms demonstrate that the rGO/Ag nanohybrid exhibits higher electrocatalytic activity in comparison to rGO sheets for methanol oxidation reaction (MOR). This enhancement is attributed to the synergetic effect produced by the presence of more active sites provided by Ag NPs anchored on a conducting network of large surface area rGO sheets.

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1. Introduction

Over the past several decades, there has been a promising progress on electrochemical devices like fuel cells because of their low-cost, environment friendly, low operating temperature and high energy conversion efficiency [1–3]. Direct methanol fuel cells (DMFCs) are considered to be one of the most attractive portable power sources due to its simplicity, abundance and high energy density [3,4]. In order to enhance the transportation of charge through this electrochemical device, a necessary support system as an electrocatalyst is required. It should be electrically conductive and resistant to corrosion in extreme electrochemical environment. Nanostructured platinum (Pt) and its alloys are invariably used as most active anode materials for methanol oxidation reactions (MOR) as they exhibit high energy conversion efficiency due to their large surface area providing more active sites [5–9]. However, high cost, lack of resources and poisoning of Pt catalyst by residual species carbon monoxide (CO) with catalyst active sites in MOR limits the widespread commercialization of the DMFCs [10,11]. Therefore, development of an efficient catalyst for DMFCs has become a challengeable task for the scientific community.

Apart from Pt, nanoparticles of Au, Ag, Pd, Rh, etc. have been found to be suitable substitute as they are benefitted by low cost, abundance, excellent catalytic activity in oxidation reduction reactions (ORR) and higher stability during MOR against CO intermediate poisoning [11–13]. But, small size and high surface energy of

the metal nanoparticles may lead to aggregation. To overcome these problems, a strategy to anchor metal nanoparticles onto a carrier like nanocarbons possessing high thermal conductivity [14,15], has been explored to stabilize small particles by suppressing the growth of metal nanoparticles through heat sink effect [16,17].

The reduced graphene oxide (rGO) has attracted attention among nanocarbons due to its 2D structure with large surface area, high thermal conductivity, excellent mechanical strength and resistance to corrosion [18]. Metal nanoparticles anchored rGO have been explored for a variety of applications demanding high catalytic activity like, lithium-ion batteries [19], light-controlled conductive switching [20], bio-imaging [21], surface enhanced Raman scattering (SERS) [22,23], dye sensitized solar cells (DSSC) [24,25] and fuel cells [26,27].

The present paper highlights the optimum anchoring of Ag NPs on rGO sheets by varying the amount of Ag NPs and sinking their excess energy through rGO sheets with larger area to produce highly catalytic Pt-free network for the realization of cost-effective fuel cells.

2. Experimental

2.1. Synthesis of rGO and rGO/Ag nanohybrid

Graphite flakes (~100 mesh, 99.95%), potassium permanganate (KMnO₄, 99%), sodium nitrate (NaNO₃, 99%), sulfuric acid (H₂SO₄, 98%), hydrogen peroxide (H₂O₂, 30%) and hydrazine hydrate (N₂H₄·xH₂O, 82%) were procured from Sigma Aldrich and used as

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such without any further purification. GO was prepared by using Hummer's method [28]. Briefly, graphite flakes (4 g) were immersed in concentrated H_2SO_4 (100 mL) and then 2 g of NaNO_3 and 12 g of KMnO_4 were slowly added and stirred continuously for 1 h in an ice bath. After removing the ice bath, deionized (DI) water was added to above solution and maintained at 98°C , followed by addition of H_2O_2 to remove impurities. Further, the solution was centrifuged at 5000 rpm for 20 min followed by washing with water so as to adjust the pH at 7. The residue GO, was then heated in an oven at 80°C for 24 h to obtain it in the powder form. The obtained GO powder was dispersed into water and was sonicated for 15 min until it became clear. After this, the solution was transferred in a round bottom flask and heated in an oil bath for 24 h and $\text{N}_2\text{H}_4\cdot\text{xH}_2\text{O}$ was used as reducing agent for the reduction of GO to form rGO. In a separate beaker, 5.0 mL of silver nitrate (AgNO_3) and 0.2 g of cetyl tri-methyl ammonium bromide (CTAB) (1.0 mM) surfactant were added into a 20 mL aq. NaBH_4 (2 mM) and stirred for 1 h to form crystalline silver nanoparticles (Ag NPs) [29]. Further, these two mixtures were heated in an oil bath at 100°C under continuous magnetic stirring for homogeneous mixing for 20 h. Finally, the product rGO/Ag nanohybrid was collected by centrifugation and washed with distilled water to remove the unbounded Ag NPs and dried under a continuous air flow.

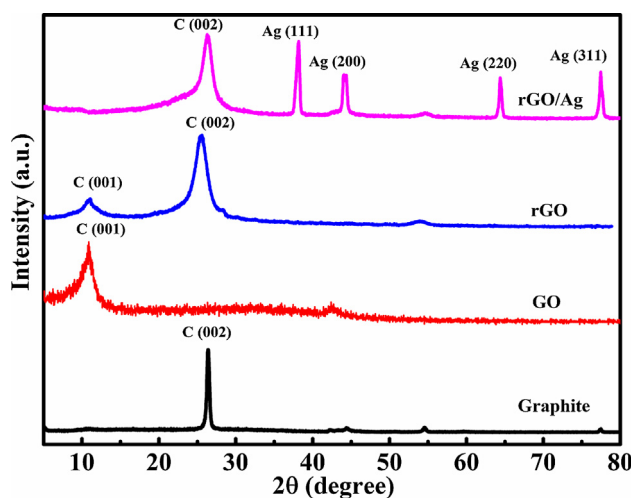


Fig. 1. XRD pattern of Graphite, GO, rGO and rGO/Ag nanohybrid.

2.2. Characterization

X-ray diffraction (XRD) study was performed on D8 FOCUS, Bruker Ettlingen using $\text{Cu K}\alpha$ line ($\lambda = 1.54 \text{ \AA}$) in the range of $5\text{--}80^\circ$. The attachment of Ag nanoparticles was confirmed by X-ray photoelectron spectroscopy (XPS) using Mac-2 electron analyzer with $\text{Mg K}\alpha$ as radiation sources. Fourier transform infrared spectroscopic (FTIR) analysis of samples was carried out on a C92035 Perkin Elmer Frontier. Raman spectroscopy was performed with Renishaw (In-Via Reflex) micro-Raman spectrometer using 514 nm argon laser as excitation source. Morphology analysis of the synthesized samples was achieved using JEOL JEM-2100 high resolution transmission electron microscope (HRTEM).

2.3. Electrochemical measurement

The electrochemical measurement was performed using Autolab potentiostat galvanostat (PGSTAT302). A standard three compartment electrochemical cell was used with glassy carbon electrode (GCE), Pt wire as the counter electrode and saturated calomel electrode (SCE) as the reference electrode. All the

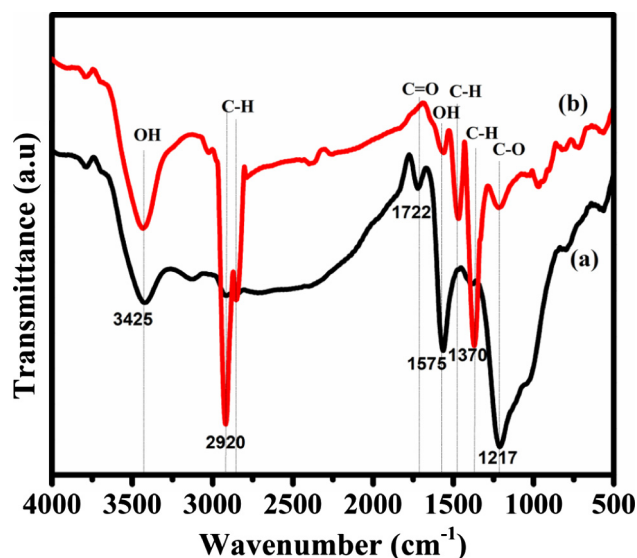


Fig. 3. FTIR spectra of (a) rGO and (b) rGO/Ag nanohybrid.

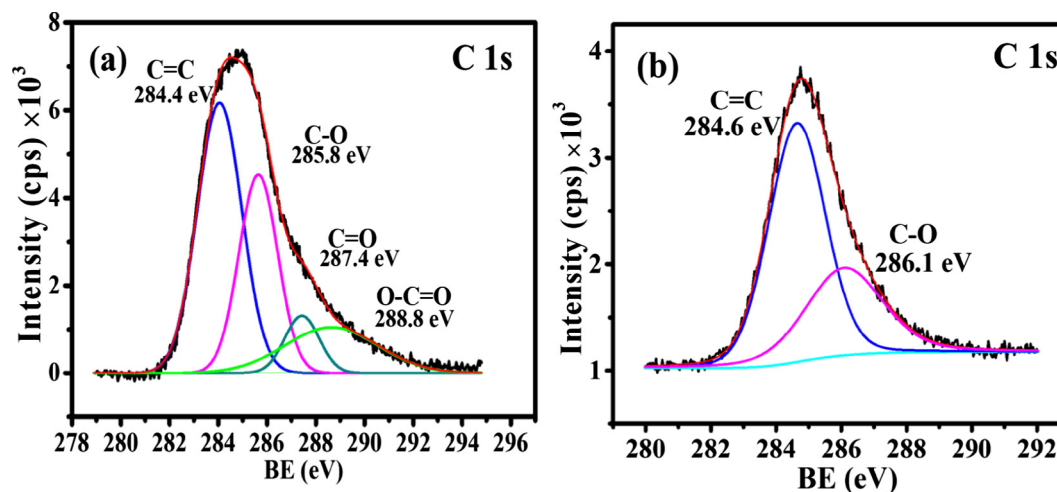


Fig. 2. XPS study of C 1s spectra of (a) rGO and (b) rGO/Ag nanohybrid.

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