Journal of Molecular Liquids 249 (2018) 61–65

Contents lists available at ScienceDirect

Journal of Molecular Liquids

journal homepage: www.elsevier.com/locate/molliq

Palladium oxide nanoparticles supported on reduced graphene oxide and gold doped: Preparation, characterization and electrochemical study of supercapacitor electrode

Vinod Kumar Gupta [a,*], Ali Fakhri [b,*], Shilpi Agarwal [a], Mahsa Naji [c]

[a] Department of Applied Chemistry, University of Johannesburg, Johannesburg, South Africa
[b] Young Researchers and Elites Club, Science and Research Branch, Islamic Azad University, Tehran, Iran
[c] Department of Materials Engineering, Karaj Branch, Islamic Azad University, Karaj, Iran

ARTICLE INFO

Article history:
Received 19 October 2017
Received in revised form 28 October 2017
Accepted 2 November 2017
Available online 04 November 2017

Keywords:
Supercapacitor
Nanocomposite
Graphene oxide
PdO nanoparticles
Au dopant

ABSTRACT

Electrochemical supercapacitors could be thought of as a major energy storage instrument for electrical devices. Nano metal oxides supercapacitors as materials with high electro-activity are attracting supreme consideration due to the best electrochemical efficiency. In this project, synthesis of pure and gold doped PdO-reduced graphene oxide nanocomposites by sonochemical and deposition-precipitation process was performed. The characterization of synthesized un-doped and Au doped PdO-RGO composites were performed by using X-ray photoelectron and Raman spectroscopy, transmission electronic microscopy, and X-ray diffraction method. The characterization results demonstrated PdO particles are embedded into the interlayer of RGO sheets. The electrochemical supercapacitive efficiency of the nanosamples was evaluated by different electrochemical analysis. Au-doped PdO-RGO nanocomposite shows enhanced specific capacitance of 253.0 Fg^{-1} at 5 mVs^{-1} and its high excellent effect was collated with PdO-RGO. The high cyclic stability and specific capacitance of Au doped PdO-RGO demonstrates to the doping of gold and good dispersion of PdO particles on RGO.

1. Introduction

Nowadays, semiconductor metal oxides, carbon materials, and conducting polymers were applied as basic pillars for electrodes. The carbonaceous substances indicate best physical and chemical properties while the polymers with conductivity properties present high pseudo capacitance, low cost, conductivity, best energy density [1–3]. The nanocomposite of these materials have been evaluated to found best electrode material with excellent cyclic stability [4,5].

Supercapacitors (SCs) are categories of energy storage devices [6]. SCs have been broadly applied in energy storage instruments which need a best rate power output [7,8]. Actually, SCs can be categorized: (i) pseudo-capacitors and (ii) electrical double-layer capacitors (EDLCs) [9,10].

In recent years, many studies have been carried out on different materials with high electro activity to modify the efficiency of SCs [11]. Therefore, the carbonaceous nanomaterials have been investigated for EDLCs in light of their best properties [12,13]. However, EDLCs have the best pore-size and surface area and the inability. Then, pseudocapacitors with transition metal oxides materials can present excellent specific capacitance and energy storage density [14–19].

Among carbonaceous nanomaterials, one material is a carbon-atoms flat-monolayer with lattice packing has incurred wide consideration due to its best conductivity, best chemical stability and highly specific surface area [20,21]. Moreover, graphene is also good react with to other nanomaterials such as metal oxide nanostructure as a best conductive material [21–23].

In this work, the new supercapacitive material such as undoped and Au doped PdO-RGO composite synthesized. The electrochemical measurement performed in aqueous Na_2SO_4, because the Na_2SO_4 electrolyte has high stability and low resistivity.

2. Material and methods

2.1. Reagents substances

Raw materials were procured from Sigma-Aldrich, Ltd.

2.2. Characteristic apparatus

The morphological information was investigated by using A transmission electron microscopy (JEM-2100F, 200 kV), and X-ray

* Corresponding authors.
E-mail addresses: vinodg@uj.ac.za, vinodfcy@gmail.com (V.K. Gupta), ali.fakhri88@yahoo.com (A. Fakhri).

https://doi.org/10.1016/j.molliq.2017.11.016
0167-7322/

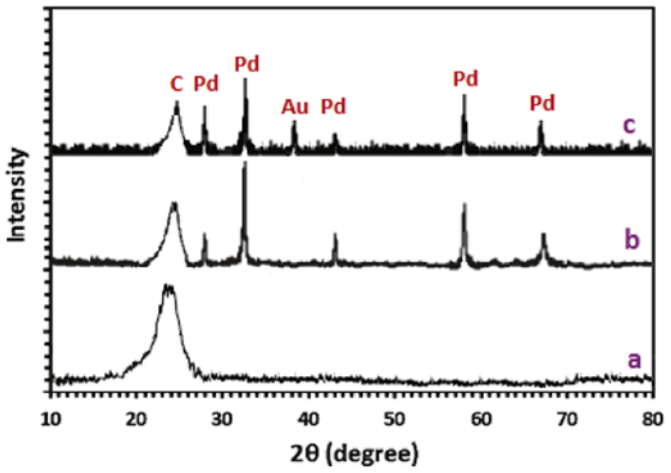

Fig. 1. XRD patterns of reduced graphene oxide (RGO) (a), PdO-RGO nanocomposites (b), and Au doped PdO-RGO nanocomposites (c).

diffractometer Philips X'Pert method. Raman spectroscopy analysis were evaluated with LabRAM HR spectrometer (Ar ion CW laser (514.5 nm) as the excitation source). The XPS spectra were investigated by a Kratos Axis Ultra DLD XPS system.

2.3. Preparation of pure and Au doped PdO-RGO nanocomposites

The modified Hummer's method was used to prepared Nanosheets of Graphene oxide (GO) [24,25]. RGO prepared to 100 mL of 50% N_2H_4 was added into the GO suspension, stirred and refluxed in a silicon oil bath at 100 °C for 12 h. Then, the prepared RGO was treated with DI water and ethanol and dried in 25 °C.

The PdO-RGO nanocomposites were produced using by sonochemical method. The 50 mg RGO were added separately, to $Pd(NO_3)_2 \cdot 2H_2O$ aqueous solutions dissolved in 1 mL 1-butyl-3-methylimidazolium tetrafluorobo-rate ([bmim]BF_4) (100 mL). The mixtures ultrasonically treated (480 W cm^{-2}, 24 kHz). The mixture temperature was constant at 50–55 °C.

Gold deposition of PdO-RGO nanocomposites was carried out using a simple deposition-precipitation method. Then, 1 g of the PdO-RGO nanocomposites was dispersed in 100 mL aqueous solution of $HAuCl_4.3H_2O$. The pH was adjusted to 9 using a 0.42 M urea solution (T: 80 °C). The reaction was then carried out for another 16 h (T: 80 °C). The precipitate was separated and washed with deionized water. The solid was calcined at 300 °C.

2.4. Electrochemical study

Electrochemical tests were performed with three electrodes. Working, reference and counter electrode we nickel foam with active material, saturated calomel electrode (SCE), platinum sheet, respectively. The disodium sulfate with concentration 1 M was chosen as electrolyte. Cyclic voltammograms tests and other test were performed using by Princeton P4000 electrochemical working station (scan rates 5–80mVs^{-1}; potential range–0.2-0.8 V). The potentiostatic signal amplitude and frequency range for EIS was 5 mV and 0.01 Hz–10 kHz, respectively. The galvanostatic charge-discharge was tested with different voltage under a range of current densities, from 0.2 to 4.0 Ag^{-1}.

3. Results and discussion

3.1. Characterization of the pure and Au doped PdO-RGO nanocomposites

As displayed by the XRD pattern of samples (Fig. 1), the representative diffraction peaks of Au doped PdO-RGO nanocomposites are located at 28.5°, 32.6°, 36.7°, 40.23°, 57.3°, and 67.4°, which are well assigned to the phase planes of the tetragonal PdO (JCPDS Card No. 75-584) and the pure Au (JCPDS-04-0784). Fig. 1a, GO showed a sharp diffraction peak at $2\theta = 10.8°$, thereby suggesting the complete removal of graphite [26,32].

The representative TEM images of pure and Au doped PdO-RGO nanocomposites are shown in Fig. 2. Fig. 2A shows that a large amount of PdO particles are embedded in to the interlayer of RGO sheets. It can be seen, the sample of doped Au on nanocomposites has agglomeration status of particles.

The Raman spectra of pure and Au doped PdO-RGO nanocomposites are shown in Fig. 3. As can be seen, the ~1320 cm^{-1} and ~1580 cm^{-1} bands for *D* band and *G* band are the defects and disorder mode in the RGO, and the sp^2-bonded vibration from carbon atoms (hexagonal lattice of graphite). The *D′* band (1600 cm^{-1}) was showed the disorder induced effect. Then, peak intensity ratio (I_D/I_G) indicates concerning the disorder information of the graphitic layers. The I_D/I_G value of the pure and Au doped PdO-RGO nanocomposites were 1.80 and 1.51, respectively. Moreover, the intensity ratio value of GO was 0.73 that indicates the progress of GO reduction was fine [27].

X-ray photoelectron analysis was used to investigation of data related to electronic state of the surface region. Fig. 4 demonstrates clear peaks of Au 4f, C 1 s, O 1 s, Pd 3p, and Pd 3d are perceived for Au doped PdO-RGO nanocomposites spectrum. The high resolution XPS spectrum (Fig. 4b) demonstrates Pd $3d_{5/2}$ and Pd $3d_{3/2}$ with binding energy at 337.5 and 342.9 eV, respectively.

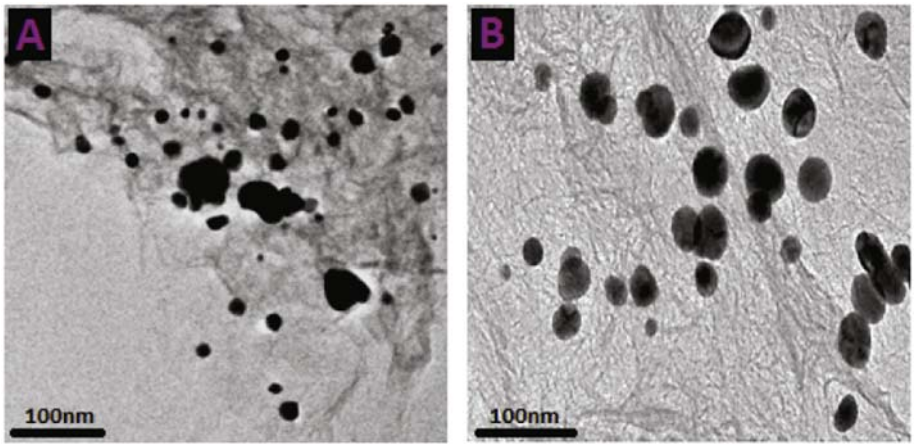

Fig. 2. TEM images of PdO-RGO nanocomposites (A), and Au doped PdO-RGO nanocomposites (B).