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Pulse electrosynthesis of novel wormlike gadolinium oxide nanostructure and its nanocomposite with conjugated electroactive polymer as a hybrid and high efficient electrode material for energy storage device

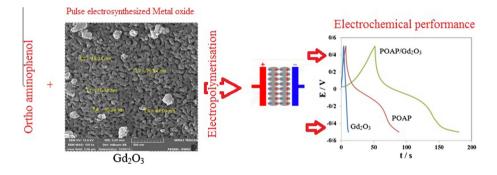
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ABSTRACT

An effective approach for increasing the life cycle of pure p-type conductive polymers is combining conventional conductive polymers and nanomaterials to fabricate hybrid electrodes. In this paper, Gadolinium oxide (Gd_2O_3) has first been synthesized using pulse electrochemical approach. Hybrid POAP/Gd₂O₃ films have then been fabricated by POAP electropolymerization in the presence of Gd_2O_3 nanoparticles as active electrodes for electrochemical supercapacitors. Surface and electrochemical analyses have been used for characterization of Gd_2O_3 and POAP/Gd₂O₃ composite films. Different electrochemical including galvanostatic charge discharge experiments, cyclic voltammetry and electrochemical impedance spectroscopy have been applied to study the system performance. Specific capacitance, specific energy and specific power of the composite film are calculated 300 F·g⁻¹, 41.66 W h·kg⁻¹ and 833.22 W·kg⁻¹ respectively. This work introduces new nanocomposite materials for electrochemical redox capacitors with such advantages as the ease of synthesis, high active surface area and stability in an aqueous electrolyte.

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1. Introduction

Electrochemical supercapacitors are the charge-storage devices having high power density and long cyclic life. The main materials





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that have been studied for the supercapacitor electrode are (i) carbons, (ii) metal oxides and (iii) polymers [1–4]. The conductive polymers are considered the most promising material in the supercapacitors. The properties of conducting polymers can be greatly enhanced by forming composites between the conducting polymer and other materials such as carbon (including carbon nanotubes) [5], inorganic oxides and hydroxides [6,7], graphene and other metal compounds [8–14]. The composite materials have improved conductivity and better cyclability, mechanical stability, specific capacitance and processability [8–13]. Some of the most famous CPs includes polyaniline, polypyrrole, polythiophene and their derivatives [15-18]. Polyaniline (PANI) is one of the most important conducting polymers because of its unique electrical, optical, and optoelectrical properties, as well as its ease of preparation and its excellent environment stability [16]. Aminophenols are interesting members of the class of substituted anilines. The hydroxyl group in the phenyl ring can be oxidized to quinine and quinine can be reduced again. POAP gives a surface film of interesting electrochemical and electrochromic properties when it is electropolymerized in acidic solution. This film is electroactive in aqueous and non-aqueous solutions containing protons but no response is observed at pH-value higher than pH 7. The variety of results for conductivity of the POAP film reported in the literature [13,19,20] show that the electrochemical response of POAP is strongly influenced by the experimental procedure used to produce the polymer film, dopant anions and the purity starting monomer. Electrochemical activity of conductive polymer in dedoped state could be a serious problem because of the typical volumetric shrinkage during ejection of counter ions (doped ions). Furthermore, the Conductance of conductive polymer was very low at dedoped state, which would result in high ohmic polarization of supercapacitor, which would reduce the reversibility and stability of supercapacitor. In order to solve the problem, a synergistic effect of conductive polymer-inorganic nanocomposite has been presumed upon to be applied in electrode materials for supercapacitor [21–27]. Recently many transition metal oxides have been investigated for pseudocapacitive charge storage in aqueous electrolytes. The behavior of these materials depends upon the structure of the material, hydration properties, and the electrolyte [28–30].

Our goals in this paper were increasing the capacitance of POAP electrode by using Gd_2O_3 nanostructures to form a composite electrode and moreover increase the cycle ability of the electrode. The complementary properties of both components generate a synergistic effect to enhance the electrochemical performance. It is an aim of the present work to investigate POAP/ Gd_2O_3 composite as supercapacitor electrode materials with several analytical tools such as field emission scanning electron microscopy, cyclic voltammetry, galvanostatic charge–discharge, and electrochemical impedance technique. The electrochemical tests demonstrate that the POAP/ Gd_2O_3 composite is a promising material in the application of energy storage electrode.

2. Experimental

2.1. Reagent and materials

All the chemical materials used in this work, obtained from Merck Chemical Co., were of analytical grade and used without further purification. Double distilled water was used throughout the experiments.

2.2. Characterization

All electrochemical experiments were carried out by a Potentiostat/galvanostat (Ivium V21508, Vertex). A conventional three electrode cell with an Ag/AgCl reference electrode (Argental, 3 M KCl) was used in order to carry out the electropolymerization of the POAP. A platinum wire and a carbon paste electrode was used as the counter and working electrodes respectively. Morphological investigations of the compounds were carried out by using FESEM analysis (Mira 3-XMU). The observed morphologies were microanalysed by energy dispersion of X-rays (EDX) at a working distance of 15 mm and 15 kV electron energy. Samples have been coated by Au before any analysis. X-ray diffraction (XRD) patterns were obtained from an X-ray diffractometer (PANalytical X'Pert-Pro) with a Cu K α monochromatized radiation source. The transmission electron microscopy (TEM) was performed using a CEM 902A ZEISS transmission electron microscope, with an accelerating voltage of 80 kV.

2.3. Preparation of Gd_2O_3 wormlike nanostructure via cathodic pulse electrochemical deposition

According to previous reported works, chemical route such as sol gel and thermal decomposition used to preparation of Gd₂O₃. In this work green route (electrosynthesis) has been used to synthesis of Gd₂O₃ nanostructure. The electrochemical depositions were performed in the reverse pulse voltage mode in a conventional three-electrode cell. Stainless steel and graphite were used as working and counter electrodes respectively. Aqueous bath electrolyte solutions containing $Gd(NO_3)_3 6H_2O$ (0.005 M) and Hexamine as a additive agent (0.005 M) were used in the electrodeposition process. Reverse pulse electroplating was preformed via square wave potential by applying $I_P = 10 \text{ mA} \cdot \text{cm}^{-2}$ and duty cycle 50 ($T_r = T_f = 10 \text{ ms}$) at room temperature. Furthermore cathode potential respect to reference electrode was adjusted about -0.6 V and continuously was traced by multiscope in the mentioned value. Parameters of reverse pulse deposition are defined as follows [13]:

 $\gamma(\text{Duty cycle}) = (T_{\text{forward}}) / (T_{\text{forward}} + T_{\text{reverse}})$

 $I_{average} = I_{peak} \times \gamma$

After electrodeposition the steel electrodes were rinsed several times with deionized water and then obtained powders were dried at RT for 48 h. The deposited gel was scraped by a non-metallic scraper, and subjected to heat-treatment. Heat-treatment of gels was conducted at 780 °C in dry air atmosphere for 3 h through electrical furnace. The furnace was programmed at a rate of 10 °C/min.

2.4. Synthesis of POAP and POAP/Gd₂O₃ nanocomposite

POAP/Gd₂O₃ composites were prepared by electropolymerization in a solution containing 0.01 M monomer, 0.5 M HClO₄, 0.1 M LiCO₄, 5.0×10^{-3} M sodium dodecyl sulfate (SDS) and 1% of Gd₂O₃ on the surface of the modified working electrode. POAP electrode was synthesized in same procedure without Gd₂O₃. Electropolymerizations were conducted by 40 consecutive cycles at the sweep rate of 50 mV·s⁻¹ in the potentials between -0.2and 0.9 V. The mass of polymer and composite films was approximated assuming a current efficiency for the electropolymerization process of 100%, using Faraday's law.

3. Results and discussion

SEM and EDX have been applied for analysis of the electrosynthesized compound. EDX spectrum and SEM image of Gd_2O_3 have been shown in Figs. 1 and 2 respectively. As evident from SEM Download English Version:

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