



Preparation of graphene/vanadium oxide nanocomposite monolith and its electrochemical performance



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ABSTRACT

Graphene/vanadium oxide nanocomposite (G/V₂O₅) monolith is prepared via a simple hydrothermal process. Owing to the intimate contact between the V₂O₅ nanobelts and graphene nanosheets in the monolith, the nanocomposite shows excellent electric conductivity, and therefore makes the electrode–electrolyte contact better and Li⁺ diffusion faster. A high specific capacitance of 163 Fg^{−1} has been achieved for G/V₂O₅ electrode in 0.5 mol L^{−1} K₂SO₄ solution. The G/V₂O₅ nanocomposite exhibits excellent cyclic performance with nearly 80% capacity retention at a current density of 5 A g^{−1} in a testing range of 1000 cycles. Moreover, G/V₂O₅ nanocomposite exhibits excellent discharge properties and cycle stability as an anode material for lithium ion batteries. The initial capacity is 1100 mAh g^{−1} and the reversible capacity of 530 mAh g^{−1} is maintained after 100 cycles at a current density of 50 mA g^{−1}.

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1. Introduction

With the depletion of fossil fuels, there is an urgent need for efficient, clean, and sustainable source of energy, as well as new technology associated with energy conversion and storage [1]. It is demonstrated that some of the most effective and practical technologies for electrochemical energy conversion and storage are fuel cells, lithium ion batteries (LIBs), and supercapacitors also called electrochemical capacitors [2–4]. The main challenges for supercapacitors and LIBs are achieving high capacity, excellent cycle performance and rate ability. To overcome the obstacles mentioned above, one of the most intensive approaches is the developing of new materials for electrodes, and the other is hybridizing the electrode materials by adding electrochemically active materials into carbon-based electrode materials for supercapacitors and LIBs [3,5].

Many transition metal oxides, such as MnO₂, Fe₃O₄, SnO₂, V₂O₅ and so on, have been employed as electrode materials for supercapacitors and LIBs [6–9]. Among the transition metal oxides containing metal atoms capable of various valence states, V₂O₅ is one of the most promising electrode materials for supercapacitors and LIBs due to its high energy density, ease of synthesis, low cost and abundant resources [10–13]. V₂O₅-based electrode materials, however, suffer from poor cycle stability and low rate capability

due to the poor electrical conductivity (10^{−2}–10^{−3} S cm^{−1}), slow Li ion diffusion rates (10^{−13} cm² s^{−1}) and the large specific volume changes upon cycling [14]. It is reported that nanostructured V₂O₅ have shown improved Li ion diffusion rates. However, the rate and cycle performances of the V₂O₅ are unsatisfied due to the low electronic conductivity [15]. Therefore, fabricating V₂O₅/carbon materials, have been developed to improve the structure integrity and electrical conductivity of V₂O₅-based electrode materials [16].

Up to now, carbon materials such as graphene (G), carbon black, and carbon nanotubes (CNT) with high electrical conductivity have been widely chosen to improve the conductivity and stability of V₂O₅ for supercapacitors and LIBs [17,18]. Among these reported V₂O₅/carbon materials, some materials (such as V₂O₅·xH₂O/CNT film [19], intertwined CNT/V₂O₅ nanowire nanocomposites [20], hierarchical V₂O₅/CNT composites [21] and so on) show excellent performance for electrochemical energy storage due to the unique intertwined structure. Compared with other carbon materials, G is emerging as one of the most appealing materials because of its unique superior electrical conductivity, excellent mechanical flexibility, and high thermal and chemical stabilities [22]. G facilitates electron transport and electrolyte ion/Li⁺ diffusion of anchored host materials, and thus enhances electrochemical capacitive performance and lithium storage. Some V₂O₅/G composites for supercapacitors and LIBs have been prepared. It is known that the monolith form electrode materials have intriguing advantages over powdered form such as high electrical and thermal conductivities, high density storage, no intergranular barriers, more well-accessible macroporous channels and easy

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handling when used for supercapacitors and LIBs [23,24]. However, there are rare reports on the G/V₂O₅ nanocomposites monolith with novel interpenetrated structure for supercapacitors and LIBs.

In the present work, G/V₂O₅ nanocomposite monolith with G amount of 25% was prepared in a mixture of ammonium vanadate, acetic acid and graphite oxide (GO) by one-step hydrothermal technology. The hydrothermal treatment made the reduction of GO to G and the formation of V₂O₅ nanobelts. Meanwhile, the G sheets decorated with V₂O₅ nanobelts simultaneously self-assembled into monolith with interpenetrated structure. Owing to this novel structure, the G/V₂O₅ nanocomposite showed higher specific capacitances than those of G and V₂O₅ electrodes for supercapacitors and LIBs. Moreover, G/V₂O₅ nanocomposite showed better cycle performance than that of V₂O₅ electrode.

2. Experimental

2.1. Material preparation

GO was fabricated from crude flake graphite (Qingdao Aoke Co.) by a modified Hummers method [25]. The as-prepared GO was then treated by ultrasonication treatment using a KQ-600kDE digital ultrasonic cleaning device (600 W, 80% amplitude) in a water bath for 1 h, and GO homogeneous dispersion (1.0 mg mL⁻¹) was obtained. In a typical synthesis, 0.3 g NH₄VO₃ powder was added into 40 mL GO dispersion, followed by stirring for 10 min. Then, 4 mL acetic acid was added to the above suspension. After being vigorously stirring for another 10 min, the orange liquid dispersion was transferred into an autoclave and was heated at 180 °C for 12 h. Finally, the resulted G/V₂O₅ nanocomposite monolith was removed, dialyzed with distilled water until the pH=7 and freeze-dried for 24 h. In comparison with G/V₂O₅ nanocomposite monolith, pure V₂O₅ and G were also prepared using the same procedure.

2.2. Characterization

X-ray diffraction (XRD) measurements were carried out using a D/Max-3c X-ray diffractometer with Cu Kα (λ=0.154 nm), using an operation voltage and current of 40 kV and 30 mA, respectively. A Quanta 200 environmental scanning electron microscope (SEM) and transmission electron microscope (TEM) (JEM2010-HR) were used to observe the morphology of the obtained materials. The surface electronic states about the obtained materials were investigated by X-ray photoelectron spectroscopy (XPS; AXIS ULTRA Kratos Analytical Ltd.). The G content in the nanocomposite was determined by weighting the residual sediment after the G/V₂O₅ nanocomposite was dissolved in a 6 mol L⁻¹ HCl solution. An IVIUMSTAT electrochemical workstation (Ivium Technologies BV Co., Holland) was used for the electrochemical measurement.

2.3. Electrochemical measurement

Electrodes used for supercapacitor were prepared by mixing the electroactive material, acetylene black and polyvinylidene fluoride (0.02 g mL⁻¹, in *N*-methyl-2-pyrrolidone) in a mass ratio of 75:20:5 to obtain slurry. Then the slurry was pressed onto the nickel foam current collector (2 cm²) and dried at 110 °C for 12 h. After drying, the coated mesh was uniaxially pressed to make the electrode material adhere to the current collector completely. The loading mass of the active material was about 3 mg. The electrochemical test of the individual electrode was performed in a three-electrode cell, in which platinum foil and saturated calomel electrode (SCE) electrodes were used as counter and reference electrodes, respectively. The specific capacitance *C* (F g⁻¹) of the electrode was determined by means of galvanostatic charge-discharge cycles as follows:

$$C = \frac{It}{(\Delta V)m} \quad (1)$$

where $\Delta V = (V_{\max} - V_{\min})$, V_{\max} is the potential at the end of charge and V_{\min} at the end of discharge (V), m is the active mass of the electrode (kg), I is the applied current (A) and t is the time of the discharge stage (s).

Electrodes used for LIBs consist of a test material (V₂O₅, G and G/V₂O₅), acetylene black and polytetrafluoroethylene (PTFE) in a weight ratio of 80:10:10, and pasted on pure Cu mesh. The prepared working electrodes were dried in a vacuum oven at 100 °C over 12 h to remove the solvent. These electrodes had a solid loading of 5.0 mg cm⁻². Cells were constructed in a glove box in argon atmosphere under a dew point below -65 °C. The electrolyte was 1 mol L⁻¹ LiPF₆ dissolved in a mixture of dimethyl carbonate, diethyl carbonate and ethylene carbonate (1:1:1 by volume), and the separator was microporous polypropylene film. The electrochemical measurements were performed at 25 °C using coin cells (CR2430) with pure lithium foil as the counter and reference electrode. The cells were tested by galvanostatic charge/discharge tests. The charge/discharge tests were carried out on LAND battery program-control test system (CT 2001A, Wuhan Jinnuo Electronic Co., Ltd., of China) at a constant current density of 50 mA g⁻¹ in a voltage window of 0.05–3.0 V.

3. Results and discussion

In this work, a G/V₂O₅ nanocomposite monolith is obtained by one-step hydrothermal technology. However, the G and V₂O₅ monoliths are absent in the control-experimental. So, the schematic for preparation of the G/V₂O₅ nanocomposite monolith is demonstrated in Fig. 1. GO with abundant function groups, including hydroxyl, carboxyl and carbonyl, can be easily dispersed in H₂O. When NH₄VO₃ and acetic acid were added to the system, a V₂O₅ colloid was formed in the existence of GO. After the hydrothermal treatment at 180 °C for 12 h, the GO was reduced

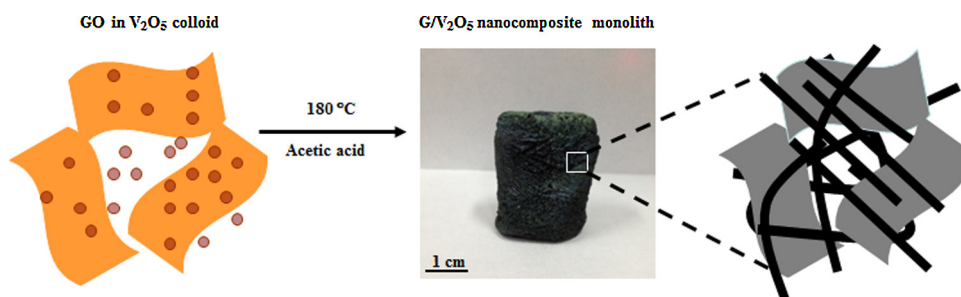


Fig. 1. The schematic for preparation of the G/V₂O₅ nanocomposite monolith.

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