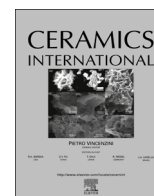




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# Synthesis of reduced graphene oxide/tungsten trioxide nanocomposite electrode for high electrochemical performance



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## ABSTRACT

A facile and well-controllable reduced graphene oxide/tungsten trioxide (rGO/WO<sub>3</sub>) nanocomposite electrode was successfully synthesized via an electrostatic assembly route at 350 rpm for 24 h. In this study, hexagonal-phase WO<sub>3</sub> (h-WO<sub>3</sub>) nanofiber was well distributed on rGO sheets by applying optimal processing parameters. The as-synthesized rGO/WO<sub>3</sub> nanocomposite electrode was compared with pure h-WO<sub>3</sub> electrode. A maximum specific capacitance of 85.7 F g<sup>-1</sup> at a current density of 0.7 A g<sup>-1</sup> was obtained for the rGO/WO<sub>3</sub> nanocomposite electrode, which showed better electrochemical performance than the WO<sub>3</sub> electrode. The incorporation of WO<sub>3</sub> into rGO could prevent the restacking of rGO and provide favourable surface adsorption sites for intercalation/de-intercalation reactions. The impedance studies demonstrated that the rGO/WO<sub>3</sub> nanocomposite electrode exhibited lower resistance because of the superior conductivity of rGO that improved ion diffusion into the electrode. To evaluate the contribution of WO<sub>3</sub> to the rGO/WO<sub>3</sub> nanocomposite, the influence of mass loading of WO<sub>3</sub> on the capacitance was investigated.

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## 1. Introduction

The global energy demand has increased due to rapid population growth. According to International Energy Outlook 2013 (IEO2013), the U.S. Energy Information Administration (EIA) estimated that the world energy consumption will increase by 56% from 524 quadrillion British thermal units (Btu) to 820 quadrillion Btu between 2010 and 2040. The depletion of energy sources and increase in energy demand have urged researchers worldwide to study the electrochemical energy storages system, such as batteries, conventional dielectric capacitors and fuel cells [1]. Supercapacitors are one of the electrochemical capacitors that have attracted much research interest because of their long cycle life, high power density, excellent reversibility, environment friendly and higher safety [2].

Supercapacitors can be classified into two types, namely electrical double-layer capacitor (EDLC) and pseudocapacitor [3]. EDLCs involve the absorption and desorption of ions between an electrode and electrolyte interface to form a double layer. The amount of charge stored in an EDLC is based on the surface area, porosity and layers of carbon materials, such as activated carbon,

carbon nanotubes, carbon aerogels and graphene [4]. Pseudocapacitors generate capacitance by transferring electrons from an electrode to electrolyte interface through redox reactions. Conducting polymers and transition metal oxides are widely used as active materials in pseudocapacitors. A key weakness of conducting polymers and transition metal oxides is their mechanical instability and poor stability during cycling [5,6]. Nonetheless, transition metal oxides are considered ideal materials because of their various oxidation states for efficient redox reaction [7]. Although pseudocapacitors exhibit higher capacitance than EDLCs, they suffer from low power density and poor cycling stability. Therefore, hybrid capacitors combined with carbon-based and transition metals oxide materials were introduced to maintain cycle life stability and increase the energy density [8].

Various transition metal oxides (RuO<sub>2</sub>, ZnO, MnO<sub>2</sub>, V<sub>2</sub>O<sub>5</sub>, IrO<sub>2</sub>, Co<sub>3</sub>O<sub>4</sub>, and WO<sub>3</sub>) are electrochemically active and have been widely investigated [9–11]. RuO<sub>2</sub> is the most suitable material for supercapacitors because of its high specific capacitance [12]. However, RuO<sub>2</sub> is very expensive and toxic in nature [13]. In recent year, WO<sub>3</sub>, an n-type semiconductor, has attracted research interest because of its high intrinsic densities, low toxicity, electrochemical redox characteristic and low cost [14]. Numerous techniques have been applied to synthesize WO<sub>3</sub>, i.e. sol-gel, thermal evaporation, solvothermal method, chemical vapour deposition and hydrothermal method [15–18]. The hydrothermal method offers several advantages, such as inexpensive and simple

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**Table 1**  
Electrochemical performances of WO<sub>3</sub>-carbon composite electrodes.

Methods of synthesis	Experimental conditions	Types of capacitor	Specific Capacitances (F · g <sup>-1</sup> )/Current density (A · g <sup>-1</sup> )	Ref.
Hydrothermal	Precursor: Na <sub>2</sub> WO <sub>4</sub> ·2H <sub>2</sub> O and GO Capping agent: Oxalic acid Acid: 3 M HCl Time: 2 h Temp.: 100 °C	Symmetric capacitor	• WO <sub>3</sub> -H <sub>2</sub> O- rGO: <b>244/1</b>	[24]
Reflux	Precursor: Na <sub>2</sub> WO <sub>4</sub> ·2H <sub>2</sub> O and graphene nanosheets (GNS) Capping agent: - Acid: 0.5 M HNO <sub>3</sub> Time: 5 h Temp.: 95 °C	Symmetric capacitor	• (WO <sub>3</sub> -WO <sub>3</sub> ·H <sub>2</sub> O)- GNS: <b>140.8/0.3</b>	[25]
Hydrothermal	Precursor: Na <sub>2</sub> WO <sub>4</sub> ·2H <sub>2</sub> O and carbon nanofibers (CNF) Capping agent: NaCl Acid: 3 M HCl Time: 24 h Temp.: 180 °C	Asymmetric capacitor	• WO <sub>3</sub> -CNF: <b>254/0.5</b>	[26]
Hydrothermal	Precursor: K <sub>2</sub> WO <sub>4</sub> ·2H <sub>2</sub> O and activated carbon (AC) Capping agent: H <sub>2</sub> O <sub>2</sub> Acid: 6 M HCl Time: 6 h Temp.: 160 °C	Asymmetric capacitor	• WO <sub>3</sub> -AC: <b>358.58/0.5</b>	[27]

experiment setup, low energy consumption and low processing time [19–21]. However, WO<sub>3</sub> exhibits low electronic conductivity and a limited potential window, which hinders its application [22]. If WO<sub>3</sub> is incorporated with highly conductive graphene, it will improve the conductivity of WO<sub>3</sub> and increase the capacitive behaviour of materials [23]. Table 1 summarizes the electrochemical performances of WO<sub>3</sub> and its hydrates with other conductive carbon materials.

Reduced graphene oxide (rGO) has been reported as a promising material because of its distinctive mechanical, optical, electronic and electrochemical properties [28]. Thus, rGO has been widely used in solar cells [29], corrosion protection [30,31], optoelectronic devices [32,33] and bioelectric sensory devices for early detection of leukemia and DNA [34–36]. rGO is formed by the exfoliation of graphene oxide (GO) from stacking graphene sheets into individual or several graphene layers via chemical reduction. Unfortunately, rGO agglomerates or restacks into graphite because of its strong van der Waals force [37]. In such conditions, the actual capacitance of graphene sheets is lowered. Hence, the attachment of transition metal oxides such as ZnO, NiO, In<sub>2</sub>O<sub>3</sub>, MnO<sub>2</sub> and WO<sub>3</sub>, onto reduced graphene sheets is important to prevent aggregation [38–40].

In this study, rGO nanoparticles were synthesized using modified Hummers' method. WO<sub>3</sub> nanofibers were prepared through hydrothermal treatment using NaCl as a capping agent. A simple and efficient electrostatic assembly method was used to incorporate WO<sub>3</sub> into rGO nanoparticles to form rGO/WO<sub>3</sub> nanocomposites. This study aimed to improve the electrical conductivity problem of WO<sub>3</sub> and aggregation problem of rGO.

## 2. Experimental method

### 2.1. Chemicals and materials

Graphite powder, ammonium paratungstate (APT) and hydrazine solution (35 wt% in H<sub>2</sub>O) were obtained from Sigma

Aldrich (Steinheim, Germany). Sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) (95–97%), phosphorus pentoxide (P<sub>2</sub>O<sub>5</sub>), potassium persulfate (K<sub>2</sub>S<sub>2</sub>O<sub>8</sub>), sodium sulfide (Na<sub>2</sub>SO<sub>3</sub>), sodium chloride (NaCl) and potassium hydroxide (KOH) were purchased from Merck (Darmstadt, Germany). Potassium permanganate (KMnO<sub>4</sub>) was gifted from Friendmann Schmidt Chemical (Woodpark, Australia). Hydrochloric acid (HCl) (37%) and hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) (30%) were purchased from R&M Chemicals (Essex, UK). The chemicals were used as received without any purification. Deionized water (18.2 MΩ) was used throughout this study.

### 2.2. Pretreatment of graphite powder

Graphite powder was pretreated before oxidation to prevent incompletely oxidized GO as a final product [41]. In a typical procedure, 30 mL of concentrated sulfuric acid was poured into a round bottom flask followed by 6 g of K<sub>2</sub>S<sub>2</sub>O<sub>8</sub>, 6 g of P<sub>2</sub>O<sub>5</sub> and 4 g of graphite powder. The mixture was heated at 80 °C in an oil bath under mechanical stirring for 6 h. The mixture was allowed to cool to room temperature, before being diluted with 2 L of deionized water and kept overnight. The precipitate was filtered and dried in a vacuum desiccator overnight.

### 2.3. Synthesis of rGO

GO was synthesized using graphite powder (from Section 2.2) as the precursor by modified Hummers' method [42]. The details of the preparation method are described in our previous work [43]. The resulting GO was reduced to rGO via a chemical reduction route using hydrazine as the reducing agent. Briefly, 1 g of graphite oxide was dissolved in 500 mL of deionized water and then sonicated for 30 min to form a supernatant GO. The solution was adjusted to pH 10 using 5 M KOH. Approximately, 0.25 mL of hydrazine was added into the above solution and refluxed at 95 °C for 24 h. The mixture was filtered, washed with deionized water and dried in an oven for 24 h to obtain rGO.

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