



# Preparation and characterization of porous carbon from expanded graphite for high energy density supercapacitor in aqueous electrolyte



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## HIGHLIGHTS

- Activated carbon made from expanded graphite dispersed in polyvinylpyrrolidone.
- KOH activation and carbonization expanded graphite solid precipitate.
- Produced carbon had a specific surface area of  $457 \text{ m}^2 \text{ g}^{-1}$ .
- Activated expanded graphite electrodes exhibit good electrochemical performance.
- Device exhibited excellent floating stability for 120 h.

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## ABSTRACT

In this work, we present the synthesis of low cost carbon nanosheets derived from expanded graphite dispersed in Polyvinylpyrrolidone, subsequently activated in KOH and finally carbonized in Ar/H<sub>2</sub> atmosphere. Interconnected sheet-like structure with low concentration of oxygen (9.0 at.%) and a specific surface area of  $457 \text{ m}^2 \text{ g}^{-1}$  was obtained. The electrochemical characterization of the carbon material as supercapacitor electrode in a 2-electrode configuration shows high specific capacitance of  $337 \text{ F g}^{-1}$  at a current density of  $0.5 \text{ A g}^{-1}$  as well as high energy density of  $37.9 \text{ Wh kg}^{-1}$  at a power density of  $450 \text{ W kg}^{-1}$ . This electrical double layer capacitor electrode also exhibits excellent stability after floating test for 120 h in 6 M KOH aqueous electrolyte. These results suggest that this activated expanded graphite (AEG) material has great potential for high performance electrode in energy storage applications.

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## 1. Introduction

The energy industry is currently facing a global crisis due to the rising cost associated with the high energy demand, a growing population and serious depletion of fossils fuels which raises some environmental issue such as global warming. Thus, there is an urgent need to explore alternative renewable energy resources as well as storage devices with high power and energy densities. In other words, the alternative energy sources are expected to be clean, low cost, environmentally friendly and sustainable [1–3]. Furthermore,

new technology has to be implemented for the storage of the energy that can be released during high demand and at any time. In the search for high energy density and high power density energy storage devices, electrochemical technologies demonstrate the aptitude to provide the means for electrical energy storage. Amongst the various electrochemical energy storage devices, supercapacitors have drawn much interest due to their extremely high power density, good cycling stability and fast charge-discharge rate compared to conventional capacitors and lithium-ion batteries which possess a low power density and a short cycle life [4–6]. However, the relatively low energy density of supercapacitors has not yet met the requirements of electrical systems, ranging from portable electronics to hybrid electric vehicles [7,8]. Hence, all research efforts are geared towards improving on the energy density of supercapacitors without sacrificing their high power density and high cyclability. Generally, supercapacitors can be classified into two categories

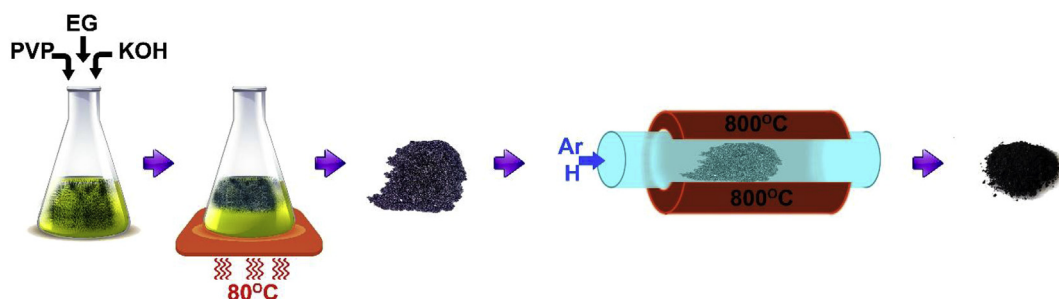
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according to their different energy storage mechanisms: Electrical double-layer capacitors (EDLCs) and pseudocapacitors (RuO and MnO) [4,9]. EDLCs energy storage mechanism is based on electrical double layer charge accumulation at the interface between the electrode and electrolyte. Advances in the field of EDLCs research have shown that the high electrochemical performance of these devices is highly dependent on the development of active electrode materials and also on the electrolyte used. In other words, materials with high micropore volume and good representation of the pore structure are needed to improve on the energy density. Carbon-based materials with high specific surface area (SSA) have been widely studied as electrode materials for EDLCs owing to their good electrochemical performances which include reasonably high specific capacitance and long-term cyclability, excellent physico-chemical properties, high electrical conductivity. In addition, their synthesis is generally easy and cost effective. While activated carbon is the material of choice for commercial supercapacitors, other forms of carbon materials such as carbide-derived carbons (CDCs) [10], onion-like carbons (OLCs) [11], carbon nanotubes (CNTs) [12] and graphene [13] are currently being considered as next generation EDLC electrodes. In general, the electrochemical performance of carbon based EDLC device is highly dependent on its SSA, pore size and pore size distribution (PSD) [14]. Therefore, it is necessary to control these parameters during the production of the carbon materials. This is usually done through physical activation using steam or CO<sub>2</sub> on one hand or through the traditional chemical activation using activating agents such as KOH, ZnCl<sub>2</sub>, and H<sub>3</sub>PO<sub>4</sub>, on the other hand. Recently, highly porous carbon nanosheets made from a three-dimensional graphene/polymer-based hydrogel with exceptional properties was reported by Zhang et al. [15]. The Hydrogel was activated with KOH and the porous materials produced demonstrated a superior supercapacitor performance in ionic liquid. Since the report of Zhang et al., several other reports based on similar method for production of porous carbons have since been published [16–19]. Herein we explore a two-step synthesis of highly porous carbon using Polyvinylpyrrolidone (PVP) and expanded graphite (EG) as carbon precursors. The choice of PVP is based on its ability to disperse and exfoliate carbon material such as graphite [20]. The obtained carbon with unique interconnected sheet-like morphology designated as AEG which stands for activated expanded graphite, exhibits high specific surface area (457 m<sup>2</sup> g<sup>-1</sup>) and specific capacitance (337 F g<sup>-1</sup>). High energy density value of 37.9 Wh kg<sup>-1</sup> at a power density of 450 W kg<sup>-1</sup> was obtained. These excellent electrochemical performances were attributed to the unique porous and interconnected morphology of the carbon nanosheets which could facilitate a fast charge transport mechanism.

## 2. Experimental

Scheme 1 presents the preparation process of the AEG material.



**Scheme 1.** Schematic diagram of the preparation process of AEG.

Firstly, graphite sample (grade ES 250 B5 from Qingdao Kropfmuehl Graphite) was expanded using a microwave oven at an irradiation power of 300 W for 5 min 1 g of the expanded graphite (EG) was dispersed in 100 ml of 10 wt% Polyvinylpyrrolidone (PVP) and the mixture was sonicated for 12 h. 5 g of KOH was then added to the solution, and the mixture was further stirred for 2 h at 60 °C. The solid precipitate obtained was collected and dried at 70 °C for 12 h. The dried solid precipitate was then placed in a horizontal tube furnace which was ramped from room temperature to 800 °C at 5 °C/min under argon and hydrogen gas flow and kept at this temperature for 2 h of carbonization. This procedure transforms the EG solid precipitate into flakes of carbon material denoted as activated expanded graphite (AEG). The black powder obtained was washed with 1 M HCl to remove the remaining KOH and subsequently with deionized water and dried at 60 °C.

## 3. Structural and electrochemical characterization

The morphology of the samples was studied using a Zeiss Ultra Plus 55 field emission scanning electron microscope (FE-SEM) operated at an accelerating voltage of 2.0 kV. Transmission electron microscopy (TEM) analysis was performed at 200 kV on a JEOL JEM-2100F microscope with field-emission gun. TEM samples were prepared by dispersing the samples in ethanol and which were dropped on a lacey carbon grid. X-ray diffraction (XRD) was recorded using an XPERT-PRO diffractometer (PANalytical BV, the Netherlands) with theta/theta geometry. Qualitative phase analysis of samples was conducted using the X'pert Highscore search match software at room temperature. Raman spectroscopy analysis of the sample was performed using a T64000 micro-Raman spectrometer from HORIBA Scientific equipped with a triple monochromator system to eliminate contributions from the Rayleigh line. All the samples were excited with a 514 nm line of an Ar laser with a power of 12 mW to avoid any thermal effect. Nitrogen adsorption-desorption isotherms were measured at −196 °C using a Micromeritics TriStar II 3020 (version 2.00) analyzer. All the samples were degassed at 180 °C for more than 12 h under high vacuum condition. The surface area was calculated with the Brunauer-Emmett-Teller (BET) method from the adsorption branch in the relative pressure range ( $P/P_0$ ) of 0.01–0.2. X-ray photoelectron spectroscopy (XPS) was used to determine the chemistry of the carbon sample. A Physical Electronics VersaProbe 5000 instrument was used which employs a 100 μm monochromatic Al-Kα to irradiate the sample surface. Photoelectrons were collected by a 180° hemispherical electron energy analyzer. Samples were analyzed at a 45° angle between the sample surface and the path to the analyzer. Survey spectra were taken at pulse energy of 117.5 eV, with a step size of 0.1 eV, which was used to obtain the elemental analysis of the powders. High-resolution spectra of C1s, N1s, and O1s regions were taken at pulse energy of 23.5 eV, with a step size

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