

Short communication

Nomex-derived activated carbon fibers as electrode materials in carbon based supercapacitors

K. Leitner^{a,*}, A. Lerf^b, M. Winter^a, J.O. Besenhard^a, S. Villar-Rodil^c,
F. Suárez-García^c, A. Martínez-Alonso^c, J.M.D. Tascón^c

^a Institute for Chemical Technology of Inorganic Materials, TU Graz University of Technology, Stremayrgasse 16, A-8010 Graz, Austria

^b Walther-Meißner-Institut, Walther-Meißnerstraße 8, D-85748 Garching, Germany

^c Instituto Nacional del Carbón, CSIC, Apartado 73, 33080 Oviedo, Spain

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Abstract

Electrochemical characterization has been carried out for electrodes prepared of several activated carbon fiber samples derived from poly (*m*-phenylene isophthalamide) (Nomex) in an aqueous solution. Depending on the burn-off due to activation the BET surface area of the carbons was in the order of 1300–2800 m² g⁻¹, providing an extensive network of micropores. Their capability as active material for supercapacitors was evaluated by using cyclic voltammetry and impedance spectroscopy. Values for the capacitance of 175 F g⁻¹ in sulfuric acid were obtained. Further on, it was observed that the specific capacitance and the performance of the electrode increase significantly with increasing burn-off degree. We believe that this fact can be attributed to the increase of surface area and porosity with increasing burn-off.

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1. Introduction

In recent years, activated carbons have found big attraction as electrode material for the storage of energy in supercapacitors due to their very high surface area, simple processability and low costs [1–3]. Chemical and physical methods of carbon activation are well known and allow producing materials, which are defined in terms of surface area and pore size distribution [4–8]. When fibrous precursors are used activated carbon fibers are obtained, which exhibit high adsorption/desorption rates and narrow pore size distribution. Using fibrous materials gives an additional profit from the construction point of view and thus offer a number of advantages over conventional powdered activated carbons [9–16].

Our previous work has shown that physically activated carbon fibers derived from poly (*m*-phenylene isophthalamide) (Nomex, Fig. 1) have a very homogeneous (micro)-pore

size distribution and can be prepared with very high pore volumes and/or ultrahigh surface areas up to 2600 m² g⁻¹ [17,18]. This seems to be an excellent prerequisite for the use of such fibers in electrochemical supercapacitors [19]. In this work we will compare the electrochemical performance of different Nomex-based activated carbon fiber materials and evaluate whether an application for supercapacitors is promising.

2. Experimental

2.1. Material

The starting material was commercially available Nomex aramid fiber in a variety known as Crystalline Nomex (T450 2.2 dtex.). Various series of activated carbon fibers were prepared from either fresh Nomex (N), or Nomex pre-impregnated with different amounts of H₃PO₄ (NP). The impregnation ratio (calculated as the weight gain after

* Corresponding author. Tel.: +43 316 873 8292; fax: +43 316 873 8272.
E-mail address: klaus.leitner@tugraz.at (K. Leitner).

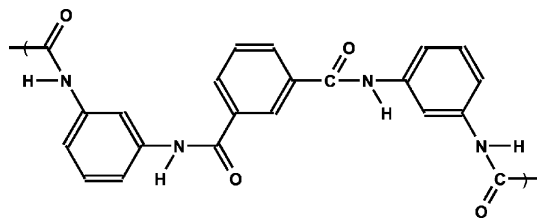


Fig. 1. Chemical structure of the precursor Nomex.

impregnation relative to the mass of Nomex) amounted to 1 and 7 wt.%, and the samples are named NP1 and NP7, respectively. The burn-off degrees of samples were calculated from the final mass (after pyrolysis and activation) relative to the initial mass of pyrolysed Nomex. Thus, N-76 is an ACF obtained from fresh Nomex pyrolysed and then activated in CO_2 to 76% burn-off, whereas NP7-72 is an ACF obtained from Nomex impregnated with 7 wt.% H_3PO_4 and activated in CO_2 to 72% burn-off. As concerns the activating agent, samples NP7-72, N-76 and NP1-90 were activated with carbon dioxide, whereas sample N-42 was activated with water vapour. The impregnation process as well as the pyrolysis/activation step is described in detail in previous reports [17,18]. Fig. 2a is an SEM micrograph of sample NP1-90, which shows that the diameter of the fibers is in the order of $10\ \mu\text{m}$. The fibers have a bilobular section, which is more easily appreciated in the higher-resolution image of a single fiber, shown in Fig. 2b. The bilobular morphology is already present in the parent Nomex polymer, and is maintained along with pyrolysis and activation treatments.

2.2. Electrochemical techniques and instrumental details

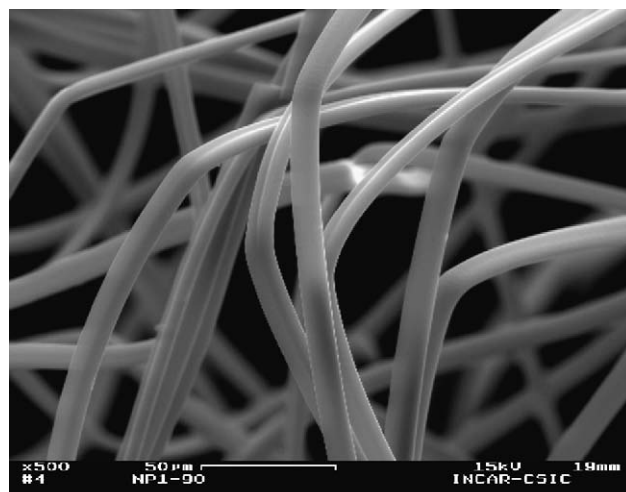
Cyclic voltammograms (CVs) were recorded with a Zahner IM6 measurement system in a potential range of -600 to $+300\ \text{mV}$ versus $\text{Hg}/\text{Hg}_2\text{SO}_4$. Cyclic voltammetry experiments started at open circuit potential (OCP) and were carried out at room temperature ($25 \pm 2\ ^\circ\text{C}$). CVs were recorded at three different scan rates (5 , 10 and $20\ \text{mV s}^{-1}$) to obtain information about the rate behavior of electrical double layer formation.

To obtain more information about the frequency behavior of the electrodes electrochemical impedance spectroscopy (EIS) was performed. For EIS the same measurement system was used. Therefore, at $-100\ \text{mV}$ versus $\text{Hg}/\text{Hg}_2\text{SO}_4$ an ac amplitude of $5\ \text{mV}$ was applied and data were collected in a frequency range of $10\ \text{kHz}$ to $5\ \text{mHz}$.

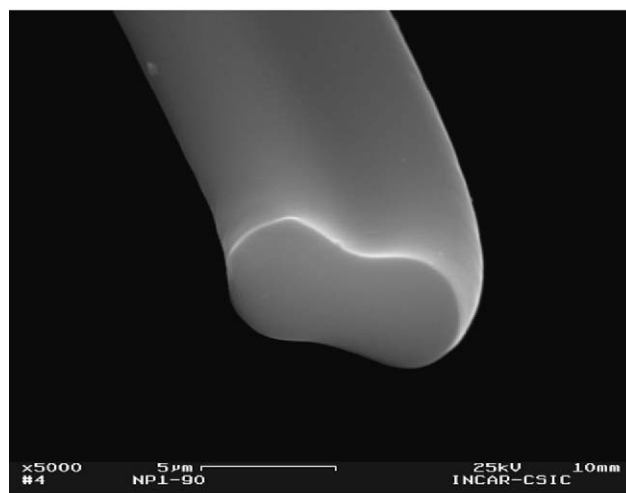
All electrochemical experiments have been performed in a three-electrode glass cell with a carbon counter electrode and an $\text{Hg}/\text{Hg}_2\text{SO}_4$ reference electrode using $5.25\ \text{M}$ sulfuric acid as the electrolyte.

2.3. Preparation of electrodes

The fibers were milled using an agate mortar and pestle to a fine powder and mixed with up to 10% PVdF (Aldrich) as



(a)



(b)

Fig. 2. SEM micrographs of NP1-90 sample: general overview (a); detailed view of a single fiber, showing the bilobular section (b).

binder for 1 h using a magnetic stirrer. Even after extensive milling, a fibrous morphology of the powder remains and is well observable under the light microscope. *N*-methyl pyrrolidinone (NMP) was slowly added to the resulting powder until a paste-like consistency was obtained. The paste was further homogenised using a spatula. The resulting active material was applied on the current collector with a doctor blade. A $10\ \text{mm} \times 10\ \text{mm}$ titanium grid was used as current collector. Each electrode was pressed five times for $60\ \text{s}$ (ca. $10^6\ \text{N m}^{-2}$) between filter paper to remove excess NMP. Finally, the electrodes were dried under vacuum for $24\ \text{h}$ at $120\ ^\circ\text{C}$ and afterwards weighted to estimate the mass of active material. All calculations of specific capacitance are referring to the total mass of active material (carbon and binder). Before the electrochemical measurements, all electrodes were vacuum-wetted with electrolyte in a desiccator for $30\ \text{min}$ in order to allow the electrolyte to penetrate into pores.

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