



Carbon aerogels as electrode material for electrical double layer supercapacitors—Synthesis and properties

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ABSTRACT

This paper constitutes a description of technological research the aim of which was to design a symmetric supercapacitor dedicated for the system of quality of electrical energy improvement (supply interruption, voltage dip). The main task was to use the carbon aerogel technology as the efficient method for production of electrode material with desirable properties. Carbon aerogels were prepared by carbonization of resorcinol–formaldehyde (RF) polymer gels. RF-gels were synthesized by curing polycondensation and by the inverse emulsion polymerization of resorcinol with formaldehyde, followed by microwave drying. The morphostructural characteristics of the carbon aerogels were investigated by atomic force microscopy (AFM) and the N_2 adsorption (BET method).

The electrochemical properties were characterized by means of cycle voltammetry, galvanostatic charging/discharging, and self-discharge.

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1. Introduction

Nowadays, the quality of electrical energy has become one of the most important problems in energetic industry. The presence of high advanced electronic and electro-technical machines, whose number is constantly increasing, necessitates a high level of the quality of electricity. However, the increasing number of active receivers of energy has a negative influence on the quality of electricity. It is especially noticeable in the case of: electric machines, devices modifying current frequency, high-power receivers, etc. Additionally, more and more frequently occurring connections from various energy sources to the common electrical circuit also requires a homogeneous quality. Under all these conditions high-power systems, which are capable of active current characteristic shaping, become essential. In these systems capacitors are very important ingredients which due to large amount of controlled energy have to be replaced by supercapacitors. In this sort of applications special properties of supercapacitors are utilized, other than the ones being currently under development worldwide. These devices should be symmetric and characterized by very short response time, should operate at high current intensity, even at the expense of self-discharge. These properties can be obtained by the presence of active groups red-ox on the surface of electrode material. Presented descriptions constitute part of the technological research leading to the valuation of possibilities to produce such

materials using the carbon aerogels technique. The main aim of our investigation was to determine whether it was possible to control and design properties of the final carbon material of electrodes in the phase of production of its polymer precursor (Tables 1 and 2).

Carbon aerogels belong to the special class of nanostructure carbons prepared via the pyrolysis of organic polymer precursors at elevated temperatures under inert atmospheres [1].

More importantly, the texture of aerogels (surface area and pore size distribution) can be modified as a function of different synthesis parameters e.g.: selection of the synthesis route; type of the precursor (reactants, solvent, catalyst); the curing and drying methods and the pyrolysis conditions [2]. Even though the properties of carbon aerogels can be enhanced by a choice of activation conditions, the structure of organic precursors significantly influences the final carbon materials [3,4].

Theoretically, the storage capacity (and charging speed) in EDLCs is proportional to the surface area of the porous carbon electrodes. Therefore, the correlation between a capacitance and pore structure has been widely investigated by many researchers. These results, reported in the literature, show that the electrochemical capacitance is generally proportional to the specific surface area for the same kind of carbon materials, which have been prepared with the same preparation method just by changing the preparation parameters.

However, according to the literature data, the capacitance depends not only on the surface area but also on the carbon structure, pore volume, pore size distribution, particle size, electrical conductivity, the surface functional groups of the electrode materials as well as the electrolyte composition [5–11].

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Table 1
Preparation conditions of RF-gels via polycondensation.

Sample	pH	Gelation temperature (°C)	Gelation time (h)	Heat treated	
				Temperature (°C)	Time (h)
RF0	6	20	24	30	15
				50	13

Table 2
Preparation conditions of the RF-gels via inverse emulsion polymerization.

Sample	Surfactant	Concentrations of RF in cyclohexane (%)	pH	Temperature (°C)	Time (h)	Stirring speed (rpm)
RFSL	Ludwik	10	6	60	6	300

Carbon aerogels are mostly obtained through reactions of sol-gel with resorcinol (1,3-dihydroksybenzene) and formaldehyde. This reaction was described by Pekala in 1987 and it is so far the best known and examined reaction of achieving carbon aerogels of desired structure. The synthesis of these materials is usually based on the polycondensation of resorcinol with formaldehyde using Na_2CO_3 as catalyst (C). Supercritical drying of RF-gels followed by a pyrolysis treatment usually gives rise to mesoporous carbon aerogels [12,13]. In this method, the control of meso- and micro-porosity takes place through the proper choice of molar ratios of resorcinol to catalyst (R/C) and resorcinol to water (R/W).

However, this process of producing carbon aerogels as suggested by Pekala is time-consuming and expensive because it requires the employment of supercritical conditions in the process of solvent removal. Therefore, many researchers seek other methods of synthesis of carbon materials which are characterized by controlled texture. These methods include solvent removal in freeze-drying conditions [14,15], with the employment of nitrogen [16], drying in ambient conditions [17,18], or with the use of microwaves [19,20]. Some other methods include modifications of the synthesis conditions, namely, the usage of a silicon matrix [21,22] or synthesis in emulsion in the presence of surfactant [15,23,24].

The aim of this study was the comparison of the influence of the synthesis method of resorcinol-formaldehyde (RF) polymer gel precursors on electrochemical characteristics of carbon aerogel electrodes for the application of EDL supercapacitor. In this study only the synthesis method was changed while the molar ratios (R/C) and (R/W) were maintained constant.

2. Experimental

2.1. Materials

Resorcinol (98%) was purchased from Aldrich Chemical Co. Formaldehyde (36–38% in water, stabilized with methanol), sodium carbonate (anhydrous), potassium hydroxide and cyclohexane was purchased from POCH. Ludwik was purchased from Inco-Veritas S.A. All reagents were used without further purification.

2.2. Preparation of carbon aerogels

RF-gels were synthesized by the polymerization of resorcinol (R) and formaldehyde (F), by using sodium carbonate as a basic catalyst (C) and deionized water (W) as a solvent. The molar ratios of resorcinol to formaldehyde (R/F), resorcinol to catalyst (R/C) and the molar ratio of resorcinol to water (R/W) were fixed at 0.5 mol/mol, 100 mol/mol and 0.08 mol/mol, respectively.

Two methods of synthesis of RF-gels were used: a sol-gel polycondensation and an inverse emulsion polymerization.

In the first method, resorcinol ($\text{C}_6\text{H}_4(\text{OH})_2$) was initially dissolved in deionized water, followed by adding sodium carbonate (Na_2CO_3) and formaldehyde (HCHO) into the solution, which was stirred by a magnetic stirrer. After gelation was performed, the sample was heat treated.

In the other method, spherical RF-gel particles were synthesized by emulsion polymerization; viscous RF-sols obtained by the first method were dispersed into cyclohexane solution containing

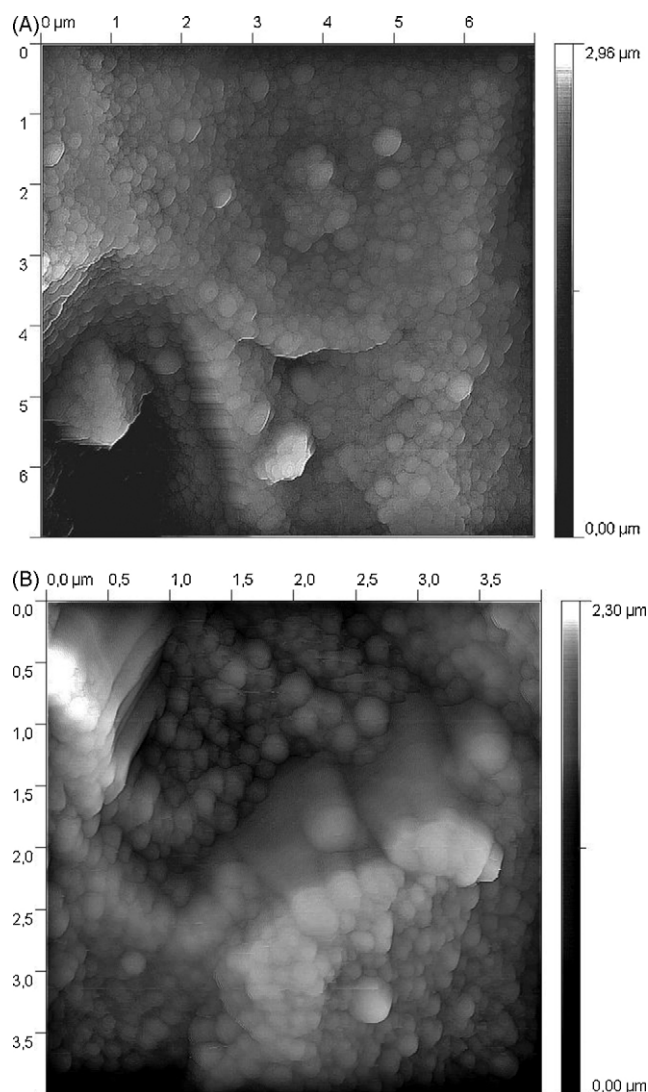


Fig. 1. (A) AFM planar image of RFO polymer precursor after drying (projected area $49 \mu\text{m}^2$). (B) AFM planar image of RFSL polymer precursor after drying (projected area $16 \mu\text{m}^2$).

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