

# Microporous carbonaceous materials prepared from biowaste for supercapacitor application



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## ABSTRACT

Agricultural waste is an abundant and cheap source of carbonaceous materials. In this work we showed that, using this source, it is possible to realize activated carbon (AC) suitable for electrochemical double capacitors (EDLCs). The AC obtained from agricultural waste is microporous and displays high specific capacitance in both, conventional and innovative electrolytes.

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## 1. Introduction

Electrochemical double layer capacitors (EDLCs), also known as supercapacitors, are presently considered among the most important electrochemical storage devices [1–4]. The commercial available EDLCs are comprised of electrodes based on activated carbons (AC) and electrolytic solutions containing a conducting salt, typically tetraethylammonium tetrafluoroborate ( $\text{Et}_4\text{NBF}_4$ ), which is dissolved in an organic solvent, e.g. acetonitrile (ACN) or propylene carbonate (PC) [5–8]. In these systems the energy is stored at the interface electrode–electrolyte, through the formation of a double layer. Thanks to this storage process EDLCs can be charged–discharged in seconds, they display high power ( $10 \text{ kW kg}^{-1}$ ) and a cycle life in the order of hundred thousand/million of cycles [1–4]. These features allow the use of EDLCs in a large number of applications, ranging from electronics, energy harvesting or hybrid vehicles [5].

In the last years many efforts have been made toward the realization of cheap and high performance EDLCs and, with the aim of realize such a goal, one of the most important aspects appears to be the introduction of advanced active materials. Due to their physicochemical properties and its relative low cost, activated carbon is the state-of-the art active material in commercial EDLCs.

For the development of advanced ACs suitable for supercapacitor application, both the raw material as well as the

synthetic process are of great importance. In order to prepare high amounts of ACs for commercial EDLCs, inexpensive and abundant sources should be preferably selected. For these reasons, organic precursors such as coconut shell [9], sugar cane bagasse [10], oil palm kernel shell [11] and seaweed [12], have been used for the synthesis of AC. These materials have shown advantageous characteristics as high specific surface area and a pore size distribution on the order of micro- and mesopores. Nowadays, commercialized ACs for being used in EDLCs are obtained from some of these sources, e.g. coconut shell.

It is worth to mention that when these organic sources are used as precursors, an important task is the optimization of the synthesis process. As a matter of fact, for the realization of ACs with high specific and/or volumetric capacitance and high capacitance retention at high current densities, the surface chemistry and surface morphology need to be carefully tailored in order to be highly compatible with the electrolytic solutions.

During the last years, different types of alternative electrolytes have been proposed, e.g. based on ionic liquids (ILs) [13,14]. It has been shown that the AC developed for organic or aqueous electrolytes might do not display good performance in these electrolytes. Therefore, the realization of AC suitable for these innovative electrolytes appears of importance.

In this work, we report about the preparation of activated carbon from agricultural waste. In rural regions, e.g. Münsterland area in Germany (which is the region where this work has been carried out), there is a large production of agricultural waste, which in many cases is not further used and has to be disposed. This waste represents a relatively inexpensive and easily available

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source for the production of carbonaceous materials. Taking this point into account, we decided therefore to use this agricultural waste for the synthesis of ACs suitable for EDLCs. In the first part of the paper the synthetic process used for the realization of the ACs is described in detail. Afterwards, the morphological and structural characterization of the produced materials is reported. Finally, the electrochemical performance of EDLCs realized using the ACs produced from biowaste as active material in combination with PC-based electrolytes is described.

## 2. Experimental

### 2.1. Materials synthesis

The preparation of activated carbons was done following a procedure similar to that described N.R. Khalili et al. [15]. Initially, the organic precursor, in this case agricultural waste, was washed and dried in an oven at 110 °C for 24 h. Subsequently, the sample was crushed mechanically using an agate mortar. The crushed sample was introduced in a water bath with  $\text{ZnCl}_2$  ( $\text{ZnCl}_2$ /Organic waste mass ratio of 0.75) at 85 °C for 7 h. After chemical activation, the sample was dried at 110 °C for 36 h and then crushed again into a smaller particle size. A thermal activation of the chemical activated sample was carried out in a nitrogen atmosphere at 800 °C for 2 h, with a temperature ramp of 5 °C min<sup>-1</sup>. Afterwards, the sample was removed from the tubular furnace, crushed and rinsed several times using 1.2 M HCl and distilled water to remove excess of zinc chloride and residual inorganic mater. After this washing procedure, the sample was dried at 80 °C overnight. In order to homogenize the particle size, the sample was ball milled using a speed of rotation in the range of 400 to 200 rpm for 1 h. Finally, the carbonaceous material followed a thermal treatment in a tubular furnace in an argon atmosphere at 800 °C for 2 h, with a temperature ramp of 2 °C min<sup>-1</sup>. The resulting sample was labeled as 1AC. Another AC was prepared with the same procedure using another agricultural waste material with a slightly different composition. This second AC is labeled 2AC. Fig. 1 shows a schematic representation of the synthetic pathway described above.

### 2.2. Physicochemical characterization

The morphology of the carbon powders was investigated by Scanning Electron Microscopy (SEM), using an AURIGA FIB-SEM from ZEISS. The samples were dispersed in ethanol by ultrasound and two or three drops of the dispersed powder were deposited on the sample holder and dried in an oven at 60 °C before analysis.

The elemental composition of the carbons was determined by CHN- and XPS analysis. For CHN analysis a CHNO-Rapid (Heraeus) was used. By combusting a small amount of carbon in excess oxygen atmosphere and qualitative analysis of the combustion product, the composition of the sample can be calculated.

An AXIS ULTRA HSA Kratos was utilized using the Al K $\alpha$  monochromatic beam (1486.7 eV). Core level spectra (pass energy of 20 meV) were used to evaluate the atomic concentration of the species present at the surface of the samples. The sample powders were dispersed onto adherent carbon film. The data were collected at room temperature and typically the operating pressure in the analysis chamber was kept below 10<sup>-9</sup> Torr. Data analyses were performed with the Casa XPS-Kratos, where every spectrum was corrected according to C 1s at 284.5 eV (graphite like carbon).

Information about the specific surface area as well as the porous structure of the prepared materials was obtained by an ASAP 2020 Surface Area and Porosity Analyzer (Micromeritics). To this, all samples were degassed at 350 °C for 6 h under vacuum before the adsorption experiment was started. The specific surface area (SSA) of the carbons was determined from N<sub>2</sub> adsorption isotherm using the BET equation and the DFT model (cumulative DFT surface area). The total micropore volume ( $V_{\text{micro}}$ ) and the average pore diameter ( $L_0$ ) were calculated by applying the Dubinin-Radushkevich (DR) equation to the N<sub>2</sub> isotherm by assuming slit-shaped pores [16]. The total pore volume (TPV) was obtained from the N<sub>2</sub> adsorption at  $P/P_0 = 0.99$  and the volume of the mesopores ( $V_{\text{meso}}$ ) was estimated by subtracting the total micropore volume from the total pore volume.

### 2.3. Electrochemical characterization

Composite electrodes containing the synthesized activated carbons were prepared following a procedure similar to that described in reference [17]. The weight ratio between the active material (1AC and 2AC), the conductive additive (Super C65, Timcal) and the binder (Sodium carboxymethyl-cellulose, CMC 2000, Walocell) was equal to 90:5:5. The electrode mass loading was 5–6 mg cm<sup>-2</sup> and the electrode area was 1.13 cm<sup>2</sup>.

The electrodes containing the investigated carbons were tested in two different electrolytes: 1 M Et<sub>4</sub>NBF<sub>4</sub> in PC and 1.5 M N-methyl-N-butyl pyrrolidinium bis[(trifluoromethyl) sulfonyl] imide (PYR<sub>14</sub>TFSI) in PC. The organic salt Et<sub>4</sub>NBF<sub>4</sub> (Sigma-Aldrich) and the ionic liquid PYR<sub>14</sub>TFSI (Iolitec, Germany) were dried under vacuum and stored in a glove box. The propylene carbonate (Sigma-Aldrich) was used as received.

Electrochemical test were carried out in a two electrodes configuration Swagelok® type cell. The cells were assembled in an Argon-filled glove box with oxygen and water contents lower than

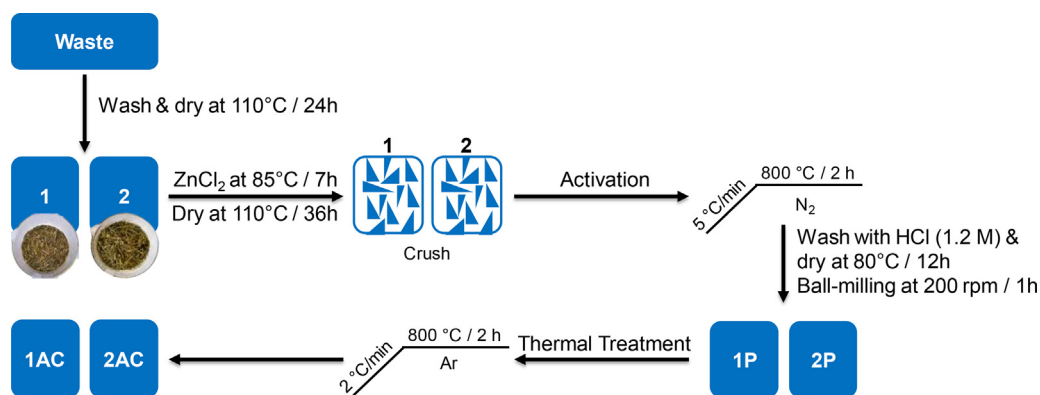


Fig. 1. Scheme of the used synthesis route.

1 ppm. For the working and the counter electrode, two identical activated carbon electrodes were used (symmetric EDLCs). As separator, a Whatman GF/D glass microfiber filter soaked with 100  $\mu\text{L}$  of the corresponding electrolyte was used.

The electrochemical tests were performed at 20 °C using a VMP multichannel potentiostatic–galvanostatic system (Biologic Science Instrument, France). Cyclic voltammetry were performed at 20 mV s<sup>−1</sup> while galvanostatic charge–discharge tests were carried out using current densities ranging from 5 to 100 mA cm<sup>−2</sup>. The values of capacitance of the total active material in the cell (C), the equivalent series resistance (ESR), and the coulombic efficiency have been calculated as indicated in Ref. [17,18].

### 3. Results and discussion

Fig. 2 shows the SEM images of the activated carbons 1AC and 2AC at two different magnifications. As shown in the figure, both carbons have irregular shapes and sizes, with most of the particles in the order of micrometers and a small amount of particles in the nanometer scale. At higher magnifications (Fig. 2c and d), both carbons exhibit a porous structure with a 3D network. These characteristics are matching the structural definition for activated carbons given by B.R. Stoner et al. [19].

Table 1 compares the chemical composition of 1AC and 2AC obtained from CHN and XPS analysis. As indicated from the elemental analysis, the carbon content of 1AC equals to 91.1%, while that of the 2AC appears significantly lower (74.7%). On the other hand, the oxygen content of 1AC, which is equal 6.3%, appears significantly lower than that of 2AC, which is of 20.9%. The results obtained using the XPS analysis, which are referring to the elemental content on the carbon surface, appear slightly different. As a matter of fact, while in case of 1AC the results are rather comparable, in case of 2AC the amount of oxygen results in less than half of that observed with the CHN analysis. Taking this outcome into account, it is reasonable to suppose that while 1AC displays a rather homogenous chemical environment, in the case of

**Table 1**

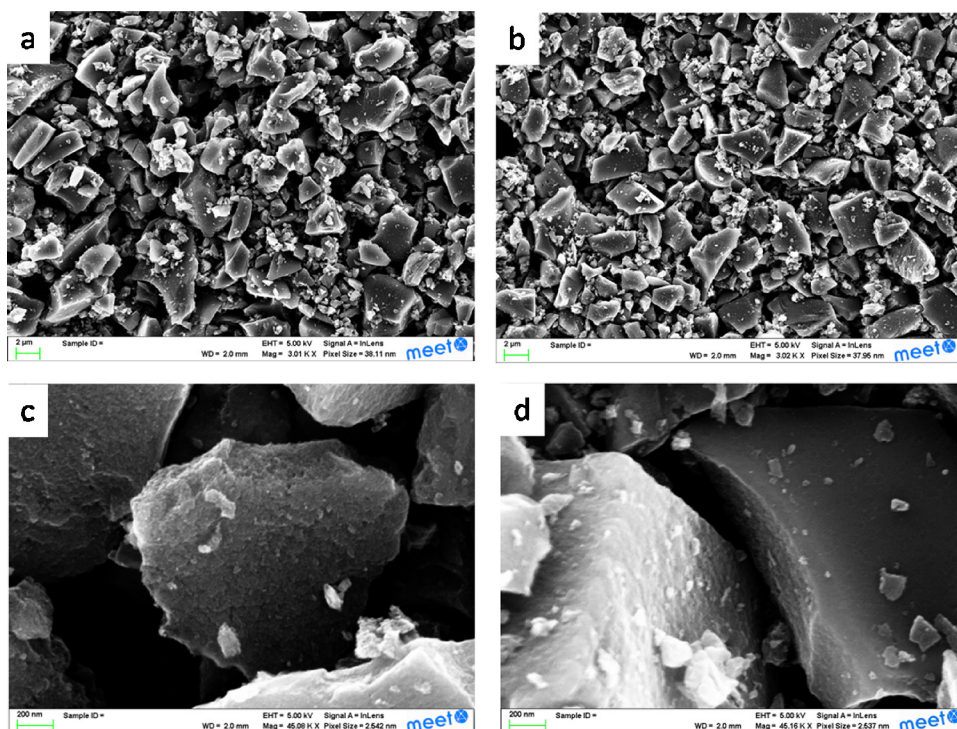
Elemental analysis and XPS data for 1AC and 2AC.

Sample	C <sub>CHN</sub> /%	O <sub>CHN-diff</sub> /%	N <sub>CHN</sub> /%	H <sub>CHN</sub> /%	C <sub>XPS</sub> /%	O <sub>XPS</sub> /%	N <sub>XPS</sub> /%
1AC	91.11	6.34	1.78	0.77	94.40	4.60	1.00
2AC	74.70	20.90	2.91	1.49	89.40	8.10	2.50

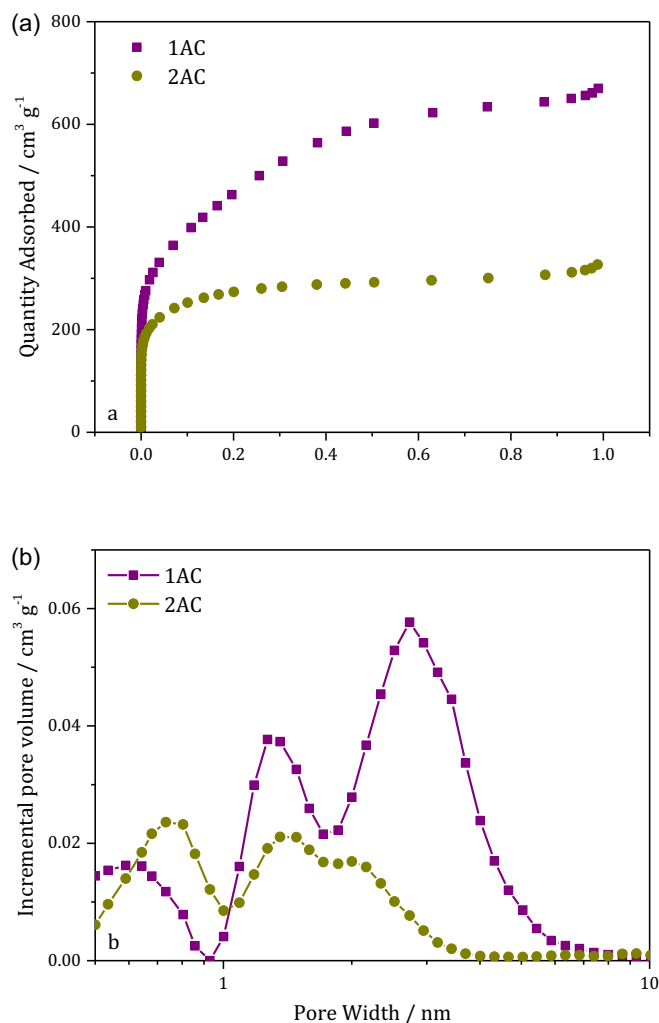
2AC there are differences in term of elemental composition between the bulk and the surface of the material.

Fig. 3 reports the data obtained from the adsorption isotherms and porosity measurements. As shown in Fig. 3a, both carbons display N<sub>2</sub> adsorption isotherms of type I, indicating that both of them are microporous materials [20]. Nevertheless, it has to be noticed that 1AC displays higher adsorption capacity and hence, a higher BET specific surface area with respect to 2AC (1635 m<sup>2</sup> g<sup>−1</sup> and 970 m<sup>2</sup> g<sup>−1</sup> for 1AC and 2AC, respectively). The same trend is also observed when the DFT model is used to evaluate the specific surface area (see Table 2). As shown in the figure, at low relative pressure values the adsorption isotherm of carbon 1AC exhibits a relative open curvature, indicating a wider pore size distribution. On the other hand, at the same relative pressure range, the 2AC sample exhibits a more perpendicular curve and a closer curvature, characteristic of highly microporous materials [21]. These observations are in agreement with the pore size distribution of 1AC and 2AC. As shown in Fig. 3b the sample 1AC has a wider pore distribution, with 23% of mesoporosity, while the sample 2AC presents only 8% of its porosity in the mesopore range and the rest of its pores in the micropore range. In the past it has been widely reported that the use of ZnCl<sub>2</sub> as activation agent leads to the formation of carbons with high surface area and a micro- and mesoporous structure [22]. The characteristics of the activated carbon considered in this work seem to confirm this finding.

Fig. 4 compares the cyclovoltammetric profiles of EDLCs containing 1AC and 2AC as the active materials in the electrolytes 1 M Et<sub>4</sub>NBF<sub>4</sub> in PC (Fig. 4a) and 1.5 M PYR<sub>14</sub>TFSI in PC (Fig. 4b).

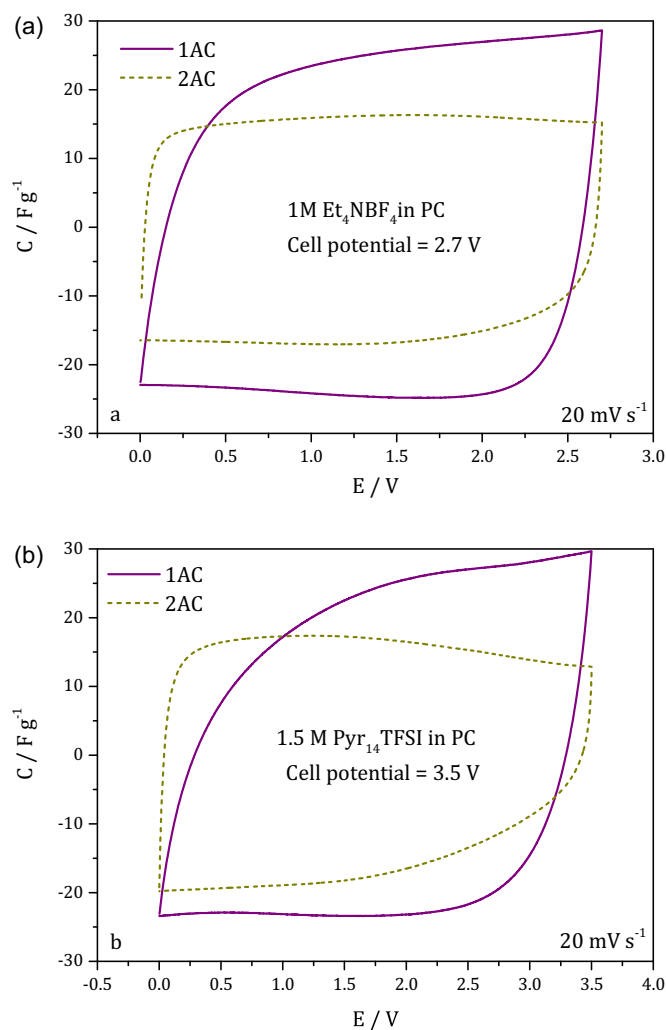


**Fig. 2.** SEM pictures of 1AC carbon (a and c) and 2AC carbon (b and d) at different magnifications.



**Fig. 3.** (a)  $N_2$  adsorption isotherms of 1AC and 2AC at 77 K, (b) Pore size distribution of 1AC and 2AC.

In both electrolytes, a scan rate of  $20 \text{ mV s}^{-1}$  was used for the experiments. As shown in the figure, all EDLCs display a quasi-rectangular shape and specific capacitance values (referring to the total active mass of both electrodes) between 15 and  $20 \text{ F g}^{-1}$ , in both investigated electrolytes. These values of specific capacitance are similar to those reported for other lab-made activated carbons [23]. Moreover, these values are also comparable with that of commercially available AC, e.g. Norit Super 30 [24]. Among the two carbons, 1AC displays the highest capacitance in both electrolytes. Nevertheless, it has to be noticed that the EDLC containing this carbon displays a deviation with respect to the ideal capacitive behavior when the potential scan is reversed at 0 V. This behavior is especially important with the 1.5 M  $\text{Pyr}_{14}\text{TFSI}$  in PC electrolyte. Since the average pore size of 1AC is big enough to allow the entrance of the electrolyte ions into the carbon structure, the



**Fig. 4.** Cyclic voltammetry of 1AC and 2AC in (a) 1 M  $\text{Et}_4\text{NBF}_4$  in PC and (b) 1.5 M  $\text{Pyr}_{14}\text{TFSI}$  in PC at  $20 \text{ mV s}^{-1}$ .

insertion and adsorption of the ions of the electrolyte into the carbon pores should not be the main reason for the behavior reported above. A possible explanation for this behavior could be the high electronic resistance of the investigated electrode. It has to be noticed that since the aim of this work was to verify the exploitation of agricultural waste as source of AC suitable for EDLCs, we did not optimize the electrode preparation. Consequently, the quality of the electrodes might affect the behavior of the devices, especially at relatively high scan rates and/or current densities. The EDLC containing 2AC display lower specific capacitance but, as clearly shown in Fig. 4, a more ideal behavior compared to the systems containing 1AC as the active material. The larger distortion from the ideal rectangular shape when using 1.5 M  $\text{Pyr}_{14}\text{TFSI}$  in PC as electrolyte, however, can also be observed for this EDLC. This behavior is obviously related to the viscosity of this electrolyte, which is higher than that of the conventional 1 M  $\text{Et}_4\text{NBF}_4$  in PC [25].

**Table 2**  
Textural characterization of 1AC and 2AC.

Sample	BET-area/ $\text{m}^2 \text{ g}^{-1}$	DFT-area/ $\text{m}^2 \text{ g}^{-1}$	TPV/ $\text{cm}^3 \text{ g}^{-1}$	$V_{\text{micro}}/\text{cm}^3 \text{ g}^{-1}$	$V_{\text{meso}}/\text{cm}^3 \text{ g}^{-1}$	$L_0/\text{nm}$
1AC	1635	1001	1.036	0.798	0.238	3.69
2AC	970	707	0.505	0.460	0.045	2.62



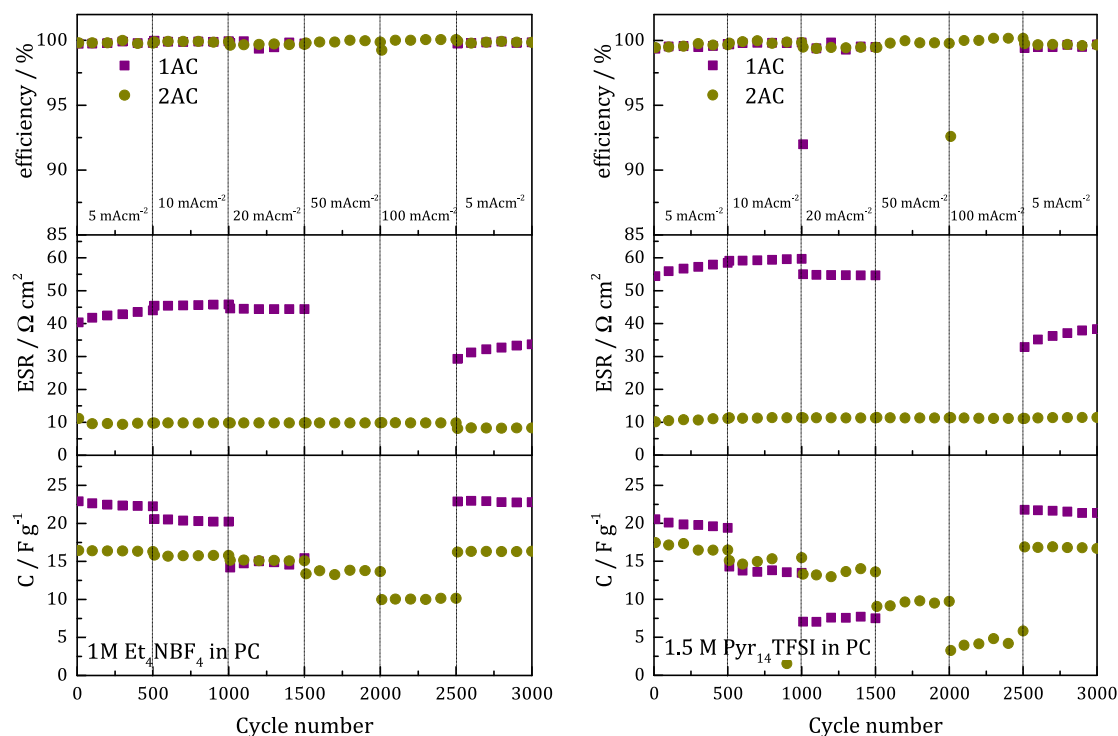


Fig. 5. Efficiency (%), ESR ( $\Omega \text{ cm}^2$ ) and C ( $\text{F g}^{-1}$ ) at different currents obtained for galvanostatic charge/discharge measurements.

Fig. 5 displays the efficiency, the electrical series resistance (ESR) and the specific capacitance of the EDLCs containing 1AC and 2AC as the active materials, as obtained during galvanostatic charge-discharge tests carried out using current densities ranging from 5 to  $100 \text{ mA cm}^{-2}$ . As shown, the efficiency of the charge-discharge process of EDLCs containing the materials is close to 100% for all applied currents densities and in both considered electrolytes. The ESR of the devices containing 1AC is, in both electrolytes, higher than  $40 \Omega \text{ cm}^{-2}$ . This high ESR confirms that the quality of the electrode containing 1AC could be certainly significantly improved, e.g. reducing the resistance at the interface between the electrode material and the current collector [5]. On the other hand, the 2AC sample exhibits ESR values in the order of  $10 \Omega \text{ cm}^{-2}$  in both electrolytes. Those values are in agreement with other activated carbon materials previously reported by our group [24]. As shown in the figure, the EDLC containing 1AC as active material display an important capacitance fade through the different applied currents in both electrolytes, with a complete polarized system at 50 and  $100 \text{ mA cm}^{-2}$ . The EDLCs containing the 2AC as the active material display a much higher capacitance retention. As shown, at  $50 \text{ mA cm}^{-2}$  the devices display capacitance retention of 84% and 59% in 1 M Et<sub>4</sub>NBF<sub>4</sub> in PC and 1.5 M Pyr<sub>14</sub>TFSI in PC, respectively. Although it has been reported that high amount of oxygen, as those present in 2AC, might affect the capacitance retention [26], it is interesting to notice that the capacitance retention shown by the 2AC-based EDLCs are not very dissimilar to those previously reported for other self-made carbons [9].

#### 4. Conclusions

In order to realize carbonaceous materials suitable for EDLCs, the search of appropriate sources of active material appears of importance. As a matter of fact, although the raw materials only make up a small amount of the total costs of AC production,

between 1.3% to 2.9% depending on the used process [27,28], the possibility to use of abundant and easily available sources is certainly representing a positive aspect for the overall production process.

Agricultural waste, the raw material that has been considered for the preparation of AC in this work, is produced on a daily basis and it can be considered as a highly abundant material, which can be fairly cheap in areas with a high concentration of agriculture. Most likely, the use of this material would not have a tremendous impact on the cost of EDLC. Nevertheless, taking into account the fact that large part of this agricultural waste is typically disposed, its use as source for carbonaceous materials could be of interest in view of the realization of sustainable processes. In this work we showed that using this source it is relatively easy to realize activated carbons suitable for EDLCs. The ACs obtained from these materials are microporous and display good specific capacitance in conventional as well as in innovative electrolytes. The performance of EDLCs containing AC made out of agricultural waste is promising, although further improvements are certainly needed. In the future it will be important to optimize the preparation of electrodes containing these AC in order to reduce the ESR. Once the electrode optimization will be done, it will be possible to evaluate more precisely the advantages and limits related to the use of AC made out of this green and largely available source.

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