



# Performance of carbon–carbon supercapacitors based on organic, aqueous and ionic liquid electrolytes

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## ARTICLE INFO

### Article history:

Received 23 December 2009  
Received in revised form 19 March 2010  
Accepted 26 March 2010  
Available online 1 April 2010

### Keywords:

Electrochemical double-layer capacitor  
Ionic liquid  
Electrolyte

## ABSTRACT

Properties of capacitors working with the same carbon electrodes (activated carbon cloth) and three types of electrolytes: aqueous, organic and ionic liquids were compared. Capacitors filled with ionic liquids worked at a potential difference of 3.5 V, their solutions in AN and PC were charged up to the potential difference of 3 V, classical organic systems to 2.5 V and aqueous to 1 V. Cyclic voltammetry, galvanostatic charging/discharging and impedance spectroscopy were used to characterize these capacitors. The highest specific energy was recorded for the device working with ionic liquids, while the highest power is characteristic for the device filled with aqueous H<sub>2</sub>SO<sub>4</sub> electrolyte. Aqueous electrolytes led to energy density an order of magnitude lower in comparison to that characteristic of ionic liquids.

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## 1. Introduction

Electrochemical capacitors are energy storage systems [1–3] which may be used in portable devices, hybrid vehicles and back-up systems [4,5]. The term electrochemical capacitor or supercapacitor [6] refers to electrochemical double-layer capacitors (EDLCs) [7–9], redox capacitors [10–12] and electronically conductive polymer-based capacitors (ECPs) [13–16]. In EDLCs energy is stored in the electric double-layer formed at the electrode/electrolyte interface. While redox capacitors and ECP based capacitors are still at the research phase, EDLCs are commercialized as computer power supply systems and developed as power supply systems in hybrid cars [17]. EDLCs consist of two active materials; i.e. a high surface carbon electrode and electrolyte. The electrolyte may be aqueous [18], organic (where usually acetonitrile (AN) or propylene carbonate (PC) are applied as solvents [19–33]), a solvent-free ionic liquid [34–37] or polymer gel electrolyte [38–42]. Inspection of original papers and review articles [43–46] suggests that most of EDLC research is devoted to the preparation of new carbons and studies of their performance as electrode materials. Moreover, specific capacitance of the carbon/electrolyte system is usually expressed in Farads per mass of carbon and often treated as an individual property of carbon [43–46]. Only few papers are devoted to the comparison of electrolytes [47,48]. The general aim of this work is to compare properties of EDLCs based on different types of

electrolytes – aqueous, organic and ionic liquids, working with the same carbon electrode.

## 2. Experimental

### 2.1. Chemicals

Acetylene black (AB) from Alfa Aesar (Johnson Matthey) and activated carbon cloth ACC-507-25 (2500 m<sup>2</sup> g<sup>-1</sup>) from Kynol®. Acetonitrile (Fluka, >99.9%) was distilled through a Vigreux column sieves. Propylene carbonate (PC, Merck) was distilled under reduced pressure. Both AN and PC were stored over A3 molecular sieves. The activated carbon cloth was dried before use at 180 °C for 24 h. N-methyl-N-propyl-piperidinium bromide (MPPipBr) was synthesised from N-methyl piperidine (Aldrich) and bromopropane (Aldrich) in chloroform. Obtained MPPipBr was purified by crystallization in 2-propanol (P.O.Ch., Poland) after addition of tetrahydrofuran (P.O.Ch., Poland). N-methyl-N-propyl-piperidinium bis(trifluoromethanesulphonyl)imide (MrPipNTf<sub>2</sub>) was obtained from MPPipBr by metathesis with lithium bis(trifluoromethanesulphonyl)imide (LiNTf<sub>2</sub>, Fluka) in an aqueous solution. N-methyl-N-propylpyrrolidinium bromide (MPPyrBr) was obtained from N-methylpyrrolidinium (Aldrich) and bromopropane (Aldrich) in acetonitrile. After acetonitrile evaporation the remaining crystals were dissolved in 2-propanol (P.O.Ch., Poland) and after the addition of tetrahydrofuran (P.O.Ch., Poland) MPPyrBr was precipitated. N-methyl-N-propylpyrrolidinium bis(trifluoromethanesulphonyl)imide (MPPyrNTf<sub>2</sub>) was obtained from MPPyrBr by metathesis with LiNTf<sub>2</sub> in an aqueous medium.

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The resulting liquid salt was dried in a vacuum at 50 °C for 10 h. The water content determined by the Karl Fisher method was below 0.2 wt.%. Tetraethylammonium tetrafluoroborate ( $\text{Et}_4\text{NBF}_4$ , Aldrich), 1-ethyl-3-methylimidazolium tetrafluoroborate ( $\text{EMImBF}_4$ , Fluka) and 1-ethyl-3-methylimidazolium bis(trifluoromethanesulfonyl)imide ( $\text{EMImNTf}_2$ , Iolitec) were used as received.

## 2.2. Measurements

Conductivity measurements of liquid electrolytes were performed in a glass cell (having thermostating jacket) with Pt working electrodes (cell constant  $4.81 \text{ cm}^{-1}$ ). Cyclic voltammetry curves were recorded with the  $\mu\text{Autolab}$  electrochemical system (Eco-Chemie, the Netherlands). The ACC/electrolyte/ACC coin-type two electrode, symmetric EDLC cells, were assembled from two ACC sheets (covered with acetylene black layer), separated by a Whatmann glassy-fiber GF/A separator, in an adapted 0.5" Swagelok® connecting tube. Activated carbon cloth electrodes as well as the separator were soaked with the electrolyte in a dry argon atmosphere in a glove box. Capacitors were conditioned before measurements in a thermostatic chamber at 60 °C. Impedance spectra were taken with the Atlas 98 EII electrochemical system (Atlas-Sollich, Poland) in a frequency range of 100 kHz to 0.01 Hz, potential perturbation of 10 mV. Before impedance measurements, each EDLC cell was galvanostatically charged/discharged 10 times (current 10 mA) and discharged before impedance spectra had been taken. Equivalent series resistance of capacitors was deduced from the deconvolution of impedance spectra (software package ZView, Scribner Co.). Galvanostatic charging/discharging experiments were conducted with the use of the ATLAS 0461MBI multichannel electrochemical system (Atlas-Sollich, Poland). Capacitances of the devices were calculated from the slope of charging/discharging curves.

## 3. Results and discussion

### 3.1. Electrolyte conductivity

Conductivity of  $\text{MPPipNTf}_2$ ,  $\text{MPPyrNTf}_2$ ,  $\text{EMImNTf}_2$  and  $\text{EMImBF}_4$  solutions in molecular liquids (ML): acetonitrile (AN) and propylene carbonate (PC) at 25 °C is shown in Fig. 1. Conductivity of neat, solvent-free ionic liquids (ILs) is of the order of  $1.5\text{--}15 \text{ mS cm}^{-1}$  ( $1.5 \text{ mS cm}^{-1}$  for the low conductive  $\text{MPPipNTf}_2$ ) and  $15.5 \text{ mS cm}^{-1}$  for  $\text{EMImBF}_4$ . Conductivity of IL+ML mixtures

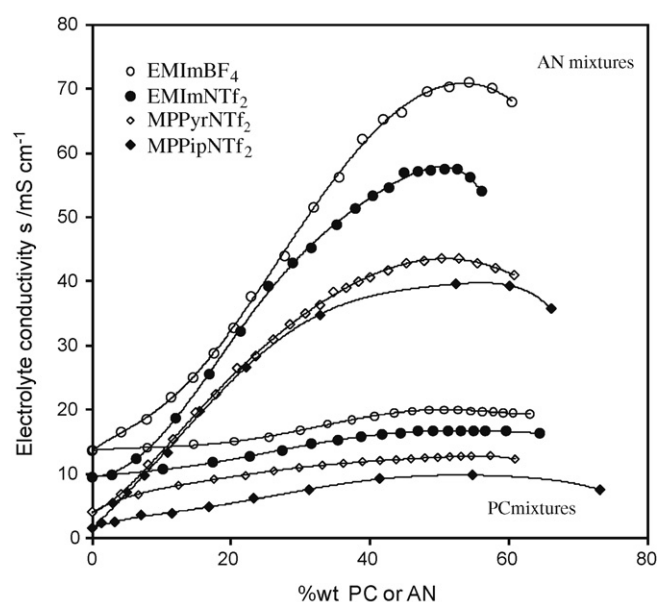


Fig. 1. Specific conductivity dependencies of ionic liquid mixtures with propylene carbonate (PC) and acetonitrile (AN).

increases with the increasing amount of ML, goes through a maximum and decreases with a further dilution, with the maximum at ca. 50 wt.% of molecular solvent. This is not unexpected, as in ionic liquid + molecular liquid mixtures (simply solutions of a liquid salt in a molecular solvent) ionic liquid ions are separated by neutral solvent molecules which increases ILs dissociation and hence, the number of charge carriers [49,50]. In addition, viscosity of IL+ML systems is lower in comparison to that of neat ILs. The latter effect may be seen in the case of ILs in AN solutions, which are much more conductive (up to  $71 \text{ mS cm}^{-1}$ ) in comparison to IL solutions in a more viscous PC (not more than ca.  $20 \text{ mS cm}^{-1}$ ). Table 1 contains conductivity of four ILs and IL+ML systems, together with the conductivity of conventional organic and aqueous electrolytes applied in EDLCs:  $\text{Et}_4\text{NBF}_4$  solution in AN (ca.  $60 \text{ mS cm}^{-1}$ ), aq. 29 wt.% KOH ( $540 \text{ mS cm}^{-1}$ ) and aq. 30 wt.%  $\text{H}_2\text{SO}_4$  ( $730 \text{ mS cm}^{-1}$ ) [36]. For comparison, organic solutions of lithium salts, for use in lithium-ion batteries, which may also be applied in EDLCs, show a conductivity of ca.  $10\text{--}20 \text{ mS cm}^{-1}$ , similar to that characteristic of most conductive ionic liquids [36].

Table 1

Parameters of EDLCs: electrolyte specific conductivity  $\sigma$ , mass of activated carbon cloth in electrodes  $m(\text{ACC})$ , equivalent series resistance  $R_s$ , capacitance  $C$  and applied voltage  $U$ .

Electrolyte	$\sigma$ [ $\text{mS cm}^{-1}$ ]	$m(\text{ACC})$ [mg]	$R_s$ [ $\Omega$ ]	$C$ [F]	$U$ [V]	
$\text{MPPipNTf}_2$	1.5	6.4	6.6	58.0	0.42	3.5
$\text{MPPipNTf}_2/\text{PC}$ (51 wt.%)	11.6	6.2	6.4	12.8	0.48	3.0
$\text{MPPipNTf}_2/\text{AN}$ (52 wt.%)	39.7	6.5	6.6	6.0	0.48	3.0
$\text{MPPyrNTf}_2$	4.1	6.5	6.3	30.2	0.45	3.5
$\text{MPPyrNTf}_2/\text{PC}$ (57 wt.%)	13.4	6.6	6.5	12.0	0.49	3.0
$\text{MPPyrNTf}_2/\text{AN}$ (51 wt.%)	43.6	6.5	6.1	5.5	0.42	3.0
$\text{EMImNTf}_2$	9.5	6.4	6.2	15.4	0.48	3.0
$\text{EMImNTf}_2/\text{PC}$ (52 wt.%)	16.8	6.5	6.6	6.2	0.48	3.0
$\text{EMImNTf}_2/\text{AN}$ (51 wt.%)	57.5	6.3	6.2	2.3	0.52	3.0
$\text{EMImBF}_4$	15.5	6.3	6.3	6.0	0.55	3.0
$\text{EMImBF}_4/\text{PC}$ (56 wt.%)	19.8	6.5	6.2	5.4	0.54	3.0
$\text{EMImBF}_4/\text{AN}$ (54 wt.%)	71.0	6.6	6.8	2.4	0.42	3.0
$\text{Et}_4\text{NBF}_4$ in PC – 1 M	14.5	7.0	7.1	7.8	0.55	2.5
$\text{Et}_4\text{NBF}_4$ in AN – 1 M	59.9	7.0	6.8	2.0	0.55	2.5
$\text{H}_2\text{SO}_4$ (30 wt.%)	750	7.0	7.0	0.16	0.67	1.0
KOH (29 wt.%)	540	7.1	7.1	0.55	0.53	1.0

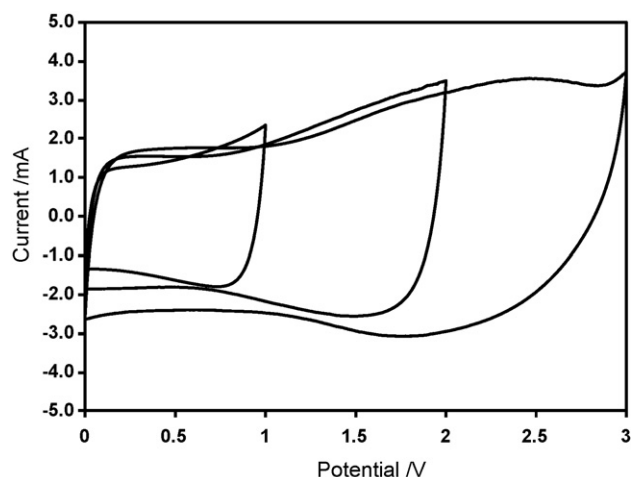


Fig. 2. Cyclic voltammetry of EDLC based on neat EMImNTf<sub>2</sub> ionic liquid. Mass of electrodes 6.4 and 6.2 mg (ACC 507-25 Kynol®). Scan rate 5 mV s<sup>-1</sup>.

### 3.2. EDLC cyclic voltammetry

Fig. 2 presents typical shape of current/potential curves obtained from cyclic voltammetry measurements (EDLCs based on neat EMImNTf<sub>2</sub> as an electrolyte). When the capacitor is charged up to a potential difference of 1 V, typical of aqueous systems, a box-like shape of the cv curve indicates a capacitor-like behavior of the device. When the device is charged to higher potential differences, the box-like shape of cv curves is still preserved, however, the highest voltage applied (3 V) results in some distortion of the ideal shape, suggesting electrochemical instability of the AC/IL interface. Data on electrochemical stability of ionic liquids at different electrode materials, such as Pt, W and glassy carbon (GC) are available in the literature. The measurements were usually performed in three electrode cells, applying different reference electrodes. Stability of ionic liquids is reported to be in a broad range from 2 to 6V [36]. The electrochemical stability of EMImNTf<sub>2</sub> determined at glassy carbon electrode is ca. 4.5 V. However, the same electrolyte working together with different electrode material (in this case with an activated carbon) may show reduced stability in comparison to that determined at a glassy carbon. Fig. 3 shows cv curves recorded for a series of EDLCs working with a neat MPPipNTf<sub>2</sub> ionic liquid as well as its mixtures (solutions) with acetonitrile (AN) and propylene carbonate (PC).

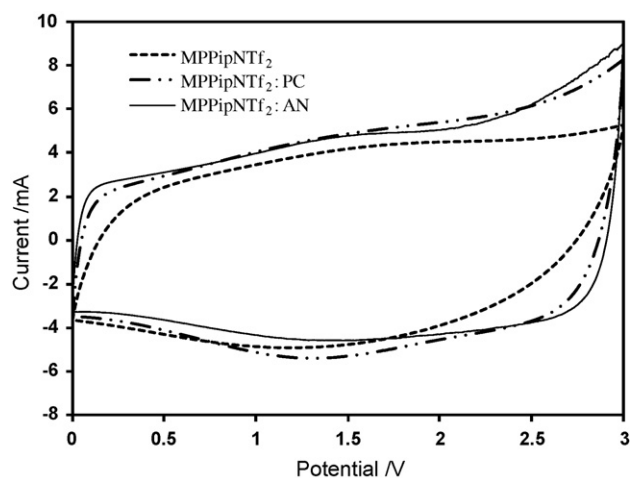


Fig. 3. The effect an addition of molecular liquids to ionic liquid on EDLC performance. Mass of electrodes ca. 6–7 mg each. Electrolytes: neat MPPipNTf<sub>2</sub> as well as its mixtures with acetonitrile (AN) and propylene carbonate (PC). Scan rate 5 mV s<sup>-1</sup>.

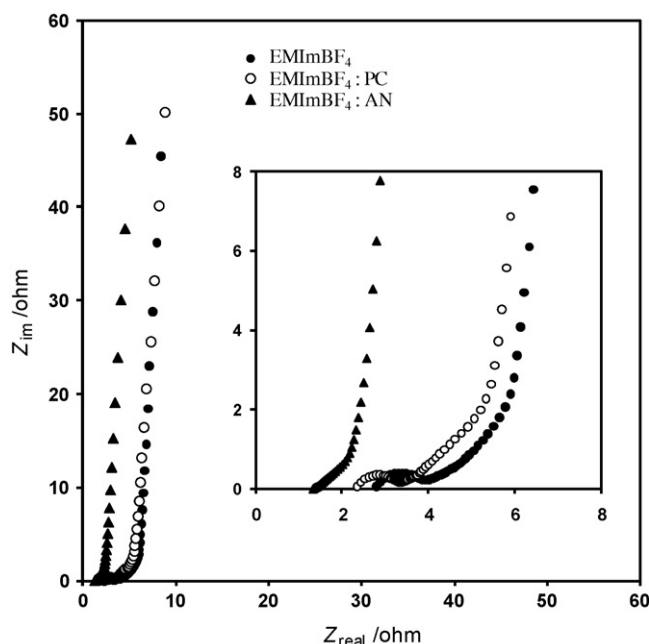
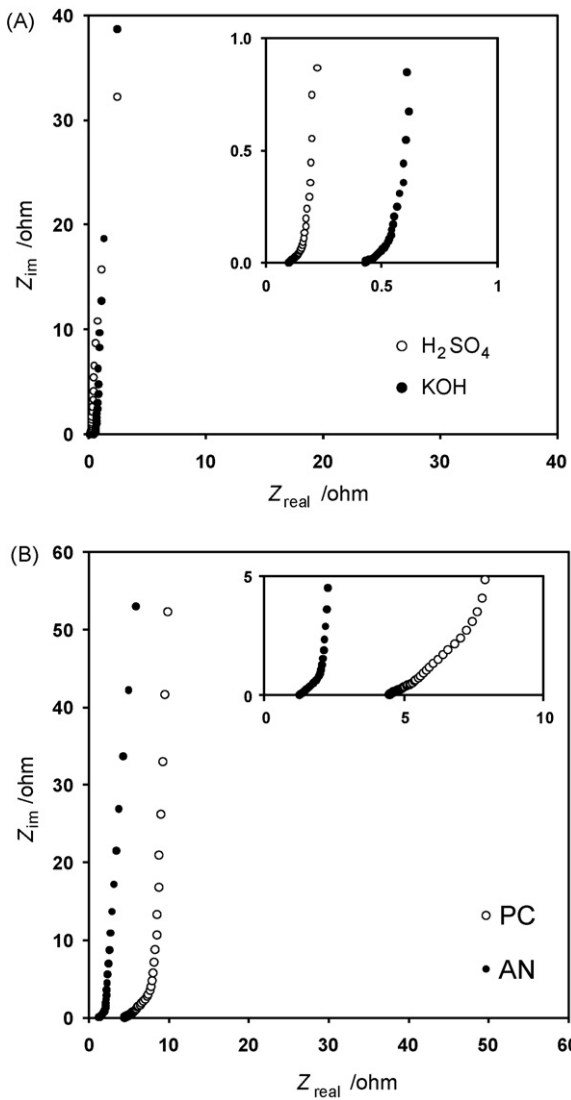


Fig. 4. Impedance spectra of EDLCs filled with neat EMImBF<sub>4</sub> and its solutions in AN or propylene carbonate (PC).

lene carbonate (PC), charged up to 3 V. In this case the box-like shape is preserved even at relatively high voltage differences (the electrochemical stability of MPPipNTf<sub>2</sub> determined at the GC electrode is ca. 6V). However, the effect of molecular solvent addition on the curves shape may also be seen.

### 3.3. Impedance studies

Fig. 4 shows impedance spectra of EDLCs based on neat EMImBF<sub>4</sub> and its solutions in AN and PC (ca. 50 wt.%). All the three plots, shown in the whole frequency range (100 kHz to 0.01 Hz) may be approximated by almost vertical lines shifted along the real axis by the value of the device series resistance ( $R_s$ ). However, when the plot is shown in a different scale, without the lowest frequencies, a semicircle at highest frequencies may be seen in the case of EMImBF<sub>4</sub> and EMIm BF<sub>4</sub> + PC electrolyte. Replacing neat ionic liquid EMImBF<sub>4</sub> ( $\sigma = 15.5 \text{ mS cm}^{-1}$ ) by its similarly conductive solutions in PC ( $19.8 \text{ mS cm}^{-1}$ ) does not bring about changes of the impedance plot shape. However, the semicircle cannot be seen on the impedance spectrum of the capacitor working with a much more conductive EMImBF<sub>4</sub> + AN electrolyte ( $71 \text{ mS cm}^{-1}$ ). Fig. 5a and b show corresponding impedance plots for classical aqueous (H<sub>2</sub>SO<sub>4</sub> and KOH) as well as organic (Et<sub>4</sub>NBF<sub>4</sub> in AN and PC) electrolytes. In all cases (aqueous, organic and ionic liquid electrolytes) the almost vertical line (at lower frequencies) typical of capacitors, follows a line of a slope close to 45°. Typically, capacitors filled with low conductive electrolytes (ILs and their solutions in PC) gave the impedance curve of the type: (semicircle)–(45° line)–(vertical line), while the more conductive systems (aqueous and solutions in AN) yielded curves without the semicircle (45° line)–(vertical line) (Fig. 4). The electrolyte resistance influences not only the shape of the plot but also the value of ohmic resistance at which the vertical line cuts off the real resistance axis. In general, impedance spectrum of EDLCs consists of three frequency-sensitive regions showing a characteristic shape of the  $Z'' = f(Z')$  curve. The semicircle present at high frequencies is due to (i) electrode porosity and (ii) the charge transfer resistance of possible pseudocapacity contributing to the total observed capacity. The electrolyte resistance is in series with the latter resistance. The middle-frequency

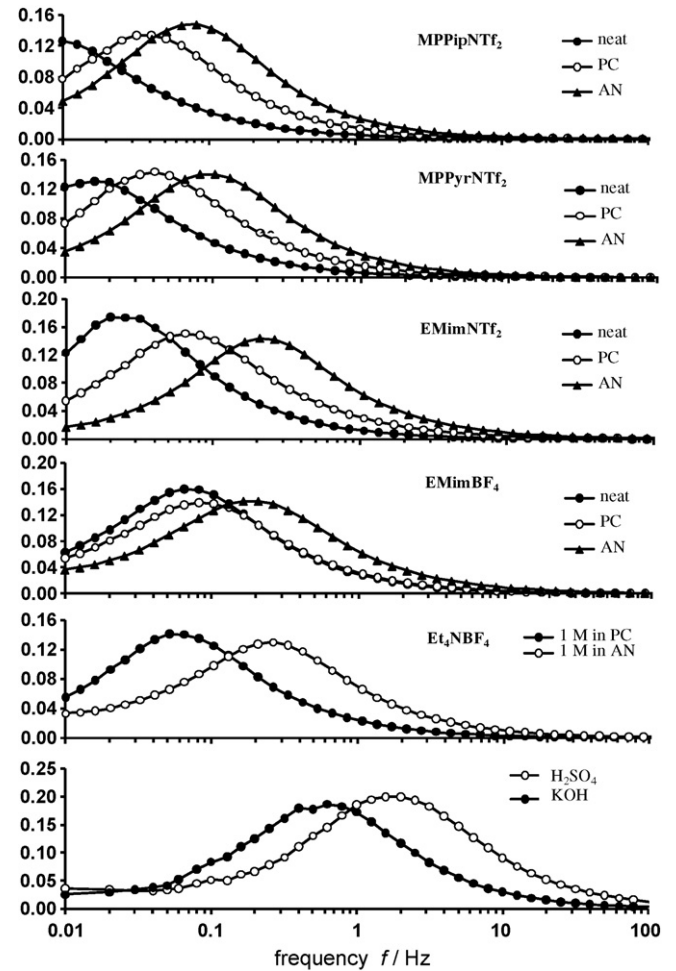


**Fig. 5.** Impedance spectra of EDLCs filled with (A) aqueous electrolytes (KOH and  $\text{H}_2\text{SO}_4$ ) and (B)  $\text{Et}_4\text{NBF}_4$  solution in propylene carbonate (PC) or acetonitrile (AN).

region, represented by the  $45^\circ$  line is rather due to the frequency dependent resistance  $R(\omega)$  associated with the electrolyte penetration of the electrode pores [1,51]. Semicircle loops may also be observed at the high-frequency region, due to the electrode/current collector interfacial resistance, inter-particle resistance or a passive layer created on current collectors [52–54]. Generally, the high-frequency semicircle may disappear when (i) the resistivity of the current collector interface is decreased by its modification [51] or (ii) the electrolyte conductivity is increased [27] (this effect is illustrated by Figs. 4 and 5).

#### 3.4. EDLC time constant and relaxation time

The low frequency, near-vertical line present at impedance plots represents the capacity of the device. However, an ideal capacitor in series with a resistor is frequency independent and represented by a line of a slope equal to 1. The deviation from the ideal slope (i.e. dependence on frequency) may be approximated by a ‘phase-constant element’ or by a capacitance described as a complex number. Frequency dependent capacitance  $C(\omega)$  can be expressed



**Fig. 6.** Dependence of imaginary capacitance of EDLCs on frequency.

as follows [55]:

$$C(\omega) = C_{\text{real}}(\omega) - jC_{\text{im}}(\omega) = \frac{-Z_{\text{im}}(\omega)}{\omega|Z(\omega)|^2} - j\frac{Z_{\text{real}}(\omega)}{\omega|Z(\omega)|^2} \quad (1)$$

where  $Z_{\text{rel}}$ ,  $C_{\text{rel}}$  and  $Z_{\text{im}}$ ,  $C_{\text{im}}$  are real and imaginary parts of impedance and capacitance, respectively. Fig. 6 presents a typical frequency dependency of imaginary capacitance as a function of frequency (in the logarithmic scale) for a number of electrolytes studied. It can be seen that the maxima are shifted towards higher frequencies in the following sequence: ionic liquid < organic electrolyte < aqueous electrolyte. The maximum of such a  $C'' = f(\omega)$  curve corresponds to the time-constant  $\tau = R_s C$  characterizing EDLC power [56]. The time constant for the EDLC working with MPPyrNTf<sub>2</sub> ionic liquid is ca. 14 s, while the corresponding value for the EDLC filled with the most conductive aqueous  $\text{H}_2\text{SO}_4$  electrolyte is only 0.1 s. On the other hand, frequency at the maximum,  $f_0$ , called relaxation frequency, leads to the relaxation time  $t_0 = f_0^{-1}$ . At frequencies below  $f_0$ , most of the device maximum capacity is reached [27]. Table 2 presents the relaxation frequency and time for EDLCs with electrolytes studies. The relaxation time  $t_0$  is useful in the characterization of EDLCs from the electric point of view, similarly as in the characterization of electric power systems. Any equivalent circuit describing supercapacitors consists at least of a resistor and a capacitor, or a number of resistors and capacitors. At high frequencies EDLC behaves like a resistor, while at lower frequencies rather like a series combination of capacitors and resistances. The capacitor is a reactive element of the circuit, leading

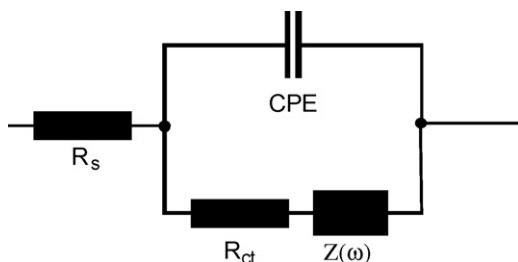
**Table 2**A comparison of EDLC parameters: time-constant  $\tau$ , relaxation frequency  $f_o$ , relaxation time  $t_o$ ,  $\tau/t_o$  ratio, specific energy  $E_{sp}$  and maximum power  $P_{max}$ .

Electrolyte	$\tau$ [s]	$f_o$ [Hz]	$t_o$ [s]	$\tau/t_o$	$E_{sp}$ [kJ kg <sup>-1</sup> ]	$P_{max}$ [kW kg <sup>-1</sup> ]
MPPipNTf <sub>2</sub>	24.4	0.010	100	4	198	4.1
MPPipNTf <sub>2</sub> /PC (51 wt.%)	6.1	0.032	31	5	171	14.0
MPPipNTf <sub>2</sub> /AN (52 wt.%)	2.9	0.082	12	4	165	28.7
MPPyrNTf <sub>2</sub>	13.6	0.016	63	5	215	7.9
MPPyrNTf <sub>2</sub> /PC (57 wt.%)	5.9	0.041	25	4	168	14.3
MPPyrNTf <sub>2</sub> /AN (51 wt.%)	2.3	0.102	10	4	150	32.5
EMImNTf <sub>2</sub>	7.4	0.026	39	5	171	11.6
EMImNTf <sub>2</sub> /PC (52 wt.%)	3.0	0.065	15	5	165	27.7
EMImNTf <sub>2</sub> /AN (50 wt.%)	1.2	0.208	5	4	187	78.3
EMImBF <sub>4</sub>	3.3	0.065	15	5	196	30.0
EMImBF <sub>4</sub> /PC (56 wt.%)	2.9	0.082	12	4	191	32.8
EMImBF <sub>4</sub> /AN (54 wt.%)	1.0	0.208	5	5	141	69.1
Et <sub>4</sub> NBF <sub>4</sub> in PC – 1 M	4.3	0.051	19	5	122	14.2
Et <sub>4</sub> NBF <sub>4</sub> in AN – 1 M	1.1	0.263	4	3	125	56.6
H <sub>2</sub> SO <sub>4</sub> (30 wt.%)	0.11	2.000	1	5	24	111.6
KOH (29 wt.%)	0.3	0.630	2	5	19	32.0

to the time difference between the voltage and the current. Consequently, some of the supplied power is not available for the EDLC (a part of the energy returns to the power source). The effect results in energy dissipation and may be described by the power factor  $P_f = P/S$ , where  $P$  is the real power (in Watts) and  $S$  is the apparent power (in Amps  $\times$  Volts) [55]. Finally, an EDLC characterized by a lower power factor requires higher currents to transfer a given quantity of real power than a circuit with a high power factor. At low values of the power factor, more apparent power needs to be transferred from the source to get the same real power  $P$  on the device. Data collected in Table 2 suggests a strong influence of the electrolyte on the relaxation time, which is consistent with the literature [27]. High conductive electrolytes are characterized by lower relaxation times.

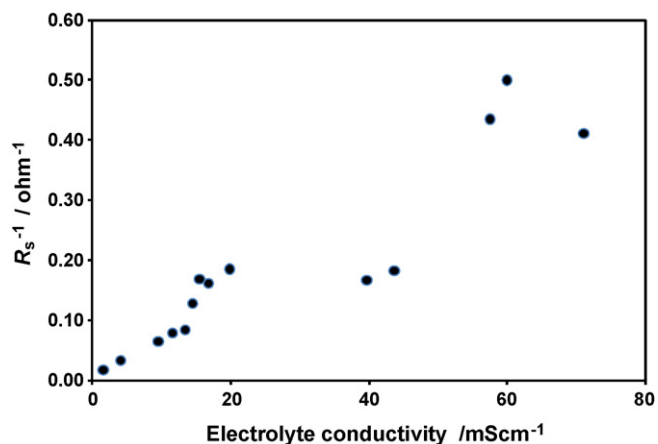
### 3.5. Specific energy and power density

The ratio of the specific energy which may be accumulated in two capacitors depends on their capacitance, but mainly on the voltage which may be applied:  $E = CU^2/2$ . Maximum power of the device is independent of the capacity, but depends on the voltage and the equivalent series resistance:  $P = U^2/4R_s$ . Values of  $R_s$  were obtained from deconvolution of EDLCs impedance plots using equivalent circuit presented in Scheme 1 and are collected in Table 2 together with calculated specific energy and specific power of devices. The series resistance of EDLCs having similar electrodes is determined mainly by the conductivity  $\sigma$  of the electrolyte contained both in the carbon material and in the separator. It must be taken into account that the series resistance of the porous electrode, such as the one used here, is not related directly to the electrolyte conductance ( $R_s \neq \sigma^{-1}$ ), due to the random orientation of the pores, but it is proportional to the  $\sigma^{-1}$  value (Fig. 7).

**Scheme 1.** Equivalent circuit representing EDLCs.

Capacitors working with two ionic liquids were charged to 3.5 V, their solutions in AN and PC as well as EMImNTf<sub>2</sub> were charged up to the potential difference of 3 V, classical organic systems to 2.5 V and aqueous to 1 V. It can be seen from the table that the highest specific energy is typical of devices working with the most stable ionic liquids, which may be charged to potential differences of 3.5 V (ca. 200 kJ kg<sup>-1</sup>), while the highest power is characteristic of the device filled with aqueous H<sub>2</sub>SO<sub>4</sub> electrolyte. On the other hand, the most powerful aqueous capacitors show the lowest energy capacity – ca. one order of magnitude lower in comparison to that characteristic of the device working with ionic liquids. Good compromise between high specific energy and possible power is obtained using solutions of room temperature ionic liquids in AN; the specific energy is still high (150–190 kW kg<sup>-1</sup>) with an acceptable maximum power (30–80 kW kg<sup>-1</sup>).

The specific capacity of devices changes with time in long-term cyclic. Typically, the initial specific capacity dropped after about 700–800 cycles to an approximately constant value. The capacity loss is high in the case of capacitors working with aq. H<sub>2</sub>SO<sub>4</sub> electrolyte (ca. 20% after 800 cycles) and much lower (5–10%) in the case of organic electrolytes (including ionic liquids). The observed loss of capacity is probably due to pseudo-faradaic processes, which can take place at the electrolyte/carbon interface.

**Fig. 7.** EDLCs equivalent series resistance,  $R_s^{-1}$ , as a function of electrolytes conductivity.

#### 4. Conclusions

1. Properties of a capacitor working with the same carbon electrodes and three types of electrolytes: aqueous, organic solutions and ionic liquids, were compared. Capacitors working with ionic liquids can be charged to a potential difference of 3.5 V, their solutions in AN and PC to 3 V, classical organic systems to 2.5 V and aqueous to 1 V.
2. The highest specific energy was recorded for the device working with ionic liquids, while the highest power is characteristic of the device filled with aqueous H<sub>2</sub>SO<sub>4</sub> electrolyte. Aqueous electrolytes led to energy density lower one order of magnitude in comparison to that characteristic of ionic liquids.
3. A good compromise between high specific energy and possible power is found when solutions of room temperature ionic liquids in AN are used, as specific energy is high and maximum power density is still acceptable.

#### Acknowledgements

The work was supported by grant DS31-178/09. The gift of activated carbon fabric ACC-507-25 from Kynol Europa GmbH is gratefully acknowledged.

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