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Review

Supercapacitors Based on Carbon Materials and Ionic Liquids

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Este artigo descreve o desempenho de supercapacitores construídos a partir de diferentes materiais de carbono. São descritos também o princípio geral de supercapacitor e a fórmula associada com sua operação. A função da nanotextura de carbono é enfatizada, selecionando carbonos preparados por técnicas de moldagem. Foi demonstrado que, principalmente, microporos e pequenos mesoporos são importantes para carregar a dupla camada elétrica, entretanto, a interligação de poros é crucial para a propagação de cargas. A presença de heteroátomo na rede de carbono, por exemplo nitrogênio, é útil por causa das reações faradaicas reversíveis adicionais. Foi encontrada uma dependência linear entre os valores de capacitância e a quantidade de nitrogênio. Líquidos iônicos são atualmente considerados como uma nova geração de eletrólitos com propriedades interessantes, por exemplo, alta voltagem de operação. Um aumento na voltagem de supercapacitores (acima de 3V) foi obtido pela aplicação de líquidos iônicos baseados em sais de fosfônio.

This paper presents performance of supercapacitor built from different carbon materials. The general principle of supercapacitor and formula connected with its operation are also described. The role of carbon nanotexture has been underlined selecting carbons prepared by template techniques. It has been demonstrated that mainly micropores and small mesopores play important role for charging of electrical double layer, however, interconnectivity of pores is crucial for charge propagation. The presence of heteroatom, e.g. nitrogen in the carbon network is profitable because of additional reversible faradaic reactions. A linear dependence of capacitance values *vs* nitrogen content has been found. Ionic liquids are presently considered as a novel generation of electrolyte with interesting properties, e.g. a high operating voltage. An increase of supercapacitor voltage (over 3V) has been realized by application of ionic liquids based on phosphonium salt.

Keywords: supercapacitor, template carbon, activated carbon, heteroatom, ionic liquid, phosphonium salt

1. Introduction

The main factors that dictate the selection of carbon for many electrochemical applications are its accessibility, low cost, easy processability, as well as different forms attainable (powder, fibers, foams, fabrics, composites)[1] and adaptable porosity[2] with various surface functionality.[3] Carbon electrodes are well polarizable, chemically stable in different solutions (acidic, basic, aprotic) and in a wide range of temperature. The amphoteric character of carbons, both electron donor/ acceptor and with the simultaneous presence of acidic/basic surface groups allows the electrochemical properties of materials based on this element to be extensively varied.

The properties of carbon materials are strongly affected by the preparation method. Hence, the selection of the synthesis route, type of precursor, heating rate, atmosphere and temperature of pyrolysis allows to control the final product but also to design carbons for a demanded practical target. Different carbon materials (active carbons, aerogels, xerogels, nanotubes.....) have been already tested for electrochemical capacitors called also supercapacitors.[4] Generally highly developed surface area, which is characteristic for microporous carbons, is necessary for formation of electrical double layer but presence of mesopores are crucial if a high propagation of charge is demanded especially for transportation system during acceleration where power peak is required as well as during braking for energy recovery.

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The aim of this paper is to highlight the key features necessary for an optimal design of carbons for electrode materials of supercapacitors. A new generation of electrolyte called ionic liquids will be also considered for electrochemical capacitor application.

2. Supercapacitors

Electrochemical double-layer capacitors (EDLC) are high power energy sources very attractive for hybrid application because of fast energy delivery and long-durability.[4-6] Charge storage in EDLC is mainly based on electrostatic attraction, i.e. pure double-layer charging. Performance of electrochemical capacitor based on activated carbon as electrode material is presented in Figure 1. The higher surface, the higher capacitance should be, if all the pores are accessible for ions and carbon material has a good wettability.

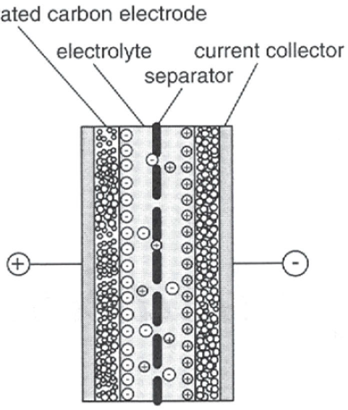

Figure 1. Performance of electrochemical capacitor.

It is noteworthy that each electrode can be treated as a single capacitor (C_1) and (C_2) and the total capacitance will depend on the electrode with a smaller value according to equation:

$$\frac{1}{C} = \frac{1}{C_1} + \frac{1}{C_2}$$

An increase of the capacitor voltage (U) causes a significant enhancement of power (P) and energy (E) because of the following dependences (where Rs stands for an equivalent series resistance):

$$E = \tfrac{1}{2}CU^2 \quad \text{and} \quad P = \frac{U^2}{4R_S}$$

Generally, in aqueous electrolytic solutions the capacitor voltage cannot exceed 1V, hence, it is clear that organic solutions present significant advantages over water medium, giving higher energy and power. Unfortunately, organic electrolytes mainly based on acetonitrile have lower conductivity, in turn, lower capacitance values.

The main electrode materials for capacitor are still activated carbons (AC) but it is not so easy to control their porosity, the size of pores and micro/meso ratio. Additionally the pores in AC are quite randomly connected. For optimal performance of capacitor it is necessary to adapt size of pores for dimensions of ions to ensure high capacitance values and a good charge propagation especially at high current loads. It is clear that template method seems to be a perfect way to obtain carbons with well-tailored pores.

3. Templated Carbons for Supercapacitor Application

The template carbonization is a novel strategy to obtain a controlled nanotexture of carbon network.[7-9] Silica matrix of defined pores and walls is selected for infiltration of carbon precursor in gas (e.g. propylene) or liquid phase, e.g. sucrose, furfuryl alcohol, pitch.[10-13]

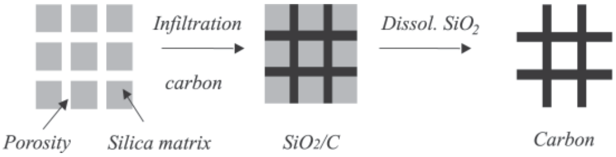

Figure 2. Scheme of template method for preparation of carbon material.

Among different silica materials, MCM-41, MCM-48, SBA-15, SBA-16 were the most often used as a host for infiltration of carbon precursor.[10-13] During carbonization process the walls of silica are transformed almost exactly into pores of carbon whereas silica pores become the carbon walls. By selecting a suitable SiO_2 matrix, the well sized carbon, which corresponds to the negative replica of the template could be prepared after dissolution of matrix by hydrofluoric acid. Carbon materials with an organized interconnected porous texture, a high specific surface area, a uniform pore size distribution and micropore/mesoporous character is obtained. The characteristics of carbon depend on the nature of carbon precursor and the silica template. Using propylene for silica infiltration the template carbon almost preserves the specific surface area of host. In the case of sucrose as precursor the additional microporosity is formed because of auto-activation due to evolved carbon dioxide.

Carbons prepared from MCM-48 and sucrose have the highest specific surface area (2000 m^2g^{-1}) and the capacitance values of 202 F g^{-1} in acidic (1 mol L^{-1} H_2SO_4), 180 F g^{-1} in alkaline (6 mol L^{-1} KOH) and 115 F g^{-1} in organic medium (1 mol L^{-1} $TEABF_4$ in acetonitrile) were measured.[10,11] Correlation of physical parameters calculated from adsorption isotherms of template carbons with capacitance data is shown in Table 1. Microporosity strongly determines the capacitance values.

For this series of template carbons, measurements of CO_2 adsorption has been also performed.[11] It is well known that only the amount of pores below 0.7 nm (ultramicropores) is estimated by this method. It was pointed out that CO_2 adsorption especially well correlates with capacitance values in aqueous as well as in organic electrolyte (Figure 3). Such dependence suggests that the major part of the electrical double layer is charged with non-solvated ions. It is well known that the micropores (ultramicropores <0.7 nm and supermicropores 0.7–2 nm) play an essential role for ions adsorption whereas the mesopores are necessary for their quick transportation to the bulk of material. Hence, an optimal performance is expected in the case of a nanostructured carbon of high specific surface area and well balanced micro/mesoporosity.

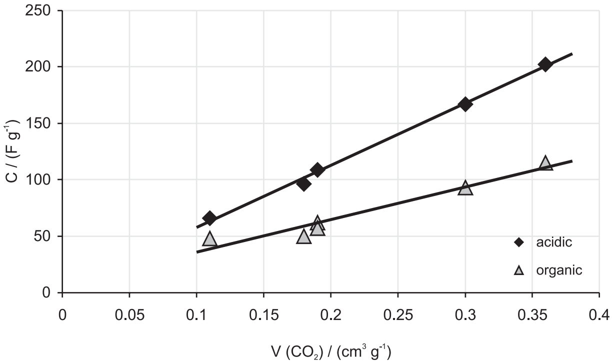

Figure 3. Capacitance values in aqueous and organic media of the carbons synthesized with different carbon precursors and silica matrices (Adapted from Reference 11).

Interesting results about the role of mesopores in template carbons have been obtained using SBA-16 as silica matrix and polyfurfuryl alcohol as carbon source. SEM images in Figure 4 show two examples of carbons obtained C1 and C2. The carbon particles have almost a perfectly spherical shape that replicates the morphology of the silica used as template.[13] Nitrogen adsorption isotherms and the pore size distribution show the highly mesoporous character of the template carbons.

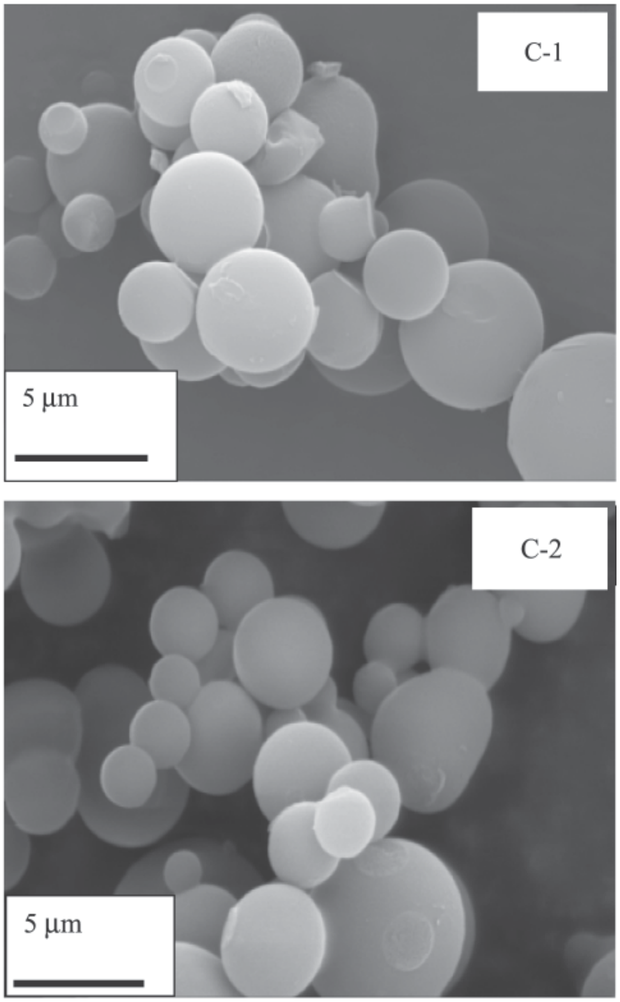

Figure 4. SEM images of mesoporous carbons from SBA16 and polyfurfuryl alcohol (Adapted from Reference 13).

Electrochemical measurements on these two types of mesoporous carbons used as capacitor electrodes proved that the carbon with a smaller average pore centered at

Table 1. Characteristics of template carbons using MCM-48 and SBA-15 as silica and propylene (Pr), sucrose (S) and pitch (P) as carbon sources

Template carbon	TSA / (m^2 g^{-1})	V_{N2} / (cm^3 g^{-1})	V_{CO2} / (cm^3 g^{-1})	Pore size / (nm)	C_{acidic} / (F g^{-1})	$C_{organic}$ / (F g^{-1})
CPr48	850	0.25	0.19	3.7	109	62
CS48	2000	0.58	0.36	2.7	202	115
CP48	1300	0.29	0.18	2.4	96	50
CPr15	713	0.21	0.11	3.4	66	48
CS15	1470	0.45	0.30	3.1	167	93
CP15	923	0.25	0.19	2.8	787	57

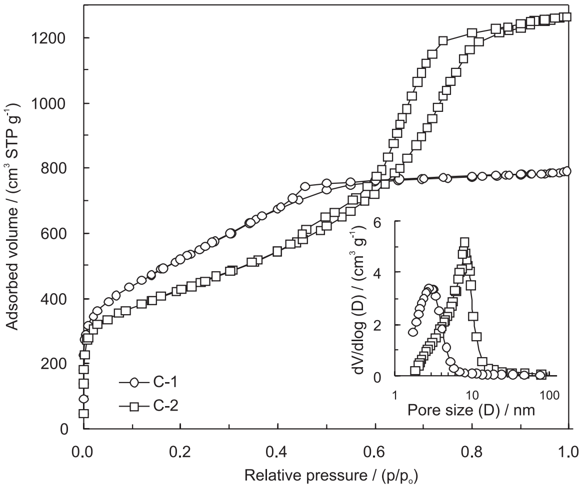

Figure 5. Nitrogen adsorption/desorption isotherms at 77K. Inset: pore size distributions of the template carbons (Adapted from Reference 13).

3 nm presents definitively a better capacitor performance than carbon with 8 nm average pore size. Galvanostatic charge/discharge in organic medium (Figure 6) presents a perfect performance of capacitor.

An example of voltammetry experiments at scan rate of potential from 2 to 20 mV s^{-1} for carbon C1 in organic medium (1 mol L^{-1} TEABF$_4$ in acetonitrile) is shown in Figure 7. Perfect square shape of characteristics proves a good charge propagation in this material.

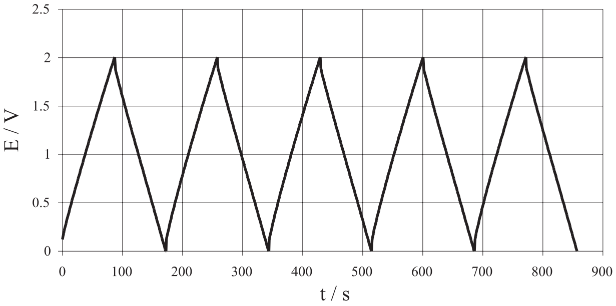

Figure 6. Galvanostatic charge/discharge (1A g^{-1}) of the carbon C1 in TEABF$_4$ in AN (Adapted from Reference 13).

For practical application a good cyclability of capacitor is very important. Cycling of capacitors in organic electrolyte was performed with 500 mA g^{-1} current load over 3000 cycles. Impedance spectra show the capacitor behavior before and after cycling. The curves are almost overlapped that proves lack of aggravation with cycling.

The novel mesoporous carbons seem to be especially adapted for organic medium where the diffusion of large-size ions (cation *ca.* 7.4 Å and anion *ca.* 4.9 Å) can proceed more easily. Additional presence of supermicropores in carbon C1 improves performance of capacitor. However, it was proved that 3 nm size of mesopores is sufficient and the most useful for a perfect ions motion. It seems that pores with higher size have a limited use for efficient charging of EDL. Well balanced micro/mesoporosity is crucial for a good capacitor performance. Additionally, the higher mesopore volume, the lower density of material, that diminishes the volumetric energy of these advanced carbons. It is noteworthy to mention the high cost of template carbons, which are perfect for fundamental research but cannot practically replace activated carbons. However, they are perfect reference carbons of strictly controlled porosity.

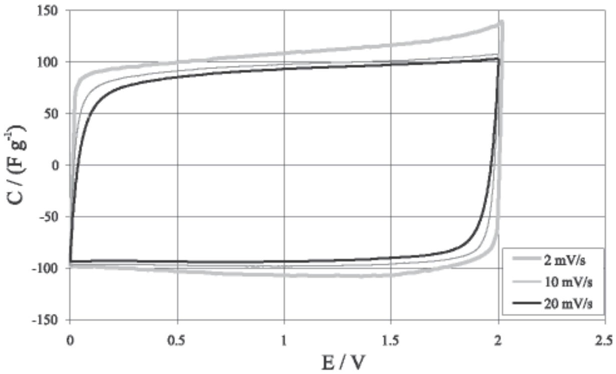

Figure 7. Voltammetry characteristics of capacitor built from C1 at different scan rates (Adapted from Reference 13).

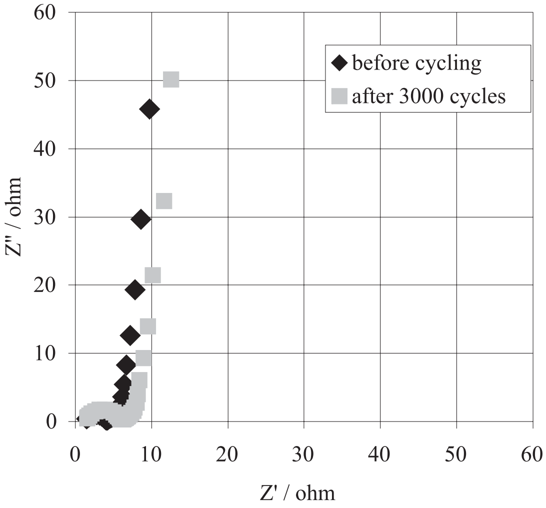

Figure 8. Nyquist plot for capacitor built from template carbon C1 before and after cycling (Adapted from Reference 13).

Finally, apart of size of pores adapted for dimensions of electrolyte ions it has been also proved that for a quick and efficient charge propagation interconnectivity between the pores play a great importance due to excellent mobility of ions.[12,13]

Apart of application of carbon materials with controlled porosity a great deal of attention is devoted to study the effect of heteroatom in carbon network on capacitance properties.

4. Role of Heteroatoms in Carbon Network for Capacitor Performance

The functional groups present at the surface of carbons used as an electrode in EDLC can considerably enhance the capacitance through additional faradaic reactions generally called pseudocapacitance effects. Therefore, instead of developing the specific surface area, an alternative is to introduce pseudo-faradaic properties by doping of carbon through foreign elements. The pseudocapacitance induced by oxygen functionalities due to close association of oxygen with carbon surface is the most often phenomenon.

The presence of different heteroatoms (oxygen, nitrogen) substituted to carbon in the graphene layers or occurring as functional groups has been considered for changing the electronic properties which can further influence the electrochemical performance. The foreign atoms modify the electron donor/acceptor properties of the graphene layers, and are consequently expected to affect the charging of electrical double layer and to give pseudo-capacitance faradaic reactions. The main dopant studied in literature is nitrogen, part of N can be substituted to carbon (“lattice nitrogen”), and the other can be chemically bound to organic molecules (“chemical nitrogen”).

Enrichment in nitrogen has been reported as an interesting route for improvement of capacitance behaviour.[14-19] Nitrogen can be incorporated into carbon network by different ways. It was proved that ammoxidation of carbon sample can bring useful effect on capacitance properties.[14]

The effect of substitutional nitrogen in the graphitic network on the pseudo-capacitance properties has been also studied by using carbon precursors in the form of polymers already rich in nitrogen. A series of nitrogen enriched carbons have been prepared by carbonization of polyacrylonitrile (PAN) or oxidized poly(4-vinylpyridine) cross-linked with 25 wt% of divinylbenzene (PVPox) and their blends with pitch (P). Then, they have been activated by steam to give activated carbons which were tested in two electrode capacitors.[16,17] The detailed nanotextural characteristics together with elemental composition expressed by N/C, O/C and N/O atomic ratios and the potential of zero charge were investigated. The voltammetry characteristics for the activated carbon from PAN show a perfect rectangular shape at 2 mV s^{-1} and 20 mV s^{-1}. The capacitance values of the nitrogenated carbons are strongly affected by the nature of the electrolyte and the electronic structure induced by nitrogen. A careful analysis of the results obtained for samples with comparable nanotextural properties, *i.e.* $S_{BET} \approx 800$ m^2 g^{-1} and comparable micropore volume, shows no remarkable differences in organic medium, whereas capacitance is proportional to the N content (Figure 9) in alkaline and especially in acidic medium. It is noteworthy that basic character of carbons is also proportional to nitrogen content. For example, carbon with 1.9 wt% N has $pH_{pzc} = 7.4$, whereas for 7.2 wt% N pH_{pzc} reaches value of 9.3. The enhancement of the capacitance values in H_2SO_4 medium is interpreted by pseudo-faradaic reactions due to the nitrogen functionality, such as those illustrated in the equations:

$$C^*{=}NH + 2\,e^- + 2\,H^+ \leftrightarrow C^*H\text{-}NH_2$$

$$C^*\text{-}NHOH + 2\,e^- + 2\,H^+ \leftrightarrow C^*\text{-}NH_2 + H_2O$$

where C* stands for the carbon network.

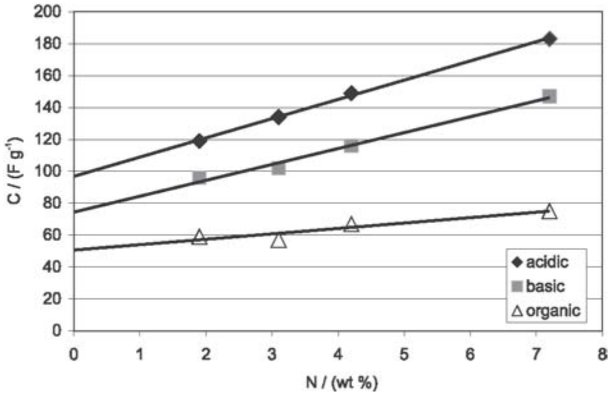

Figure 9. Capacitance values versus content of nitrogen in N-riched carbon materials.

It is clear that noticeable capacitance values can be obtained when heteroatoms are incorporated in the network of activated carbons, even if their specific surface area is quite moderate. The contribution of this pseudocapacitive effect due to nitrogen will be higher for the composites prepared at lower temperatures, e.g. 700 °C, because they have a higher nitrogen content than those obtained at 900 °C. It is well known that over 850 °C the amount of nitrogen in carbon drastically diminishes.[15] The second reason could be related to the nanotextural reorganisation depending on temperature. Some pores may be closed at 900 °C and become less accessible for the electrolyte ions than for the composites prepared at 700 °C.

These kinds of materials open new insights for the development of high performance materials. It also confirms that the pseudo-faradaic effects due to heteroatoms are an additional factor which, beside the nanotextural parameters, may contribute to the capacitance properties.

5. Ionic Liquids as Electrolyte for Supercapacitor

An increase of the capacitor voltage causes a significant enhancement of power and energy. Replacing an aqueous electrolytic solution by an organic one is an easy way to increase the capacitor voltage but obviously the capacitance values are lower. Practically, aprotic electrolytes most often based on acetonitrile or propylene carbonate as solvents allow to operate a capacitor only to 2.3 V. A further enhancement of capacitor voltage can be realized by the application of ionic liquids (ILs) which are characterized by a definitively higher decomposition potential.

ILs are defined as thermally stable salts synthesized by combining bulky organic cation with a wide variety of anions. ILs are generally liquid at room temperature and their properties strongly depend on the type of cation and anion as well as on the length of the alkyl group. An interest for ILs is connected with their unique physicochemical properties such as high thermal stability, negligible vapor pressure and good electrochemical stability. However, decomposition potential was mostly evaluated on glassy carbon or other metallic surface but not on porous carbon materials which are frequently used as efficient capacitor materials. Application of carbon materials of developed surface area significantly decreases the operating potential range. Additionally, a good wetting of highly porous carbon by ILs is complicated due to their high viscosity. It is possible to overcome the ILs viscosity by operating the capacitor at higher temperature.[20] A rapid increase of conductivity with temperature for a few ionic liquids with different anions is presented in Figure 10.

It can be observed that for the same cation, *i.e.* 1-ethyl-3-methylimidazolium, conductivity values are strongly affected by the type of anions. IL with BF_4^- anion shows the best conductivity.

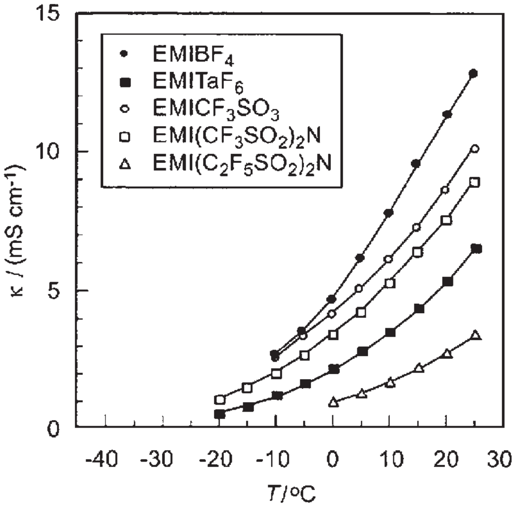

Figure 10. Dependence of conductivity versus temperature for various ILs with the same cation.

Generally ILs family is dominated by imidazolium, pyridinium and quaternary ammonium salts. Salts containing quaternary phosphorous cation are listed but not widely used in electrochemical applications.

The aim of investigation was to show that the operating voltage of carbon based capacitors can be increased to 3.4 V by application of ILs phosphonium salts. Carbon used as electrode material for all the investigation with ILs was obtained by KOH activation of commercially available carbon. Activation was performed at 850 °C with a ratio KOH:C = 4:1. Nitrogen adsorption isotherms at 77 K for the obtained carbon is shown in Figure 11.

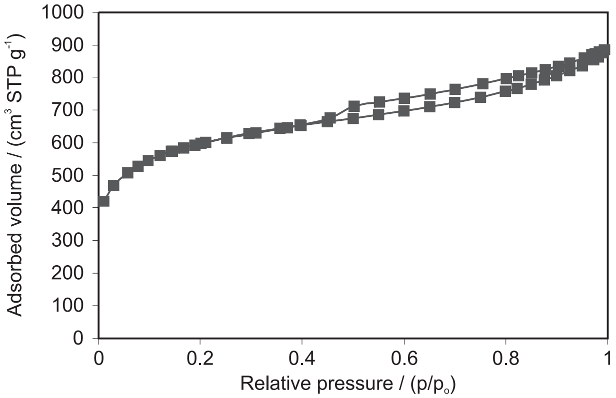

Figure 11. Nitrogen adsorption/desorption isotherms of KOH activated carbon.

Carbon is basically microporous with a developed surface area of 2070 $m^2 g^{-1}$ and a high micropore volume (V_{micro} = 0.8 $cm^3 g^{-1}$) but rich in mesopores. External surface area connected with mesopores was 374 $m^2 g^{-1}$, hence, carbon was adapted for relatively big size of ions.

Two phosphonium salts $[(C_6H_{13})_3P(C_{14}H_{29})]$ $[Tf_2N]$ and $[(C_6H_{13})_3P(C_{14}H_{29})]$ $[(CN)_2N]$ products of CYTEC Canada Inc. have been used as electrolytes components.[21] IL with (trifluoromethylsulfonyl)imide, *i.e.* (Tf_2N) anion is called here IL1 whereas IL with dicyanamide $(CN)_2N$ anion stands for IL2. The semi-structural drawings of both ILs are shown in Figure 12 whereas the structural formula of trihexyl-(tetradecyl) phosphonium cation is seen in Figure 13.

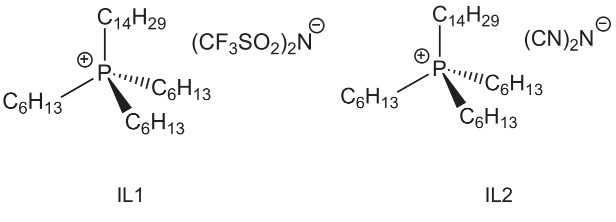

Figure 12. Semistructural drawings of two phosphonium salts with the same cation $[(C_6H_{13})_3P(C_{14}H_{29})]$ but different anions $[Tf_2N]$ for IL1- left and $[(CN)_2N]$ for IL2 - right.

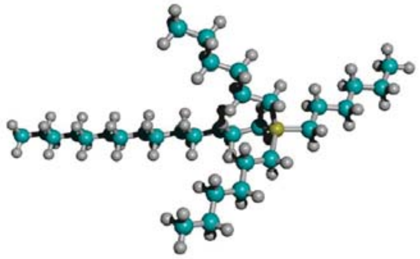

Figure 13. Structure of trihexyl(tetradecyl) phosphonium cation $[(C_6H_{13})_3P(C_{14}H_{29})]$.

Preliminary capacitance measurements of activated carbon have been performed with pure IL1 and IL2, however, due to an extremely high viscosity, the results were not satisfactory. The characteristics of the capacitors were strongly resistive, and the capacitance values quite low $C < 15$ F g^{-1}. A viscous liquid cannot easily penetrate into the microporous bulk of the electrodes. During the next step, we tried to decrease the viscosity of ILs by preparing a mixture of IL with acetonitrile.[21] The decrease of the equivalent series resistance (ESR) of capacitors built from AC and three different electrolytic compositions was confirmed by impedance spectroscopy. A drastic improvement of capacitor performance could be realized by the application of IL1 adding 17 wt% and 25 wt% of acetonitrile (AN). The further capacitor investigations were performed only with mixtures of ILs and AN.

The voltammetry experiments clearly proved the benefit of applying the new composition of ionic liquid in comparison with the conventional organic electrolyte used industrially ($TEABF_4$ in AN) as well as with an aqueous acidic solution (1 mol L^{-1} H_2SO_4). In all three capacitors, the same carbon material has been used for the preparation of the electrodes with comparable mass. The operating voltage of the supercapacitor could be significantly increased to 3.4 V in the case of the IL1/AN 25% electrolyte (Figure 14). It is noteworthy that the capacitor based on IL2 with the same amount of AN could be charged/discharged only to 3.0 V. The voltammetry curve for the IL1/AN 25% electrolyte preserves quite good rectangular shape at 5 mV s^{-1}. It is well visible that the different size of cation and anion of IL determines certain irregular shape of CV. It means that a suitable matching of pores size of carbon for the positive and negative electrodes with ILs ions size can still bring a further improvement of the capacitor performance. The voltammetry experiments at various voltage scan rate (2-50 mV s^{-1}) have been also performed in order to predict some limitations of this electrolyte. A scan rate of 50 mV s^{-1} was rather too quick for this electrolyte, however, 10- 20 mV s^{-1} is still acceptable.

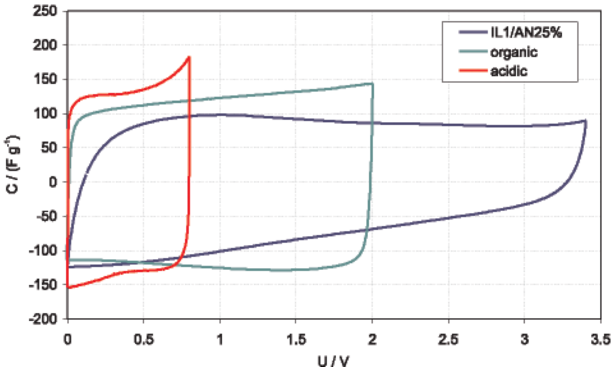

Figure 14. Voltammetry characteristics (5mV s^{-1}) for the activated carbon with IL1/AN 25%, organic and acidic electrolytic solutions.

Supercapacitor based on activated carbon (AC) as electrodes and IL1 with 25 wt% of acetonitrile supplied capacitance values of 100 F g^{-1} at a high operating voltage of 3.4 V. Such supercapacitor reached a high energy of *ca.* 40 Wh kg^{-1} and a good cyclability.

The dependence of capacitance measured from galvanostatic charge/discharge at different current loads of a supercapacitor built from AC using various ILs compositions was measured and the electrolyte with the composition IL1/AN 25% shows the best performance. The capacitor based on activated carbon and this electrolyte could be charged and discharged even at high current load of 2 A g^{-1} (*ca.* 20 mA cm^{-2}) for which the capacitance values are equal to 40 F g^{-1}.

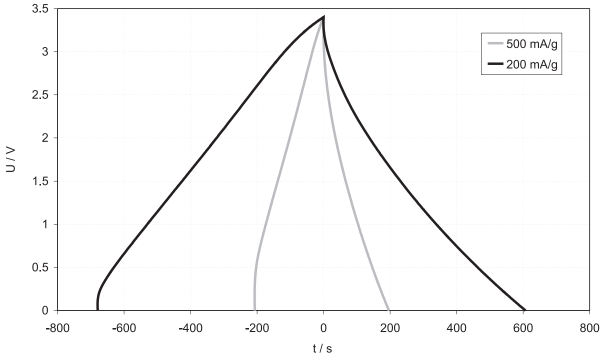

Figure 15. Galvanostatic characteristics of supercapacitor built from AC and IL1/AN 25%.

Taking into account the high operating voltage (3.4 V), we calculated the energy and power for all types of capacitor in order to establish a Ragone plot (Figure 16). For this, the data from galvanostatic charge/discharge characteristics at the current loads from 50 mA g^{-1} to 2 A g^{-1} were considered. The voltage values were taken without ohmic drop and the mass of both electrodes was used in the calculation. The Ragone plot includes also the values of energy and power obtained for the carbon AC operating in the same experimental conditions but with the acidic and conventional organic electrolyte (1mol L^{-1} $TEABF_4$ in AN). The energy of the capacitor in the electrolyte IL1/AN 25% (*ca.* 40 Wh kg^{-1}) is high and very promising for the practical application.

The cyclability of a capacitor built from activated carbon AC operating with IL1/AN 25% at 200 mA g^{-1} (*ca.* 2 mA cm^{-2}) current load was tested. Stable values of capacitance (*ca.* 70 F g^{-1}) were easily maintained over 1000 cycles. By contrast, a different electrolytic composition (IL1/AN 17%) pointed out a gradual aggravation of stability with cycling, whereas IL2 with 25% of AN supplied stable but low capacitance values (*ca.* 25 F g^{-1}).

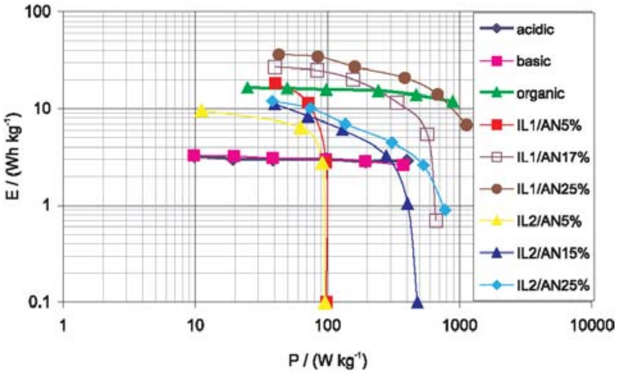

Figure 16. Energy versus power (Ragone plot) for the capacitor built from the same activated carbon but with different type of electrolyte (Adapted from Reference 21).

It seems that ILs are promising electrolyte for supercapacitors but the further studies are demanded to decrease viscosity, increase of conductivity and selection of optimal choice of cation and anion.

6. Conclusions

New trends of preparation of carbon materials for supercapacitor application have been presented. Especially template technique even if quite expensive it appears to be a perfect method to prepare the model carbons for studying the effect of pores size and their connectivity on the supercapacitor performance. The optimal pore size adapted to ions size and interconnectivity between pores are crucial for charge accumulation and ions mobility. It seems that non-solvated ions can easily take part in charging of electrical double layer.

Carbon reach in heteroatoms, e.g. nitrogen and/or oxygen gives useful pseudocapacitance effects. They might be a promising alternative for the development of high performance supercapacitors. It is noteworthy that in this case a moderate porosity is sufficient for the perfect capacitor performance. Additionally, a useful volumetric capacity can be significantly improved.

Ionic liquids are presently considered as a novel generation of electrolyte with interesting properties, e.g. a high operation voltage. A small additive of acetonitrile improved significantly supercapacitor characteristics due to decrease of ILs viscosity, however, acetonitrile is not optimal for ecological reasons. On the other hand, it seems that ILs application in enhanced temperatures are highly recommended because it overcomes their viscosity, hence, additive of solvent is not necessary. Higher temperature (40-60 °C) allows to better penetrate the bulk of carbon electrode. In practical applications, especially for transportation systems, temperatures higher than ambient are expected.

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