Nanostructured Carbons: Double-Layer Capacitance and More

by Patrice Simon and Andrew Burke

This article is concerned with the design and characteristics of electrochemical double layer capacitors (EDLCs), and in particular the role of nanostructured carbons and how they function. EDLCs have been under development for over 20 years, but it is only relatively recently that the mechanisms of energy storage in carbon film electrodes have been studied in depth and the relationships between the carbon properties and specific capacitance (F/gm) have been quantified. The first section of this article is devoted to a discussion of the double-layer energy storage mechanisms and several important recent findings that begin to explain the relationship between the pore characteristics of the carbon and specific capacitance. The later sections of the article are concerned with the present characteristics (energy density and power capability) of EDLCs using nanostructured carbons and what further improvements in device performance are likely in the future.

Nanostructured Carbons

Charge Storage Mechanism.—Electrochemical double layer capacitors (EDLCs) are electrochemical capacitors (ECs) using carbon as active materials. They store energy at the electrolyte/carbon interface through reversible ion adsorption onto the carbon surface, thus charging the so called "double-layer capacitance;" no faradaic (redox) reaction is involved in the charge storage mechanism. This capacitance initially introduced by Helmholtz and later refined by Stern and Geary[1] can be described according to Eq. 1

$$C = \varepsilon A/d \qquad (1)$$

where ε is the electrolyte dielectric constant, A the surface area accessible to ions, and d the distance between the center of the ion and the carbon surface. Double layer capacitance for carbon materials in liquid electrolytes is in the range of 5 and 20 $\mu F/cm^2$, depending on the electrolyte. To increase the charge stored, it is necessary to increase the carbon surface area; this is achieved using different carbon precursors as well as different synthesis techniques. Surface area is generally increased by the development of porosity in the bulk of carbon materials. Unfortunately, there is no simple linear relationship between the surface area and the capacitance.[2] Indeed, more than the total porous volume, it is the way this porosity is created—*i.e.*, control of the pore size as well as pore size distribution—that has a great impact on the carbon capacitance. This will be discussed in a later section ("Capacitance vs. Pore Size").

Types of Nanocarbons Used for EDLCs.—Many different carbon forms can be used as active materials in EDLC electrodes and only a few examples, which are thought to be the most representative, are given below. A recent and exhaustive review has been recently published by Pandolfo, *et al.* and that paper can be consulted for more detailed information.[3]

Activated carbon powders.—Activated carbons (ACs) are the most widely used active materials for EDLC applications, because of their high surface area and relatively low cost. As in the case of batteries, materials cost is a limiting factor for EDLC applications, thus restricting the use of expensive synthesis processes and precursors. ACs are derived from carbon-rich organic precursors by heat treatment in inert atmosphere (a carbonization process). Activated carbons can be obtained from natural sources such as fruit shells like coconut shells, and from wood, pitch, coke, or from synthetic precursors such as selected polymers. High surface area is achieved through what is called the "activation process" consisting in a partial, controlled oxidation of the carbon precursor grains; physical (high temperature treatment in oxidizing atmosphere) or chemical (hot acidic or alkaline oxidation, $ZnCl_2$ process) routes can be used. The activation process leads to the development of a porous network in the bulk of the carbon particles; micropores (< 2 nm), mesopores (between 2 and 50 nm), and macropores (larger than 50 nm size) are randomly created (Fig. 1).

Specific surface area (SSA) as high as 3000 m²/g can be achieved; however, practical useable values are in the range of 1000-2000 m²/ g. Activated carbon powders are processed into active material films by mixing with carbon blacks and organic binders; films are coated onto metallic current collectors to obtain the electrode. Most of the today's commercial devices use activated carbon-based electrodes in organic electrolytes with cell operating voltage up to 2.7 V. In such electrolytes, activated carbon capacitance reaches about 100 F/g and 50 F/cm³; in aqueous electrolytes, it can be increased up to 200 F/g but the cell voltage is limited to 0.9 V.

The pore size distribution in most activated carbon materials is not optimum because of poor pore size control in the activation process. The result is that the high surface area of the carbon cannot be fully exploited to form the double-layer.

Activated carbon fabrics.—As compared to powders, activated carbon fabrics do not require any binder addition and can be directly used as active material films. Electrical conductivity is high (200-1000 S/cm). Activated carbon fabrics are obtained from polymeric fibers such as rayon and polyacrylonitrile for example.[4] Once activated, the surface area is comparable to activated carbons, in the range of 1000-2000 m²/g, most of the porosity being developed in the mesoporous range. However, the cost of these materials is high, restricting their use in EDLCs to very specific applications.

Carbon nanotubes.—As is the case for batteries, electrochemical capacitors have been identified as a potential promising application for carbon nanotubes (CNTs). CNTs are produced from the catalytic decomposition of hydrocarbons. Depending on the synthesis parameters, single wall (SWCNTs) as well as multi-wall carbon nanotubes (MWCNTs) can be prepared, combining both fully accessible external surface area and very high electrical conductivity. However, specific capacitance values achieved with purified CNT powders are not impressive—from 20 up to 80 F/g.[3,5]
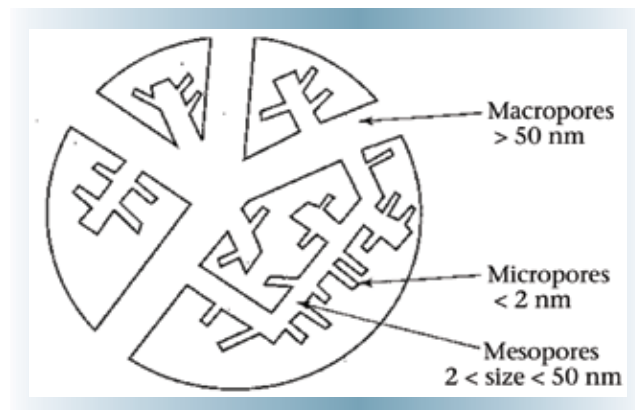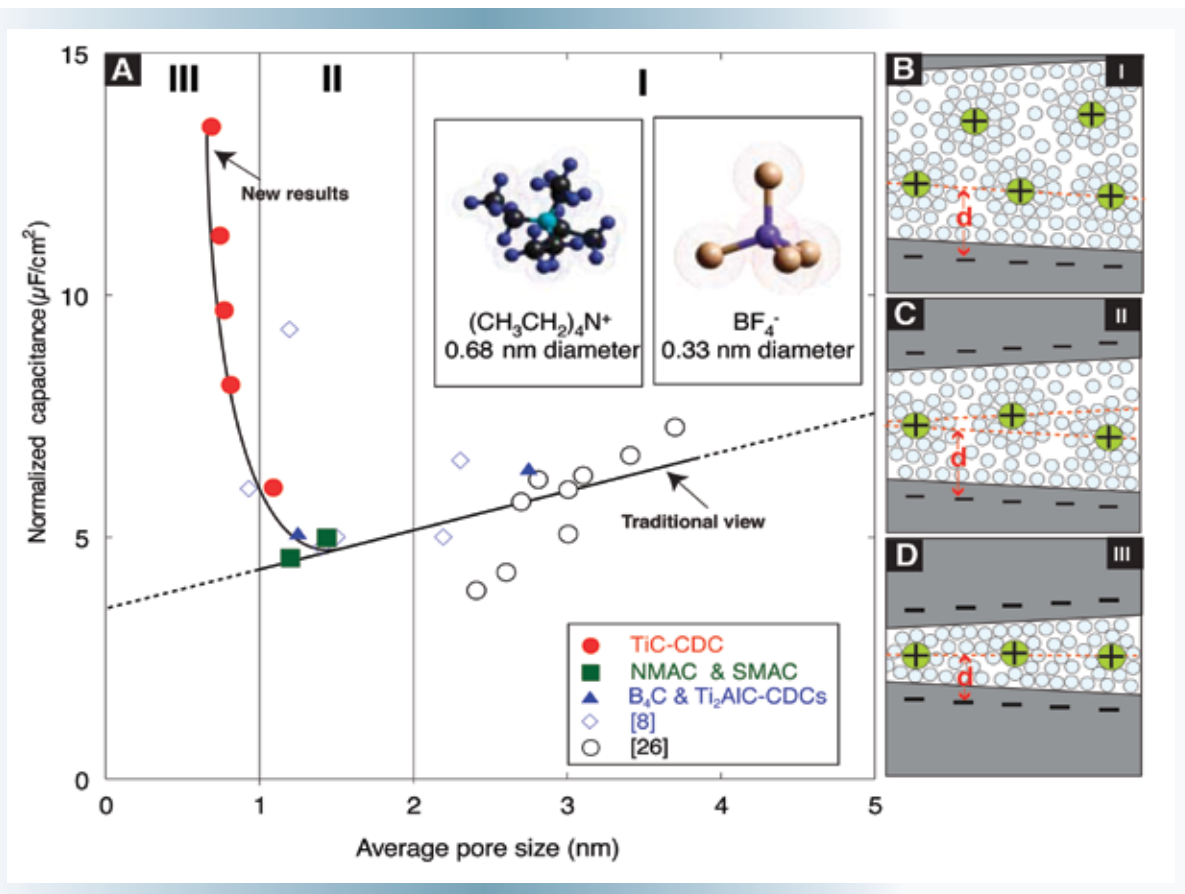

FIG. 1. *Schematic diagram of the pore size network of an activated carbon grain.*

Fig. 2. *Normalized capacitance vs. average pore size for TiC-CDC and other carbons from literature tested in the same electrolytes.*[13,16] *(From Ref. 15.)*

This has been mainly attributed to the hydrophobic property of CNT surface. Surface functionalization by introducing pseudocapacitance contribution through oxidation treatments leads to significant improvement of the specific capacitive behavior; however, cycling life tends to be limited in this case. Today, many efforts are focused on the development of a dense, nano-ordered, aligned CNT forest perpendicular to the current collector that could help to increase the capacitance by fine-tuning the inter-tube distance. Such CNT-based nano-architectured electrodes appear to be promising mainly for microelectronics applications.[5,6]

Carbon aerogels.—Carbon aerogels are prepared from the sol-gel route, for example, by the poly-condensation reaction of resorcinol and formaldehyde. A pyrolysis treatment in an inert atmosphere leads to the formation of a porous carbon aerogel with a controlled and uniform mesoporous structure (pore size between 2 and 50 nm), and a high electrical conductivity (several S/cm). Specific surface areas are in the range of 400-900 m²/g, *i.e.*, lower than the activated carbon ones. Due to this ordered and interconnected pore structure, power capabilities of carbon aerogel-based electrodes are generally high.[3] However, specific gravimetric capacitance in the range of 50 and 100 F/ g for organic and aqueous electrolytes, respectively, have

been reported in the literature, thus limiting the useable energy density of these materials.

Capacitance vs. Pore Size.—The specific capacitance of carbons has a linear dependence on surface area for low surface area, but it tends rapidly to a plateau[2,7,8] when SSA is further increased. As a consequence, the way in which the surface is developed has a great impact on the value of specific capacitance achieved. One of the key issues in designing nanostructured carbons for EDLC applications is then the understanding of the relationship between the electrolyte ion size and the carbon pore size. In other words: what is the best pore size to achieve the highest specific capacitance?

Due to the poor pore size control offered by the traditional activation processes, many recent studies have focused on the design of fine-tuned porous carbons for increasing the specific capacitance using different strategies. In this regard, the template synthesis route has appeared to be an efficient way to create controlled porosity in the mesoporous range (2 to 10 nm)[9-11] needed to maximize the capacitance, according to the long-held axiom that pores of about twice the solvated ion size were needed to optimize the charge storage.[3] Basically, the template process consists in filling the pores of an inorganic template host

matrix—the template—with a carbon precursor. After carbonization, the template is removed by acidic treatment and the carbon obtained a pore size as a mirror image of the template. However, specific capacitance values measured with such templated carbons, even if slightly higher than standard activated carbons in some cases, did not show a large increase—about 200 F/g in sulfuric acid and 100 F/g in organic electrolytes.[12] At the same time, some authors pointed out that, surprisingly, high specific capacitance could be obtained with microporous carbons. This suggested that even the small micropores (size less than 2 nm) contribute to the charge storage mechanism.[8,13,14]

The demonstration of charge storage in pores smaller than the size of solvated electrolyte ions has been recently made by using carbide-derived carbons (CDCs).[15] These materials, having a unique pore size distribution with a mean value that is tunable with better than 0.05 nm accuracy, have been used as model compounds to study ion adsorption in pore size between 0.6 and 1.1 nm. Figure 2 shows the specific capacitance normalized by BET SSA *vs.* CDC pore size, obtained in acetonitrile electrolyte containing 1.5M NEt₄BF₄ salt.[15]

The normalized capacitance decreases with decreasing pore size until a critical value is reached, unlike the traditional view, which assumed that capacitance

would continually decrease. Pores smaller than 1 nm greatly contribute to the double layer charge storage even if the size of the solvated ions is larger. This capacitance increase for pore size < 1 nm was explained by the distortion of the ion solvation shell, leading to a closer approach of the ion to the carbon surface, which by Eq.1 leads to improved capacitance. These results are particularly interesting, as they should permit the design of the next generation of high-energy and density EDLCs, by fine tuning the carbon porosity and designing carbon materials with a large volume of narrow but short pores.

Capacitor Design, Testing, and Performance

Electrode Design Considerations.— As indicated in Fig. 3, the construction of an electrochemical capacitor is much like that of a battery in that it consists of two active layers with a separator between them wetted with an electrolyte.

The electrodes in the capacitors are in general thin coatings applied to a metallic current collector. The active material is mixed with a binder to form a slurry that can be applied at a controlled thickness, rolled, and dried to form the thin, porous electrode. The thickness of the electrode is generally in the range of 100-300 microns and it has a high porosity of 65-75%. In order to achieve a low resistance, the contact resistance between the active material coating and the current collector must

be very low. This requires special attention to preparing the surface of the current collector before applying the electrode coating.[17] The performance of an electrochemical capacitor is simply related to the characteristics of the electrode material and the electrolyte used in the device. The relationship for the energy density (Wh/kg) can be expressed as

$$Wh/kg = 1/8 \ (F/g) \times V_0^2/3.6 \qquad (2)$$

where F/g is the specific capacitance of the electrode material and V_0 is the

cell voltage dependent primarily on the electrolyte used in the device. The weight of the materials in the cell, other than carbon, are neglected in Eq. 2. The power characteristics of a cell are proportional to V_0^2/R where R is the DC resistance of the device. Estimation of the resistance of a cell, including the contribution of ion diffusion in the pores and the effects of current transients in the electrodes, is not simple. However, a first approximation for the resistance can be written as
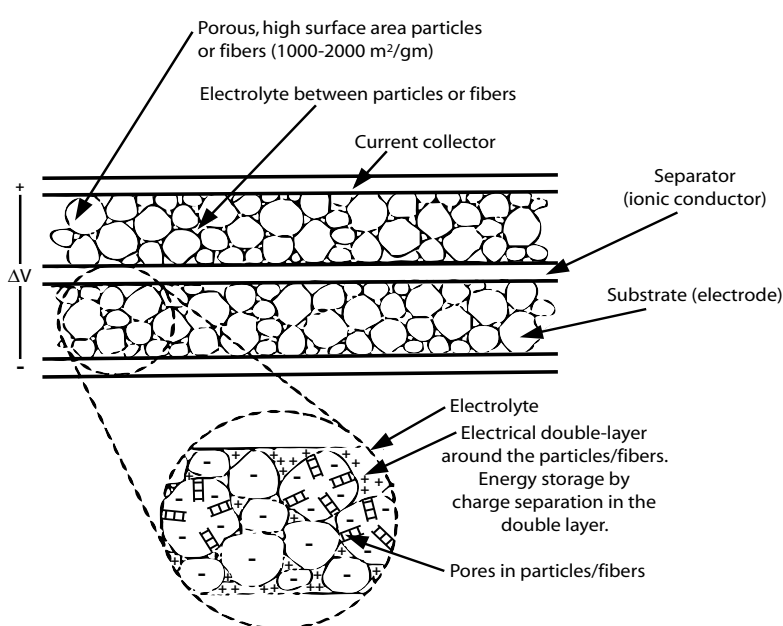
$$R = 2/3 \ t \times r'/A_x \qquad (3)$$

Table I. Properties of various materials used in electrochemical capacitor electrode materials.				
Material	**Density (g/cm³)**	**Electrolyte**	**F/g**	**F/cm³**
Carbon cloth	.35	KOH Organic	200 100	70 35
Activated carbon	0.7	KOH Organic	160 100	112 70
Aerogel carbon	0.6	KOH Organic	50-75 100-125	84
Particulate carbon from SiC	0.7	KOH Organic	175 100	122 70
Particulate carbon from TiC	0.5	KOH Organic	220 120	110 60
Anhydrous RuO₂	2.7	Sulfuric acid	150	405
Hydrous RuO₂	2.0	Sulfuric acid	650	1300
Doped conducting polymer	0.7	Organic	450	315

Properties of various electrolytes.			
Electrolyte	**Density (gm/cm³)**	**Resistivity (Ohm-cm)**	**Cell Voltage**
KOH	1.29	1.9	1.0
Sulfuric acid	1.2	1.35	1.0
Propylene carbonate	1.2	52	2.5-3.0
acetonitrile	.78	18	2.5-3.0
Ionic liquid	1.3-1.5	125 (25°C) 28 (100°C)	4.0 3.25

Porous, high surface area particles or fibers (1000-2000 m²/gm)

Electrolyte between particles or fibers

Current collector

Separator (ionic conductor)

Substrate (electrode)

Electrolyte

Electrical double-layer around the particles/fibers. Energy storage by charge separation in the double layer.

Pores in particles/fibers

Fig. 3. *Schematic diagram of an electrochemical capacitor.*

where "t" is the electrode thickness, "r´" is the resistivity (Ohm-cm) of the electrolyte, and A_x is the geometric area of the electrode. Equations 2 and 3 show the effect of key geometric parameters and material properties on cell performance. Given the internal dimensions of a device and the characteristics of electrode materials and electrolytes (Table I), its performance can be estimated with reasonable accuracy using Eqs. 2 and 3.

Testing Electrochemical Capacitors.—There are similarities and differences in the test procedures for electrochemical capacitors and high power batteries. It is customary to perform constant current and constant power tests of both types of devices as well as pulse power tests.[18] From the constant current tests, the charge capacity (capacitance in Farads) and resistance of the devices are determined. A typical constant current charge/discharge for a carbon/carbon capacitor is shown in Fig. 4.

From the constant power tests, the energy storage characteristics (Wh/kg vs. W/kg—the Ragone curve) are determined. The currents and powers to be used in the testing are selected such that the charge and discharge times are compatible with the capabilities of the devices. In the case of the capacitors, the charge/discharge times are usually in the range of 5-60 seconds. The pulse power test cycles[18,19] consisting of a sequence of short charge and discharge pulses (power density for a specified time) are meant to simulate how the devices would be used in particular applications. The relative performance of the electrochemical capacitors and power batteries can be directly assessed from the pulse power tests since both types of devices are capable of high power pulses even though the battery is not intended to be charged/discharged for long periods at these high power levels. The pulse testing of the battery is usually done at a specified partial state-of-charge as only a small fraction of the energy stored in the battery is used in the cycle tests. Of particular interest in the pulse tests is the round-trip efficiency of the devices. This is determined from the ratio of the energy out compared to the energy into the devices during the test cycle. This efficiency depends primarily by the resistance of the device and is higher for electrochemical capacitors than batteries.

Performance Characteristics Using Activated Carbons.—There are presently commercially available carbon/carbon devices (single cells and modules) using activated carbon from a number of companies.[20] These devices are suitable for high power industrial and vehicle applications. The performance of the various devices is given in Table II. The energy densities (Wh/kg) shown correspond to the useable energy from the devices based on constant power discharge tests from V_0 to ½ V_0. Peak
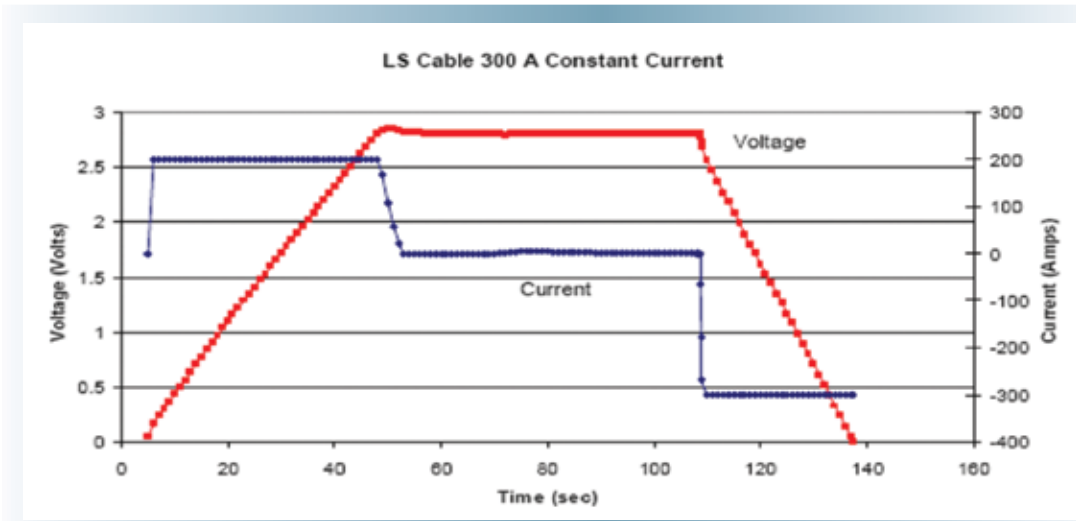

Fig. 4. *Voltage vs. time for a carbon/carbon capacitor.*

Device	V rated	C (F)	R (mOhm)	RC (sec)	Wh/kg (1)	W/kg (95%) (2)	W/kg Match. Imped.	Wgt. (kg)	Vol lit.
Maxwell*	2.7	2800	.48	1.4	4.45	900	8000	.475	.320
Apowercap**	2.7	590	.9	.53	5.0	2618	23275	.087	.062
Ness	2.7	1800	.55	1.00	3.6	975	8674	.38	.277
Ness	2.7	5085	.24	1.22	4.3	958	8532	.89	.712
Asahi Glass (PC)	2.7	1375	2.5	3.4	4.9	390	3471	.210 (estimated)	.151
Panasonic (PC)	2.5	1200	1.0	1.2	2.3	514	4596	.34	.245
LS Cable	2.8	3200	.25	.80	3.7	1400	12400	.63	.47
BatScap	2.7	2680	.20	.54	4.2	2050	18225	.50	.572
Power Sys. (PC)**	2.7	1350	1.5	2.0	4.9	650	5785	.21	.151

(1) Energy density at 400 W/kg contstant power, Vrated - 1/2 Vrated
(2) Power based on P = 9/16*(1-EF)*V2/R, EF=efficiency of discharge
* Except where noted, all the devices use acetonitrile as the electrolyte

* all devices except those with ** are packaged in metal containers
PC: Propylene Carbonate-based electrolyte

power densities are given for both matched impedance and 95% efficiency pulses. For most applications with electrochemical capacitors, the high efficiency power density is the appropriate measure of the power capability of the device. For the large devices, the energy density for the available devices is between 3.5-4.5 Wh/kg and the 95% power density is between 800-1200 W/kg.

Future Advanced Developments Using Carbon Electrodes

Continued research on electrochemical capacitors using carbon electrodes can be expected to improve their performance. These improvements will result from the utilization of better materials, more optimum design, and changes in assembly techniques. In the case of carbon/carbon devices, the primary improvements will be the use of carbon with higher specific capacitance (F/g), a cell voltage of at least 3 V, and thinner electrodes and current collectors. It seems likely that the useable energy density of commercially available capacitors using activated carbon in both electrodes (double-layer storage devices) will be increased from about 4 Wh/kg to 5-6 Wh/kg in the near future. Estimating the improvement likely for non-double-layer electrochemical capacitors (hybrid devices and those using advanced carbons) is more uncertain than for the double-layer devices.

For the advanced devices, an analytical method[21] was used to calculate the performance characteristics of devices for specified device geometry and assumed material properties of the electrodes and electrolyte. The results of the calculations are given in Table III. Also shown in the table are calculated results for carbon/carbon devices using both sulfuric acid and acetonitrile as the electrolyte. All the advanced capacitor devices have higher energy density than the carbon/carbon devices and comparable power capability. Note from Table III that the devices using aqueous electrolytes, especially sulfuric acid, have higher power capability than comparable devices using an organic electrolyte. This results from the much lower ionic resistivity of the aqueous electrolytes. The major uncertainties regarding the advanced devices are their cycle and calendar life. It is highly unlikely that the devices using battery-like electrodes will have a cycle life comparable to the activated carbon, double-layer devices (greater than 500K cycles) for deep discharges. However, a cycle life of 100K deep cycles seems possible by proper design of the battery-like electrode. For some applications, this reduced cycle life would be sufficient.

Conclusions

The characteristics of an EDLC are highly dependent on the nanostructure of the carbon used to form its thin film electrodes—that is, the average pore size and its distribution. The pore characteristics are closely related to

the precursor material and the way it is processed to form the microporous carbon used in the capacitor. The key macroscopic property of the carbon is its specific capacitance (F/gm and F/cm^3). As discussed in this article, recent advances have been made in understanding the relationships between the pore characteristics and specific capacitance and how those pore size requirements depend on the electrolyte used in the device—that is the size of the ions being transported into the micropores of the carbon. Hence the tailoring of the properties of the carbon to meet the special needs of the EDLCs can be expected to significantly increase the useable energy density of future devices from the present values of 4-5 Wh/kg to at least 6-8 Wh/kg. Further increases in energy density of EDLCs are projected if a nanostructured carbon electrode is combined with an electrode material having pseudocapacitance or surface faradaic charge storage properties. ■

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Table III. Projected characteristics of advanced electrochemical capacitors using carbon electrodes.

Type	V	Wh/kg*	Wh/L	Ohm-cm^{2}**	kW/kg 95% eff.
C/PbO$_2$/ Sulfuric acid	2.25-1.0	15.7	39.2	.12	8.9
C/NiOOH/KOH	1.6-0.6	13.9	31.5	.16	4.0
Li$_4$Ti$_5$O$_{12}$/Graphitic Carbon/ Organic	3.7-2.5	50	90	1.7	7.0
Li$_4$Ti$_5$O$_{12}$/AC Carbon/Organic	2.8-1.6	18	29	1.8	4.5
AC Carbon/ AC Carbon/Sulfuric acid	1.0-0	1.7	2.2	.17	1.2
AC Carbon/ AC Carbon/organic	2.7-1.35	5.7	7.6	.78	6.4
AC Carbon/ Graphitic carbon/organic	3.4-1.8	18	30	2.0	1.8

* unpackaged-active materials only
** neglecting contact and pore resistances

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