Different micro-mesoporous silicon carbide-derived carbons (SiC-CDC) were synthesized via gas phase chlorination at 1100 °C and thereafter activated at 900 °C and 1000 °C with H2O steam using Ar and CO2 as the carrier gases. The physical characterization data show that these materials are mainly amorphous, the structure does not change remarkably during the activation process and the surface chemistry of the differently activated and treated materials remains the same and there are no functional groups at the Si-C-D-C surface, N2, Ar and CO2 sorption measurements indicate an increase in the specific surface area and pore size distribution with increasing the activation temperature, whereas the influence of the carrier gas during synthesis is minimal. Although the specific surface areas and pore size distributions differed, the electrochemical parameters in 1 M (C2H5)3CH3NBF4 acetonitrile solution for all SiC-CDC materials were similar - specific gravimetric capacitances 130 ± 18 Fg\(^{-1}\) and volumetric capacitance 67 ± 14 Fcm\(^{-3}\) were calculated. Absolute phase angle values from ~85° to ~88° at low frequencies and very high energy and power densities 22 Wh kg\(^{-1}\) at 20 kW kg\(^{-1}\) and 12 Wh dm\(^{-3}\) at 10 kW dm\(^{-3}\) have been achieved.

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sorption experiments were performed at the boiling temperature of liquid nitrogen (−195.8 °C) using the 3Flex system (Micromeritics). In addition to nitrogen, sorption measurements were also performed with argon and carbon dioxide in order to see if there are any functional groups on the surface interacting differently with different gases (N₂ or Ar) and to get a better estimation of smaller pores with CO₂ testing gas. The sorption measurements with Ar (at −186.15 °C) and CO₂ (at 9.85 °C) were performed using the ASAP 2020 system (Micromeritics). N₂ and Ar are mostly suitable in case of micro-mesoporous materials with pore width \( d \approx 0.7 \text{ nm} \), providing reliable information on the surface available for charge storage in case of typical porous carbons. For materials with high proportion of ultramicropores, CO₂ adsorption has to be implemented for better estimation due to the fact that CO₂ can more easily access ultramicropores.\(^5\,^6\,^14\)

The specific surface area, \( S_{\text{BET}} \), and other parameters for the carbon materials were calculated according to Brunauer-Emmett-Teller (BET) theory (Table II).\(^5\,^6\,^15\,^16\) The total volume of pores (\( V_{\text{tot}} \)) was obtained at the conditions near to saturation pressure \( p/p_0 = 0.97 \). The micropore area (\( S_{\text{micro}} \)) and micropore volume (\( V_{\text{micro}} \)) were calculated using the t-potential method. The pore size distributions have been calculated applying non-local density functional theory (NLDFT) to nitrogen adsorption isotherms within the relative pressure range from 10\(^−1\) to 0.95 with new program Solution of Adsorption Integral Equation Using Splines (SAIEUS, Micromeritics).\(^5\,^13\) Compared to previously introduced modifications of NLDFT, the current application provides smoother and more realistic shape of pore size distribution for different amorphous micro-mesoporous carbon materials successfully taking into account the surface heterogeneity of CDC carbons.\(^18\)

**Electrochemical measurements.**—The EDLC electrodes were composed of an aluminum current collector and a mixture of the activated carbon materials with 4% binder (PTFE, 60% dispersion in H₂O). This mixture was laminated and roll-pressed (HS-160N, Hohsen Corporation, Japan) together to form a flexible layer of the active electrode material with thickness 100 ± 3 \text{ μm}. After drying under vacuum, the pure Al layer (2 \text{ μm}) was deposited onto one side of the CDC by the magnetron sputtering method.\(^19\,^21\) The electrolyte used was prepared from pure acetonitrile (AN, H₂O < 20 ppm), and from dry (C\(_2\)H\(_3\))\(_3\)CH\(_2\)NBF\(_4\) (Stella Chemifa). The two–electrode standard Al test cell (HS Test Cell, Hohsen Corporation) with two identical electrodes (geometric area of about 2.0 cm\(^2\)) was completed inside a glove box (Labmaster sp, MBraun; O₂ and H₂O concentrations lower than 0.1 ppm) and all electrochemical experiments were carried out at temperature \( T = 20 \degree \text{ C} \). The 25 \text{ μm} thick TF4425 (Nippon Kodoshi) separator sheet was used for mechanical separation of the working electrodes. Electrochemical characteristics of the EDLCs containing 1 M (C\(_2\)H\(_3\))\(_3\)CH\(_2\)NBF\(_4\) acetoniitrile solution have been studied by the cyclic voltammetry (CV), constant current charge/discharge (CC/CD) and the electrochemical impedance spectroscopy methods (EIS), using a SI1287 Solartron potentiostat and 1252 A frequency response analyzer over ac frequency \( (f) \) range from 1 mHz to 300 kHz at 5 mV modulation. The constant power (CP) method (using a BT2000 testing system Arbin Instruments, USA) has been used for obtaining the experimental Ragone plots.

**Results and Discussion**

**Physical characterization data.**—The XRD patterns (Fig. 1a) of the activated carbon materials show low intensity reflections, specifically (002) at 20°−26° and (100)/(101) at 43°, which refer to small graphitic areas but no significant long-range ordering in these materials. The (002) diffraction peak characterizes the existence of the parallel graphene layers, while the (100)/(101) peak is associated with the 2D in-plane symmetry along the graphite layers.\(^22\) The XRD patterns for the different materials under study in this paper are almost identical, which indicates that no structural changes take place during the H₂O steam activation process.

The Raman spectra for the synthesized materials (Fig. 1b) are very similar to the CO₂ activated materials spectra demonstrated in previous work.\(^12\) The spectra are characteristic of disordered amorphous carbon, demonstrating mainly two peaks in first order excitation region: the so-called graphite (G) peak at ∼1577 cm\(^−1\) and the disorder-induced (D) peak at ∼1388 cm\(^−1\). The G-peak corresponds to the graphite in-plane bond-stretching motion of the pairs of C atoms in sp\(^2\) configuration with E\(_2g\) symmetry. Thus, this mode does not require the presence of sixfold aromatic C rings, and it occurs at all sp\(^2\) sites, not only for those atoms located in the hexagonal aromatic carbon structure. The D-peak is a breathing mode with A\(_1g\) symmetry, which is forbidden in perfect graphite and only becomes active in the presence of disorder in the graphite structure.\(^23\) The spectrum also shows the second-order peak of D-band (2D) at ∼2670 cm\(^−1\). The increase of the 2D peak intensities can be related with the crystallographic ordering of the graphitic structure.\(^24\) The XRD and Raman data for 900° C and 1000° C SiC-CDC confirm that no significant structural changes take place and the difference between the materials activated at 900° C

**Table I.** The synthesized materials with their abbreviations and properties.

| Abbreviation | \( \text{H₂O} \) (%) | Activation time (h) | Carrier gas | Activation temperature (°C) | Activation burn-off (%) | \( \text{H₂} \) treated |
|--------------|---------------------|--------------------|-------------|-----------------------------|------------------------|-----------------------|
| St-900       | 47                  | 2                  | Ar          | 900                         | 15                     | No                    |
| St-900-H2    |                     |                    |             |                             |                        |                       |
| St-1000      | 15                  | 2                  | Ar          | 1000                        | 50                     | Yes                   |
| St-1000-H2   |                     |                    |             |                             |                        |                       |
| St-CO2-900   | 10                  | 2                  | CO₂         | 900                         | 19                     | Yes                   |
| St-1000-H2   |                     |                    |             |                             |                        |                       |
| St-CO2-900-H2| 60                  | 2                  | CO₂         | 1000                        | 60                     | Yes                   |
| St-CO2-1000  |                     |                    |             |                             |                        |                       |
| St-CO2-1000-H2|                   |                    |             |                             |                        |                       |

**Table II.** Results of the sorption measurements for the synthesized materials.

| Abbreviation | \( S_{\text{BET}} \) (m\(^2\)g\(^−1\)) | \( S_{\text{micro}} \) (m\(^2\)g\(^−1\)) | \( V_{\text{micro}} \) (cm\(^3\)g\(^−1\)) | \( V_{\text{tot}} \) (cm\(^3\)g\(^−1\)) |
|--------------|----------------------------------------|----------------------------------------|----------------------------------------|----------------------------------------|
| St-900       | 1700                                   | 1570                                   | 0.77                                   | 0.96                                   |
| St-900-H2    | 1720                                   | 1620                                   | 0.73                                   | 0.87                                   |
| St-1000      | 2410                                   | 2260                                   | 1.09                                   | 1.32                                   |
| St-1000-H2   | 2290                                   | 2180                                   | 1.00                                   | 1.35                                   |
| St-CO2-900   | 1670                                   | 1600                                   | 0.72                                   | 0.82                                   |
| St-CO2-900-H2| 1750                                   | 1660                                   | 0.74                                   | 0.88                                   |
| St-CO2-1000  | 2610                                   | 2470                                   | 1.22                                   | 1.44                                   |
| St-CO2-1000-H2|                                    | 2270                                   | 1.20                                   | 1.32                                   |
Figure 1. (a) Characteristic XRD patterns and (b) Raman spectra for initial SiC-CDC 1100 and corresponding data for H2O steam activated materials with different activation parameters, given in Table I.

and 1000°C is very minimal, showing at 1000°C only a slightly more ordered structure.

IR spectroscopy spectra are shown in Fig. 2a. It can be seen that all the materials have the same IR peaks and there is only a slight difference in the peak intensities. The same tendencies can also be seen on the XPS spectra (Fig. 2b). Therefore the results of both methods confirm that the surface chemistry of the differently activated (H2O or H2O+CO2) and treated materials remains the same. It can be explained by the activation conditions – after activation the materials have been cooled down in an Ar atmosphere starting from high temperatures, where the oxygen containing functional groups are removed and the surface stays mostly free of them. Otherwise, there should be observed a different surface chemistry between the H2 treated versus non-H2 treated materials.

According to the data in the Table II and Fig. 3a the materials have mainly microporous structure. The H2 treated materials are not separately shown in the figures, because they were almost identical to the non-H2 treated versions, i.e. the H2 treatment has a very minimal influence on the specific surface area and pore size distribution. The specific surface area and pore volumes are similar or slightly higher compared to the previously studied CO2 activated materials.12 The pore size distributions show that the materials are mainly microporous, but a small increase in mesoporous area with increasing the activation temperature from 900°C to 1000°C, can be observed. The comparison of sorption measurements data using N2, Ar and CO2 is shown in Fig. 3b. As expected, the data for CO2 measurement shows a bigger peak below 1 nm, i.e. for the smallest pores, whereas Ar and N2 show more peaks within a bit wider range (Fig. 3b). These tendencies were same for all the material studied in this work. Also there is small dependence of the peak position on the gas used, which indicates that there might be specific adsorption interactions between the used gas and the carbon surface. However, the pore size distribution plots shape depends on the model used for fitting the adsorption data. Nevertheless, the comparison of N2, Ar and CO2 adsorption confirms that the difference between H2 treated and H2-untreated materials is negligible.

Cyclic voltammetry data.—The cyclic voltammetry (CV) curves expressed as specific gravimetric capacitance (Cm) and volumetric capacitance (Cv) vs. cell potential (∆E) are presented in Figs. 4a–4f. The CVs were measured within various electrode potential regions at potential scan rates (ν) from 1 to 500 mV s⁻¹. In addition to the activated materials prepared and tested in this work, data for the initial un-activated carbon, denoted as SiC-CDC 1100 and the CO2 activated material (with the best electrochemical properties) denoted as SiC-CDC 1100 A3, from our previous work, are shown for comparison.12

The current density, j (obtained using the flat-cross section geometric surface area), measured at fixed scan rate has been used for...
calculation of the medium capacitance values according to Eq. 1:

\[ C = j^n. \]  

Eq. 1 is exactly correct if the capacitance, \( C \), is constant (\( C \neq f(\Delta E) \)) and the series resistance \( R_s \rightarrow 0 \), if \( j \rightarrow 0 \). Thus, Eq. 1 can be used to obtain the capacitance values only within the region of slow \( v \) if the values of current are small, as the ohmic potential drop, i.e. IR–drop, is negligible only under these conditions and the current response is essentially equal to that of a pure capacitor.\(^{25,26}\) In a symmetrical two–electrode system the specific gravimetric capacitance \( C_m \) (F g\(^{-1}\)) for one activated \( \nu \) carbon electrode can be obtained as follows:

\[ C_m = \frac{2C}{m}. \]  

where \( m \) is the weight in g cm\(^{-2}\) per one activated carbon electrode, assuming that the positively and negatively charged electrodes have the same capacitance at fixed \( \Delta E \). The volume of electrodes has been used for calculation of the volumetric capacitance values (Figs. 4c and 4f).

All the activated carbon materials have nearly mirror image symmetry of the current responses about the zero current line, obtained at potential scan rates \( v \leq 500 \) mV s\(^{-1}\) and \( \Delta E \leq 3.0 \) V (Figs. 4a–4f). Measuring up to 3.4 V cell potential the materials continue showing quite good capacitive behavior (Fig. 4e). Although the materials differ in their activation and treatment parameters and because of that also in their specific surface areas, their CV curves are very similar and for all the materials differ quite good capacitive behavior (Fig. 4e).

Figure 3. (a) Differential pore size distribution vs. pore width plots obtained using the SAIEUS method\(^{16,17}\) and (inset) nitrogen sorption isotherms for initial SiC-CDC 1100 and for H\(_2\)O steam activated materials (noted in Table I) and (b) differential pore size distribution vs. pore width plots obtained using the SAIEUS method\(^{16,17}\) for different gases (N\(_2\), Ar, CO\(_2\)).
Figure 4. Cyclic voltammograms expressed as specific gravimetric (a, b, d, e) and volumetric capacitance (c, f) vs. cell potential dependencies for untreated SiC-CDC 1100, the CO2 activated material denoted as SiC-CDC 1100 A3, and H2O steam activated materials (noted in Table I) at potential scan rate 5 mV s\(^{-1}\) (a), at potential scan rate 50 mV s\(^{-1}\) (b), (volumetric capacitance) at potential scan rate 5 mV s\(^{-1}\) (c), at potential scan rate 500 mV s\(^{-1}\) (d) and for St-1000 at different potential scan rates specific gravimetric capacitance (e) and volumetric capacitance (f).

A small semicircle at higher \(ac\) frequencies \((f > 300 \text{ Hz})\). Based on systematical analysis of EDLC data, it is known that the semicircle shape depends on the adsorption kinetics of ions at the porous carbon electrode and on the series resistance of a material and mass transfer resistance inside a macroporous carbon structure. In addition, the very high frequency semi-circle can also be caused by the artifacts of the measurement system as well as by formation of a passive layer, covering the (positively charged) aluminum current collector, which introduces so-called electrostatic capacitor in series with the two series-connected double layer capacitors. In the initial SiC-CDC 1100 (untreated) EDLCs system, with very high microporosity, but without mesopores in the carbon particles, the mass transfer limited processes prevail at \(f = 1 \text{ mHz}\) and there is no ideal capacitive behavior even at \(f = 1 \text{ mHz}\).12

The specific series capacitance values, \(C_m\), calculated from the \(Z'\) vs. \(Z''\) plots at \(ac\) frequency \(f = 1 \text{ mHz}\) are shown in Table III and Fig. 6b. The obtained \(C_m\) values are in the range from 123 \(\text{F g}^{-1}\) to 141 \(\text{F g}^{-1}\), which is slightly higher than the values obtained for the CO\(_2\) activated materials.12 The \(C_m\) values are in a good agreement with the values obtained using CV and CC methods. Data in Fig. 6c show the phase angle \(\theta\) vs \(ac\) frequency dependencies. The phase angle values for the steam activated materials are
Table III. Calculated specific capacitance (gravimetric and volumetric) values from cyclic voltammetry ($\Delta E = 3.0$ V; $v = 5$ mV s$^{-1}$), constant current ($\Delta E = 3.0$ V; $j = 1$ A g$^{-1}$) and impedance spectroscopy measurements ($\Delta E = 3.0$ V; $f = 1$ mHz).

| Electrode material | Cyclic voltammetry | Constant current | Impedance spectroscopy |
|--------------------|--------------------|------------------|------------------------|
|                    | ($F$ g$^{-1}$)     | ($F$ cm$^{-3}$)  | ($F$ g$^{-1}$)         | ($F$ cm$^{-3}$)         |
| St-900             | 134                | 77               | 116                    | 67                     |
| St-900-H2          | 126                | 65               | 112                    | 57                     |
| St-1000            | 142                | 81               | 118                    | 67                     |
| St-1000-H2         | 138                | 71               | 118                    | 60                     |
| St-CO2-900         | 125                | 73               | 117                    | 69                     |
| St-CO2-900-H2      | 132                | 70               | 113                    | 61                     |
| St-CO2-1000        | 124                | 64               | 115                    | 60                     |
| St-CO2-1000-H2     | 130                | 57               | 115                    | 59                     |

Figure 5. Constant current charge/discharge data at current density $j = 0.1$ A g$^{-1}$ (a) and at current density $j = 1$ A g$^{-1}$ (b) for H$_2$O steam activated materials (noted in Table I).

Figure 6. (a) Nyquist plot at cell potential $\Delta E = 3.0$ V, (b) specific gravimetric series capacitance $C_m$ and (c) volumetric series capacitance vs. ac frequency dependencies and (d) phase angle vs. ac frequency plots for untreated SiC-CDC 1100, the CO$_2$ activated material denoted as SiC-CDC 1100 A312 and H$_2$O steam activated materials (noted in Table I).
Figure 7. (a, b) Gravimetric Ragone plots and (c, d) volumetric Ragone plots for untreated SiC-CDC 1100, the CO2 activated material denoted as SiC-CDC 1100 A312 and H2O steam activated materials (noted in Table I), respectively, for the EDLCs completed using CDC electrodes in 1 M (C2H5)3CH3NBF4 acetonitrile solution, obtained from constant power tests within the cell potential range from 1.5 V to 3.0 V (a, c) as well as from 1.7 V to 3.4 V (b, d).

within the range from $-85^\circ$ to $-87^\circ$ ($f > 5$ mHz). Small drop in the absolute phase angle value at low frequencies (to $-80^\circ$ at $f = 1$ mHz) can be caused by some water residues and other electrochemically active components in the system, as well as by the non-equilibrium adsorption of ions into (ultra)micropores. Nevertheless the obtained values are again somewhat higher from these established for the CO2 activated systems results.

**Ragone plots.**—The gravimetric (Figs. 7a, 7b) and volumetric (Figs. 7c, 7d) Ragone plots (specific energy, $E$, vs. specific power, $P$, dependencies)28,33 calculated to the total material weight (except the mass of cases) or volume of two electrodes have been obtained from constant power test within the cell potential range from 1.5 V to 3.0 V (Figs. 7a, 7c) as well as from 1.7 V to 3.4 V (Figs. 7b, 7d). All the activated materials show again quite similar results and behavior. The Ragone plots were compared also with the CO2 activated materials previously studied. Confirming the results from other methods here again, very high energy densities at high power densities have been observed, however independent of activation temperature and method applied. It is interesting to mention, that there is no difference between H2 treated and H2 non-treated materials. Thus, steam activation and Ar treatment simplifies the synthesis of SiC-CDC materials noticeably.

**Conclusions**

Different micro-mesoporous carbide-derived carbons (CDC) were synthesized from SiC powders via gas phase chlorination at 1100 °C and thereafter activated at 900 °C and 1000 °C with steam using Ar and CO2 as the carrier gases. The physical characterization data from XRD and Raman spectroscopy show that these materials are mainly amorphous, but containing small graphitic crystallites. However, the crystallographic structure does not change remarkably during the steam activation process at 900 °C and 1000 °C. IR spectroscopy and XPS data confirm that the surface chemistry of the differently activated and treated materials remains the same and there are no functional groups at the SiC-CDC surface, due to the treatment at high temperature in Ar atmosphere. The low-temperature N2, Ar and CO2 sorption measurements indicate an increase in the specific surface area and pore size distribution with increasing the activation temperature from 900 °C to 1000 °C, whereas the influence of the carrier gas during synthesis (Ar vs. CO2) is minimal.

The electrochemical parameters were obtained using cyclic voltammetry, constant current charge/discharge, electrochemical impedance spectroscopy and constant power discharge methods. It has been suggested earlier that a large specific surface area of carbons is the most important parameter leading to a high capacitance values. However, the results of this paper clearly demonstrated that although the specific surface area increases significantly and also the pore size distribution expands, it has a very weak influence on the electrochemical parameters, which are quite similar for all the steam activated materials – specific gravimetric capacitances $130 \pm 18$ F g$^{-1}$ or volumetric capacitances $67 \pm 14$ F cm$^{-3}$. Very high absolute phase angle values from $-85^\circ$ to $-88^\circ$ at low frequencies have been calculated indicating nearly ideally polarizable systems.

Very high energy and power densities (22 Wh kg$^{-1}$ at 20 kW kg$^{-1}$ and 12 Wh dm$^{-3}$ at 10 kW dm$^{-3}$) have been calculated for SiC-CDC steam activated materials based EDLCs.

However the comparison of current data with CO2 activated materials based EDLCs from previous work shows that the steam activation is more effective, giving slightly better results with a shorter activation time. This can be explained by the higher reactivity of steam at high temperatures. The small differences between the results of steam
activation and the steam + CO₂ activation shows that at higher temperatures both CO₂ and H₂O compete for the same carbon reaction (active) sites.

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