Aqueous Synthesis of Ordered Mesoporous Carbon via Self-Assembly Catalyzed by Amino Acid

An-Hui Lu,* Bernd Spliethoff, and Ferdi Schüth

Max-Planck-Institut für Kohlenforschung, D-45470 Mülheim an der Ruhr, Germany

Received February 4, 2008. Revised Manuscript Received June 2, 2008

In this manuscript, we demonstrate a synthesis of ordered mesoporous carbon via a self-assembly of resorcinol-formaldehyde polymer and surfactant F127 in aqueous phase in the presence of glutamic acid as the catalyst. It was found that the amounts of glutamic acid and surfactant F127 in the synthesis are important in determining the structural ordering of the resultant mesoporous carbons. The textural parameters of the obtained ordered mesoporous carbon are tunable simply by varying the pyrolysis temperature. The process has the advantage of being a single step self-assembly approach and only involves the use of simple organic precursors and water.

1. Introduction

Structurally ordered mesoporous carbon materials exhibit high surface area, large pore size, and electronic conductivity that make them interesting for applications in catalysis, adsorption, and energy storage/conversion devices.[1–3] The first synthesis of ordered mesoporous carbon was achieved in 1999 via the nanocasting route, that is, using ordered mesoporous silica (MCM-48) as the hard template, the pores of which were impregnated with a carbon precursor, followed by pyrolysis and template removal.[4] Following the nanocasting strategy, various ordered/structured mesoporous carbon materials have been synthesized, based on the high variety of mesoporous silica structures, which are usually synthesized through the cooperative assembly of inorganic precursors and amphiphilic surfactants, involving either a hydrothermal or an evaporation-induced self-assembly (EISA) pathway.[5–7] The nanocasting pathway is a powerful tool for the creation of nanostructured materials. However, it is also associated with severe disadvantages, such as the need to prepare the hard template (porous silica) and possibly incompatibility between the template removal method and the materials which should be cast.

Thus, in parallel to nanocasting, also self-assembly methods were pursued to widen the range of materials accessible via this technique. As a result of these efforts, the synthesis of ordered mesoporous carbon directly synthesized via the use of surfactant as the structure-directing agent has been reported, similar to the synthesis of ordered mesoporous silica. As a successful example, Dai et al. developed a stepwise assembly approach to prepare well-ordered mesoporous carbon films.[8] The carbon precursor molecules are spatially arranged into well-defined nanostructures by the self-assembly of block copolymer (polystyrene-*block*-poly(4-vinylpyridine). A hexagonally packed carbon-channel array whose orientation is normal to the carbon film surface has been prepared. As an alternative approach, Tanaka et al. have used a spin coating method to deposit an ordered carbon film from resorcinol-formaldehyde (RF), block copolymer Pluronic F127, and triethylorthoacetate (EOA) on a silicon substrate.[9] The synthesis involves the use of EOA as the carbon coprecursor in addition to RF which was dissolved in water and ethanol media in the presence of HCl as the acid catalyst. In this synthesis, EOA plays a crucial role in obtaining the well-ordered mesostructure. Ordered mesoporous polymers and carbon frameworks with the 2D hexagonal (*p*6*mm*), 3D cubic (*Ia*3*d* and Im3*m*), and lamellar structures have also been synthesized using prepolymerized phenol-formaldehyde as the carbon precursor and triblock copolymer F127, P123, and F108 as structure-directing agents via EISA in alcohol phase under nearly neutral conditions or in aqueous phase under basic conditions.[10–13] Highly ordered mesoporous carbons with a cubic structure of *Im*3*m* symmetry have been obtained in a basic medium of ethanol solution via the EISA pathway where Pluronic F108 was used as a structure-directing agent and RF sol as a carbon precursor.[14]

---

* Corresponding author. E-mail: lu@mpi-muelheim.mpg.de. Fax: +49-0208 306 2995.

(1) Taguchi, A.; Schüth, F. *Microporous Mesoporous Mater.* **2005**, *77*, 1.
(2) Schüth, F. *Angew. Chem., Int. Ed.* **2003**, *42*, 3604.
(3) de A., A.; Soler-Illia, G. J.; Sanchez, C.; Lebeau, B.; Patarin, J. *Chem. Rev.* **2002**, *102*, 4093.
(4) Ryoo, R.; Joo, S. H.; Jun, S. *J. Phys. Chem. B* **1999**, *103*, 7743.
(5) Ying, J. Y.; Mehnert, C.; Wong, M. S. *Angew. Chem., Int. Ed.* **1999**, *38*, 56.
(6) Lu, A.-H.; Schüth, F. *Adv. Mater.* **2006**, *18*, 1793.
(7) Yang, H.; Zhao, D. *J. Mater. Chem.* **2005**, *15*, 1217.
(8) Liang, C. D.; Hong, K. L.; Guiochon, G. A.; Mays, J. W.; Dai, S. *Angew. Chem., Int. Ed.* **2004**, *43*, 5785.
(9) Tanaka, S.; Nishiyama, N.; Egashira, Y.; Ueyama, K. *Chem. Commun.* **2005**, 2125.
(10) Meng, Y.; Gu, D.; Zhang, F.; Shi, Y.; Yang, H.; Li, Z.; Yu, C.; Tu, B.; Zhao, D. *Angew. Chem., Int. Ed.* **2005**, *44*, 7053.
(11) Zhang, F.; Meng, Y.; Gu, D.; Yan, Y.; Yu, C.; Tu, B.; Zhao, D. *J. Am. Chem. Soc.* **2005**, *127*, 13508.
(12) Huang, Y.; Cai, H.; Yu, T.; Zhang, F.; Zhang, F.; Meng, Y.; Gu, D.; Wan, Y.; Sun, X.; Tu, B.; Zhao, D. *Angew. Chem., Int. Ed.* **2007**, *46*, 1089.
(13) Wan, Y.; Shi, Y.; Zhao, D. *Chem. Mater.* **2008**, *20*, 932.
(14) Liu, C.; Li, L.; Song, H.; Chen, X. *Chem. Commun.* **2007**, 757.

10.1021/cm800362g CCC: $40.75

The above-mentioned syntheses of ordered mesoporous carbon mostly involve ethanol as the organic solvent, and the mesoporous carbons are always obtained in the form of films by the evaporation induced self-assembly method. Although such a direct synthesis of ordered mesoporous carbon is a great improvement compared to the nanocasting pathway, it is difficult to quantitatively synthesize ordered mesoporous carbon by the EISA method which involves the evaporation of large amounts of organic solvent. In the following we report a solution to this problem, that is, a facile one-pot synthesis approach in the aqueous phase under mild reaction conditions toward the synthesis of ordered mesoporous carbon via self-assembly. The crucial difference to previous work is the presence of an amino acid as the catalyst which catalyzes both the formation of the resorcinol-formaldehyde resin and the assembly of the mesostructure. The synthesis involves the use of F127 as the structure-directing agent, glutamic acid as the catalyst, and resorcinol and formaldehyde as the carbon precursors; the pH of the reaction solution is controlled at 3. The phase separation occurs within 10 h at 60 °C. At higher reaction temperature, the phase separation period is shortened (for instance, to about 2 h at 80 °C). The solid product, a light yellow glue-like precipitate, can easily be recovered by decanting the aqueous solution. After further aging at 90 °C for 48 h, a glass-like bright red solid is formed. After carbonization of the initial hydrogel, an ordered mesoporous carbon can be obtained. A great advantage of this process is that no metal or halogenide ions are involved, and only hydrocarbon and water are present in the reaction system. This results in the formation of a high purity carbon, which could be beneficial for some applications.

2. Experimental Section

2.1. Synthesis Procedure. In a typical synthesis, 1.0 g of resorcinol (Aldrich), 0.12 g of glutamic acid (Fluka), and 0.8 g of F127 (Aldrich) were dissolved in 100 mL of H_2O at 60 °C under strong stirring. Subsequently, 1.48 g of 37 wt % formaldehyde (Fluka) was added to the above clear solution. After about 10 h, some white-yellow particles were formed through phase separation. To complete the reaction, the solution was maintained at 60 °C for 4 days. Finally, the light yellow glue-like precipitate was collected and dried at 90 °C for 48 h to form a crystal-like solid of bright red color. The composite was pyrolyzed at 350, 500, 700, or 850 °C for 2 h, with a heating rate of 1 °C min^{-1} under an argon atmosphere, to generate the ordered mesoporous carbons. For comparison, the influences of the amount of catalyst and surfactant on the carbon structures were also investigated. Details are described together with the results in the next section.

2.2. Characterization. Thermogravimetic analysis was performed on a NETZSCH STA 449C thermobalance. Low angle X-ray diffraction patterns of samples were recorded with a STADI P diffractometer (Stoe) in the Bragg−Brentano (reflection) geometry. The nitrogen sorption measurements were performed on an ASAP 2010 (Micromeritics). The BET surface area was calculated from the adsorption data in the relative pressure interval from 0.04 to 0.2. Pore size distribution (PSD) curves were calculated by the BJH (Barrett−Joyner−Halenda) method from the adsorption branch. The total pore volume was estimated from the amount adsorbed at a relative pressure of 0.99. The transmission electron microscopy images of the ordered mesoporous carbons were obtained with a Hitachi HF 2000 transmission electron microscope operated at 200 keV. Scanning electron microscopy investigations were carried out with a Hitachi S-3500N instrument equipped with an OXFORD energy dispersive X-ray analysis (EDX) system. The FT-IR spectra of samples were collected on a Magna-IR 750 Nicolet FT-IR spectrometer using an ATR cell. X-ray photoelectron spectra (XPS) were recorded with a Kratos His spectrometer, equipped with a dual anode (Mg/Al) and a monochromatic Al source.

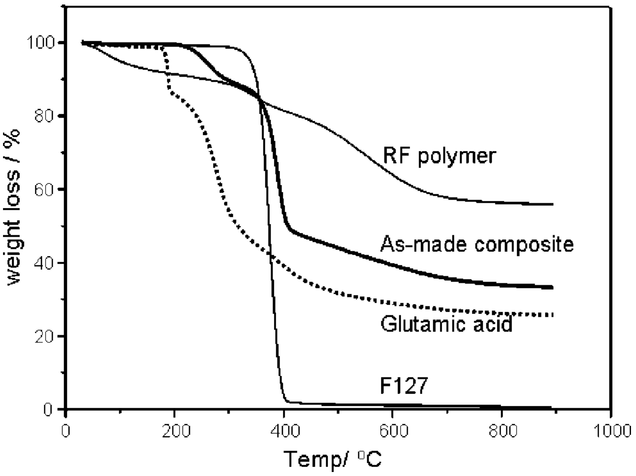

Figure 1. TG analyses of RF polymer, as-made composite, glutamic acid, and F127. Heating rate 3 °C/min to 900 °C under argon.

3. Results and Discussion

The pyrolysis behavior of the as-made composite was monitored by TG at a heating rate of 3 °C/min under argon. For comparison, the reactants, such as F127, glutamic acid, and RF polymer (which was prepared by NaOH as the catalyst and washed intensively), were also analyzed under the same pyrolysis conditions. The TG curves of those samples are compiled in Figure 1. The TG curve of F127 shows a sharp weight loss in the temperature range of 300−400 °C, and F127 can be completely decomposed before 400 °C. Glutamic acid exhibits three pronounced weight loss steps, that is, approximately 14 wt % in the temperature range of 175−190 °C, approximately 32 wt % in the temperature of 190−300 °C, and approximately 20 wt % weight loss in the temperature range of 300−450 °C. After further increasing the pyrolysis temperature, glutamic acid was not decomposed completely, and at the end, approximately 26 wt % carbon residue was left. The RF polymer shows a gradual weight loss up to 700 °C. Over the whole temperature range, the RF polymer undergoes decomposition and condensation reactions,[15] and finally a carbon residue of approximately 56 wt % is left. For the as-made composite, a pronounced weight loss of approximately 11 wt % occurs in the temperature range of 200−300 °C, which can be assigned to the decomposition of the material by release of water and other small molecules. This decomposition stage is probably related to the presence of glutamic acid, since by XPS no nitrogen signal in the as-made composite was detected. In the range of 300−410 °C, the weight loss of the as-made composite is roughly 40 wt %, which is mostly attributed to the decomposition of

---

[15] Pekala, R. W. *J. Mater. Sci.* **1989**, *24*, 3221.

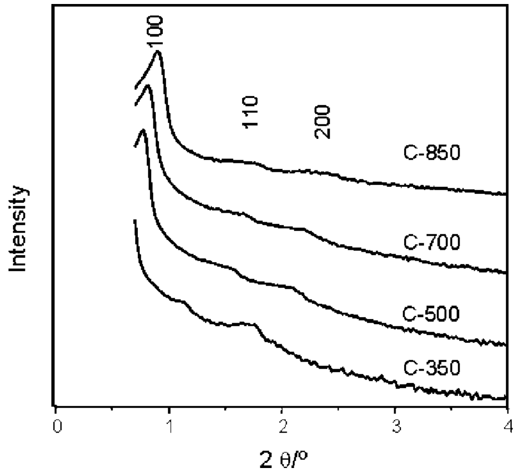

Figure 2. Low angle XRD patterns of ordered mesoporous carbon samples.

F127. When the temperature exceeds 410 °C, further condensation of the formed mesostructure results in approximately 16 wt % weight loss, which is due to release of hydrocarbons, oxygen, and other fragments. According to the information obtained from TG analyses, final pyrolysis temperatures of 350, 500, 700, and 850 °C, mainly corresponding to the decomposition temperature of F127 and RF, were selected to carbonize the as-made composite, in order to trace the pore structure development of the products. The obtained carbons were labeled C-350, C-500, C-700, and C-850, respectively.

Low angle X-ray diffraction (XRD) patterns of the pyrolyzed samples at different temperatures are shown in Figure 2. Basically, all the samples show well-resolved reflections, and the patterns can be assigned to an ordered structure of hexagonal symmetry with *p*6*mm* space group. The as-made composite pyrolyzed at 350 °C only shows (110) and (200) reflections; the (100) reflection is beyond the low angle cutoff of our instrument. With increasing thermal treatment temperature, the corresponding reflections are shifted to higher angles, which is due to the structural shrinkage resulting from pyrolysis. The unit cell parameters of samples C-350, C-500, C-700, and C-800 were calculated to be 15.5, 13.1, 12.4 and 11.3 nm, respectively.

The structures of the as-made composite and the pyrolyzed samples were characterized by TEM analysis. As seen in Figure 3, structural order of the as-made composite is almost impossible to observe, due to the weak contrast between the RF polymer and the surfactant F127. With increasing pyrolysis temperature, the structural regularity of the sample gradually appears, resulting from the increasing contrast between the open pore and the carbon framework, due to the decomposition of the surfactant F127 and condensation of the RF skeleton. Figure 3 shows the sample C-500, having very clear hexagonal structural ordering, and the structures viewed parallel and perpendicular to the pores clearly give evidence for the presence of mesopores and the carbon walls. The mesopore diameter and the pore wall thickness were estimated from the TEM images to be 6.2 and 7.0 nm, respectively. The noncrystallized carbon wall is also contributing to the meso- or microporosity, which can be determined by sorption measurement; see below. The center-to-center distances of adjacent channels are approximately12.7 nm which is almost identical to the distance estimated from the low angle X-ray diffraction pattern. Combining the X-ray pattern and the TEM observation, it can be concluded that porous carbons produced from RF/F127 composites are structurally ordered on the mesoscale.

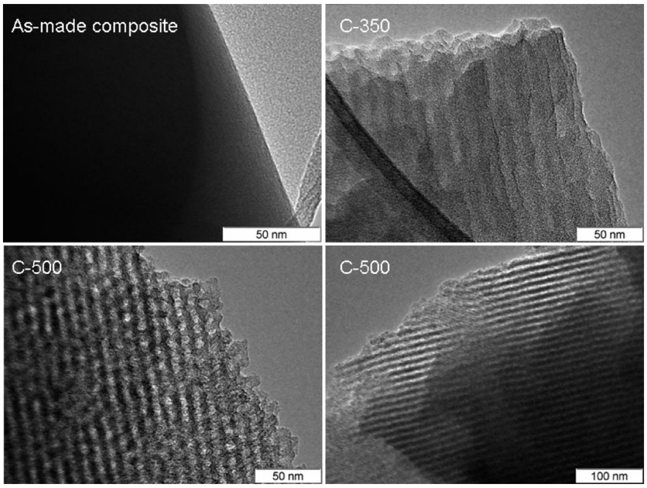

Figure 3. TEM images of as-made composite and samples pyrolyzed at 350 and 500 °C.

The textural properties of the samples were determined by recording their nitrogen sorption isotherms. As seen in Figure 4a, all the isotherms of the carbonized samples are of type IV with a clear hysteresis loop, indicating mesoporous character. Their textural parameters are listed in Table 1. The specific surface area, pore diameter, and volume adsorbed/g are varying, depending on the thermal treatment temperatures. C-350 has the lowest surface area and pore volume, because the framework is still between polymer and carbon. Lower thermal treatment temperature results in less decomposition of the polymer and thus less developed porosity. With increasing pyrolysis temperature, the surfactant F127 decomposes to a large extent, thus giving access to the open porosity. In the same temperature range, also the RF framework starts to decompose to carbon species to generate much more abundant porosity. As seen in Table 1, sample C-500 shows the highest specific surface area and pore volume, that is, 725 m^2/g and 0.63 cm^3/g, respectively. With further increase in pyrolysis temperature, the pore volume shows a clearly decreasing trend, and the specific surface area stays almost constant. This is attributed to the combined effects from the structural shrinkage and further developed porosity by the thermal decomposition of the carbon framework. As seen in Figure 4b, the BJH (Barrett−Joyner−Halenda) pore size distributions of all the samples are very narrow, and the pore sizes of the samples decrease continuously with increasing treatment temperature, from 6.9 nm for C-350 to 5.0 nm for C-850, which is due to the structural shrinkage resulting from the pyrolysis process. This is also consistent with the XRD measurements. (Note: Sample C-850 has the closure of the hysteresis around $p/p_0 = 0.42$, which is due to instability of the meniscus. In this case, using the desorption branch to calculate the pore sizes would lead to an untrue reflection of the pore size distribu-

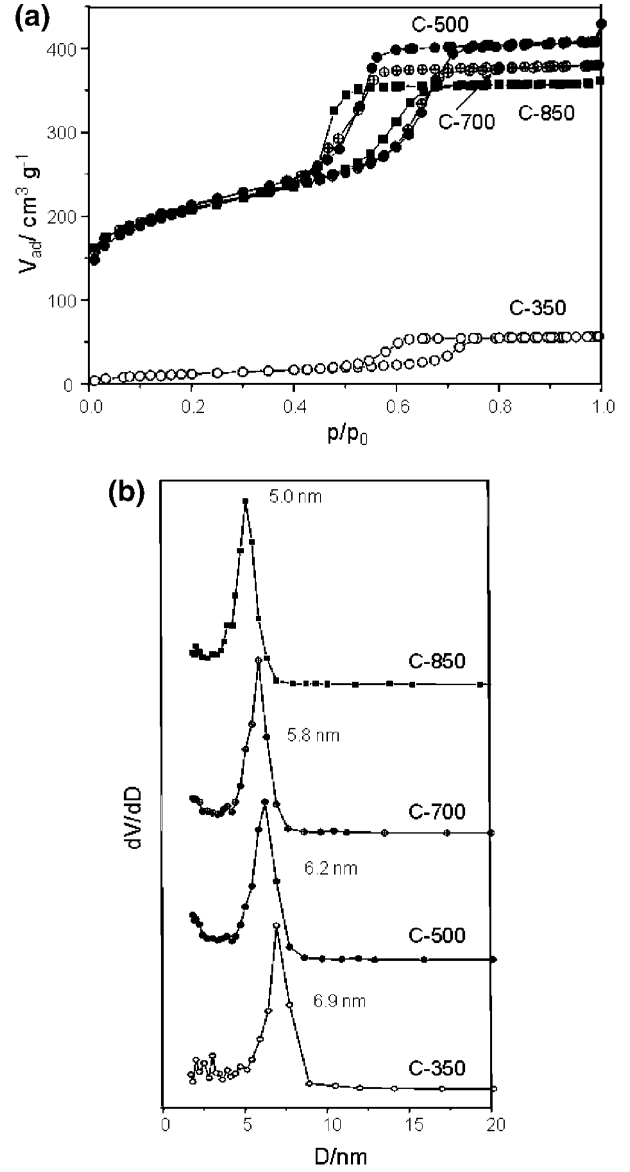

Figure 4. (a) Nitrogen sorption isotherms and (b) pore size distributions of ordered mesoporous carbons

tion.[16] Thus, to avoid the misinterpreting the pore sizes, the adsorption branches were used to determine the pore size distributions of this series samples.)

To the best of our knowledge, it is the first time that amino acid (glutamic acid) was used in the direct synthesis of ordered mesoporous carbon via a self-assembly process. The amount of glutamic acid used influences the carbon structure, as discussed in the following. The amount of glutamic acid was set at 0.06 g ("GL") and 0.360 g ("GH") in the synthesis, while the other parameters were maintained at the values used for the previously discussed sample. The as-synthesized polymers were pyrolyzed at 700 °C, resulting in the carbon products C-GL and C-GH, respectively. The XRD patterns of C-GL and C-GH are shown in Figure 5. It can be seen that the XRD pattern of C-GH displays a reflection at $2\theta = 0.84°$, indicating this sample has certain structural ordering but not as good as that of sample C-700. In contrast to this, the XRD pattern of C-GL displays no detectable reflections, indicating that there is no structural ordering of this sample. These results reveal that the amount of glutamic acid is crucial for determining the quality of the self-assembly structure and in turn the structural ordering of a porous carbon.

As displayed in Figure 6a, the nitrogen sorption isotherms of both samples C-GL and C-GH exhibit type IV shape with a clear capillary condensation step in the relative pressure range 0.4−0.8, indicating that these samples have mesopores. The pore size distributions (Figure 6a, inset) show that sample C-GL contains mesopores with a size of 3−6 nm, and sample C-GH contains mesopores mainly with a pore size at 4.7 nm. By looking at the textural parameters of these two samples compiled in Table 2, one can see that sample C-GL prepared using a lower amount of glutamic acid has relatively high specific surface area, larger pore volume, and broader pore size distribution than sample C-GH prepared using a higher amount of glutamic acid. The aforementioned data clearly reveal that mesoporous carbons can be obtained from the RF/F127/glutamic acid/water reaction system. However, mesoporous carbon, like C-700 with ordered structure, was synthesized only at optimized amounts of glutamic acid. As known, RF polymer can form a rigid three-dimentional network under acidic or basic conditions. Thus, by the conventional polymerization process, it is difficult to form ordered assembly structure of RF/F127, because RF most likely tends to cross-link with itself rather than to assemble around the micelle of F127. Here, in contrast to that, it is likely that the polymerization of RF and the assembly between RF and F127 proceed in a cooperative manner at a certain amount of glutamic acid.

As template, the surfactant determines the final structure of the self-assembled composite under a certain concentration.[17] Here, the influence of the amount of surfactant F127 used on the carbon structure was investigated as well. Carbon products synthesized using 0.4 g and 1.5 g of F127 and pyrolyzed at 700 °C were named as C-FL and C-FH, respectively. As seen in Figure 5, the XRD pattern of sample C-FH prepared using a higher amount of surfactant F127 shows one pronounced reflection at $2\theta = 0.84°$; however, other reflections are not detectable, indicating less structural ordering in this sample. The XRD pattern of C-FL (not shown) prepared using a lower amount of F127 exhibits no detectable reflections, revealing that this sample has a disordered structure. The mesoporous character of these two samples was verified by their nitrogen sorption isotherms, as displayed in Figure 6b, and their textural parameters listed in Table 2. The pore size distributions (in Figure 6b, inset) show that samples C-FH and C-FL have mesopore sizes mainly in the ranges of 3.9−5.8 nm and 5.8−6.9 nm, respectively. In comparison, C-700 shows a more ordered structure and has a much narrower pore size distribution mainly at 5.8 nm, due to the optimized RF to F127 ratio.

In general, the polymerization of resorcinol and formaldehyde can be catalyzed either by acid or base. We found that mesoporous carbons can be synthesized via a self-assembly process, if glutamic acid is used as catalyst. If

---

(16) Kruk, M.; Jaroniec, M. *Chem. Mater.* **2001**, *13*, 3169.

(17) Meng, Y.; Gu, D.; Zhang, F.; Shi, Y.; Cheng, L.; Feng, D.; Wu, Z.; Chen, Z.; Wan, Y.; Stein, A.; Zhao, D. *Chem. Mater.* **2006**, *18*, 4447.

Table 1. Textual Parameters of Ordered Mesoporous Carbons[s]

sample	pyrolysis temp. (°C)	S_{BET} (m^2 g^{-1})	V_{tot} (cm^3 g^{-1})	D_{pore} (nm)	wall thickness (nm)	*a* (nm)
C-350	350	47	0.087	6.9	8.6	15.5
C-500	500	725	0.630	6.2	6.9	13.1
C-700	700	724	0.586	5.8	6.6	12.4
C-850	850	714	0.554	5.0	6.3	11.3

[s] S_{BET}: apparent surface area calculated by BET method. V_{tot}: total pore volume at $p/p_0 = 0.99$. D_{pore}: pore sizes calculated from the adsorption branch. *a*: unit cell parameter. The wall thickness was calculated as the difference between *a* and D_{pore}.

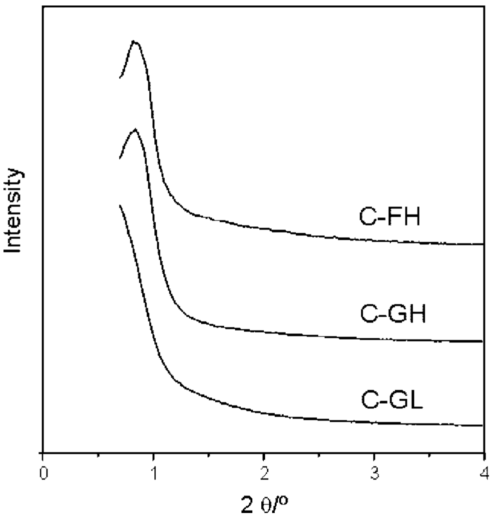

Figure 5. Low angle XRD patterns of ordered mesoporous carbons prepared using different amounts of glutamic acid and F127.

lysine is used instead as basic catalyst, while leaving all other reaction conditions identical as for sample C-700, spherical microporous carbons were produced instead of mesostructured carbon, as can be seen in Figure 7. Neither XRD nor TEM (data not shown) gives any indication of the presence of ordered structures on the mesoscale. The final pH of the solution in the lysine catalyzed reaction was about 3.19, which was measured by CyberScan pH300/310. This level of acidity might be generated by formaldehyde (contains or generates formic acid) and resorcinol in the aqueous solution. Since the pH values of the glutamic acid and the lysine catalyzed reaction system are quite similar, apparently, the glutamic acid plays an important role in initiating the self-assembly between the RF polymer and the surfactant F127. It is likely that glutamic acidic catalyzes the formation of an RF polymer with linear structure instead of a three-dimensional rigid structure catalyzed by lysine. The soft linear RF species obtained with glutamic acid can more easily assemble into the ordered structure under the influence of the F127 template. Under acidic conditions, it is likely that the RF polymer interacts with the polyethylene (PEO) chains of F127 through hydrogen bonding.[18] To obtain more insight in the structural development of the resulting carbons and to verify if glutamic acid molecules are incorporated in the as-made composite, ^{13}C NMR, FT-IR, and XPS were used to characterize this series of samples. The ^{13}C NMR spectrum (see the Supporting Information) of the as-made composite clearly shows the presence of glutamic acid in the as-made polymer composite as revealed by the peaks around 175 and 179 ppm which are attributed to the −COOH group. As seen in Figure 8, by comparing the FTIR spectra of glutamic acid, F127, RF polymer, and the as-made composite, one can conclude that the band around 1730 cm^{-1} present in the as-made composite can be assigned to the C−O stretching vibration of an H-bonded carboxylic acid group, indicating the formation of intermolecular H-bonds between organic molecules. Since the final pH of the reaction system is about 3, the amine group of glutamic acid can be protonated. Thus, the band at 1506 cm^{-1} in the spectra of as-made polymer and glutamic acid can be assigned to the symmetrical NH_3^+ bending. This also indicates that glutamic acid is incorporated into the as-made polymer, in line with the NMR result. With increasing pyrolysis temperature, though the FT-IR spectra of the samples exhibit broad bands, the major features around 1600, 1445, and 1240 cm^{-1}, corresponding to the stretching skeletal vibration of C=C and the stretching vibrations of C−O and C−H, are maintained. The spectrum of C-850 is

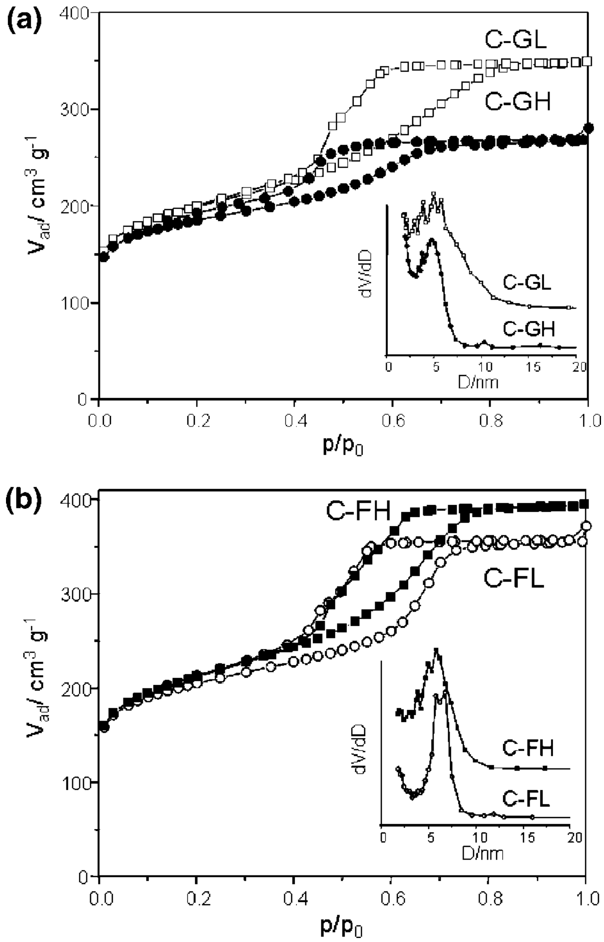

Figure 6. (a) Nitrogen sorption isotherms of carbons prepared using different amounts of glutamic acid and (b) nitrogen sorption isotherms of carbons prepared using different amounts of F127; insets are their pore size distributions.

---

(18) Liang, C.; Dai, S. *J. Am. Chem. Soc.* **2006**, *128*, 5316.

Table 2. Textual Parameters of Ordered Mesoporous Carbons Prepared Using Different Amounts of Glutamic Acid and F127[a]

sample	glutamic acid (g)	F127 (g)	temp (°C)	S_{BET} (m^2 g^{-1})	V_{tot} (cm^3 g^{-1})	D_{pore} (nm)
C-GL	0.060	0.8	60	686	0.538	3.0−6.0
C-GH	0.360	0.8	60	634	0.414	4.7
C-FH	0.120	1.5	60	734	0.608	3.9−5.8
C-FL	0.120	0.4	60	705	0.550	5.8−6.9

[a] Note: Temp is the reaction temperature for the aqueous synthesis. The others are the same as noted in Table 1.

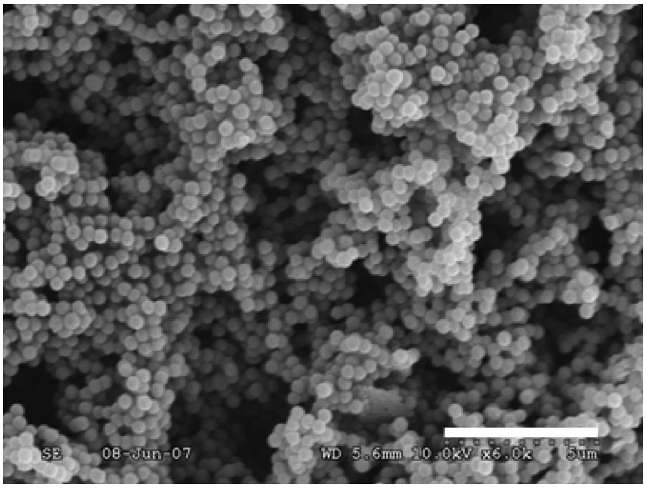

Figure 7. SEM image of carbon spheres prepared by lysine catalyzed polymerization of resorcinol-formaldehyde polymer (scale bar 5 μm).

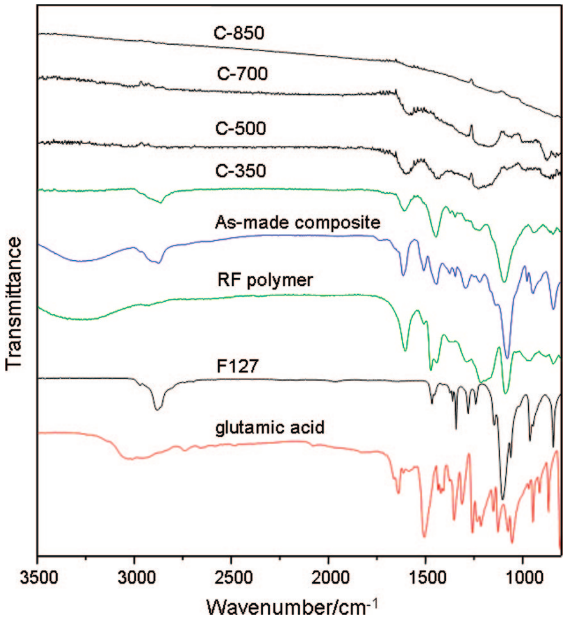

Figure 8. FT-IR spectra of RF polymer, glutamic acid, F127, and as-made composite and the pyrolyzed samples at different temperatures.

rather featureless because the developed carbon framework results in strong adsorption of the IR radiation, so that almost no sample information is obtained.

XPS is a very sensitive technique to characterize the surface of a material. If all the reactants, such as resorcinol, formaldehyde, glutamic acid, and F127, react with each other to form the final polymer composite, the molar percentage of glutamic acid in the composite would be about 4%. To check for nitrogen in the sample, which could only result from the glutamic acid, the as-made composite was ground into a fine powder to expose more surface for the XPS analysis. The XPS spectra (not shown here) do not give any indication for nitrogen in the sample. However, elemental analysis indicates that 1.7 wt % of nitrogen exists in the sample. That indicates that glutamic acid molecules are present in the as-made mesostructures but below the detection limit of XPS on the surfaces. Depth profiling by sputtering with Ar$^+$ ions was attempted, but the composite decomposed under these conditions, severely contaminating the spectrometer. Attempts to detect the nitrogen species by XPS were therefore discontinued. It was also attempted to synthesize a mesostructure without glutamic acid as the catalyst, but without success. Based on the above experiments, we conclude that the glutamic acid on one hand acts as a catalyst to polymerize resorcinol and formaldehyde but on the other hand must initiate the interaction between RF polymer and F127 through hydrogen bonding, resulting in the self-assembly of RF and F127 into a mesostructure. Thus, glutamic acid plays an important role in obtaining ordered mesoporous carbon via self-assembly in the system of resorcinol-formaldehyde, F127, and water.

A number of other acids were studied as well in this reaction under otherwise identical conditions as used for the synthesis of C-700. Use of acetic acid did not result in the formation of an ordered structure. Oxalic acid, on the other hand, led to the precipitation of a material with one low angle reflection in the XRD pattern, indicating the formation of a disordered structure. It can not be excluded that optimization of the synthesis for these alternative acids to also result in the formation of more ordered structures would be possible. However, so far this was not successful, and it could be a rather lengthy process, if successful at all.

4. Conclusion

In this study, the synthesis of ordered mesoporous carbon via a self-assembly approach in aqueous phase in the presence of glutamic acid as the catalyst has been described. The pore structure parameters of the obtained ordered mesoporous carbon are tunable simply by varying the pyrolysis temperature. The process has the advantage of being a single step self-assembly approach and only involves the use of simple organic precursors and water. Moreover, the synthesis can also be easily scaled up to produce ordered mesoporous carbon with monolithic shape in large amounts.

Acknowledgment. The authors would like to thank the basic funding provided by the Institute. We are highly grateful to Mr. Bongard for SEM, Dr. Zibrowius for NMR, and Dr. Weidenthaler for XPS measurements.

Supporting Information Available: TEM, XRD, XPS, and NMR (PDF). This material is available free of charge via the Internet at http: //pubs.acs.org.

CM800362G