Kilogram-scale synthesis of ordered mesoporous carbons and their electrochemical performance

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ABSTRACT

An efficient post-cure approach has been demonstrated for the kilogram-scale synthesis of high-quality ordered mesoporous carbons (OMC) by using triblock copolymer Pluronic F127 as a template, phenolic resol as a carbon precursor and polyurethane foam as a sacrificial scaffold through an organic–organic self-assembly. The effects of the concentration and the loading amount of resol on the mesostructure of the carbons are systematically investigated. The small-angle X-ray scattering, nitrogen sorption and transmission electron microscopy results reveal that the resultant OMC in kilogram-scale quantities possesses high surface area (~690 m² g⁻¹), large pore volume (~0.45 cm³ g⁻¹) and uniform, large pore size (~4.5 nm) as well as thick pore walls (~6.5 nm). The OMC exhibits good electrochemical performance of about 130 F g⁻¹ in KOH electrolyte.

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1. Introduction

Ordered mesoporous carbons (OMC) have attracted much attention not only because of the peculiar structures and unique chemical properties, but also the wide applications for super-capacitor, adsorption and separation, hydrogen storage and lithium batteries [1–5]. Practical application of the OMC is still in an early stage and has been inhibited by its insufficient supply. Therefore, mass production of the OMC is greatly desired before promoting its commercialization and development into high-tech applications.

Typically, two approaches are developed to fabricate the OMC. One is the hard template method [6–10] which is fussy and of a high cost due to tedious synthesis procedures, thus this method is not suitable for industrial production. The other method is the organic-organic self-assembly approach [11–14], which involves the polymerization of carbon precursors around the hydrophilic domains of the soft template by hydrogen bonds under a heat treatment and the decomposition of the template during the carbonization process, realized by solvent-annealing strategy, solvent evaporation induced self-assembly (EISA) strategy or hydrothermal treatment [11,15–17]. Besides, self-assembly approach combining with some sacrificial templates such as anodic alumina membrane and colloidal crystal is an efficient method to synthesize hierarchically porous carbons [18–21]. Recently, an EISA procedure on polyurethane (PU) foam scaffolds for fabricating porous carbons draw more and more attentions. The EISA process is simple and convenient to control for the reproducible and high-yield synthesis of mesoporous carbons except for the required large space vessels [22]. PU foam scaffolds can provide abundant surfaces for solvent evaporation as well as loading carbon precursors. What important is that it can be almost completely decomposed in inert gas [22–24]. These advantages of PU foam endow it a suitable sacrificial scaffold for the OMC synthesis. This strategy overcomes the limitation of necessary interfaces supplied by large space vessels and makes the organic-organic self-assembly approach a feasible route for the OMC mass production. However, the porosities of the carbons fabricated on the PU foam are needed to be...
improved for further applications. Besides, to the best of our knowledge, there have been no reports on the kilogram-scale fabrication of the OMC by using commercial chemicals, which is of great significance for industrial production.

Here, we report a novel post-cure method for the kilogram-scale synthesis of the OMC based on the commercial chemicals. Post-cure by increasing the solidification temperature and time is demonstrated to be an efficient approach for high-quality OMC with well-ordered 2D mesostructure, high surface area (-690 m² g⁻¹), large pore volume (~0.45 cm³ g⁻¹), and uniform pore size (~4.5 nm) as well as thick pore walls (~6.5 nm). A systematic study on the synthesis conditions including the concentration and the loading amount of resol in a broad range is carried out. Furthermore, the electrochemical properties of the OMC used as electrodes of supercapacitors are also exploited.

2. Experimental

2.1. Chemicals

Commercial PU foam was purchased from the Shanghai Gaoxin Foam Factory and its density was 13 kg m⁻³. Commercial phenolic resol (reddish-brown transparent thick liquid, solid content of 50 wt.%, viscosity of 8–10 mPa s) was purchased from the Aerzi Co., Commercial triblock copolymer F127 (EO106PO70EO106, \( M_w = 12600 \)) was purchased from the BASF Co., and commercial ethanol (chemically pure) was purchased from Shanghai Chemical Co. All chemicals were used as received without any further purification.

2.2. Kilogram-scale synthesis of OMC

Kilogram-scale OMC was synthesized by organic-organic self-assembly on the PU foam scaffold through a solvent EISA approach as reported previously [22]. Briefly, 1.0 kg of copolymer F127, 1.0 kg of resol and 1.0 kg of ethanol were mixed and stirred for 3 h at room temperature. The obtained homogeneous solution was coated onto 0.16 kg PU foam. After the ethanol evaporated completely at 25 °C for 8 h, the sample was heated at 100 °C for 24 h and post-cured at 150 °C for 24 h to obtain the as-made sample, and then was carbonized at 700 °C for 3 h under \( N_2 \) to get the carbon denoted as OMC-150. When other post-cure temperatures of 100, 130 and 180 °C were utilized, the resultant carbons were denoted as OMC-100, OMC-130 and OMC-180, respectively. To determine the influence of post-curing, carbon sample without post-cure treatment was also produced, which was denoted as OMC-0. Other synthesis parameters including the concentration and the loading amount of resol were also investigated by fixing the post-cure temperature at 150 °C and varying the weight ratio of copolymer F127, ethanol, resol and PU, the resultant carbons were assigned as OMC-m-n (m represents the resol concentration of resol/ethanol, wt.%; n represents the resol loading amount of resol/foam, g/g). When F127/resol/ethanol/PU (weight ratio) = 1:1:0.125–8:0.625, the obtained carbon materials were assigned as OMC-m-1.6. When F127/resol/ethanol/PU (weight ratio) = 1:1:0.5:0.069–0.625, the obtained carbon materials were assigned as OMC-67-n.

2.3. Measurements

The small-angle X-ray scattering (SAXS) data were collected on a Nanostar U small-angle X-ray scattering system using Cu Kα radiation at 40 kV and 35 mA. The \( d \)-spacing values and unit cell parameters were calculated from the formula \( d = 2\pi q^{-1} \) and \( a = 2d_{100}(\sqrt{3})^{-1} \), respectively. Transmission electron microscopy (TEM) experiments were carried out on a JEOL 2100 microscope operated at 200 kV. The ground samples for TEM measurements were suspended in ethanol and supported onto a carbon-coated copper grid. Scanning electron microscopy (SEM) images were collected with a Philips XL30 electron microscope operated at 20 kV. Field-emission scanning electron microscopy (FE-SEM) images were collected on the Hitachi S-4800 field emission scanning electron microscope and the samples without further gold-spraying treatment were used for the observations. \( N_2 \) sorption isotherms were measured on a Micromeritics Tristar 3000 analyzer at -196 °C. Before the measurements, all samples were degassed at 180 °C in vacuum for at least 6 h. The Brunauer-Emment-Teller (BET) method was utilized to calculate the specific surface areas. The pore size distributions were derived from the adsorption branches of isotherms using the Barrett-Joyner-Halenda (BJH) model. The C, H and N contents were measured on a Vario EL III element analyzer.

2.4. Electrochemical measurements

For preparing a working electrode, a mixture containing 85 wt.% active materials, 10 wt.% acetylene black, and 5 wt.% polytetrafluoroethylene were well mixed and ground together to form homogeneous slurry. The slurry was then pressed onto nickel foam that served as a current collector. The electrochemical experiments were carried out using a three-electrode cell, employing platinum as the counter electrode, \( Hg/HgO \) (0.052 V vs. the normal hydrogen electrode) as the reference electrode and a 6 M KOH solution as the electrolyte. The cyclic voltammetry (CV) and the galvanostatic charge–discharge (GC) tests were performed using electrochemical analyzer, CHI 606B under ambient condition.

3. Results and discussion

3.1. Influences of the post-cure on OMC

The as-made sample without post-cure has one strong and two weak scattering peaks shown in its SAXS pattern (Fig. 1A(a)), ascribed to the 100, 110 and 200 planes of a 2-D hexagonal mesostructure [25]. After carbonization, the obtained OMC-0 also possesses three well-resolved scattering peaks (Fig. 1B(a)), indicating ordered structure. The calculation from SAXS reflects unit cell parameters of 16.3 and 9.8 nm for the as-made sample and OMC-0, respectively, implying serious resin framework shrinkage of ~40% during carbonization. \( N_2 \) sorption isotherms of OMC-0 show type IV isotherms with an un conspicuous hysteresis loop (Fig. 2A(a)), indicating a low mesoporosity. A calculation based on the adsorption branch indicates that only 19% of the BET surface area is derived from mesopores. Besides, a very small
pore size of \( \sim 2.1 \) nm is observed from the pore size distribution (Fig. 2B(a)) and the TEM images (Fig. 3(a and b)). The big framework shrinkage also leads to a thick pore wall of 7.7 nm. The reason of the low-quality may be that the resin framework of the as-made sample is not rigid, thus during carbonization the serious framework shrinkage leads to the low mesoporosity and small pore size.

In order to improve the quality of the OMC, a post-cure is performed for the OMC from 100 to 180 °C [26]. Here, a sample post-cured at 150 °C is taken as an example. Compared with the as-made sample without post-cure, the one with post-cure at 150 °C shows only one clear scattering peak (Fig. 1A(d)), which may be attributed to the low contrast between the surfactant and resin resulted from the increased polymerization of the resin framework. The unit cell parameter calculated from the SAXS measurement is 16.3 nm, which is same as the as-made sample without post-cure. This indicates the post-cure has little effect on the unit cell parameters of the as-made samples. However, after carbonization at 700 °C, more resolved SAXS pattern (Fig. 1B(d)) with four well-resolved scattering peaks indexed as the 100, 110, 200 and 210 planes of a 2-D hexagonal mesostructure can be clearly observed for the resultant OMC-150, compared with OMC-0, indicating a well ordered mesostructure. The calculated unite cell parameter of OMC-150 is 11.1 nm and much larger than that (9.8 nm) of OMC-0. Correspondingly, the shrinkage is only 33% and much smaller than that (40%) of the as-made sample without post-cure. The results suggest that the resin framework is enhanced by the post-cure process and can well resist the framework shrinkage during the carbonization. This proves post-cure can be in favor of the framework stability and promote the mesostructure. Typical type-IV isotherms with H1 hysteresis loop at the evident capillary condensation steps are observed from the N2 sorption isotherms of OMC-150 (Fig. 2A(d)), indicating ordered mesostructure. A narrow pore size distribution (Fig. 2B(d)) with a mean value of \( \sim 4.5 \) nm is calculated from the adsorption branch based on the BJH model. Furthermore, OMC-150 shows a high BET surface area of 687 m² g⁻¹ and 50% of the BET surface area from the mesopores. The calculated pore wall of OMC-150 is 6.6 nm which is smaller than that (7.7 nm) of OMC-0 as a result of a smaller framework shrink-
age. Obviously, the pore size and the mesoporosity are much larger than that of OMC-0. The N2 sorption results indicate that post-cure can greatly improve the mesoporosity as well as the mesoporous structure. TEM measurement was also exploited to reveal the mesostructure of OMC-150. Typical stripe-like and hexagonal arrays viewed from the [0 0 1] and [1 1 0] directions can be clearly observed (Fig. 3(c and d)), indicating ordered hexagonal mesostructures. The lattice parameter and pore size estimated from the TEM images are about 11.0 and 4.5 nm, respectively, in agreement with the SAXS and N2 sorption results. Altogether, these results confirm post-cure can effectively promote the mesoporosity and lead to high-quality OMC.

Other post-cure temperatures were also elaborately investigated. All the as-made samples show the similar SAXS patterns (Fig. 1A(b, c and e)) as that of the one with post-cure at 150 °C. All the as-made samples have the same unit cell parameter of 16.3 nm except that with post-solidification at 180 °C which exhibits a unit cell parameter of 15.6 nm (~4% in contraction). The result implies that a slightly shrinkage occurred on the framework during post-cure treatment at 180 °C. After carbonization at 700 °C, well ordered mesostructure can also be observed for OMC-100, OMC-130 and OMC-180 from their SAXS patterns (Fig. 1B(b, c and e)) and the calculated unite cell parameters are 10.5, 11.0 and 10.8 nm, respectively. Obviously, the unite cell parameters are all larger than that (9.8 nm) of OMC-0, further indicating post-cure is an efficient approach to strengthen the framework and resist shrinkage. The N2 sorption isotherms of OMC-100, OMC-130 and OMC-180 (Fig. 2A(b, c and e)) all show the similar shape as that of OMC-150. Moreover, the capillary condensation steps of OMC-130 and OMC-150 are steeper than that of carbons with post-cure at a relative low (100 °C) and high (180 °C) temperature, implying more uniform mesopores. The pore size distributions of all carbons are shown in Fig. 2B. OMC-130 shows the same pore size of 4.5 nm as that of OMC-150. And relative small pore sizes of 4.0 and 3.6 nm are observed for OMC-100 and OMC-180, respectively, which is in accordance with capillary condensation steps at lower relative pressures. Compared with a relative small pore size of ~2.1 nm of OMC-0, large pore sizes for the carbons with post-solidification can be clearly observed, indicating post-cure can effectively promote the mesopore structure. Besides, the wall thicknesses of 6.5, 6.5 and 7.2 nm for OMC-100, OMC-100, and...
130 and OMC-180 are all smaller than that (7.7 nm) of OMC-0, according with the small framework shrinkage after post-cure. The BET surface areas and pore volumes are shown in Table 1. Evidently, the mesopore BET surface areas are all about half of the total surface area and much larger than that of OMC-0, implying post-cure definitely improves the mesoporosity. Along with the post-cure temperature increase from 100 to 150 °C, the BET surface areas increase gradually and reach 687 m² g⁻¹ of OMC-150. However, with further increase the temperature to 180 °C, a low BET surface area of 599 m² g⁻¹, a small pore volume of 0.34 cm³ g⁻¹ and a thick pore wall of 7.2 nm can be observed, which attributed to the large framework shrinkage during post-cure in accordance with the SAXS results. These results mentioned above indicate post-cure is an effective method and the obtained carbons with post-cure have high quality with well ordered mesostructures, high mesoporosities and large mesopore sizes. Moreover, among the post-cure temperatures from 100 to 180 °C, 150 °C is confirmed to be the optimum.

### 3.2 Influences of the resol concentration on OMC

Influences of the resol concentration (resol/(resol + ethanol), wt.%) on the OMC are investigated with the weight ratio of resol and foam is fixed to 1.6. With the resol concentration increasing from 11%, 33%, 67% to 89%, all the SAXS patterns of the obtained OMC-11-1.6, OMC-33-1.6, OMC-67-1.6, and OMC-89-1.6 show three resolved scattering peaks indexed as 100, 110 and 210 reflections, indicating 2-D hexagonal mesostructures (Fig. 4A). The unit cell parameters are calculated to be 11.0, 11.0, 10.9 and 11.1 nm for the OMC with an increasing concentration of the resol, respectively. The results indicate the resol concentration has little effect on the mesostructure. The N₂ sorption isotherms of the carbons with different resol concentration all exhibit type-IV curve with a capillary condensation step at P/P₀ = 0.4–0.6 and good H₁ hysteresis loop (Fig. 5A), implying the ordered mesostructures as well as the narrow pore size distributions. The BET surface areas are 470, 504, 472 and 493 m² g⁻¹ for OMC-11-1.6, OMC-33-1.6, OMC-67-1.6, and OMC-89-1.6, respectively. Simultaneously, the pore volumes are 0.31, 0.34, 0.32 and 0.34 cm³ g⁻¹. This further implies the resol concentration unconspicuously affects the physicochemical properties of the carbon materials and which was further confirmed by the pore size distributions (inset in Fig. 5A) with similar main value of about 4.3 nm and the similar wall thickness of about 6.5 nm (Table 1). These results indicate that high-quality OMC can be obtained in a wide range of the resol concentration. This benefits practical manipulation and economizes the cost by reducing the solvent. On the basis of our experimental results, when the resol concentration increases to 89%, it becomes difficult for the completely permeating of the mixture solution into the foam. Thus, the resol concentration of 67% is adopted in the following studies considering the convenient manipulation as well as the cost.

### 3.3 Influences of the resol loading amount on OMC

It is necessary to make full use of the large surface provided by the foam and thus to economize the cost of the foam which can not be recycled. Here, under the condition of the resol concentration mentioned above, the structure evolutions of the OMC are investigated by adjusting the resol loading amount (resol/foam, g/g). The SAXS patterns (Fig. 4B) of the carbons with different resol loading amount all show three well-resolved scattering peaks of a 2-D ordered hexagonal mesostructure. Their first peak occurred almost at the same q value and the calculated unite cell parameters are 10.9 nm. This suggests the ordered mesostructure are repeatedly assembled in a wide range of the resol loading amount. It should be noted that when the resol loading amount is up to 130, the saturation could be realized in the PU foam (36.0 g mixed solution per 1.0 g foam), and the obtained OMC-67-14.4 still has a well-ordered mesostructure with large-domain

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**Table 1 – Physicochemical properties of ordered mesoporous carbons.**

<table>
<thead>
<tr>
<th>Sample code</th>
<th>Unit cell parameter a¹ (nm)</th>
<th>BET surface area¹ (m² g⁻¹)</th>
<th>Micropore area² (m² g⁻¹)</th>
<th>Pore size³ (nm)</th>
<th>Pore volume² (cm³ g⁻¹)</th>
<th>Wall thickness⁴ (nm)</th>
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<tr>
<td>OMC-0</td>
<td>9.8</td>
<td>435</td>
<td>352</td>
<td>2.1</td>
<td>0.22</td>
<td>7.7</td>
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<tr>
<td>OMC-100</td>
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<td>596</td>
<td>314</td>
<td>4.0</td>
<td>0.38</td>
<td>6.5</td>
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<tr>
<td>OMC-130</td>
<td>11.0</td>
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<td>326</td>
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<td>0.44</td>
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<td>OMC-150</td>
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<td>0.45</td>
<td>6.5</td>
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<td>3.6</td>
<td>0.34</td>
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<td>OMC-11-1.6</td>
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<td>4.3</td>
<td>0.34</td>
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<td>OMC-67-4.8</td>
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<td>0.43</td>
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<td>OMC-67-8.0</td>
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<td>338</td>
<td>4.5</td>
<td>0.44</td>
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<td>OMC-67-14.4</td>
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<td>650</td>
<td>347</td>
<td>4.5</td>
<td>0.45</td>
<td>6.4</td>
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</table>

¹ Calculated from SAXS results.
² Calculated by the BJH model from sorption data in a relative pressure range from 0.02 to 0.1.
³ Calculated by the V-t method.
⁴ Calculated by the BJH model from the adsorption branches of the isotherms.
⁵ Calculated by the formula \( h = a - D \), where \( a \) represents the unite cell parameter and \( D \) represents the pore diameter determined by the BJH model.
typical stripe-like and hexagonal arrays viewed from the [1 1 0] and [0 0 1] directions from the TEM images (Fig. 3(e and f)). FE-SEM images (Fig. 3(g and h)) further reveal long-range stripe-like and hexagonal arrays with open windows. N₂ sorption isotherms of the carbons with different resol loading amount (Fig. 5B) show the similar shape of typical type-IV isotherms. H₁ hysteresis loops with obviously capillary condensation steps at $P/P₀ = 0.5–0.6$ can also be clearly observed, which suggests uniform mesopores and cylinder channels [17]. It should be noted that the adsorption and desorption branches of all carbons are well closed at the low relative pressures, suggesting well opened pore channels in agreement with the high-resolution FE-SEM results. With the resol loading amount increase from 1.6 to 14.4, the BET surface areas were increased from 472 m² g⁻¹ of OMC-67-1.6 to 650 m² g⁻¹ of OMC-67-14.4 and the pore volumes increased from 0.32 to 0.45 cm³ g⁻¹. When a low resol loading amount of 1.6 is adopted, the obtained OMC-67-1.6 has a relative lower BET surface area (Table 1). The reason may be that the big ratio of residue derived from PU foam contributes a very small surface area. With the increase of the resol loading amount, the weight ratio of the PU foam residue in the final carbons decreases which could increase the BET surface areas eventually. Narrow pore size distributions (inset in Fig. 5B) with the similar mean value of 4.5 nm calculated from the adsorption branch are clearly observed for OMC-67-4.8, OMC-67-8.0 and OMC-67-14.4, which is larger than that (4.2 nm) of OMC-67-1.6 with lower resol loading amount of 1.6. This may be due to a thinner coating layer leads to bigger shrinkage during carbonization. These results indicate that along with the increase of the resol loading amount even up to the saturation of the PU foam, mesoporous carbons can still have ordered 2-D mesostructures with high BET surface areas and pore volumes, open and big pore sizes. The wide tunable range of resol loading amount is propitious to convenient manipulation.

The as-made OMC-67-14.4 (Fig. 6a) shows dark-brown color because of the oxidation and cross-linkage of the resol during solidification for a long time and a high temperature. SEM image of the PU foam scaffolds shows that the scaffolds consist of irregular quasi-prism struts and some membranes (Fig. 6b). The 3-D interconnected struts build a cellular structure with
the pore diameter of about 100–500 μm. When a low resol loading amount of 1.6 is used, OMC-67-1.6 has 3-D skeletal structure similar to that of the PU foam scaffolds (Fig. 6c), implying a well copy of the foam scaffolds. Along with the increase of the resol loading amount, new cell membranes based on the adjacent struts begin to form and further construct cellular cavities with increasing wall thickness (Fig. 6(d–f)). The results indicate PU foam is a good supporting scaffold which provides large space to accommodate resol with the loaded resol amount almost 14 times heavier than that of the PU foam.

The element analysis (Table 2) shows that the OMC contains N derived from the PU foam, implying some residue of the foam are remained. The OMC-11-1.6, OMC-33-1.6, OMC-67-1.6 and OMC-89-1.6 with different resol concentrations exhibit almost the same weight ratio of C, H and N. This indicates resol concentration has little effect on the element composition of the final carbons. The N content gradually decrease from 2.9% of OMC-67-1.6 to 0.6% of OMC-67-14.4 with the increasing resol loading amount from 1.6 to 14.4, suggesting a decreasing weight ratio of the residue from foam in the final carbons which also leads to the increasing BET surface areas.

### 3.4. Electrochemical performance

The electrochemical capacitive performance of the large-quantitative OMC with different resol loading amount was also investigated. The CV curves (Fig. 7A) of OMC-67-1.6, OMC-67-4.8, OMC-67-8.0 and OMC-67-14.4 measured at a low scan rate of 5 mV s⁻¹ all display well-symmetric and rectangular shape, indicating electric double-layer capacitor performances. In addition, at a high scan rate of 100 mV s⁻¹, the CV curves (Fig. 7B) can still preserve a relatively good rectangular profile. This indicates all the OMC with different resol loading amount possess desirable electric performance due to the well-ordered and open mesopore structure which is favorable for the fast electrolyte transport.

The GC curves (Fig. 7C) of OMC-67-1.6, OMC-67-4.8, OMC-67-8.0 and OMC-67-14.4 conducted at 0.5 A g⁻¹ show that all the responses are almost linear and symmetric. The calculated specific capacitances are 113, 130, 126, 132 F g⁻¹, respectively, which is in accordance with the increasing BET surface areas. The results indicate the N content in carbons has no significant influence on the capacities. To further demonstrate this point, GC curves at different current densities were also conducted

<table>
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<th>Sample code</th>
<th>C</th>
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Obviously, the capacitances increase slightly for the OMC with an increasing resol loading amount at all the current densities from 0.5 to 15 A g\(^{-1}\). The capacitance retentions are calculated to be 60%, 64%, 67% and 68% for OMC-67-1.6, OMC-67-4.8, OMC-67-8.0 and OMC-67-14.4 when the current density increased by 30 times. This demonstrates that all the OMC with an increasing resol loading amount have good rate capability. These results indicate that the OMC fabricated by commercial chemicals is very promising as electrode materials for wide application in electrochemical area.

4. Conclusions

A novel post-cure method is developed for synthesizing high-quality OMC in kilogram-scale utilizing only commercial chemicals. Post-cure evidently promotes the mesoporosities of carbons, especially for mesoporous size and surface area. It is found that high-quality OMC can be obtained in a wide range of the concentration and the loading amount of resol. The kilogram-scale OMC with highly ordered 2-D hexagonal mesostructure, large surface area (~690 m\(^2\) g\(^{-1}\)), large pore volume (~0.45 cm\(^3\) g\(^{-1}\)) and uniform pore size (~4.5 nm) show a specific capacitance of about 130 F g\(^{-1}\) in 6.0 M KOH electrolyte at a current density of 0.5 A g\(^{-1}\). We believe that the kilogram-scale production of high-quality OMC will promote their wide applications in other areas, including batteries, storage and catalysis.

Acknowledgments

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