Supporting Information

Porous Carbon Nanofiber-Sulfur Composite Electrodes for Lithium/Sulfur Cells

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Table 1. BET measurement results and conductivity of different samples.

<table>
<thead>
<tr>
<th>Samples</th>
<th>BET total surface area (m² g⁻¹)</th>
<th>Pore volume (cm³ g⁻¹)</th>
<th>Conductivity (S cm⁻¹)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Porous CNFs</td>
<td>123.05</td>
<td>0.27</td>
<td>1.8</td>
</tr>
<tr>
<td>Porous CNFs-42 wt% S</td>
<td>7.39</td>
<td>0.04</td>
<td>0.52</td>
</tr>
<tr>
<td>Porous CNFs-60 wt% S</td>
<td>2.20</td>
<td>0.01</td>
<td>0.17</td>
</tr>
</tbody>
</table>

Figure S1. Electrospun PAN/PMMA bicomponent nanofibers.
Figure S2. SEM images of porous CNF-S nanocomposites with different surface morphologies and structures before thermal treatment in Ar environment.
Figure S3. TEM images of porous CNF-S nanocomposites with different morphologies and structures before thermal treatment in Ar environment.

Figure S4. XRD patterns of porous CNF-S nanocomposites before (a) and after thermal treatment in Ar environment (b) at 155 °C for 12h, and (c) at 155 °C for 12h, then at 160 °C for 6h.
Figure S5. XRD pattern of pure porous CNFs prepared from carbonizing electrospun PAN/PMMA bicomponent nanofibers.

Figure S6. TGA curve of porous CNF-S nanocomposites recorded in N₂ with a heating rate of 10 °C min⁻¹ before (a) and after the thermal treatment in Ar environment at 155°C for 12h (b), further thermal treatment at 160 °C for 6h (c).
Figure S7. (a, d) TEM images of porous CNF-S nanocomposites thermally treated at 155°C for 12h, and the corresponding carbon (b, e) and S (c, f) elemental maps showing the homogeneous distribution of S in porous CNFs.
Figure S8. CV curve of a porous CNF-S nanocomposite electrode at 0.05 mV s\(^{-1}\) scanning rate; (b) Galvanostatic charge/discharge profiles of porous CNF-S nanocomposite electrode at 0.02C rate; (c) cycling performance of porous CNF-S nanocomposite electrode at a constant current rate of (c) 0.05C, and (d) 0.1C, after an initial activation processes at 0.02C for 2 cycles; (e-f) Reversible capacity vs. current density (rate capability) for porous CNF-S nanocomposite electrode. All the cells were cycled in the potential window from 1.0 to 3.0 V. The porous CNF-S nanocomposites were thermally treated in Ar environment at 155 °C for 12h with a S content of 60 wt% (The overall S content of the electrode was 42 wt%).
Figure S9. (a) Galvanostatic charge/discharge profiles of porous CNF-S nanocomposite electrode at 0.02C rate; (b) cycling performance of porous CNF-S nanocomposite electrode at a constant current rate of (c) 0.05C, and (c) 0.1C, after an initial activation processes at 0.02C for 2 cycles; The porous CNF-S nanocomposite electrodes were directly used after the synthesis via solution-based chemical reactions. The S content of the nanocomposite was 76 wt% (The overall S content of the electrode was 53.2 wt%).