Influence of the pyrolizing temperature on textural properties of the templated mesoporous carbon materials

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Overview

• Synthesis procedure (hard template method)
• Characterization:
  – N\textsubscript{2}-sorption
  – Small Angle X-ray Scattering (SAXS)
  – Scanning Electron Microscopy (SEM)
  – Raman spectroscopy
  – IR spectroscopy
• Conclusions
Synthesis procedure - hard template method

I

SC700
SC800
SC900
SC1000

SBA-15

IMPREGNATION
Carbon precursor

POLYMERIZATION
100 °C, 6 h
160 °C, 6 h

SBA-15/poly-glycerol

II

SCx

X=700, 800, 900, 1000 °C
5 h, N₂

PIROLISYS

C700
C800
C900
C1000

SC700
SC800
SC900
SC1000

S700
S800
S900
S1000

Sx
S=silica

III

CARBON REMOVAL

C=C=carbon

SILICA DISSOLUTION

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Characterization: N$_2$ sorption

- type IV isotherms characterizing mesoporous materials with cylindrical pores
- second step of desorption branch $\rightarrow$ blocked pores
- at high temperatures $\rightarrow$ reduced porosity $\rightarrow$ structural shrinkage
- type IV isotherms characterizing mesoporous materials with slit-like pores
- same isotherm shape → not so big differences in their textural properties
- at high temperature → narrow and uniform pore size distribution, no shrinkage
Characterization: N\textsubscript{2} sorption

- type IV isotherms characterizing mesoporous materials with cylindrical pores
- capillary condensation step moves to lower P/P\textsubscript{0} \rightarrow decrease of pore size
- higher pirolysing temperature \rightarrow smaller pores \rightarrow structural shrinkage
## Characterization: N\textsubscript{2} sorption results

<table>
<thead>
<tr>
<th>Sample</th>
<th>$S_{\text{BET}}$, m\textsuperscript{2} g\textsuperscript{-1}</th>
<th>$V_{\text{tot}}$, cc g\textsuperscript{-1}</th>
<th>$S_{\text{mic}}$, m\textsuperscript{2} g\textsuperscript{-1}</th>
<th>$V_{\text{mic}}$, cc g\textsuperscript{-1}</th>
<th>$d_p$, nm</th>
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<tbody>
<tr>
<td>SBA-15</td>
<td>728</td>
<td>1.07</td>
<td>196</td>
<td>0.079</td>
<td>9.3</td>
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<tr>
<td>S700</td>
<td>430</td>
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<td>56</td>
<td>0.019</td>
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<tr>
<td>S800</td>
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<td>0.55</td>
<td>34</td>
<td>0.011</td>
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<tr>
<td>S900</td>
<td>353</td>
<td>0.54</td>
<td>30</td>
<td>0.008</td>
<td>6.3; 7.2</td>
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<tr>
<td>S1000</td>
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<td>28</td>
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<tr>
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<td>314</td>
<td>0.38</td>
<td>48</td>
<td>0.018</td>
<td>3.9; 6.6</td>
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<tr>
<td>SC800</td>
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<td>3; 3.6; 5.6</td>
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<tr>
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<td>0</td>
<td>0</td>
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<tr>
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<td>1398</td>
<td>1.25</td>
<td>97</td>
<td>0.035</td>
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</table>
Characterization: SAXS

- three well resolved peaks → p6mm Hexagonal symmetry
- the (100) peak shifts to higher 2θ values → decrease of unit cell parameter and interplanar distance → decrease of pore size → confirmation of structural shrinkage
Characterization: SAXS

- not so well resolved peaks → only the first two could be distinguished
- the (100) peak shifting is not so large as in the case of composites → almost no change of unit cell parameter and interplanar distance → no pore size changing → no structural shrinkage
Characterization: SAXS

- three well resolved peaks → p6mm Hexagonal symmetry
- the (100) peak shifts to higher 2Θ values → decrease of unit cell parameter and interplanar distance → decrease of pore size → confirmation of structural shrinkage

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Characterization: SEM

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Characterization:

RAMAN

IR

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Conclusions

- The influence of the pyrolizing temperature on the textural properties of mesoporous carbon has been successfully demonstrated.

- It has been shown that the silica shrinkage plays an important role in their synthesis.

- Textural properties of the templated carbon material could be fine tuned by controlling synthesis conditions with regard to the choice of pyrolizing temperature.

**Future work:** applications as filtration systems in dialysis as adsorbents in haemoperfution.
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Thank you for your attention!