The influence of particle size of amino-functionalized MCM-41 silicas on CO₂ adsorption

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Supporting Information

Figure S.I.1. XRD patterns between 2 and 7 °2θ of MCM-41 micro (a), MCM-41 nano (b), P_MCM-41 micro (a’) and P_MCM-41 nano (b’).
Figure S.I.2. TGA curves of MCM-41 micro (a), MCM-41 nano (b). The measurements were done under Argon flow (20 ml/min) from 20 to 1100°C (1°C min⁻¹ heating rate).

Figure S.I.3. FTIR spectra of CO₂ adsorbed (Pmax = 60 mbar) at 35°C on P_MCM-41 micro (a-aIV) and P_MCM-41 nano (b-bIV). The arrows indicate decreasing CO₂ pressure until vacuum. Spectra are reported after subtraction of the spectrum recorded before CO₂ interaction, used as a background.
Figure S.I.4. Heat Flow from DSC-TGA Analysis for MCM-41 micro (Frame A) and MCM-41 nano (Frame B) samples.

Figure S.I.5. TPD curves for P_MCM-41 micro and P_MCM-41 nano sample at 2 ml/min flow rates. CO₂ partial pressure: 0.1 bar.

Integrating the area under the TPD curves, the value of irreversible chemisorbed fraction can be achieved. The obtained values of irreversible fractions respectively for P_MCM-41 micro and P_MCM-41 nano samples are 0.16 and 0.06 mmol·g⁻¹.
Table 1. Irreversible fractions obtained with TPD, TGA and Volumetric Analysis for the grafted MCM-41 materials.

<table>
<thead>
<tr>
<th>Sample</th>
<th>TPD irreversible fraction [mmol/g]</th>
<th>TGA irreversible fraction [mmol/g]</th>
<th>Volumetry irreversible fraction [mmol/g]</th>
</tr>
</thead>
<tbody>
<tr>
<td>P_MCM-41 micro</td>
<td>0.16</td>
<td>0.16</td>
<td>0.16</td>
</tr>
<tr>
<td>P_MCM-41 nano</td>
<td>0.06</td>
<td>0.07</td>
<td>0.10</td>
</tr>
</tbody>
</table>

Table 2. CO₂ Adsorption Capacity, reversible and irreversible fractions obtained with TGA analysis for the grafted MCM-41 materials.

<table>
<thead>
<tr>
<th>Sample / Temperature</th>
<th>CO₂ Adsorption Capacity [mmol/g]</th>
<th>reversible fraction [%]</th>
<th>irreversible fraction [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>P_MCM-41 micro / 20°C</td>
<td>0.63</td>
<td>64.9</td>
<td>35.1</td>
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<tr>
<td>P_MCM-41 micro / 35°C</td>
<td>0.60</td>
<td>72.8</td>
<td>27.2</td>
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<tr>
<td>P_MCM-41 micro / 50°C</td>
<td>0.58</td>
<td>78.6</td>
<td>21.4</td>
</tr>
<tr>
<td>P_MCM-41 micro / 70°C</td>
<td>0.55</td>
<td>86.6</td>
<td>13.4</td>
</tr>
<tr>
<td>P_MCM-41 micro / 90°C</td>
<td>0.49</td>
<td>93.3</td>
<td>6.7</td>
</tr>
<tr>
<td>P_MCM-41 nano / 20°C</td>
<td>0.96</td>
<td>87.4</td>
<td>12.6</td>
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<tr>
<td>P_MCM-41 nano / 35°C</td>
<td>0.79</td>
<td>90.7</td>
<td>9.3</td>
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<tr>
<td>P_MCM-41 nano / 50°C</td>
<td>0.60</td>
<td>95.4</td>
<td>4.6</td>
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<tr>
<td>P_MCM-41 nano / 70°C</td>
<td>0.38</td>
<td>96.0</td>
<td>4.0</td>
</tr>
<tr>
<td>P_MCM-41 nano / 90°C</td>
<td>0.21</td>
<td>99.1</td>
<td>0.9</td>
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</table>