Electronic Supplementary Information

**Nanoscale MIL-101 supported RhNi nanoparticles: an efficient catalyst for hydrogen generation from hydrous hydrazine**

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Table S1. Characteristics of MOFs with different dimensions and Rh\textsubscript{58}Ni\textsubscript{42}@MIL-101.

<table>
<thead>
<tr>
<th>Sample</th>
<th>wt%</th>
<th>Surface Area (m\textsuperscript{2} g\textsuperscript{-1})</th>
<th>Pore volume (m\textsuperscript{3} g\textsuperscript{-1})</th>
<th>Pore size (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>MIL-101-50 nm</td>
<td></td>
<td>3913.2</td>
<td>2.135</td>
<td>3.183</td>
</tr>
<tr>
<td>MIL-101-100 nm</td>
<td></td>
<td>3420.8</td>
<td>1.869</td>
<td>3.176</td>
</tr>
<tr>
<td>MIL-101-200 nm</td>
<td></td>
<td>2232.8</td>
<td>1.195</td>
<td>2.995</td>
</tr>
<tr>
<td>Rh\textsubscript{58}Ni\textsubscript{42}@MIL-101</td>
<td>7.46</td>
<td>1538.2</td>
<td>0.922</td>
<td>1.178</td>
</tr>
</tbody>
</table>

Scheme S1. The potential pathway of the catalytic mechanism.
Figure S1. Low-angle Powder X-ray diffraction patterns of MIL-101 with different dimensions: (a) 200 nm; (b) 100 nm; (c) 50 nm.

Figure S2. XPS spectra for catalyst Rh\textsubscript{58}Ni\textsubscript{42}@MIL-101 (a) survey spectrum and (b) Cr\textsuperscript{2p}.
Figure S3. Magnetic effect test of the as-synthesized Rh$_{58}$Ni$_{42}$@MIL-101 catalyst.

Figure S4. MS profile for the gases released from the decomposition reaction of hydrous hydrazine in aqueous NaOH solution (0.5 M) over Rh$_{58}$Ni$_{42}$@MIL-101 (catalyst = 0.100 g; N$_2$H$_4$H$_2$O = 0.1 mL) at 50 °C.
Figure S5. MS profile for the gases released from the decomposition reaction of hydrous hydrazine in aqueous NaOH solution (0.5 M) over RhNi/C (catalyst = 0.100 g; N₂H₄H₂O = 0.1 mL) at 50 °C.

Figure S6. TEM of RhNi NPs with different dimensions.
Figure S7. Rh$_{38}$Ni$_{42}$@MIL-101 catalyst after the canalization reaction of 5 runs toward the decomposition of hydrous hydrazine: (a) SEM; (b) TEM; (c) N$_2$ sorption isotherms; (d) low-angle powder X-ray diffraction pattern.