Electronic Supplementary Information

Rapid detemplation of nanozeolite $\beta$:
microwave-assisted Fenton-like oxidation

Yuanyuan Hu, Yahong Zhang* and Yi Tang

The detailed calculation of $H_2O_2/TEA^+$ and $Fe^{3+}/H_2O_2$ are showed as follow:

For the as-prepared nanozeolite $\beta$:

$$\frac{H_2O_2 (mol)}{TEA^+ (mol)} = \frac{V_{H_2O_2} ml \times 1.1g/ml \times 30\%}{\frac{34g/mol}{150mg \times 0.8 \times 10^{-3}}} = 11.89V_{H_2O_2}$$ (1)

Herein, the amount of $TEA^+$ in the solution is determined by the volume of as-prepared $\beta$ nanozeolite solution and the mass of TEAOH in the synthesis receipts, which means that $TEAOH(g)/Nanozeolite_{as-prepared(g)} = 0.8$ and the mass of $\beta$ nanozeolite added in the system is 150 mg. $V_{H_2O_2}$ presents the addition volume of $H_2O_2$ in the as-prepared $\beta$ nanozeolite system.

For the washed $\beta$ nanozeolite:

$$\frac{H_2O_2 (mol)}{TEA^+ (mol)} = \frac{V'_{H_2O_2} ml \times 1.1g/ml \times 30\%}{\frac{34g/mol}{150mg \times 0.2 \times 10^{-3}}} = 42.06V'_{H_2O_2}$$

The mass of $TEA^+$ in the washed nanozeolite solution was determined by the TG analysis (Fig. 2b-I). It was indicated (Fig. 2b-I) that $TEA^+$ in the micropores of the as-prepared $\beta$ could reach about 20 wt%, i.e., $TEA^+(g)/Nanozeolite_{washed(g)} = 0.2$, and the mass of $\beta$ nanozeolite added in the system is 150 mg. $V'_{H_2O_2}$ presents the addition volume of $H_2O_2$ in the washed $\beta$ nanozeolite system.
\[
\frac{Fe^{3+} (\mu mol)}{H_2O_2 (mol)} = \frac{V_{Fe^{3+}} \mu L \times 0.2 mol/L}{V_{H_2O_2} (V_{H_2O_2}) ml \times 1.1 g/ml \times 30\%} = 20.606 \frac{V_{Fe^{3+}}}{V_{H_2O_2} (V_{H_2O_2}) 34 g/mol}
\]

\(V_{Fe^{3+}}\) presents the addition volume of Fe (NO\(_3\))\(_3\) of 0.2 M in the as-prepared (or washed) \(\beta\) nanozeolite system.

Table S1 Fe/Si ratios in Fenton-treated \(\beta\) with different compositions before and after acid wash step.

<table>
<thead>
<tr>
<th>Sample (^a)</th>
<th>8</th>
<th>5</th>
<th>9</th>
<th>10</th>
</tr>
</thead>
<tbody>
<tr>
<td>(Fe^{3+}/H_2O_2) (10(^{-6}) mol/mol)</td>
<td>15.45</td>
<td>9.27</td>
<td>4.64</td>
<td>2.32</td>
</tr>
</tbody>
</table>

\(Fe/Si\) before acid wash\(^b\) | 2.6\(\times\)10\(^{-3}\) | 2.1\(\times\)10\(^{-3}\) | 1.9\(\times\)10\(^{-3}\) | 1.5\(\times\)10\(^{-3}\)

\(Fe/Si\) after acid wash\(^b\) | n.d.\(^c\) | n.d. | n.d. | n.d. |

\(^a\): the No. of sample and reaction condition are corresponding to those in Table 1. \(^b\): The \(Fe/Si\) ratios of nanozeolites were detected by EDX (Philips XL 30). \(^c\): n.d. = not detected.

Fig. S1 SEM image of calcined \(\beta\) nanozeolite.
Fig. S2 XRD patterns of as-prepared β (a), Fenton-treated β (b) and calcined β (c).
Fig. S3 MS signals of H₂O (m/z = 18), CO (m/z = 28), CO₂ (m/z = 44) and NO₂ (m/z = 46) evolved when the as-prepared β (a, c, e, g) and Fenton-treated β (b, d, f, h) heated from 25 to 480 °C in air.
Fig. S4 FTIR spectrum of the Fenton-treated sample by an electric oven at 170 °C for 120 min. The C-H stretching (2987 and 2880 cm\(^{-1}\)) and bending (1483 and 1396 cm\(^{-1}\)) vibrations corresponding to SDAs are still observed.