Realizing Toluene Deep Mineralization by Coupling Nonthermal Plasma and Nitrogen-Enriched Hollow Hybrid Carbon

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Catalyst Characterizations

The X-ray diffraction (PXRD) curves of samples were collected by diffractometer (D8-Focus Bragg-Brentano, Bruker, Germany) with a Cu Kα radiation (scan rate 0.142224 °/s at 40 kV and 40 mA). N2 adsorption-desorption data were obtained with a SSA-6000 specific surface area aperture analyzer (Builder, China) at -196 °C. Before the measurements, the catalysts were degassed under vacuum at 250 °C for 3 h. The specific surface areas (S_{BET}) of catalysts were calculated by the Brunauer-Emmett-Teller (BET) equation according to N2 adsorption branch at P/P0 = 0.05-0.20. The pore size distribution (PSD) curves were obtained from the adsorption branch by using the Barrett-Joyner-Halenda (BJH) method and the Horvath-Kawazoe (HK) method. The total pore volumes (V_t) were estimated from the amount of N2 adsorbed at P/P0 = 0.99. The micropore volumes (V_{mic}) were calculated by using the t-plot method. The surface morphology of catalysts was investigated using a field emission scanning electron microscopy (FE-SEM, JEOL 7800F, Japan). Structural details and chemical composition were observed using a high resolution transmission electron microscopy (HR-TEM, FEI Tecnai G2F30, USA) equipped with an energy dispersive X-ray spectrometer (EDS) analysis system operated at 300 kV. X-ray photoelectron spectroscopy (XPS) of the catalysts were carried out on a Kratos AXIS Ultra DLD instrument (Kratos, UK) with Mg Kα radiation (hν = 1253.6 eV). The XPS data were analyzed by using the CASA XPS software, and binding energies were calibrated using the containment carbon (C 1s = 284.8 eV). Fourier transform infrared (FTIR) spectra were recorded by the potassium bromide/sample flakelet method using a Tensor 37 spectrometer (Bruker Optics,
Germany) in spectral range of 4000-400 cm\(^{-1}\) at ambient temperature with resolution of 4 cm\(^{-1}\). \textit{In situ} diffuse reflectance infrared Fourier transform spectroscopy (\textit{in situ} DRIFTS) experiments of the toluene adsorption and oxidation by ozone over the N-HHC were carried out on a Tensor 37 spectrometer equipped with a Mercury Cadmium Telluride (MCT) detector cooled by liquid N\(_2\) at a resolution of 4 cm\(^{-1}\) from 4000-600 cm\(^{-1}\). Before the tests, the N-HHC was heated at 573 K for 30 min in N\(_2\) to remove surface impurities, and then adsorb toluene at 30 and 100 °C for 35 min reach adsorption saturation, respectively. The toluene oxidation reactions with O\(_3\) under different temperatures (30 and 100 °C) started when the toluene was adsorption saturation on N-HHC.

The temperature-programmed reduction of H\(_2\) (H\(_2\)-TPR) was performed in a quartz reactor connected to a thermal conductivity detector (TCD) with H\(_2\)-He mixture (5 vol\% H\(_2\) by volume, 30 mL/min) as a reductant. Before the reduction, 50 mg catalyst was pretreated in pure He (40 mL/min) at 300 °C for 1 h. And then, it was cooled to the room temperature. Finally, the reduction was carried out from room temperature to 800 °C with a rate of 10 °C/min.
**Table S1** Comparison of energy yield and toluene decomposition with previous literature works.

<table>
<thead>
<tr>
<th>SIE (J/L)</th>
<th>$\eta_{\text{toluene}}$ (%)</th>
<th>EY (g/kW·h)</th>
<th>Ref.</th>
</tr>
</thead>
<tbody>
<tr>
<td>101</td>
<td>78.2</td>
<td>12.0</td>
<td>This work</td>
</tr>
<tr>
<td>278</td>
<td>90.1</td>
<td>5.0</td>
<td>This work</td>
</tr>
<tr>
<td>262</td>
<td>91.1</td>
<td>5.2</td>
<td>14</td>
</tr>
<tr>
<td>473</td>
<td>94.9</td>
<td>2.8</td>
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<td>93</td>
<td>41.9</td>
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<tr>
<td>424</td>
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<td>1.4</td>
<td>16</td>
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<tr>
<td>100</td>
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<td>3.3</td>
<td>17</td>
</tr>
<tr>
<td>400</td>
<td>94.5</td>
<td>1.7</td>
<td>17</td>
</tr>
<tr>
<td>211</td>
<td>29.8</td>
<td>6.3</td>
<td>38</td>
</tr>
<tr>
<td>809</td>
<td>92.1</td>
<td>5.1</td>
<td>38</td>
</tr>
<tr>
<td>81</td>
<td>18.0</td>
<td>2.2</td>
<td>39</td>
</tr>
<tr>
<td>347</td>
<td>56.0</td>
<td>1.8</td>
<td>39</td>
</tr>
</tbody>
</table>
Figure S1 Schematic diagram of the experiment.
Figure S2 (a) Toluene degradation efficiency and (b) energy yield as a function of SIE.
Figure S3 Photos of the discharge zones of post-NTP-catalytic system with different specific input energies.
Figure S4. Curves computed from the oscilloscope for the discharge zones of post-NTP-catalytic system with different specific input energies: (a) 28 J/L, (b) 101 J/L, (c) 174 J/L, (d) 217 J/L, (e) 278 J/L, (f) 357 J/L and (g) 444 J/L.
Figure S5 (a) FE-SEM, (b,c) TEM, and (d) HAADF-STEM images of ZIF-8; (e) Elemental EDX mappings and (f) EDS spectra of ZIF-8.

Note: Fig. S5 illustrates ZIF-8 polyhedral morphology (TEM and FE-SEM images) along with its elemental composition (EDX mapping and EDS spectra). The characterizations revealed that dodecahedron shape ZIF-8 (Figs. S5b,c) possesses uniform distribution of C, N, O and Zn elements (Figs. S5d-f).
Figure S6 FE-SEM images of Mn-ZIF-8 with different amounts of Mn(NO₃)₂: (a) 0 mmol, (b) 5 mmol, (c) 10 mmol, (d) 15 mmol, and (e) 20 mmol.

Note: For different Mn-ZIF-8 synthesized by different Mn/Zn molar ratio, it is clear from their FE-SEM images that the crystal size of Mn-ZIF-8 increases with the increasing of Mn/Zn molar ratio (Fig. S6). Such crystal size enlargement is mainly due to the fact that some Zn²⁺ in ZIF-8 were replaced by Mn²⁺ with larger ionic radius, making Mn-ZIF-8 with larger unit cells [37].
Figure S7 (a) FE-SEM and (b,c) TEM images of Mn-ZIF-8; (d) HAADF-STEM image and elemental mappings of Mn-ZIF-8; (e) EDS spectra of Mn-ZIF-8.

**Note:** Fig. S7 illustrates Mn-ZIF-8 polyhedral morphology (TEM and FE-SEM images) along with its elemental composition (EDX mapping and EDS spectra). The characterizations revealed that dodecahedron shape Mn-ZIF-8 (Figs. S7b,c) possesses uniform distribution of C, N, O and Zn elements (Figs. S7d-f). Furthermore, the well-dispersion of Mn in Mn-ZIF-8 is also perceivable (Fig. S7d,e).
Figure S8 (a) FE-SEM, (b,c) TEM and (d) HAADF-STEM images and elemental mappings of Mn-ZIF-8@PDA;

Note: A layer of polydopamine (PDA) was grown over Mn-ZIF-8, utilizing ammonia as catalyst [38]. Cube-like morphology of the Mn-ZIF-8@PDA core-shell sample was clearly exhibited in Fig. S8a-c. From its elemental mappings (Fig. S8d), successful deposition of the PDA shell on the Mn-ZIF-8 core is realized from the intensified EDX signal of carbon element.
Synthesis of N-HHCs

N-HHC-1. In a typical preparation, 0.2 g of Mn-ZIF-8 and 0.55 g of F127 are dispersed in 25 mL of deionized water by ultrasonication and stirring to form an emulsion solution. 2.5 mL of 1,3,5-trimethylbenzene (TMB) and 0.5 mL of ammonium hydroxide (25-28%) are dispersed in 20 mL of anhydrous ethanol by stirring to form a clear solution. Then the two solutions were mixed under stirring for 2 min, which was subsequently poured into 15 mL of deionized water containing 0.3 g of dopamine hydrochloride and stirred for 48 h at room temperature. The black powders were obtained by centrifugation, washed with anhydrous ethanol for several times, and dried in vacuum at 80 °C for 12 h. The powder of Mn-ZIF-8@PDA was placed in a tube furnace and heated to 600 °C for 2 h under N₂ stream with a heating rate of 1 °C/min to obtain the N-HHC-1.

N-HHC-2. 0.2 g of Mn-ZIF-8 and 0.55 g of F127 are dispersed in 25 mL of deionized water by ultrasonication and stirring to form an emulsion solution. 2.5 mL of 1,3,5-trimethylbenzene (TMB) and 9.0 mL of ammonium hydroxide (25-28%) are dispersed in 20 mL of anhydrous ethanol by stirring to form a clear solution. Then the two solutions were mixed under stirring for 2 min, which was subsequently poured into 15 mL of deionized water containing 0.3 g of dopamine hydrochloride and stirred for 48 h at room temperature. The black powders were obtained by centrifugation, washed with anhydrous ethanol for several times, and dried in vacuum at 80 °C for 12 h. The powder of Mn-ZIF-8@PDA was placed in a tube furnace and heated to 600 °C for 2 h under N₂ stream with a heating rate of 1 °C/min to obtain the N-HHC-2.
**N-HHC-3.** 0.2 g of Mn-ZIF-8 is dispersed in 25 mL of deionized water by ultrasonication and stirring to form an emulsion solution. 2.5 mL of 1,3,5-trimethylbenzene (TMB) and 3.0 mL of ammonium hydroxide (25-28%) are dispersed in 20 mL of anhydrous ethanol by stirring to form a clear solution. Then the two solutions were mixed under stirring for 2 min, which was subsequently poured into 15 mL of deionized water containing 0.3 g of dopamine hydrochloride and stirred for 48 h at room temperature. The black powders were obtained by centrifugation, washed with anhydrous ethanol for several times, and dried in vacuum at 80 °C for 12 h. The powder of Mn-ZIF-8@PDA was placed in a tube furnace and heated to 600 °C for 2 h under N₂ stream with a heating rate of 1 °C/min to obtain the N-HHC-3.

**N-HHC-4.** 0.2 g of Mn-ZIF-8 and 0.55 g of F127 are dispersed in 25 mL of deionized water by ultrasonication and stirring to form an emulsion solution. 3.0 mL of ammonium hydroxide (25-28%) is dispersed in 20 mL of anhydrous ethanol by stirring to form a clear solution. Then the two solutions were mixed under stirring for 2 min, which was subsequently poured into 15 mL of deionized water containing 0.3 g of dopamine hydrochloride and stirred for 48 h at room temperature. The black powders were obtained by centrifugation, washed with anhydrous ethanol for several times, and dried in vacuum at 80 °C for 12 h. The powder of Mn-ZIF-8@PDA was placed in a tube furnace and heated to 600 °C for 2 h under N₂ stream with a heating rate of 1 °C/min to obtain the N-HHC-4.

**N-HHC-5.** 0.2 g of Mn-ZIF-8 and 0.55 g of F127 are dispersed in 25 mL of deionized water by ultrasonication and stirring to form an emulsion solution. 0.5 mL of
1,3,5-trimethylbenzene (TMB) and 3.0 mL of ammonium hydroxide (25-28%) are dispersed in 20 mL of anhydrous ethanol by stirring to form a clear solution. Then the two solutions were mixed under stirring for 2 min, which was subsequently poured into 15 mL of deionized water containing 0.3 g of dopamine hydrochloride and stirred for 48 h at room temperature. The black powders were obtained by centrifugation, washed with anhydrous ethanol for several times, and dried in vacuum at 80 °C for 12 h. The powder of Mn-ZIF-8@PDA was placed in a tube furnace and heated to 600 °C for 2 h under N₂ stream with a heating rate of 1 °C/min to obtain the N-HHC-5.

**N-HHC-6.** 0.2 g of Mn-ZIF-8 and 0.55 g of F127 are dispersed in 25 mL of deionized water by ultrasonication and stirring to form an emulsion solution. 5.0 mL of 1,3,5-trimethylbenzene (TMB) and 9.0 mL of ammonium hydroxide (25-28%) are dispersed in 20 mL of anhydrous ethanol by stirring to form a clear solution. Then the two solutions were mixed under stirring for 2 min, which was subsequently poured into 15 mL of deionized water containing 0.3 g of dopamine hydrochloride and stirred for 48 h at room temperature. The black powders were obtained by centrifugation, washed with anhydrous ethanol for several times, and dried in vacuum at 80 °C for 12 h. The powder of Mn-ZIF-8@PDA was placed in a tube furnace and heated to 600 °C for 2 h under N₂ stream with a heating rate of 1 °C/min to obtain the N-HHC-6.
**Table S2** Synthesis conditions of N-HHCs materials.

<table>
<thead>
<tr>
<th>Sample</th>
<th>NH$_3$·H$_2$O (mL)</th>
<th>F127 (g)</th>
<th>TMB (mL)</th>
</tr>
</thead>
<tbody>
<tr>
<td>N-HHC</td>
<td>3.0</td>
<td>0.55</td>
<td>2.5</td>
</tr>
<tr>
<td>N-HHC-1</td>
<td>0.5</td>
<td>0.55</td>
<td>2.5</td>
</tr>
<tr>
<td>N-HHC-2</td>
<td>9.0</td>
<td>0.55</td>
<td>2.5</td>
</tr>
<tr>
<td>N-HHC-3</td>
<td>3.0</td>
<td>0</td>
<td>2.5</td>
</tr>
<tr>
<td>N-HHC-4</td>
<td>3.0</td>
<td>0.55</td>
<td>0</td>
</tr>
<tr>
<td>N-HHC-5</td>
<td>3.0</td>
<td>0.55</td>
<td>0.5</td>
</tr>
<tr>
<td>N-HHC-6</td>
<td>3.0</td>
<td>0.55</td>
<td>5.0</td>
</tr>
</tbody>
</table>
a) Effect of ammonia on N-HHC

Figure S9 FE-SEM images of Mn-ZIF-8@PDA and its corresponding N-HHCs prepared with different volumes of ammonium hydroxide: (a,d) 1.0 mL (Mn-ZIF-8@PDA-1, N-HHC-1), (b,e) 3.0 mL (Mn-ZIF-8@PDA, N-HHC), and (c,f) 9.0 mL (Mn-ZIF-8@PDA-2, N-HHC-2).
Figure S10 (a) $\text{N}_2$ adsorption-desorption isotherms of N-HHCs prepared with different volumes of ammonium hydroxide and the corresponding (b) micropore and (c) mesopore size distributions.

Table S3 Textural property of prepared N-HHCs with different volumes of NH$_3$·H$_2$O.

<table>
<thead>
<tr>
<th>Sample</th>
<th>NH$_3$·H$_2$O (mL)</th>
<th>$S_{\text{BET}}$ (m$^2$/g)</th>
<th>$S_{\text{Lang}}$ (m$^2$/g)</th>
<th>$S_{\text{mic}}$ (m$^2$/g)</th>
<th>$V_t$ (cm$^3$/g)</th>
<th>$V_{\text{mic}}$ (cm$^3$/g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>N-HHC-1</td>
<td>0.5</td>
<td>965</td>
<td>1261</td>
<td>730</td>
<td>0.89</td>
<td>0.30</td>
</tr>
<tr>
<td>N-HHC</td>
<td>3.0</td>
<td>1159</td>
<td>1481</td>
<td>904</td>
<td>1.11</td>
<td>0.37</td>
</tr>
<tr>
<td>N-HHC-2</td>
<td>9.0</td>
<td>1474</td>
<td>1903</td>
<td>1134</td>
<td>1.45</td>
<td>0.46</td>
</tr>
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</table>

Note: By increasing the volume of NH$_3$·H$_2$O from 0.5 to 9.0 mL, the morphology of Mn-ZIF-8@PDA and their N-HHCs does not demonstrate any egregious change (Fig. S9).
However, the specific surface area of various N-HHCs (i.e., N-HHC-1, N-HHC and N-HHC-2 represents the adding amount of NH$_3$·H$_2$O is 0.5, 3.0 and 9.0 mL, respectively) increases with the increasing of NH$_3$·H$_2$O, in which the total pore volume also follows the same variation trend (Table S3). As shown in Fig. S10a, all N-HHCs exhibit types I and IV combined adsorption isotherms with type H4 hysteresis at $P/P_0 = 0.5$-1.0, corresponding to their micro-meso-macroporous composite structures [40]. The pore size distribution (PSD) curves (Fig. S10b,c) further confirm that the micropores, mesopores and macropores are co-existence in N-HHCs. In addition, the width of hysteresis loop increases with the increasing of NH$_3$·H$_2$O attributing to the rapid nucleation in the early stage of the dopamine hydrochloride precursor, conducing to the formation of mesostructure [39].
b) Effect of F127 and/or TMP on N-HHC

**Figure S11** FE-SEM images of Mn-ZIF-8@PDA and its corresponding N-HHCs prepared in the presence of F127 and/or TMB: (a,d) 0.55 g F127 and 2.5 mL TMB (Mn-ZIF-8@PDA, N-HHC), (b,e) 2.5 mL TMB (Mn-ZIF-8@PDA-3, N-HHC-3), and (c,f) 0.55 g F127 (Mn-ZIF-8@PDA-4, N-HHC-4).
**Figure S12** (a) $N_2$ adsorption-desorption isotherms of N-HHCs prepared in the presence of F127 and/or TMB and the corresponding (b) micropore and (c) mesopore size distributions.

**Table S4** Textural property of prepared N-HHCs with different additives.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Additive</th>
<th>$S_{BET}$ (m²/g)</th>
<th>$S_{Lang}$ (m²/g)</th>
<th>$S_{mic}$ (m²/g)</th>
<th>$V_t$ (cm³/g)</th>
<th>$V_{mic}$ (cm³/g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>N-HHC</td>
<td>F127+TMB</td>
<td>1159</td>
<td>1481</td>
<td>904</td>
<td>1.11</td>
<td>0.37</td>
</tr>
<tr>
<td>N-HHC-3</td>
<td>TMB</td>
<td>285</td>
<td>401</td>
<td>200</td>
<td>0.39</td>
<td>0.09</td>
</tr>
<tr>
<td>N-HHC-4</td>
<td>F127</td>
<td>357</td>
<td>467</td>
<td>272</td>
<td>0.28</td>
<td>0.11</td>
</tr>
</tbody>
</table>

**Note:** The role of block copolymers and pore swelling agent is immensely imperative in the formation of micro-/mesoporous particles. Mn-ZIF-8@PDA and its corresponding
N-HHCs nanosheets and truncated cubes were formed with the addition of TMB (Fig. S11b,e); however, complete Mn-ZIF-8@PDA truncated nanocubes and its corresponding N-HHC are obtainable with the addition of F127 (Fig. S11c,f). Three kinds of N-HHCs (i.e., N-HHC, N-HHC-3 and N-HHC-4 represents the amount of NH₃·H₂O added is 0.5, 3.0 and 9.0 mL, respectively), have shown comparably identical nitrogen adsorption/desorption isotherms and PSD curves (Fig. S12). However, both the $S_{\text{BET}}$ (1159 m²/g) and $V_i$ (1.11 cm³/g) of the prepared N-HHC with the co-adding of F127 block copolymer and TMB pore swelling agent are much larger than those of N-HHCs with just TMB (285 m²/g and 0.39 cm³/g) or just F127 (357 m²/g and 0.28 cm³/g). The results in Table S4 indicates that the F127 and TMB provide the necessary conditions to generate plenty micropores and mesopores in the synthesized N-HHC materials.
c) Effect of TMP on N-HHC

**Figure S13** FE-SEM images of Mn-ZIF-8@PDA and its corresponding N-HHCs prepared with different volumes of TMB: (a,d) 0.5 mL (Mn-ZIF-8@PDA-5, N-HHC-5), (b,e) 2.5 mL (Mn-ZIF-8@PDA, N-HHC), and (c,f) 5.0 mL (Mn-ZIF-8@PDA-6, N-HHC-6).
Figure S14 (a) N\textsubscript{2} adsorption-desorption isotherms of N-HHCs and the corresponding (b) micropore and (c) mesopore size distributions.

Table S5 Textural property of prepared N-HHCs with different amounts of TMB.

<table>
<thead>
<tr>
<th>Sample</th>
<th>TMB (Ml)</th>
<th>S\textsubscript{BET} (m\textsuperscript{2}/g)</th>
<th>S\textsubscript{Lang} (m\textsuperscript{2}/g)</th>
<th>S\textsubscript{mic} (m\textsuperscript{2}/g)</th>
<th>V\textsubscript{t} (cm\textsuperscript{3}/g)</th>
<th>V\textsubscript{mic} (cm\textsuperscript{3}/g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>N-HHC-5</td>
<td>0.5</td>
<td>248</td>
<td>350</td>
<td>179</td>
<td>0.46</td>
<td>0.08</td>
</tr>
<tr>
<td>N-HHC</td>
<td>2.5</td>
<td>1159</td>
<td>1481</td>
<td>904</td>
<td>1.11</td>
<td>0.37</td>
</tr>
<tr>
<td>N-HHC-6</td>
<td>5.0</td>
<td>343</td>
<td>477</td>
<td>272</td>
<td>0.50</td>
<td>0.10</td>
</tr>
</tbody>
</table>

Note: The effect of TMB amount on the formation of N-HHCs (i.e., N-HHC-5, N-HHC and N-HHC-6 represents the amount of TMB added is 0.5, 2.5 and 5.0 mL, respectively) was further investigated. By ten-fold increasing the TMB volume from 0.5 to 5.0 mL, the
structures of the obtained Mn-ZIF-8@PDA precursors and their corresponding hollow hybrid carbons are approximately alike (Fig. S13). Interestingly, when the added TMB amount is 2.5 mL, the carbonization of Mn-ZIF-8@PDA is led to generation of appreciable amount of micropores and mesopores compared to the condition that added TMB amount is either 0.5 or 5.0 mL. As shown in Fig. S14, all the N-HHCs exhibit typical Type I and IV hybrid isotherms, suggesting the presence of micro- and mesoporous composite structures. The specific surface areas of prepared N-HHCs are respectively calculated to be 248, 1159 and 343 m²/g for N-HHC-5, N-HHC and N-HHC-6, respectively (Table S5).
Figure S15 TEM images of (a) Mn-ZIF-8@PDA and N-enriched porous carbons prepared at different pyrolysis temperatures: (b) 400 °C, (c) 600 °C, and (d) 800 °C.
**Figure S16** FTIR spectra of prepared catalysts precursor (a) and their carbonization products (b).
Figure S17 (a) micropore and (b) meso-/macropore size distributions of ZIF-8-600, Mn-ZIF-8-600, and N-HHC.
**Figure S18** FE-SEM images of synthesized materials: (a,d) ZIF-8-600, (b,e) Mn-ZIF-8-600, and (c,f) N-HHC.
Figure S19 (a) Three nitrogen species co-exist in nitrogen-containing carbon; (b) Schematic diagram of O₃ and toluene adsorbed on oxide pyridinic-N.
Figure S20 H₂-TPR profiles of prepared catalysts.
Figure S21 GC-MS chromatogram of organic byproducts in the NTP and NTP-catalytic systems with the specific input energy of (a) 217 J/L and (b) 278 J/L (1: benzene, 2: toluene, 3: benzaldehyde, 4: 2,6-dimethyl-nonane, 5: n-undecane, 6: n-dodecane, 7: á,á-dimethyl-1-oxo-benzene hexane nitrile, 8: octadecanoic acid, 2-oxo-, methyl ester, 9: 2,6,11-trimethyl-dodecane, 10: n-pentadecane, 11: 2-methyl- naphthalene, and 12: 1-methyl- naphthalene).