Electronic Supporting Information

Highly Porous Photoluminescent Diazaborole-Linked Polymers: Synthesis, Characterization, and Application to Selective Gas Adsorption

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Materials and Methods Table of Contents

<table>
<thead>
<tr>
<th>Section S1</th>
<th>Scanning Electron Microscopy Imaging (SEM)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Section S2</td>
<td>FT-IR Spectroscopy of Starting Materials and DBLPs</td>
</tr>
<tr>
<td>Section S3</td>
<td>TGA Trace for DBLPs</td>
</tr>
<tr>
<td>Section S4</td>
<td>Solid-State $^{11}$B and $^{13}$C CP-MAS NMR Spectrum for DBLPs</td>
</tr>
<tr>
<td>Section S5</td>
<td>Excitation-Emission Spectra for DBLPs</td>
</tr>
<tr>
<td>Section S6</td>
<td>PXRD Pattern for DBLPs</td>
</tr>
<tr>
<td>Section S7</td>
<td>Low Pressure (0 – 1.0 bar) Gas Adsorption Studies</td>
</tr>
</tbody>
</table>
Materials and Methods Section S1: Scanning Electron Microscopy Imaging (SEM) for DBLPs

In order to determine the purity of products, SEM was used to scan for the morphology present in the sample. SEM samples were prepared by dispersing the material onto a sticky carbon surface attached to a flat aluminum sample holder. The samples were then platinum coated using an EMS (Electron Microscopy Sciences) 550x Sputter Coater at $1 \times 10^{-5}$ mbar of pressure in a nitrogen atmosphere for 90 seconds while maintaining 20 mA of current. Samples were analyzed on a Hitachi SU-70 Scanning Electron Microscope using the SEI detector with accelerating voltages ranging from 10 kV to 20 kV.

Figure S1: SEM images of DBLP-3, DBLP-4, and DBLP-5.

DBLP-3: Aggregated particles

DBLP-4: Aggregated particles

DBLP-5: Aggregated particles
**Materials and Methods Section S2: Thermogravimetric Analysis**

Thermogravimetric analysis was obtained using a TA Instruments TGA Q5000 analyzer with 50 μL platinum pans to assess the thermal stability of each diazaborole-linked polymer. Experiments were run at a ramp rate of 5 °C/minute under a nitrogen atmosphere.

**Figure S2**: TGA traces of DBLP-3, DBLP-4, and DBLP-5

![TGA Traces](image-url)
**Materials and Methods Section S3: FT-IR Spectroscopy of DBLPs and Starting Materials.**

**Figure S3:** FT-IR spectra of DBLPs and their starting building units. Lower panel is an expanded region from 400 to 1600 cm$^{-1}$
Materials and Methods Section S4: Solid-State $^{11}\text{B}$ and $^{13}\text{C}$ Cross-Polarization Magic Angle Spinning (CPMAS) Nuclear Magnetic Resonance

Figure S4: Solid state $^{13}\text{C}$ CP-MAS and $^{11}\text{B}$ MAS NMR spectra of DBLPs collected with a 45 degree $^{11}\text{B}$ pulse length and a recycle delay of two seconds. Chemical shifts are referenced to external neat boron trifluoroetherate. Spectra were obtained using Tecmag-based NMR spectrometer, operated at a H-1 frequency of 363 MHz, using contact time of 1 ms and delay of three seconds for CP-MAS experiments by Spectral Data Services, Inc., Champaign, IL. Asterisks represent spinning side bands.
Materials and Methods Section S5: Photobleaching experiment

Figure S5: Photobleaching experiment of DBLP-4 polymer in dimethylformamide upon exposure to UV light for 40 minutes
Figure S6: Excitation and Emission Spectra for reactants, DBLP-3, and DBLP-5 (A) and combined emission spectra for all DBLPs (B).  

(A)
**Materials and Methods Section S6: XRD-patterns for the DBLPs**

**Figure S7:** XRD-patterns for the DBLPs. Broad peak at around $2\theta = 15$ indicates the amorphous characteristics of BILPs.
**Materials and Methods Section S7: Adsorption Measurements for the DBLPs**

**Activation of polymers for gas adsorption measurements:** A sample was loaded into a 9 mm large bulb cell (from Quantachrome) of known weight and then hooked up to MasterPrep. The sample was degassed at 120 °C for 16 hours. The degassed sample was weighed precisely and then transferred back to the analyzer. The temperatures for adsorption measurements was controlled by using refrigerated bath of liquid argon (87 K), and temperature controlled water bath (273 K and 298 K). Adsorption measurements were performed on an Autosorb-iQ (Quantachrome) volumetric analyzer using adsorbates of UHP grade.

**Figure S8:** Ar adsorption isotherm for DBLP-3 (black), DBLP-4 (blue) and DBLP-5 (red) measured at 87 K. The filled markers are adsorption points and the empty markers are desorption points.
**Figure S9:** Experimental Ar adsorption isotherm for, DBLP-3 (black), DBLP-4 (blue) and DBLP-5 (red) measured at 87 K. The calculated NLDFT isotherm is overlaid as open circle. Note that a fitting error of < 1 % indicates the validity of using this method for assessing the porosity of BILPs. The fitting error is indicated.
Figure S10: Pore Size Distribution for DBLP-3 (black), DBLP-4 (blue) and DBLP-5 (red) was calculated from the Ar adsorption isotherms by the Non-Local Density Functional Theory (NLDFT) method using a cylindrical pore model.
Figure S11: BET plot for DBLP-3 (black), DBLP-4 (blue) and DBLP-5 (red) calculated from the Ar adsorption isotherm at 87 K. The model was applied from $P/P_0 = 0.04-0.16$. The correlation factor is indicated. ($W =$ Weight of gas absorbed at a relative pressure $P/P_0$).
Figure S12: Gas uptake isotherms for DBLP-3 (black), DBLP-4 (blue) and DBLP-5 (red) at 273 and 298 K.
Figure S13: Virial analysis of CO$_2$ adsorption data for DBLP-3, DBLP-4, and DBLP-5 and their isosteric heat of adsorption ($Q_{st}$).
Figure S14: Virial analysis of H₂ adsorption data for DBLP-3, DBLP-4, and DBLP-5 and their isosteric heat of adsorption ($Q_{st}$).
Figure S15: Virial analysis of CH$_4$ adsorption data for DBLP-3, DBLP-4, and DBLP-5 and their isosteric heat of adsorption ($Q_{st}$).
Figure S16: Gas sorption capacities for DBLP-3, DBLP-4, and DBLP-5 at 298 K. CO₂ (black squares), CH₄ (red circles) and N₂ (green triangle).
**Figure S17:** Adsorption selectivity of CO$_2$ over N$_2$ and CH$_4$ for DBLPs from initial slope calculations. CO$_2$ (black), CH$_4$ (red) and N$_2$ (blue) isotherms collected at and 298 K.