Electronic Supplementary Information

Integration process of CO$_2$ capture and *in situ* hydrogenation to formate with tunable ethoxyl-functionalized amidine and Rh/bisphosphine system†

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1. General experimental methods

Materials

Metal salts and ligands were obtained commercially from J&K Chemical and Aladdin without further purification. The solvents of chromatographically purity were obtained commercially and distilled prior to use. The absorbent materials were synthesized according to the references.1-7

Instruments

$^1$H NMR spectra was recorded at Bruker 400 or Varian Mercury-Plus 400 spectrometer in CDCl$_3$, D$_2$O or d$_6$-DMSO and TMS (0 ppm) was used as internal reference. $^{13}$C NMR was recorded at 100.6 MHz in CDCl$_3$ or D$_2$O and CDCl$_3$ (77.0 ppm) was used as internal reference. $^{31}$P NMR (162 MHz, CDCl$_3$) chemical shifts were measured relative to an external orthophosphoric acid standard. Infrared (IR) spectra were recorded on a Bruker Tensor 27 FT-IR spectrophotometer with KBr pellets. In situ FTIR was collected on a Mettler Toledo React IR ic10, inner silicon probe, using ic IR analysis system. The probe is placed at the bottom of the autoclave, which is constantly stirred by mechanical stirring, and the spectra are collected in situ during the whole process. ESI-MS were recorded on a Thermo Finnigan LCQ Advantage spectrometer in ESI mode with a spray voltage of 4.8 kV. GC analyses were performed on Shimadzu GC-2014, equipped with a capillary column (RTX-17, 30 m × 0.25 μm) using a flame ionization detector. GC-MS analyses were performed on SHIMADZU GCMS-QP2010 SE and GC equipped with a capillary column (Rxi-5MS, 30 m × 0.25 μm). TGA was performed on a PTC-10A TG-DTA analyzer (Japan, Rigaku) under air flow from room temperature to 700 °C with a heating rate of 10 °C/min. Elementary analysis was performed on vario ELCUBE analyzer. The scanning electron micro copy (SEM) images were taken with a JEOLJSM-6700F field emission scanning electron microscope (15 kV). The surface area was calculated using a multipoint Brunauer eEmmett e Teller (BET) model. The total pore volume was estimated at a relative pressure of 0.99, assuming full surface saturation with
nitrogen.

All of the theoretical calculations here were carried out using the GAUSSIAN 09 packages. The geometries were completely optimized by the B3LYP method in conjunction with 6-31+G(d) basis set. All the final structures were confirmed by frequency calculation to be the real minima without any imaginary frequency using the same level of theory. The solvent effect using the Conductor-like Polarizable Continuum Model (CPCM) in each case was carried out at the M06-2X/6-311++G(d,p) level. Solute-solvent interactions can have dramatic effects on molecular energies, structures, and properties, and ethanol was chosen as solvent environment. All the bond lengths are in angstroms (Å). Structures were generated using GaussView 5.0.

**Synthesis of amidine derivatives**

*Synthesis of 1,2-bis(2-bromoethoxy)ethane (BrPEG₁₅₀Br)*

\[
\text{PEG}_{150} \quad \text{OH} \quad + \quad \text{PBr}_3 \quad \xrightarrow{\text{pyridine}} \quad \text{BrPEG}_{150} \quad \text{Br}
\]

To a stirred mixture of triethylene glycol (PEG₁₅₀) (0.13 mol) and pyridine (0.51 mol) at 0 °C, phosphorus tribromide (0.103 mol, distilled) was added dropwise over 30 min. The resulting suspension was heated at 60 °C for 4 h, and then the mixture was poured into ice-water (30 mL). The lower organic layer was washed with water (5 × 20 mL), and then it was dried with MgSO₄. Finally, the target compound BrPEG₁₅₀Br was purified through distillation under reduced pressure as a light yellow liquid.

**Synthesis of PEG₁₅₀MeCl**

\[
\text{PEG}_{150} \quad \text{OH} \quad + \quad \text{SOCl}_2 \quad \xrightarrow{\text{pyridine, benzene}} \quad \text{ClPEG}_{150} \quad \text{OMe}
\]

A solution of thionyl chloride (0.45 mol) in CHCl₃ (90 mL) was added slowly over 60 min to a stirred solution of triethylene glycol monomethyl ether (0.3 mol) and pyridine (0.3 mol) in CHCl₃ (200 mL), followed by refluxing the above reaction mixture at 100 °C for 4 h, and then a yellow liquid mixture is obtained, which was
washed with water (4×125 mL), dried with MgSO₄, and concentrated under reduced pressure at 60 °C to remove CHCl₃. The crude product was purified under reduced pressure to give PEG₁₅₀MeCl as a light yellow liquid.²

**Synthesis of ionic liquids DBU₂PEG₁₅₀Br₂**

![Chemical structure of DBU₂PEG₁₅₀Br₂](image)

Under N₂, DBU (20 mmol) and BrPEG₁₅₀Br (34.4 mmol) were added to dry toluene (2 mL) in a round-bottomed flask. The mixture was heated under reflux under N₂ for 24 h, upon which two layers had formed. The flask was allowed to cool to room temperature and was then cooled to -10 °C overnight, during which time white solid or yellow oil were formed. The excess toluene was decanted, while N₂ was being passed over the product layer. The resulting product was washed with dry Et₂O and then dried in vacuo for 24 h to obtain DBU₂PEG₁₅₀Br₂ as brown oil.¹

**Synthesis of DBUOH**

![Chemical structure of DBUOH](image)

The reaction was carried out on a scale of 10 g under Ar atmosphere. DBU (1.0 equiv.) was added to the dry THF (50 mL) and it was cooled to -78 °C by liquid nitrogen-acetone bath. n-BuLi (1.6 M in hexane) (1.05 equiv.) was added dropwise into the solution at -78 °C for 20 min. After stirring for 2 h at -78 °C and for 1 h at -20 °C, a solution of propylene oxide (0.5 equiv.) in THF was added dropwise for 20 min at -10 °C. After stirring for 1 h at -10 °C, the solution was warmed up to room temperature slowly. The mixture was stirred for 2 h and hydrolyzed by deionized water (1.05 equiv.) for 30 min. The mixture was filtered and evaporated. DBUOH was obtained by fractional vacuum distillation.³
Synthesis of DBUPEG\textsubscript{150}Me

The reaction was carried out under Ar atmosphere. DBU (45 mmol, 6.84 g) was added to the dry THF (50 mL) and it was cooled to -78 °C by liquid nitrogen-acetone bath. n-BuLi (1.6 M in hexane) (47.3 mmol, 30 mL) was added dropwise into the solution at -78 °C for 20 min. After stirring for 2 h at -78 °C and for 1 h at -20 °C, a solution of PEG\textsubscript{150}MeCl (45 mmol, 8.22 g) in THF was added dropwise for 20 min at -10 °C. After stirring for 1 h at -10 °C, the solution was warmed up to room temperature slowly. The mixture was stirred for 2 h and hydrolyzed by deionized water for 30 min. The mixture was filtered and evaporated. DBUPEG\textsubscript{150}Me was obtained by column chromatography.\textsuperscript{4}

Synthesis of TMGPEG\textsubscript{150}Me

In a dry flask under N\textsubscript{2} atmosphere, 1,1,3,3-tetramethylguanidine (20 mmol, 2.30 g) and xylene (0.3 mL) were mixed together, and the system was heated up to 120 °C and maintained at this temperature for 2 h, and then PEG\textsubscript{150}MeCl (10 mmol, 1.83 g) was added dropwise over 2 h. After being stirred at 120 °C for 7 h under N\textsubscript{2} atmosphere, the resulting mixture was allowed to cool down to room temperature. The formed salt precipitates 1,1,3,3-tetramethylguanidine·HCl was filtrated out. The filtrate liquid was distilled under reduced pressure and TMGPEG\textsubscript{150}Me was thus obtained.\textsuperscript{5}

Synthesis of DBUOH@Silica
In a typical surface modification process, activated HG/T2354-92 H silica (8 g, 150 °C, under vacuum) was first treated with a refluxing anhydrous toluene solution (50 mL) of 3-glycidoxypropyltrimethoxysilane (25 mmol, 6 g). The solution was refluxed for 24 h at 120 °C under an inert N₂ atmosphere. The solution was filtered and the solid was washed subsequently with toluene, dichloromethane, and methanol, and dried under reduced pressure at 80 °C for 10 h to obtain epoxide@Silica as white powder, which was analyzed by IR spectroscopy.⁶

Then, the reaction was carried out under Ar atmosphere. DBU (30 mmol, 4.56 g) was added to the dry THF (50 mL) and it was cooled to -78 °C by liquid nitrogen-acetone bath. n-BuLi (1.6 M in hexane) (40 mmol, 25 mL) was added dropwise into the solution at -78 °C for 20 min. After stirring for 2 h at -78 °C and for 1 h at -20 °C, epoxide@Silica (13 g) was added in batches under inert atmosphere. Then, after stirring for 1 h at -10 °C, the solution was warmed up to room temperature slowly. The mixture was stirred overnight and hydrolyzed by methanol for 30 min. The mixture was filtered and evaporated to give the DBUOH@Silica as white powder, which was analyzed by IR spectroscopy and CHN elemental analysis.³

**Synthesis of DBU@PS**

The reaction was carried out under Ar atmosphere. DBU (30 mmol, 4.56 g) was added to the dry THF (50 mL) and it was cooled to -78 °C by liquid nitrogen-acetone bath. n-BuLi (1.6 M in hexane) (32 mmol, 20 mL) was added dropwise into the solution at -78 °C for 20 min. After stirring for 2 h at -78 °C and for 1 h at -20 °C,
activated chloromethylated polystyrene resin (18-19% Cl) was added to the solution under Ar atmosphere. Then, after stirring for 1 h at -10 °C, the solution was warmed up to room temperature slowly. The mixture was stirred overnight and hydrolyzed by methanol for 30 min. The mixture was filtered and evaporated to give the DBU@PS, which was analyzed by IR spectroscopy.  

2. Table S1 and Figures S1-S10  

<table>
<thead>
<tr>
<th>Amidine derivative</th>
<th>Surface area ($S_{BET}$) (m$^2$ g$^{-1}$)</th>
<th>$V_{total}$ (cm$^3$ g$^{-1}$)$^a$</th>
<th>Base loading (wt %)$^b$</th>
</tr>
</thead>
<tbody>
<tr>
<td>DBU@PS</td>
<td>27.3</td>
<td>0.09</td>
<td>20.0%</td>
</tr>
<tr>
<td>DBUOH@Silica</td>
<td>98.5</td>
<td>0.07</td>
<td>11.3%</td>
</tr>
</tbody>
</table>

$^a$ Total volume calculated at P/P$_0 =$ 0.99. $^b$ Measured by Elemental Analysis.

Figure S1: CO$_2$ capture/activation by alkanolamidine with enthalpy changes. (Calculated by M06-2X/6-311++G(d,p)/B3LYP/6-31+G(d)/PCM method in ethanol.) A: DBUOH; B: DBUH$^+$OCO$_2$; C: DBUH$^+$OCO$_2^-$ with intramolecular hydrogen bond. H: white, N: blue, O: red. N-H bond (1.10 Å) and O-H bond (1.46 Å) in the cyclic capture product C.
Figure S2 Temperature and time effect under CO$_2$ (4 MPa) and H$_2$ (4 MPa) pressure. Reaction conditions: (a) RhCl$_3$·3H$_2$O (0.01 mmol, 2.6 mg), DPEphos (0.01 mmol, 27 mg), DBU (3.3 mmol), methanol (3 mL), 16 h. (b) RhCl$_3$·3H$_2$O (0.005 mmol, 1.3 mg), DPEphos (0.025 mmol, 13.5 mg), DBU (6.6 mmol), methanol (3 mL), 60 °C.

Figure S3 FT-IR spectra of the recovered DBUOH@Silica (a) and recovered DBUOH@Silica + CO$_2$ (b).
Figure S4 TGA of DBUOH@Silica. N₂, 10 °C min⁻¹, temperature range: 0-700 °C. The weight loss did not appear until heating to 189 °C.

Figure S5 Reversible CO₂ absorption and desorption (a) and three consecutive cycles (b, c) by DBUOH@Silica with amidine content of 11.3% at 25 °C.
Figure S6 \(^1\)H (400 MHz, DMSO-\(d_6\)) and \(^{13}\)C NMR (100.6 MHz, DMSO-\(d_6\)) spectra of DBUOH@Silica.
Figure S7 $^1$H NMR spectrum for reaction mixture of CO$_2$ gas hydrogenation. Conditions: RhCl$_3$·3H$_2$O (0.005 mmol, 1.3 mg), DPEphos (0.025 mmol, 13.5 mg), DBUOH (3 mmol, 0.64 g), methanol (3 mL), H$_2$ (4 MPa), CO$_2$ (4 MPa), 60 °C, 16 h.

Figure S8 $^1$H NMR spectrum for reaction mixture of captured CO$_2$ hydrogenation. RhCl$_3$·3H$_2$O (0.005 mmol, 1.3 mg), DPEphos (0.05 mmol, 13 mg), DBUOH + CO$_2$ in tetraethylene glycol dimethyl ether (3 mmol : 2.85 mmol : 5 mmol), methanol (3 mL), H$_2$ (4 MPa), 60 °C, 16 h.
Figure S9 CG-MS analysis for reaction mixture of captured CO₂ hydrogenation (a) and gaseous CO₂ hydrogenation (b). Conditions: (a) RhCl₃·3H₂O (0.005 mmol, 1.3 mg), DPEphos (0.05 mmol, 13 mg), DBUOH + CO₂ in tetraethylene glycol dimethyl ether (3 mmol : 2.85 mmol : 5 mmol), methanol (3 mL), H₂ (4 MPa), 60 °C, 16 h; (b) RhCl₃·3H₂O (0.005 mmol, 1.3 mg), DPEphos (0.05 mmol, 13 mg), DBUOH (3 mmol, 0.64 g), methanol (3 mL), CO₂ (4 MPa), H₂ (4 MPa), 60 °C, 16 h.
**3. The Characterization data for the absorption mixture**

PEGl\textsubscript{150} (triethylene glycol, MW = 150 Da)

\( ^1\)H NMR (400 MHz, CDCl\textsubscript{3}) \( \delta \) 3.71 (t, \( ^3J = 4.8\) Hz, 4 H), 3.65 (s, 4 H), 3.59 (t, \( ^3J = 4.8\) Hz, 4 H), 3.40 (s, 2 H); \( ^{13}\)C NMR (100.6 MHz, CDCl\textsubscript{3}) \( \delta \) 72.6, 70.25, 61.50.

**PEG\textsubscript{150}MeCl**

\( ^1\)H NMR (400 MHz, CDCl\textsubscript{3}) \( \delta \) 3.44-3.45 (m, 4H), 3.67 (d, \( ^3J = 2.8\) Hz, 4H), 3.78-3.81 (m, 4H); \( ^{13}\)C NMR (100.6 MHz, CDCl\textsubscript{3}) \( \delta \) 161.30, 52.60, 48.14, 44.04, 37.21, 29.56, 28.32, 25.80, 22.29.

**BrPEG\textsubscript{150}Br**

\( ^1\)H NMR (400 MHz, CDCl\textsubscript{3}) \( \delta \) 3.73 (s, 4H), 2.66 (s, 1H), 2.30 (s, 1H); \( ^{13}\)C NMR (100.6 MHz, CDCl\textsubscript{3}) \( \delta \) 63.68.

**DBU**

\( ^1\)H NMR (400 MHz, CDCl\textsubscript{3}) \( \delta \) 3.67-3.73 (m, 12H), 3.60-3.62 (m, 4H), 3.49-3.51 (m, 8H), 2.83 (d, \( ^3J = 8.8\) Hz, 4H), 2.04 (t, \( ^3J = 5.2\) Hz, 4H), 1.68-1.71 (m, 12H); \( ^{13}\)C NMR (100.6 MHz, D\textsubscript{2}O) \( \delta \) 167.2, 70.1, 68.0, 54.8, 52.5, 48.9, 47.2, 28.0, 25.4, 22.6, 19.6; ESI-MS caled for C\textsubscript{2}H\textsubscript{4}Br\textsubscript{2}N\textsubscript{4}O\textsubscript{2} 578.18, found 210.5 [(M-2Br)/2]\textsuperscript{+}, 499.4, 501.4 [M-Br]\textsuperscript{+}.

**HOCH\textsubscript{2}CH\textsubscript{2}OH (Glycol)**

\( ^1\)H NMR (400 MHz, CDCl\textsubscript{3}) \( \delta \) 3.73 (s, 4H), 2.66 (s, 1H), 2.30 (s, 1H); \( ^{13}\)C NMR (100.6 MHz, CDCl\textsubscript{3}) \( \delta \) 63.68.

**DBU\textsubscript{2}PEG\textsubscript{150}Br\textsubscript{2}**

\( ^1\)H NMR (400 MHz, D\textsubscript{2}O) \( \delta \) 3.67-3.73 (m, 12H), 3.60-3.62 (m, 4H), 3.49-3.51 (m, 8H), 2.83 (d, \( ^3J = 8.8\) Hz, 4H), 2.04 (t, \( ^3J = 5.2\) Hz, 4H), 1.68-1.71 (m, 12H); \( ^{13}\)C NMR (100.6 MHz, D\textsubscript{2}O) \( \delta \) 167.2, 70.1, 68.0, 54.8, 52.5, 48.9, 47.2, 28.0, 25.4, 22.6, 19.6; ESI-MS caled for C\textsubscript{2}H\textsubscript{4}Br\textsubscript{2}N\textsubscript{4}O\textsubscript{2} 578.18, found 210.5 [(M-2Br)/2]\textsuperscript{+}, 499.4, 501.4 [M-Br]\textsuperscript{+}.
**DBUPEG$_{150}$Me**

$^1$H NMR (400 MHz, CDCl$_3$) δ 3.68-3.47 (m, 13H), 3.32-3.29 (m, 3H), 3.16-3.12 (s, 5H), 2.31 (s, 1H), 1.71-1.49 (m, 8H); $^{13}$C NMR (100.6 MHz, CDCl$_3$) δ 165.04, 71.73, 71.16, 70.45, 70.40, 58.87, 53.94, 48.50, 42.58, 39.13, 33.12, 29.03, 27.06, 24.32, 20.03. ESI-MS: m/z 299.4 [M+H$^+$].

**TMGPEG$_{150}$Me**

$^1$H NMR (400 MHz, CDCl$_3$) δ 2.60 (s, 6H), 2.69 (s, 6H), 3.27 (t, $^3$J = 6.4 Hz, 2H), 3.32 (s, 3H), 3.58-3.50 (m, 4H), 3.60 (s, 6H); $^{13}$C NMR (100.6 MHz, CDCl$_3$) δ 161.00, 73.23, 71.80, 70.50, 70.33, 70.22, 58.87, 49.30, 39.48, 38.68. ESI-MS: m/z 262.2 [M+H$^+$].

**DBUOH**

$^1$H NMR (400 MHz, CDCl$_3$) δ 1.01-1.07 (m, 3H), 1.69-1.56 (m, 7H), 1.94 (s, 1H), 2.35-2.45 (m, 3H), 2.49-2.61 (m, 2H), 3.25-3.27 (m, 2H), 3.30-3.48 (m, 2H), 3.57 (s, OH), 3.68-3.79 (m, 1H); $^{13}$C NMR (100.6 MHz, CDCl$_3$) δ 176.19, 64.75, 56.60, 49.35, 45.72, 45.30, 36.92, 29.73, 28.31, 27.57, 23.23, 20.42; ESI-MS: m/z 211.4 [M+H$^+$].

**3-Glycidoxypropyltrimethoxysilane**

$^1$H NMR (400 MHz, CDCl$_3$) δ 3.64-3.67 (m, 2H), 3.51 (s, 9H), 3.38-3.44 (m, 1H), 3.30-3.34 (m, 1H), 3.08 (s, 1H), 2.73 (s, 1H), 2.55 (s, 1H), 1.64 (t, $^3$J = 8.0 Hz, 2H), 0.617 (t, $^3$J = 8.4 Hz, 2H); $^{13}$C NMR (100.6 MHz, CDCl$_3$) δ 73.37, 71.24, 50.71, 50.35, 44.17, 22.66, 5.08.

**DBUOH@Silica**

$^1$H NMR (400 MHz, DMSO-d$_6$) δ 3.53 (s, OH), 3.04-3.16 (m, 8H), 2.25 (s, 2H), 1.26-1.63 (m, 11H), 0.84 (s, 3H), 0.46-0.54 (m, 3H); $^{13}$C NMR (100.6 MHz, DMSO-d$_6$) δ 160.78, 70.71, 64.17, 52.01, 48.77, 47.67, 43.24, 35.99, 29.18, 28.14, 26.37, 25.81, 22.25, 13.81, 11.85.

**DBUPEG$_{150}$Me /Glycol (1:2)+CO$_2$**

$^1$H NMR (400 MHz, CDCl$_3$) δ 1.57-1.65 (m, 6H), 1.86-1.95 (m, 1H), 2.68-2.76 (m, 2H), 3.18-3.28 (m, 7H), 3.28-3.56 (m, 22H); $^{13}$C NMR (100.6 MHz, CDCl$_3$) δ 165.76, 151.29, 71.47, 70.90, 70.13, 63.40, 58.61, 54.13, 48.36, 42.47, 37.76, 32.11, 28.58, 26.32, 23.57, 19.08.

**TMGPEG$_{150}$Me /Glycol (1:5)+CO$_2$**

$^1$H NMR (400 MHz, CDCl$_3$) δ 2.78 (t, $^3$J = 8.0 Hz, 9H), 3.16 (t, $^3$J = 6.8 Hz, 4H), 3.33 (s, 2H), 3.41-3.45 (m, 30H), 3.77 (s, 2H), 5.0 (s, 1H); $^{13}$C NMR (100.6 MHz, CDCl$_3$) δ 162.17, 158.73, 71.27, 69.83, 69.70, 69.03, 66.66, 63.00, 61.12, 58.31, 44.26, 39.25.

**DBUOH+CO$_2$**

$^1$H NMR (400 MHz, CDCl$_3$) δ 0.92-1.00 (m, 3H), 1.47-1.67 (m, 8H), 2.34-2.43 (m, 3H), 2.51-2.58 (m, 2H), 3.18-3.31 (m, 4H), 3.61 (s, 0.5H), 3.79 (s, 0.5H), 5.21 (s, OH); $^{13}$C NMR (100.6 MHz, CDCl$_3$) δ 176.19, 164.25, 63.90, 55.93, 49.19, 45.36, 45.01, 36.64, 29.48, 28.04, 26.44, 23.00, 20.41.
4. $^1$H NMR and $^{13}$C NMR Charts
1H NMR (400 MHz, CDCl₃)

13C NMR (100.6 MHz, CDCl₃)
$^{1}H$ NMR (400 MHz, CDCl₃)

$\text{NMR (100.6 MHz, CDCl₃)}$
$^1$H NMR (CDCl$_3$, 400 MHz)

$^{13}$C NMR (CDCl$_3$, 100.6 MHz)
$^1$H NMR (CDCl$_3$, 400 MHz)
$^{13}$C NMR (CDCl$_3$, 100.6 MHz)

![Chemical structure with NMR spectrum]

**Chemical Structure**

1. $^1$H NMR (CDCl$_3$, 100.6 MHz)

**NMR Spectrum**

- 77.318 ppm
- 77.204 ppm
- 77.081 ppm
- 76.883 ppm
- 71.258 ppm
- 70.406 ppm
- 70.320 ppm
- 58.868 ppm
- 48.206 ppm
- 39.479 ppm
- 38.676 ppm
1H NMR (CDCl$_3$, 400 MHz)
13C NMR (CDCl₃, 100.6 MHz)

1H NMR (400 MHz, DMSO-d₆)
$^{13}$C NMR (100.6 MHz, DMSO-$d_6$)

$^1$H NMR (CDCl$_3$, 400 MHz)
$^{13}$C NMR (CDCl$_3$, 100.6 MHz)

$^1$H NMR (CDCl$_3$, 400 MHz)
$^{13}$C NMR (CDCl$_3$, 100.6 MHz)

$^1$H NMR (CDCl$_3$, 400 MHz)
5. **IR spectra**

**DBU@PS**

![IR spectrum of DBU@PS](image)

**Epoxide@Silica**
DBUOH@Silica

DBUOH@Silica+CO₂
DBUOH

DBUOH+CO₂
$\text{TMGPEG}_{150}\text{Me}$

$\text{TMGPEG}_{150}\text{Me} + \text{Glycol} + \text{CO}_2$
6. ESI-MS
36
7. References