Supporting Information

Adamantane Ionic Liquids

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Experimental Section

**General.** $^1$H NMR and $^{13}$C NMR spectra were measured with a JEOL EX–400 (400 MHz for $^1$H and 100 MHz for $^{13}$C) spectrometer. Coupling constants ($J$ value) are reported in hertz. The chemical shifts are expressed in ppm downfield from tetramethylsilane, using residual chloroform ($\delta = 7.24$ in $^1$H NMR, $\delta = 77.0$ in $^{13}$C NMR) as an internal standard. MASS spectra were obtained on a JEOL JMS–SX102A. Water abundances were evaluated with a Karl–Fischer Moisture Titrator MKC–501, Kyoto Electronics Manufacturing, Co., Ltd. (Kyoto, Japan). Powder XRD patterns were recorded on a SHIMADZU X-ray diffractometer-6000 with high-intensity Cu Kα radiation at a scanning rate of 0.02° S$^{-1}$ in 2$\theta$ ranging from 2 to 90°.

**Materials.** Imidazolium salts with various lengths of alkyl chains were synthesized according to previous reports.$^{1,2}$ Hydroxyl forms were prepared before mixing with the carboxylates. Other reagents and solvents were commercially available and used without further purification. Synthetic scheme is shown in Scheme S1.

**Bromination of adamantane.** The procedures and characterization data of 2a,b were reported in the previous report.$^3$ Compounds 2c and 2d were commercially available and used without further purification. To the mixture of AlBr$_3$ powder (64.0 g, 0.240 mol) in bromine (100 g, 0.628 mol) adamantane (2.13 g, 15.6 mmol) was added at 0 °C. Then, the mixture was refluxed for 24 h. After quenching the reaction by adding sat. NaHSO$_3$aq. (100 mL), 1 N HCl (10 mL) was poured. The products were extracted with chloroform, and the organic layer was washed with water and brine, dried over Na$_2$SO$_4$, and filtrated. After evaporation to remove volatiles, 2a was obtained as a white solid from the crystallization in 2-propanol (28%). To the mixture of Fe powder (0.710 g, 12.7 mmol) in bromine (25 g, 0.16 mol) adamantane (2.13 g, 15.6 mmol) was added at 0 °C. Then, the mixture was
refluxed for 24 h. After reaction, purification method was similar to 2a and obtained 2b as a white solid (39%).

**Compound 3.** The typical protocol is described here\textsuperscript{4,5}: The mixture containing 2 (for 2a: 10.42 g, 23.06 mmol), 2,2'-azobis(isobutyronitrile) (AIBN, 0.909 g, 5.534 mmol), toluene (80 mL), acetonitrile (9.790 mL, 184.5 mmol), and Bu\textsubscript{3}SnH (32.22 g, 110.7 mmol) was refluxed for 5 h under Ar atmosphere. After cooling to room temperature, the products were extracted with ethyl acetate, and the organic layer was washed with 1 N NH\textsubscript{3}aq. and brine, dried over Na\textsubscript{2}SO\textsubscript{4}, and filtrated. After evaporation, the silica gel column chromatography was performed with the mixture eluent (chloroform / methanol = 30 / 1). The recrystallization with hexane and ethyl acetate afforded the compounds as a white powder (3a: 39%, 3b: 25%, 3c: 33%, 3d: 53%). 3a: \textsuperscript{1}H NMR (DMSO-\textit{d}\textsubscript{6}) \(\delta\) 1.04 (s, 12H), 1.43 (t, 8H, \(J = 7.94\) Hz), 2.41 (t, 8H, \(J = 7.82\) Hz). \textsuperscript{13}C NMR (DMSO-\textit{d}\textsubscript{6}) \(\delta\) 10.41, 33.70, 38.88, 43.53, 121.25.

**Adamantane carboxylates, Ada(CH\textsubscript{2}CH\textsubscript{2}COOH)\textsubscript{m}**. The typical protocol is described here: The mixture of 3 (for 3a: 2.289 g, 6.5735 mmol) in 6N HCl (20 mL) was refluxed for 2 days. After cooling to room temperature, the solution was poured into excess amount of ice water. Then, the precipitation was collected with filtration. The desired compound was obtained as a white powder from the recrystallization in acetonitrile (Ada(CH\textsubscript{2}CH\textsubscript{2}COOH)\textsubscript{4}: 90%, Ada(CH\textsubscript{2}CH\textsubscript{2}COOH)\textsubscript{3}: 94%, Ada(CH\textsubscript{2}CH\textsubscript{2}COOH)\textsubscript{2}: 90%, Ada(CH\textsubscript{2}CH\textsubscript{2}COOH): 94%). Ada(CH\textsubscript{2}CH\textsubscript{2}COOH)\textsubscript{4}: \textsuperscript{1}H NMR (DMSO-\textit{d}\textsubscript{6}) \(\delta\) 0.99 (s, 12H), 1.34 (t, 8H, \(J = 8.20\) Hz), 2.13 (t, 8H, \(J = 8.10\) Hz), 11.94 (s, 4H). \textsuperscript{13}C NMR (DMSO-\textit{d}\textsubscript{6}) \(\delta\) 27.80, 33.42, 37.70, 45.05, 175.14. HRMS (NBA) [M+Cl]\textsuperscript{−} calcd. 459.1791, found 459.1789.

**General procedure for the preparation of the salts.** Desired equivalent mole of bromide anion to the carboxyl groups was converted into the hydroxyl form by anion exchange...
resin (Amberlite–IRA400) in water, and neutralized with each carboxyl compound suspended in methanol (2 L). The aqueous solution was concentrated by a rotary evaporator and the residual liquid was freeze dehydrated to give the white solid. The solid was dried \textit{in vacuo} and stored in a glove box. [C(4)mim]$_4$Ada(CH$_2$CH$_2$COO)$_4$: $^1$H NMR (DMSO-d$_6$) $\delta$ 0.83 (s, 12H), 0.89 (t, 12H, $J = 7.40$ Hz), 1.18 (t, 8H, $J = 8.00$ Hz), 1.16–1.27 (m, 8H), 1.66 (t, 8H, $J = 8.20$ Hz), 1.75 (q, 8H, 7.30 Hz), 3.86 (s, 12H), 4.43 (t, 8H, $J = 7.40$ Hz), 7.72 (s, 4H), 7.79 (s, 4H), 9.69 (s, 4H). $^{13}$C NMR (DMSO-d$_6$) $\delta$ 13.24, 18.74, 31.36, 32.72, 33.43, 35.59, 41.00, 46.73, 48.35, 122.14, 123.49, 137.34, 175.89.

[C(6)mim]$_4$Ada(CH$_2$CH$_2$COO)$_4$: $^1$H NMR (DMSO-d$_6$) $\delta$ 0.84 (m, 24H), 1.25 (m, 32H), 1.68 (m, 8H), 1.76 (m, 8H), 3.85 (s, 12H), 4.16 (t, 8H, $J = 7.00$ Hz), 7.71 (s, 4H), 7.78 (s, 4H), 9.66 (s, 4H). $^{13}$C NMR (DMSO-d$_6$) $\delta$ 13.82, 21.87, 25.15, 29.40, 30.56, 32.65, 33.44, 35.54, 40.95, 46.69, 48.58, 122.15, 123.46, 137.65, 176.25.

[C(8)mim]$_4$Ada(CH$_2$CH$_2$COO)$_4$: $^1$H NMR (DMSO-d$_6$) $\delta$ 0.82–0.86 (m, 24H), 1.23 (m, 48H), 1.67 (m, 8H), 1.76 (m, 8H), 3.86 (s, 12H), 4.16 (t, 8H, $J = 7.20$ Hz), 7.71 (s, 4H), 7.80 (s, 4H), 9.86 (s, 4H). $^{13}$C NMR (DMSO-d$_6$) $\delta$ 13.89, 22.00, 25.48, 28.31, 28.45, 29.40, 31.12, 32.71, 33.54, 35.54, 40.99, 46.71, 48.58, 122.13, 123.43, 137.58, 176.09.

[C(10)mim]$_4$Ada(CH$_2$CH$_2$COO)$_4$: $^1$H NMR (DMSO-d$_6$) $\delta$ 0.82 (m, 24H), 1.23 (m, 64H), 1.67 (m, 8H), 1.76 (m, 8H), 3.85 (s, 12H), 4.15 (t, 8H, $J = 7.20$ Hz), 7.71 (s, 4H), 7.78 (s, 4H), 9.57 (s, 4H). $^{13}$C NMR (DMSO-d$_6$) $\delta$ 13.91, 22.11, 25.57, 28.47, 28.71, 28.91, 29.86, 29.58, 31.31, 32.46, 33.49, 35.51, 40.77, 46.61, 48.58, 122.23, 123.50, 137.81, 176.97.

[C(12)mim]$_4$Ada(CH$_2$CH$_2$COO)$_4$: $^1$H NMR (DMSO-d$_6$) $\delta$ 0.82 (m, 24H), 1.23 (m, 80H), 1.67 (t, 8H, $J = 7.60$ Hz), 1.76 (m, 8H), 3.86 (s, 12H), 4.16 (t, 8H, $J = 7.20$ Hz), 7.73 (s, 4H), 7.80 (s, 4H), 9.85 (s, 4H). $^{13}$C NMR (DMSO-d$_6$) $\delta$ 13.89, 22.03, 25.47, 28.35, 28.66, 28.79, 28.90, 28.96, 28.97, 29.40, 31.24, 32.73, 33.43, 35.54, 40.99, 46.73, 48.58, 122.12, 123.43, 137.56, 175.99. [C(14)mim]$_4$Ada(CH$_2$CH$_2$COO)$_4$: $^1$H NMR (DMSO-d$_6$) $\delta$ 0.82 (m, 24H), 1.22 (m, 96H), 1.76 (m, 16H), 3.86 (s, 12H), 4.16 (t, 8H, $J = 7.20$ Hz), 7.73 (s, 4H), 7.81 (s, 4H), 9.86 (s, 4H). $^{13}$C NMR (DMSO-d$_6$) $\delta$ 13.87, 22.03, 25.48, 28.36, 28.64, 28.80, 28.91, 28.96, 28.99, 29.41, 31.23, 32.37, 33.44, 35.53, 40.76, 46.60, 48.58, 122.12, 123.43,
[C(16)mim]₄Ada(CH₂CH₂COO)₄: ¹H NMR (DMSO-d₆) δ 0.82 (m, 24H), 1.22 (m, 112H), 1.69 (m, 8H), 1.76 (m, 8H), 3.86 (s, 12H), 4.16 (t, 8H, J = 7.20 Hz), 7.72 (s, 4H), 7.79 (s, 4H), 9.74 (s, 4H). ¹³C NMR (DMSO-d₆) δ 13.87, 22.03, 25.47, 28.35 ~ 39.38, 31.22, 33.42, 35.56, 46.65, 48.49, 122.13, 123.44, 137.39, 176.00.

[C(18)mim]₄Ada(CH₂CH₂COO)₄: ¹H NMR (DMSO-d₆) δ 0.84 (m, 24H), 1.22 (m, 128H), 1.76 (m, 8H), 1.92 (m, 8H), 3.83 (s, 12H), 4.13 (t, 8H, J = 7.20 Hz), 7.68 (s, 4H), 7.75 (s, 4H), 9.13 (s, 4H). ¹³C NMR (DMSO-d₆) δ 13.94, 22.07, 25.49, 28.37~29.39, 31.27, 33.47, 35.68, 46.38, 48.69, 122.20, 123.54, 137.52, 176.05.

[C(20)mim]₄Ada(CH₂CH₂COO)₄: ¹H NMR (DMSO-d₆) δ 0.80–0.87 (m, 6H), 1.23 (ddd, 2H, J = 15.00, 7.30 Hz), 3.86 (s, 3H), 4.17 (t, 2H, J = 7.20 Hz), 7.75 (s, 1H), 7.82 (s, 1H), 9.79 (s, 1H). ¹³C NMR (DMSO-d₆) δ 11.50, 13.85, 22.04, 25.49, 28.38, 28.60, 28.77, 28.83, 29.39, 31.22, 31.69, 35.52, 46.58, 122.14, 123.46, 137.49, 175.91. ¹⁵N NMR (DMSO-d₆) δ 0.82–0.88 (m, 6H), 1.22 (m, 10H), 1.74–1.82 (m, 4H), 3.86 (s, 3H), 4.17 (t, 2H, J = 7.20 Hz), 7.75 (s, 1H), 7.82 (s, 1H), 9.90 (s, 1H). ¹³C NMR (DMSO-d₆) δ 11.50, 13.85, 21.99, 25.47, 28.29, 28.42, 29.41, 31.09, 31.65, 35.49, 48.55, 122.03, 123.34, 137.44, 175.90.

[C(10)mim]₄Ada(CH₂CH₂COO)₄: ¹H NMR (DMSO-d₆) δ 0.82–0.88 (m, 6H), 1.22 (m, 14H), 1.74–1.80 (m, 4H), 3.86 (s, 3H), 4.16 (t, 2H, J = 7.60 Hz), 7.74 (s, 1H), 7.80 (s, 1H), 9.83 (s, 1H). ¹³C NMR (DMSO-d₆) δ 11.52, 13.86, 22.02, 25.45, 28.32, 28.60, 28.77, 28.83, 29.39, 31.22, 31.69, 35.52, 46.58, 122.14, 123.46, 137.49, 175.91. ¹⁵N NMR (DMSO-d₆) δ 0.81-0.88 (m, 6H), 1.21 (m, 18H), 1.75-1.82 (m, 4H), 3.73 (s, 3H), 4.17 (t, 2H, J = 7.32 Hz), 7.79 (s, 1H), 7.87 (s, 1H), 10.14 (s, 1H). ¹³C NMR (DMSO-d₆) δ 11.47, 13.83, 22.04, 25.49, 28.38, 28.67, 28.82, 28.92, 28.97, 28.99, 29.47, 31.26, 31.68, 35.42, 48.49, 122.16, 123.46, 137.91, 176.15. ¹⁵N NMR (DMSO-d₆) δ 0.81-0.88 (m, 6H), 1.21 (m, 22H), 1.75-1.81 (m, 4H), 3.87 (s, 3H), 4.17 (t, 2H, J = 7.20 Hz), 7.77 (s, 1H), 7.85 (s, 1H), 10.07 (s, 1H). ¹³C NMR (DMSO-d₆) δ 11.52, 13.84,
22.05, 25.49, 28.38, 28.68-29.03, 29.45, 31.26, 31.73, 35.45, 48.51, 122.15, 123.44, 137.81, 176.00. [C(16)mim](ArmCOO): ¹H NMR (DMSO- d₆) δ 0.82-0.87 (m, 6H), 1.22 (m, 26H), 1.74-1.80 (m, 4H), 4.16 (t, 2H, J=7.60Hz), 7.75 (s, 1H), 7.82 (s, 1H), 9.93 (s, 1H). ¹³C NMR (DMSO-d₆) δ 11.53, 13.86, 22.05, 25.48, 28.37, 28.67, 28.82-29.02, 29.43, 31.26, 31.72, 35.49, 48.54, 122.14, 123.45, 137.63, 175.88. [C(18)mim](ArmCOO): ¹H NMR (DMSO- d₆) δ 0.82–0.86 (m, 6H), 1.22 (m, 30H), 1.72-1.79 (m, 4H), 3.85 (s, 3H), 4.15 (t, 2H, J = 7.20 Hz), 7.70 (s, 1H), 7.74 (s, 1H), 9.54 (s, 1H). ¹³C NMR (DMSO-d₆) δ 11.52, 13.86, 22.03, 25.47, 28.35-29.39, 31.24, 35.57, 48.62, 122.15, 123.47, 137.29, 150.71, 175.85. [C(4)mim]Ada(CH₂CH₂COO): ¹H NMR (DMSO- d₆) δ 0.88 (t, 3H, J = 7.40 Hz), 1.18–1.25 (m, 4H), 1.37 (s, 6H), 1.59 (dd, 6H, J = 18.4, 30.4 Hz), 1.69-1.87 (m, 4H), 3.85 (s, 3H), 4.17 (t, 3H, J = 7.20 Hz), 7.73 (s, 1H), 7.80 (s, 1H), 9.76 (s, 1H). ¹³C NMR (DMSO-d₆) δ 13.30, 18.78, 28.17, 31.43, 31.61, 32.23, 35.58, 36.85, 41.48, 42.04, 48.35, 122.11, 123.50, 137.46, 176.19. [C(10)mim]Ada(CH₂CH₂COO): ¹H NMR (DMSO- d₆) δ 0.83 (t, 3H, J = 7.60 Hz), 1.19–1.26 (m, 16H), 1.37 (s, 6H), 1.59 (dd, 6H, J = 18.4, 30.4 Hz), 1.71-1.76 (m, 4H), 1.86 (s, 3H), 3.85 (s, 3H), 4.17 (t, 3H, J = 7.20 Hz), 7.76 (s, 1H), 7.86 (s, 1H), 10.10 (s, 1H). ¹³C NMR (DMSO-d₆) δ 13.84, 22.08, 25.55, 28.13, 28.19, 28.47, 28.69, 28.92, 29.53, 31.28, 31.57, 32.17, 35.45, 36.81, 41.45, 42.01, 48.51, 122.20, 123.41, 137.90, 176.24. [C(18)mim]Ada(CH₂CH₂COO): ¹H NMR (DMSO-d₆) δ 0.83 (t, 3H, J = 7.60 Hz), 1.21 (m, 32H), 1.37 (s, 6H), 1.59 (dd, 6H, J = 18.4, 30.4 Hz), 1.75 (m, 4H), 1.86 (s, 3H), 3.85 (s, 3H), 4.16 (t, 3H, J = 7.20 Hz), 7.72 (s, 1H), 7.82 (s, 1H). ¹³C NMR (DMSO-d₆) δ 13.87, 22.08, 25.55, 29.50, 31.29, 31.59, 32.11, 35.54, 36.84, 41.43, 42.02, 48.57, 122.20, 123.41, 137.76, 176.09. [C(4)mim]₂Ada(CH₂CH₂COO)₂: ¹H NMR (DMSO-d₆) δ 0.88 (t, 6H, J = 7.40 Hz), 1.02 (s, 2H), 1.18–1.33 (m, 16H), 1.48 (s, 2H), 1.68–1.77 (m, 8H), 1.89 (s, 2H), 3.86 (s, 6H), 4.17 (t, 4H, J = 7.20 Hz), 7.73 (s, 2H), 7.78 (s, 2H), 9.85 (s, 2H). ¹³C NMR (DMSO-d₆) δ 13.25, 18.76, 28.62, 31.41, 32.28, 32.38, 35.54, 36.52, 41.29, 41.74, 47.29, 48.31, 122.08, 123.51, 137.56, 176.13. [C(10)mim]₂Ada(CH₂CH₂COO)₂: ¹H NMR (DMSO-d₆) δ 0.83 (t, 6H, J = 7.40 Hz), 1.02 (s, 2H), 1.21–1.30 (m, 40H), 1.48 (s, 2H), 1.69–1.77 (m, 8H), 1.89 (s, 2H),
3.86 (s, 6H), 4.17 (t, 4H, $J = 7.20$ Hz), 7.75 (s, 2H), 7.85 (s, 2H), 10.09 (s, 2H). $^{13}$C NMR (DMSO-$d_6$) $\delta$ 13.92, 22.07, 25.52, 28.42-28.92, 29.49, 31.27, 32.26, 32.42, 35.46, 36.52, 41.31, 41.73, 47.31, 48.52, 122.12, 123.41, 137.88, 176.14.

$[C(18)mim]_2$Ada(CH$_2$CH$_2$COO)$_2$: $^1$H NMR (DMSO-$d_6$) $\delta$ 0.84 (t, 6H, $J = 7.40$ Hz), 1.02 (s, 2H), 1.22 (m, 72H), 1.48 (s, 2H), 1.75 (m, 8H), 1.89 (s, 2H), 3.85 (s, 6H), 4.15 (t, 4H, $J = 7.20$ Hz), 7.71 (s, 2H), 7.79 (s, 2H), 9.76 (s, 2H). $^{13}$C NMR (DMSO-$d_6$) $\delta$ 13.91, 22.06, 25.50, 28.40, 29.43, 31.26, 32.27, 35.57, 36.53, 40.13, 41.27, 41.73, 47.31, 48.59, 122.15, 123.49, 137.40, 175.86. $[C(4)mim]_3$Ada(CH$_2$CH$_2$COO)$_3$: $^1$H NMR (DMSO-$d_6$) $\delta$ 0.86–0.99 (m, 15H), 1.17–1.27 (m, 18H), 1.66–1.79 (m, 12H), 1.91 (s, 1H), 3.86 (s, 9H), 4.18 (t, 6H, $J = 7.20$ Hz), 7.73 (s, 3H), 7.80 (s, 3H), 9.84 (s, 3H). $^{13}$C NMR (DMSO-$d_6$) $\delta$ 13.89, 22.07, 25.52, 28.41, 28.66, 28.86, 28.91, 29.16, 29.49, 31.26, 32.53, 32.89, 35.47, 41.10, 41.41, 47.01, 48.52, 122.17, 123.41, 123.79, 176.30. $[C(10)mim]_3$Ada(CH$_2$CH$_2$COO)$_3$: $^1$H NMR (DMSO-$d_6$) $\delta$ 0.82–0.99 (m, 15H), 1.22 (m, 54H), 1.69–1.79 (m, 12H), 1.90 (s, 1H), 3.87 (s, 9H), 4.17 (t, 6H, $J = 7.20$ Hz), 7.76 (s, 3H), 7.86 (s, 3H), 10.12 (s, 3H). $^{13}$C NMR (DMSO-$d_6$) $\delta$ 13.89, 22.07, 25.52, 28.41, 28.66, 28.86, 28.91, 29.16, 29.49, 31.26, 32.53, 32.89, 35.47, 41.10, 41.41, 47.01, 48.52, 122.17, 123.41, 137.31, 176.30. $[C(18)mim]_3$Ada(CH$_2$CH$_2$COO)$_3$: $^1$H NMR (DMSO-$d_6$) $\delta$ 0.82–0.99 (m, 15H), 1.22 (m, 102H), 1.66–1.76 (m, 12H), 1.91 (s, 1H), 3.85 (s, 9H), 4.15 (t, 6H, $J = 7.20$ Hz), 7.70 (s, 3H), 7.78 (s, 3H), 9.66 (s, 3H). $^{13}$C NMR (DMSO-$d_6$) $\delta$ 13.91, 14.42, 22.06, 25.50, 28.40-29.42, 31.27, 32.49, 32.90, 35.60, 41.11, 41.40, 47.02, 48.63, 122.16, 123.47, 137.31, 156.72, 175.98.

**Differential scanning calorimetry (DSC).** DSC thermograms were carried out on a SII DSC 6220 instrument by using approximately ~10 mg of exactly weighed samples. The sample on the aluminum open pan was cooled to –120 °C at the rate of 10 °C/min under nitrogen flowing (30 mL/min) and then heated from –120 °C to 100 °C with the same rate.

**Thermogravimetric analysis (TGA).** TGA was performed on an EXSTAR TG/DTA6220, Seiko Instrument, Inc., with the heating rate of 10 °C/min up to 600 °C under nitrogen
flowing (200 mL/min). Residual water was removed by keeping on the aluminum pan at 110 °C for 1 h before the curve profiling. The decomposition temperatures ($T_d$) were determined from the onset of the weight loss.
**Scheme S1.** Synthetic scheme of the ILs$^a$

Reagents and conditions: (a) $\text{Br}_2$, $\text{AlBr}_3$ or Fe, reflux, 9 h; (b) acrylonitrile, Bu$_3$SnH, AIBN, toluene, reflux, 5 h; (c) 10 N $\text{HCl}$, reflux, 2 d; (d) imidazolium salt, methanol, r.t. 5 min.
Table S1. Glass transition temperatures of the salts determined from the DSC curves$^a$

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$^a$Obtained from the second heating curves in the DSC profiles.
Table S2. Glass transition temperatures of \([\text{C(n)mim}]_m\text{Ada(CH}_2\text{CH}_2\text{COO})_m\) (n = 4, 10 and 18)

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<sup>a</sup>Obtained from the second heating curves in the DSC profiles.
References


