

Supporting Information

for

Water-Enriched Poly(Ionic Liquid)s: High-Efficient Microreactors for the Hydrolysis of Ethylene Carbonate

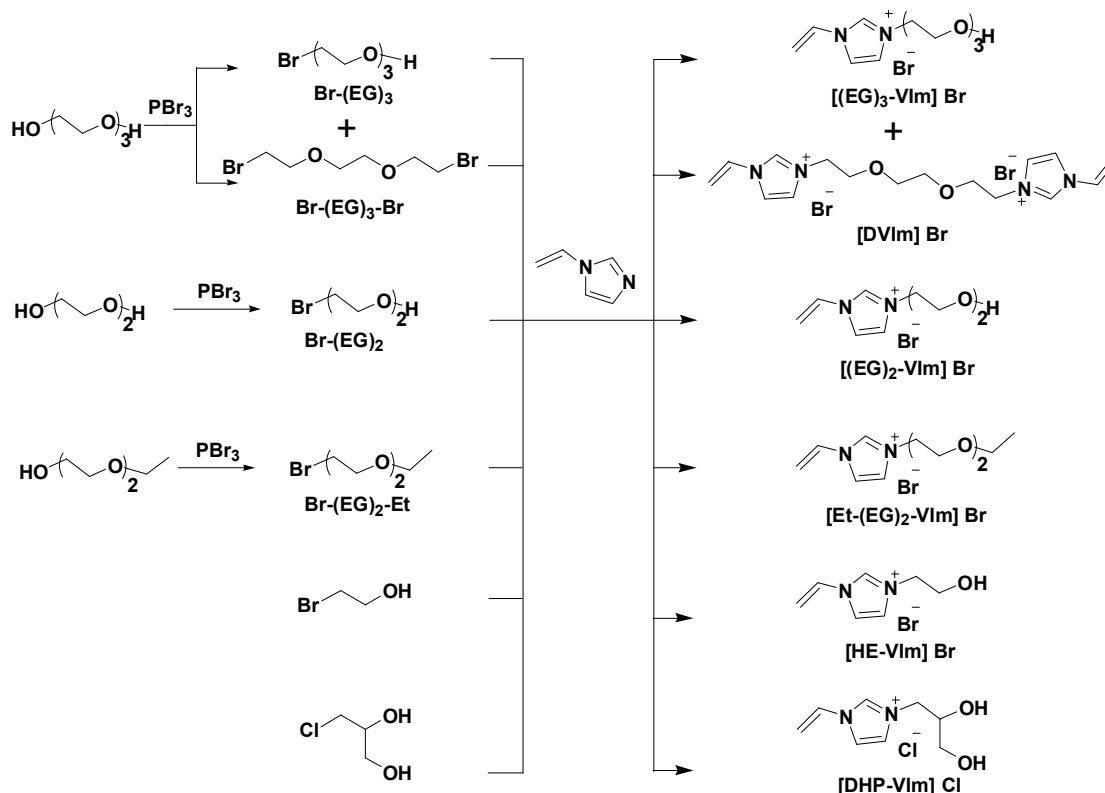
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Experimental

Synthesis of polymerizable monomers and IL type crosslinker [DVIm]Br



Scheme S1. Graphic illustration for the preparation of polymerizable monomers and ionic type crosslinker.

1. Preparation of Br-(EG)₃

Triethylene glycol (30.04 g, 0.2 mol) was dissolved in 100 mL CH₂Cl₂ into a 250-mL round-bottomed flask equipped with a magnetic stirrer and stirred under ice-bath. PBr₃ (13.54 g, 0.05 mol) was added dropwise within 1 h. Then reaction mixture was stirred for 24 h at room temperature. After the reaction, CH₂Cl₂ was removed under reduced pressure. H₂O (20 mL) was added and aqueous phase was washed with 30mL*3 CH₂Cl₂. The CH₂Cl₂ phase was concentrated and dried under vacuum. The desired product was obtained by using chromatography on silica gel and structurally characterized by NMR spectroscopy. Colorless liquid (10.87 g) was obtained in a yield of 34 %.

¹H NMR (400 MHz, CDCl₃, TMS) δ (ppm): 3.83 (t, *J* = 6.0 Hz, 2H), 3.75 (t, *J* = 4.0

Hz, 2H), 3.69 (s, 4H), 3.63 (t, $J = 4.0$ Hz, 2H), 3.49 (t, $J = 6.0$ Hz, 2H), 2.40 (s, 1H); ^{13}C NMR (100 MHz, CDCl_3 , TMS) δ (ppm): 72.51, 71.18, 70.54, 70.35, 61.81, 30.27.

2. Preparation of Br-(EG)₃-Br

Triethylene glycol (15.02 g, 0.1 mol) was dissolved in 50 mL CH_2Cl_2 into a 250-mL round-bottomed flask equipped with a magnetic stirrer and stirred under ice-bath. PBr_3 (27.07 g, 0.1 mol) was added dropwise within 1 h. Then reaction mixture was stirred for 24 h at room temperature. After the reaction, CH_2Cl_2 phase was washed with 30 mL*3 H_2O to remove excessive reagents and by-products. The CH_2Cl_2 phase was concentrated and dried under vacuum. The desired product was obtained by using chromatography on silica gel and structurally characterized by NMR spectroscopy. Colorless liquid (20.15 g) was obtained in a yield of 73 %.

^1H NMR (400 MHz, CDCl_3 , TMS) δ (ppm): 3.83 (t, $J = 6.0$ Hz, 4H), 3.69 (s, 4H), 3.49 (t, $J = 6.0$ Hz, 4H); ^{13}C NMR (100 MHz, CDCl_3 , TMS) δ (ppm): 71.27, 70.53, 30.35.

3. Preparation of Br-(EG)₂

Diethylene glycol (21.22 g, 0.2 mol) was dissolved in 100 mL CH_2Cl_2 into a 250-mL round-bottomed flask equipped with a magnetic stirrer and stirred under ice-bath. PBr_3 (13.54 g, 0.05 mol) was added dropwise within 1 h. Then reaction mixture was stirred for 24 h at room temperature. After the reaction, CH_2Cl_2 was removed under reduced pressure. H_2O (20 mL) was added and aqueous phase was washed with 30mL*3 CH_2Cl_2 . The CH_2Cl_2 phase was concentrated and dried under vacuum. The desired product was obtained by using chromatography on silica gel and structurally characterized by NMR spectroscopy. Colorless liquid (7.80 g) was obtained in a yield of 31 %.

^1H NMR (400 MHz, CDCl_3 , TMS) δ (ppm): 3.83 (t, $J = 6.0$ Hz, 2H), 3.77 (t, $J = 4.0$ Hz, 2H), 3.64 (t, $J = 4.0$ Hz, 2H), 3.50 (t, $J = 6.0$ Hz, 2H), 2.26 (s, 1H); ^{13}C NMR (100 MHz, CDCl_3 , TMS) δ (ppm): 72.15, 70.90, 61.71, 30.60.

4. Preparation of Br-(EG)₂-Et

2-(2-ethoxyethoxy) ethanol (13.40 g, 0.1 mol) was dissolved in 50 mL CH₂Cl₂ into a 250-mL round-bottomed flask equipped with a magnetic stirrer and stirred under ice-bath. PBr₃ (10.83 g, 0.04 mol) was added dropwise within 1 h. Then reaction mixture was stirred for 24 h at room temperature. After the reaction, CH₂Cl₂ phase was washed with 30 mL*3 H₂O to remove excessive reagents and by-products. The CH₂Cl₂ phase was concentrated and dried under vacuum. The desired product was obtained by using chromatography on silica gel and structurally characterized by NMR spectroscopy. Colorless liquid (14.32 g) was obtained in a yield of 73 %.

¹H NMR (400 MHz, CDCl₃, TMS) δ (ppm): 3.82 (t, *J* = 8.0 Hz, 2H), 3.69-3.67 (m, 2H), 3.61-3.59 (m, 2H), 3.57-3.47 (m, 4H), 1.22 (t, *J* = 8.0 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃, TMS) δ (ppm): 71.24, 70.69, 69.81, 66.74, 30.23, 15.16.

5. Typical procedure for synthesis of 3-R-1-vinylimidazolium salts

The polymerizable IL monomers were synthesized *via* ionization of vinyl imidazole and halides. A general procedure is as follows:

Vinyl imidazole (1.88 g, 20 mmol) and Br-(EG)₃ (4.26 g, 20 mmol) were dissolved in THF and added into a 25-mL round-bottomed flask equipped with a magnetic stirrer. The reaction mixture was heated to reflux and stirred for 24 h. After reaction, the top phase was removed, and the oily residue was washed three times with diethyl ether. The desired ionic liquid was obtained after the solvent was removed under reduced pressure distillation and structurally characterized by NMR spectroscopy. Yellow viscous fluid (5.89 g) was obtained in a yield of 96 %.

[(EG)₃-VIm]Br

¹H NMR (400 MHz, DMSO-d₆, TMS) δ (ppm): 9.49 (s, 1H), 8.23 (s, 1H), 7.92 (s, 1H), 7.36 (dd, *J* = 10.0, 14.0 Hz, 1H), 5.99 (d, *J* = 16.0 Hz, 1H), 5.43 (d, *J* = 8.0 Hz, 1H), 4.61-4.40 (m, 5H), 3.82-3.39 (m, 8H); ¹³C NMR (100 MHz, DMSO-d₆, TMS) δ (ppm): 135.65, 128.77, 123.67, 118.74, 108.70, 72.21, 69.49, 67.77, 60.12, 49.13.

[(EG)₂-VIm]Br

^1H NMR (400 MHz, DMSO- d_6 , TMS) δ (ppm): 9.62 (s, 1H), 8.28 (s, 1H), 7.97 (s, 1H), 7.39 (dd, $J = 14.0, 10.0$ Hz, 1H), 6.02 (dd, $J = 16.0, 2.4$ Hz, 1H), 5.44 (dd, $J = 8.0, 1.2$ Hz, 1H), 4.75 (s, 1H), 4.43 (t, $J = 4.0$ Hz, 2H), 3.82 (t, $J = 4.0$ Hz, 2H), 3.49 (s, 4H); ^{13}C NMR (100 MHz, DMSO- d_6 , TMS) δ (ppm): 135.69, 128.77, 123.64, 118.76, 108.66, 72.00, 67.74, 59.95, 49.15.

[Et-(EG) $_2$ -VIm]Br

^1H NMR (400 MHz, DMSO- d_6 , TMS) δ (ppm): 9.61 (s, 1H), 8.30 (s, 1H), 7.96 (s, 1H), 7.39 (dd, $J = 15.6, 8.8$ Hz, 1H), 6.03 (dd, $J = 15.6, 2.4$ Hz, 1H), 5.44 (dd, $J = 8.6, 2.2$ Hz, 1H), 4.43 (t, $J = 4.8$ Hz, 2H), 3.83 (t, $J = 4.8$ Hz, 2H), 3.58-3.56 (m, 2H), 3.47-3.45 (m, 2H), 3.41-3.36 (m, 2H), 1.07 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, DMSO- d_6 , TMS) δ (ppm): 135.65, 128.76, 123.61, 118.80, 108.70, 69.53, 68.89, 67.76, 65.46, 49.11, 15.06.

[HE-VIm]Br

^1H NMR (400 MHz, DMSO- d_6 , TMS) δ (ppm): 9.73 (s, 1H), 8.36 (s, 1H), 8.02 (s, 1H), 7.44 (dd, $J = 15.8, 8.6$ Hz, 1H), 6.09 (dd, $J = 15.6, 2.4$ Hz, 1H), 5.45 (dd, $J = 8.8, 2.4$ Hz, 1H), 5.23 (s, 1H), 4.34 (t, $J = 5.0$ Hz, 2H), 3.80 (t, $J = 4.8$ Hz, 2H); ^{13}C NMR (100 MHz, DMSO- d_6 , TMS) δ (ppm): 135.50, 128.70, 123.56, 118.86, 108.62, 59.00, 51.96.

6. Preparation of [DHP-VIm]Cl

Vinyl imidazole (1.88 g, 20 mmol) and 3-Chloro-1,2-dihydroxypropane (Cl-DHP) (3.32 g, 30 mmol) were added into a 25-mL round-bottomed flask equipped with a magnetic stirrer. The reaction mixture was stirred for 72 h at 80 °C. After reaction, the oily mixture was washed five times with chloroform. The desired ionic liquid was obtained after the solvent was removed under reduced pressure distillation and structurally characterized by NMR spectroscopy. Light yellow powder (3.57 g) was obtained in a yield of 87 %.

[DHP-VIm]Cl

^1H NMR (400 MHz, DMSO- d_6 , TMS) δ (ppm): 9.64 (s, 1H), 8.28 (s, 1H), 7.93 (s, 1H), 7.39 (dd, $J = 14.0, 10.0$ Hz, 1H), 6.03 (d, $J = 16.0$ Hz, 1H), 5.61 (s, 1H), 5.42 (d,

$J = 8.0$ Hz, 1H), 5.17 (s, 1H), 4.39 (d, $J = 12.0$ Hz, 1H), 4.18-4.13 (m, 1H), 3.84 (s, 1H), 3.65-3.45 (m, 2H); ^{13}C NMR (100 MHz, DMSO- d_6 , TMS) δ (ppm): 135.96, 128.81, 124.14, 118.50, 108.43, 69.39, 62.53, 52.44.

7. Synthesis of ionic type crosslinker [DVIm]Br

Vinyl imidazole (3.76 g, 40 mmol) and Br-(EG)₃-Br (5.52 g, 20 mmol) were dissolved in chloroform (30 mL) in a 100-mL round-bottomed flask. Then the mixture was stirred at reflux for 24 h. After reaction, the top phase was removed, and the oily residue was washed three times with diethyl ether. The desired ionic liquid was obtained after the solvent was removed under reduced pressure distillation and structurally characterized by NMR spectroscopy. White powder solid (9.09 g) was obtained in a yield of 98 %.

^1H NMR (400 MHz, DMSO- d_6 , TMS) δ (ppm): 9.74 (s, 2H), 8.37 (s, 2H), 8.01 (s, 2H), 7.48 (dd, $J = 16.0, 8.0$ Hz, 2H), 6.10 (dd, $J = 16.0, 4.0$ Hz, 2H), 5.51 (dd, $J = 8.0, 4.0$ Hz, 2H), 4.48 (t, $J = 12.0$ Hz, 4H), 3.88 (t, $J = 12.0$ Hz, 4H), 3.64 (s, 4H); ^{13}C NMR (100 MHz, DMSO- d_6 , TMS) δ (ppm): 135.59, 128.75, 123.59, 118.87, 108.77, 69.34, 67.82, 49.09.

Characterization

Table S1. CHN analysis of PILs

PILs samples	Measured value				Theoretical value
	C	N	Imidazolium	C/N	C/N
	(wt.%)	(wt.%)	(mmol/g)	(wt./wt.)	(wt./wt.)
poly[C4-VIm-AA-DVB]	52.71	9.79	3.50	5.38	5.62
poly[C4-VIm-AA-EGDMA]	50.56	8.64	3.09	5.85	5.62
poly[C4-VIm-AA-DVIm]	46.55	8.66	3.09	5.38	5.06
poly[C8-VIm-AA-DVB]	58.23	8.45	3.02	6.89	7.33
poly[C8-VIm-AA-EGDMA]	56.66	8.14	2.91	6.96	7.33
poly[C8-VIm-AA-DVIm]	51.52	9.03	3.23	5.71	6.47
poly[Et-(EG) ₂ -VIm-AA-DVB]	53.52	9.50	3.39	5.63	6.48
poly[Et-(EG) ₂ -VIm-AA-EGDMA]	51.91	9.39	3.35	5.53	6.48
poly[Et-(EG) ₂ -VIm-AA-DVIm]	45.57	8.69	3.10	5.24	5.77
poly[(EG) ₃ -VIm-AA-DVB]	50.00	8.51	3.04	5.88	6.48
poly[(EG) ₃ -VIm-AA-EGDMA]	49.08	8.03	2.87	6.11	6.48
poly[(EG) ₃ -VIm-AA-DVIm]	44.68	7.43	2.65	6.01	5.77
poly[(EG) ₂ -VIm-AA-DVIm]	45.39	8.55	3.05	5.31	5.06
poly[HE-VIm-AA-DVIm]	48.64	10.05	3.59	4.84	4.36
poly[DHP-VIm-AA-DVIm]	48.61	9.49	3.39	5.12	4.71

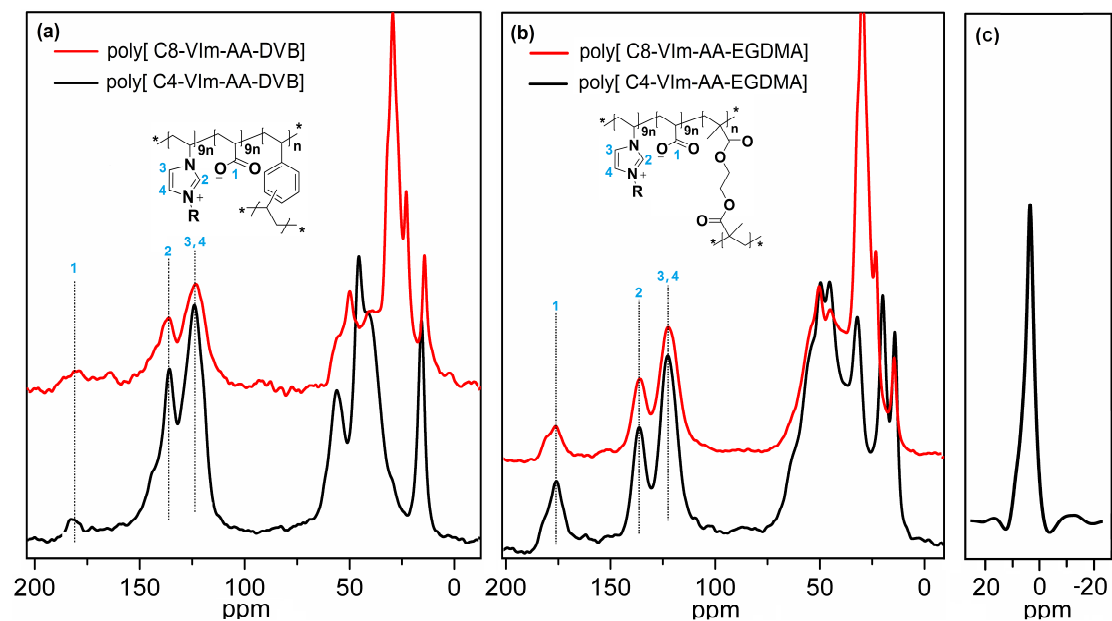


Figure S1. (a) ^{13}C MAS NMR spectra of poly[C4-VIm-AA-DVB] and poly[C8-VIm-AA-DVB]; (b) ^{13}C MAS NMR spectra of poly[C4-VIm-AA-EGDMA] and poly[C8-VIm-AA-EGDMA]; (c) ^1H MAS NMR spectrum of poly[C4-VIm-AA-DVB].

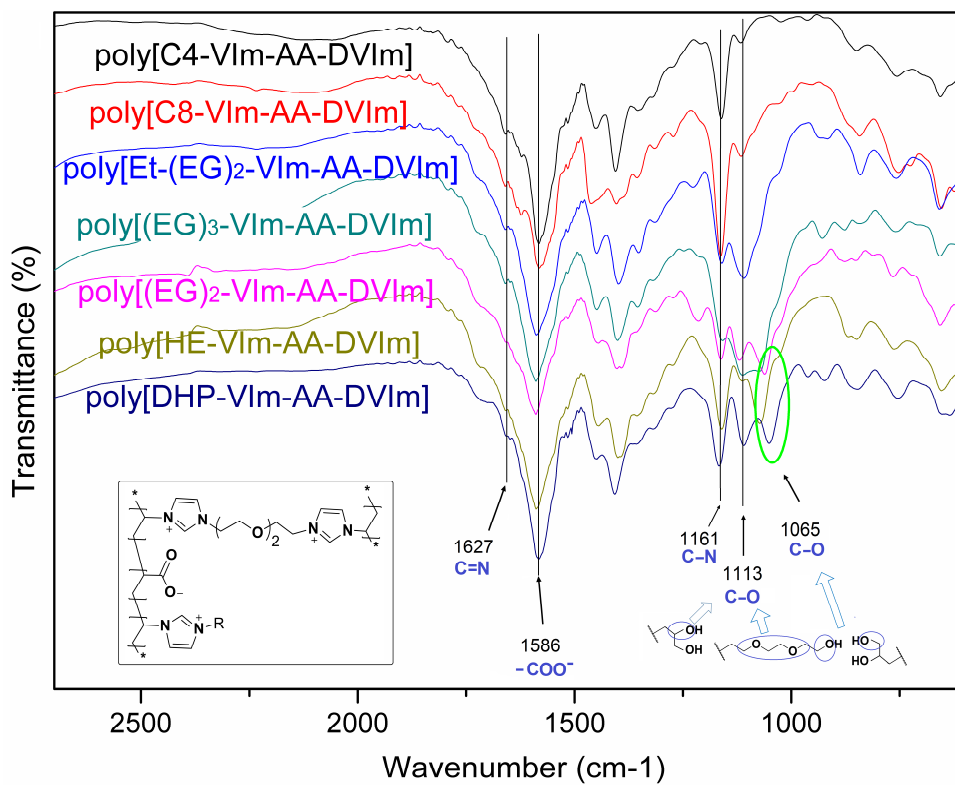


Figure S2. FT-IR spectra of poly[R-VIm-AA-DVIm].

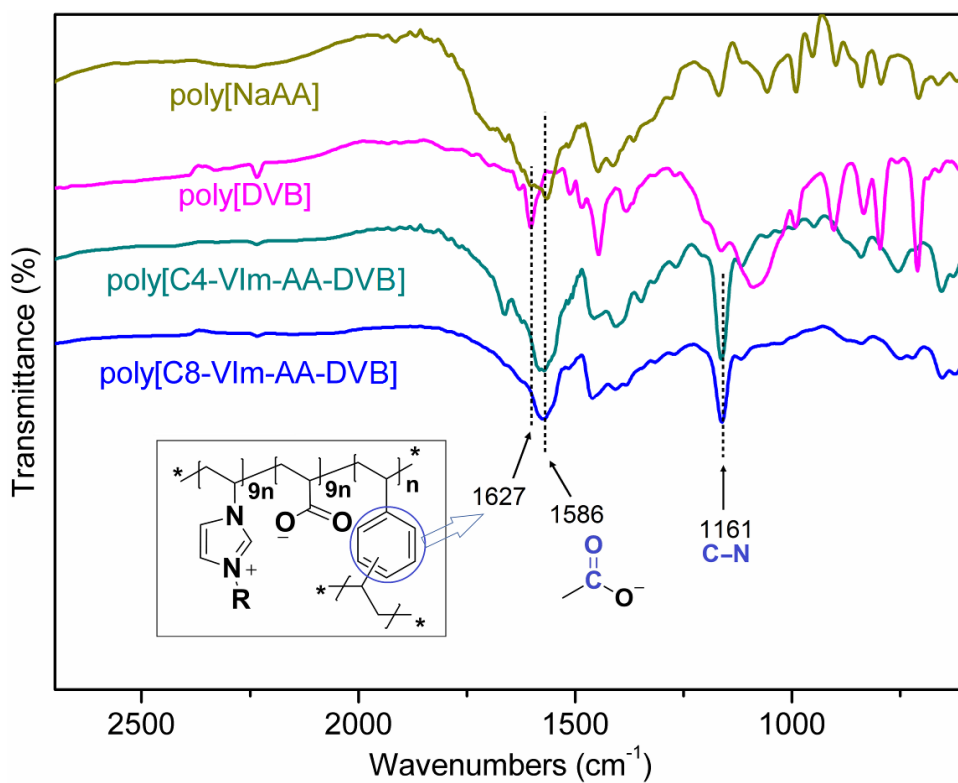


Figure S3. FT-IR spectra of poly[R-VIm-AA-DVB].

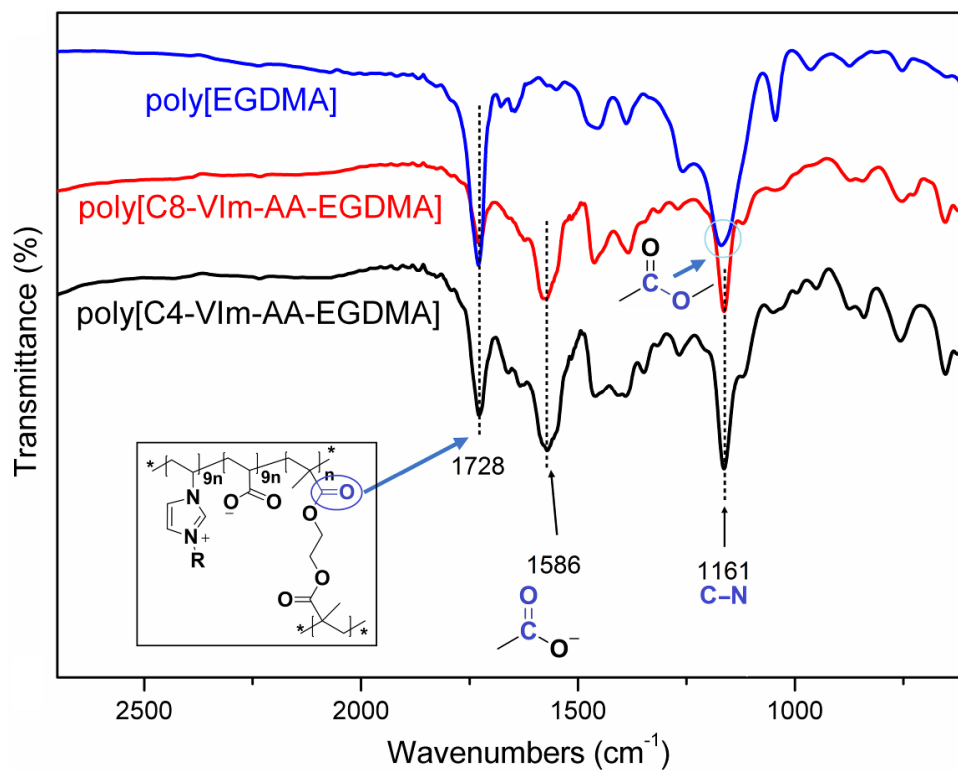


Figure S4. FT-IR spectra of poly[R-VIm-AA-EGDMA].

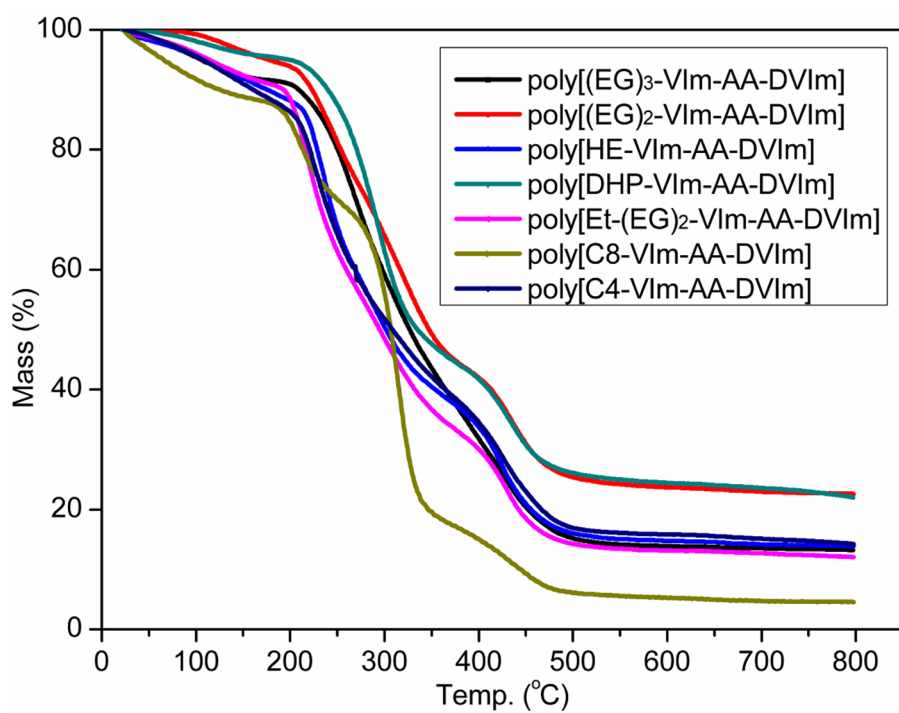


Figure S5. TGA of poly[R-VIm-AA-DVIm].

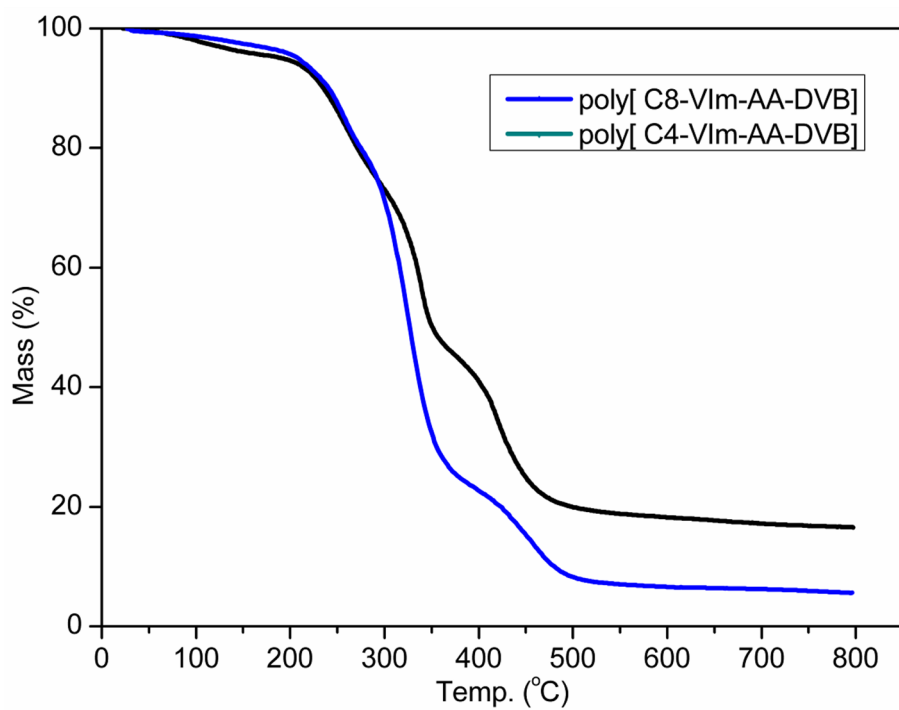


Figure S6. TGA of poly[R-VIm-AA-DVB].

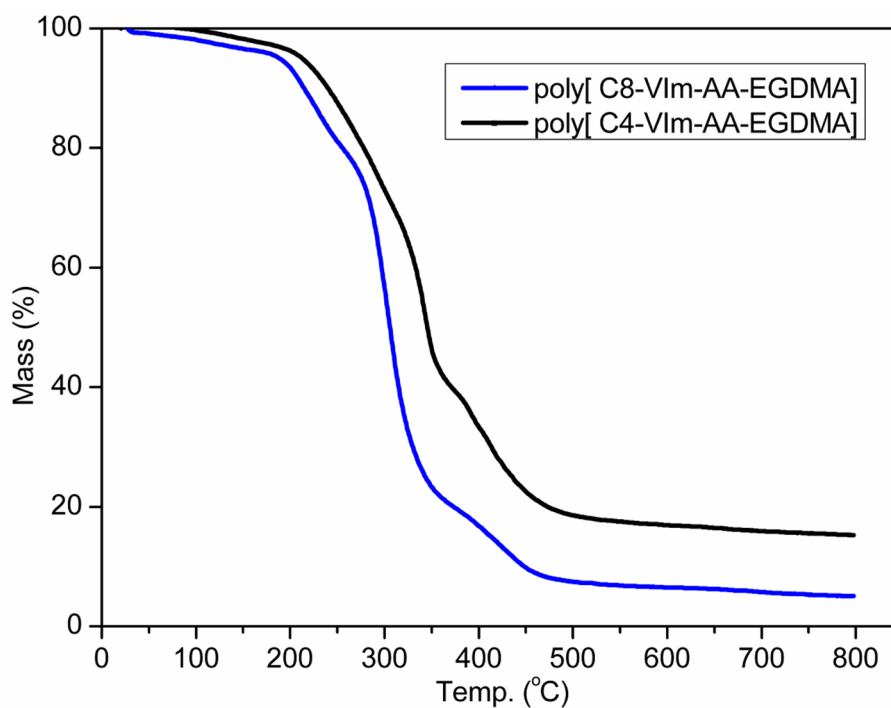


Figure S7. TGA of poly[R-VIm-AA-EGDMA].

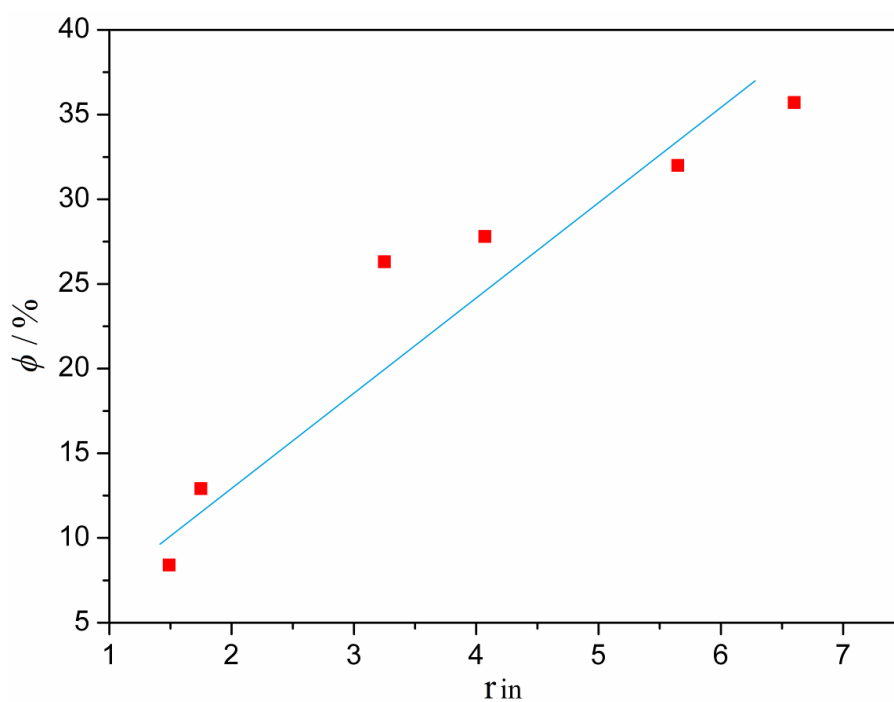


Figure S8. The positive relationship between the cross-sectional surface porosity (ϕ) and the molar ratio of water to EC inside (r_{in}) the PILs after water was sublimated by vacuum freeze-drying.

Supplementary dates

Table S2. The molar ratios of water to EC inside (r_{in}) and outside (r_{out}) the PIL phases when swelling in water/EC mixed solvents ($r=1.5$).

Entry	PILs	n(H ₂ O/EC)	
		r_{in}	r_{out}
1	poly[C8-VIm-AA-EGDMA]	soluble	-
2	poly[C8-VIm-AA-DVB]	soluble	-
3	poly[C8-VIm-AA-DVIm]	1.31	1.58
4	poly[C4-VIm-AA-EGDMA]	1.49	1.54
5	poly[C4-VIm-AA-DVB]	1.75	1.47
6	poly[C4-VIm-AA-DVIm]	3.25	1.50
7	poly[Et-(EG) ₂ -VIm-AA-DVIm]	3.54	1.52
8	poly[(EG) ₃ -VIm-AA-DVIm]	4.07	1.45
9	poly[(EG) ₂ -VIm-AA-DVIm]	4.10	1.50
10	poly[HE-VIm-AA-DVIm]	5.59	1.52
11	poly[DHP-VIm-AA-DVIm]	6.05	1.49

Table S3. The water enrichment ability of PILs in different H₂O/EC mixed solvents.

Entry	PILs	E _w in the H ₂ O/EC mixed solvents			
		$r = 1.0$	$r = 2.0$	$r = 3.0$	$r = 5.0$
1	poly[C8-VIm-AA-DVIm]	0.95	0.99	1.02	0.98
2	poly[C4-VIm-AA-EGDMA]	1.08	1.20	1.31	1.09
3	poly[C4-VIm-AA-DVB]	1.02	1.24	1.26	1.09
4	poly[C4-VIm-AA-DVIm]	2.99	2.19	1.72	1.21
5	poly[Et-(EG) ₂ -VIm-AA-DVIm]	2.45	2.21	1.69	1.25
6	poly[(EG) ₃ -VIm-AA-DVIm]	3.25	2.63	1.99	1.32

With the increase of water amount, the water enrichment ability (E_w) of PILs was decreased gradually. Similar phenomenon was reported in other literatures. [1, 2]

Table S4. The molar ratio of water to EC inside PIL microreactors when swelling in the H₂O/EC mixed solvents.

Entry	PILs	r_{in} in the H ₂ O/EC mixed solvents				
		$r = 1.0$	$r = 1.5$	$r = 2.0$	$r = 3.0$	$r = 5.0$
1	poly[C8-VIm-AA-DVIm]	0.97	1.31	2.12	3.45	4.99
2	poly[C4-VIm-AA-EGDMA]	1.12	1.49	2.22	3.96	5.20
3	poly[C4-VIm-AA-DVB]	0.94	1.75	2.77	3.99	5.52
4	poly[C4-VIm-AA-DVIm]	3.08	3.25	4.12	4.96	5.98
5	poly[Et-(EG) ₂ -VIm-AA-DVIm]	2.44	3.54	4.71	5.36	6.03
6	poly[(EG) ₃ -VIm-AA-DVIm]	3.59	4.07	4.98	5.65	6.60

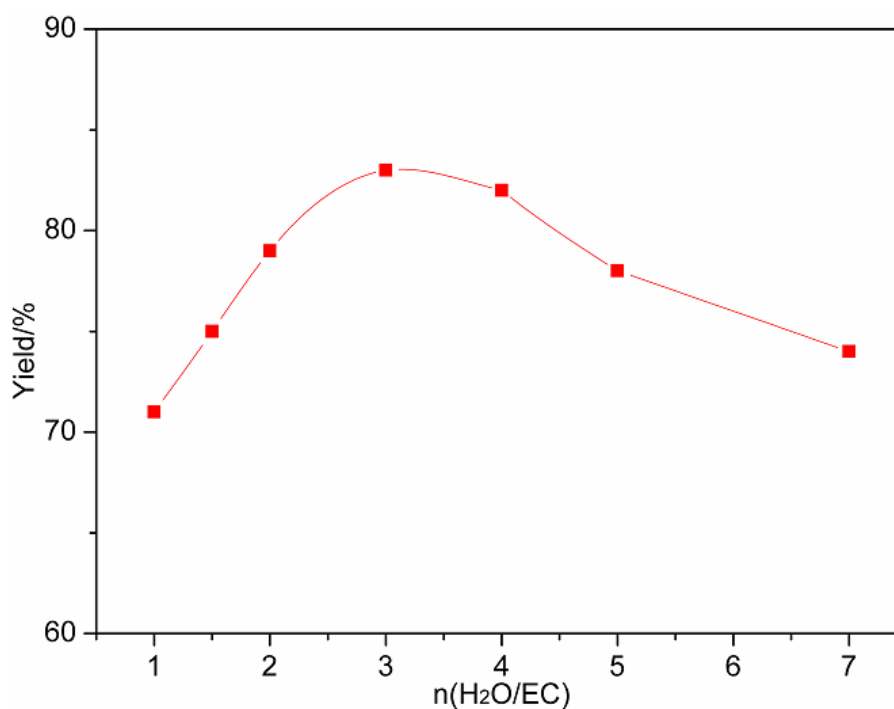
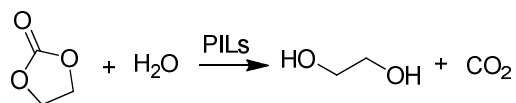


Figure S9. Effect of molar ratio of H₂O to EC from 1.0 to 7.0 on the catalytic efficiency of homogeneous catalyst [BMIm]OAc. Reaction conditions: EC (20mmol), [BMIm]OAc (0.05 mmol), 0.4 MPa CO₂, 100 °C, 3 h.

Table S5. Optimization of the reaction conditions.

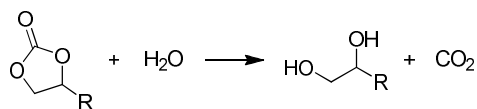
Entry ^a	Temp./°C	Time/h	n(H ₂ O/EC)	Catalyst amount/mmol% ^b	Yield/% ^c
1	140	0.5	1.5	0.5	>99
2	120	0.5	1.5	0.5	88
3	120	1	1.5	0.5	95
4	120	3	1.5	0.5	>99
5	100	3	1.5	0.5	86
6	100	3	1.0	0.5	70
7	100	3	2.0	0.5	86
8	100	3	3.0	0.5	84
9	100	3	5.0	0.5	82
10	100	3	7.0	0.5	81
11	100	1	1.5	0.5	40
12	100	3	1.5	0.5	86
13	100	5	1.5	0.5	98
14	100	5	1.5	0.8	99
15	100	5	1.5	0.25	80
16	100	5	1.5	-	trace
17 ^d	100	5	1.5	0.5	97
18 ^e	100	5	1.5	0.5	81

^a Reaction conditions: EC (20 mmol), 0.4 MPa CO₂; ^b Catalyst: poly[(EG)₃-VIm-AA-DVIm], the mole amount of catalysts was based on imidazolium; ^c GC yield; ^d 0.4 MPa N₂; ^e Atmospheric pressure. The CO₂ pressure of 0.4 MPa decreased the possibly of loss of water with CO₂ release. When reaction was carried out at atmospheric pressure of CO₂, the yield of EG was decreased. [3]

Table S6. Comparison of catalytic performances between swelling PILs and other heterogeneous catalysts for the hydrolysis of EC. ^a

Catalyst	W _{catal.} /g	n(H ₂ O/EC)	T/°C	Time /h	Yield /%	r _m /g _{EC} ·g _{catal.} ⁻¹ ·h ⁻¹
S-[bpim][HCO ₃] ^b	0.127	2	120	3	80	3.70
			(140)		(99.7)	(4.61)
Al ₂ O ₃ ^c	0.050	2	120	2	55	9.68
			(140)		(98)	(17.25)
[{Cu(imi) ₂ } ₃ As ₃ Mo ₃ O ₁₅]}·H ₂ O ^d	0.036	5	95	8	97	5.93
poly[(EG) ₃ -VIm-AA -DVIm]	0.028	1.5	100	5	98	12.32
			(120)	(1)	(97)	(60.97)

^a EC (20 mmol); ^b Ref. [4]; ^c Ref. [5]; ^d Ref. [6].

Table S7. Synthesis of vicinal diols from cyclic carbonates catalyzed by swelling PILs.

Entry ^a	Carbonate	Cat. ^b	Temp./°C	Time/h	Yield/% ^c	Select./% ^c
1		poly[C4-VIm-AA-DVB]	110	3	97	>99
2		poly[C4-VIm-AA-EGDMA]	120	3	86	>99
3		poly[C4-VIm-AA-DVB]	120	5	95	>99

^a Reaction conditions: Cyclic carbonate (20 mmol), H₂O (30 mmol), 0.4 MPa CO₂, 3 h, catalyst (0.1 mmol based on imidazolium); ^b The optimized catalyst was different for the hydrolysis of different cyclic carbonates due to the fact that water enrichment ability of each PILs was different;

^c Determined by GC.

1,2-Butanediol: ¹H NMR (400 MHz, CDCl₃, TMS) δ (ppm): 3.66-3.63 (m, 2H), 3.46-3.42 (m, 1H), 3.31 (d, *J*=8.0 Hz, 2H), 1.50-1.43 (m, 2H), 0.96 (t, *J* = 12.0 Hz, 3H). ¹³C NMR (100 MHz, CDCl₃), δ (ppm): 73.75, 66.40, 26.02, 9.95.

3-Butene-1,2-diol: ¹H NMR (400 MHz, CDCl₃, TMS) δ (ppm): 5.88-5.79 (m, 1H), 5.34 (dd, *J* = 18.0, 4.0 Hz, 1H), 5.21 (dd, *J* = 10.0, 4.0 Hz, 1H), 4.24 (s, 1H), 3.67-3.63 (m, 1H), 3.54-3.46 (m, 3H). ¹³C NMR (100 MHz, CDCl₃), δ (ppm): 136.69, 116.59, 73.32, 66.21.

Table S8. The swelling ability and water enrichment ability of poly[(EG)₃-VIm-AA-DVIm] before and after reused for five times.

Entry	Cata.	Q _{water}	Ew (r = 1.5)
1	poly[(EG) ₃ -VIm-AA-DVIm]	39	2.81
2	After recycled	32	2.66

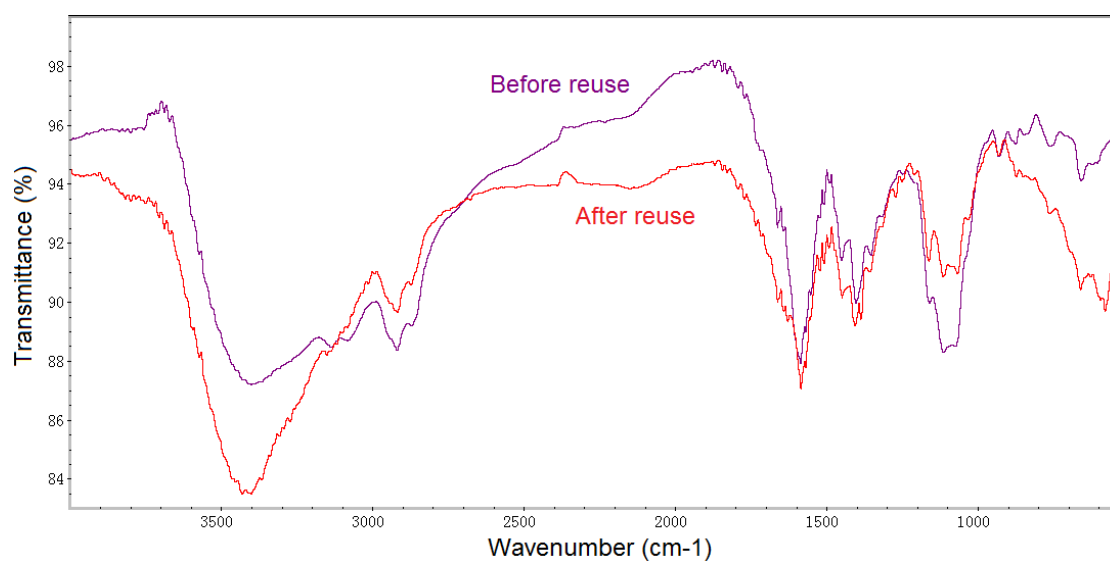


Figure S10. The FT-IR spectra of poly[(EG)₃-VIm-AA-DVIm] before and after reused for five times.

References

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