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Supporting Information for

Polycarbazole-SEBS-Crosslinked AEMs Based on Two Spacer Polymers for High-Performance AEMWE

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Experimental Section

Synthesis of polycarbazole 4

9-(6-Bromohexyl)-9*H*-carbazole (5.00 g, 15.14 mmol) and 1,1,1-trifluoroacetone (2.71 mL, 30.28 mmol) were poured into a dry 50-mL two-neck round-bottom flask with a magnetic stirrer under a nitrogen atmosphere and then dissolved in dichloromethane (15 mL). After the mixture was fully dissolved, the solution was cooled to 0°C. Next, trifluoromethanesulfonic acid (13.37 mL, 151.4 mmol) was slowly added to the solution with continuous stirring. The color of the solution changed from transparent to dark brown. After 48 hours, the viscous solution was poured into a large amount of methanol, and the precipitated polymer was washed with methanol several times to remove any residual reactants. The obtained polymer was dried in a vacuum oven at 80°C for 24 hours, yielding polycarbazole 4 as a white fiber (6.27 g, 97.6%); $\delta_{\rm H}$ (400 MHz, CDCl₃) 8.31–8.08 (2H, s, H₁), 4.36–4.05 (2H, s, H₄), 3.41–3.21 (2H, t, H₉), 2.18–2.08 (3H, s, H₁₀), 1.95–1.66 (4H, broad signal, H_{5,8}), 1.52–1.26 (4H, broad signal, H_{6,7}).

Synthesis of dimethylamine-functionalized polycarbazole 2

The polycarbazole 4 (5.00 g, 11.78 mmol) was poured into a 250-mL two-neck round-bottom flask with a magnetic stirrer under a nitrogen atmosphere and dissolved in NMP (50 mL). After the polymer was fully dissolved, dimethylamine (17.67 mL, 35.34 mmol) was added. The reaction mixture was heated to 70°C and was left for 12 hours at this temperature. The reaction mixture was cooled to room temperature and poured into a 0.5 M KOH solution. After that, the precipitated polymer was poured onto filter paper and washed with deionized (DI) water several times to remove residual reactants. The obtained polymer was dried in a vacuum oven at 80°C for 24 hours to produce the dimethylamine-functionalized polycarbazole **2** as a white powder (4.26 g, 93.1%); $\delta_{\rm H}$ (400 MHz, CDCl₃) 8.27–8.17 (2H, s, H₁), 4.32–4.15 (2H, s, H₄), 2.41–2.33 (2H, t, H₉), 2.33–2.27 (6H, s, H₁₁), 2.22–2.11 (3H, s, H₁₀), 1.93–1.80 (2H, broad signal, H₅), 1.58–1.47 (2H, quintet, H₇), 1.45–1.31 (4H, broad signal, H_{6.8}).

Preparation and characterization of quaternary ammonium-functionalized SEBS (QA-SEBS)

Bromohexyl SEBS 3 (0.9 g) and HPLC-grade chloroform (15 mL) were placed in a 20-mL

vial. After the polymer had completely dissolved, the solution was added to a glass Petri dish through a cotton filter before drying at room temperature (r.t.) for 24 h. The membrane was peeled from the Petri dish and immersed in deionized (DI) water to remove residual solvent. Next, the membranes were soaked in trimethylamine (TMA) solution at 40°C for 24 h. Then, the membrane was washed with DI water to remove excess TMA. Finally, the membrane was immersed in 1 M KOH solution for at least 12 h at room temperature to exchange Br⁻ for OH⁻ and washed with DI water more than three times to remove excess KOH before any measurements were performed. All the prepared solutions were purged with N₂ gas before the measurements.

The complete quaternization of the bromohexyl SEBS with the QA group was confirmed by the disappearance of the C–Br stretching peak at 642 cm⁻¹ in the IR spectrum of QA–SEBS (**Fig. S4c**).

Characterization and measurements

The chemical structures of the synthesized dimethylamine-functionalized polycarbazole **2** and bromohexyl SEBS **3** were identified via ¹H NMR spectroscopy and Fourier transform infrared (FTIR) spectroscopy. ¹H NMR spectra were obtained using a 400 MHz NMR instrument (Agilent 400-MR) using CDCl₃ as a reference. FTIR spectra were obtained using a PerkinElmer Spectrum Two ATR-FTIR spectrometer. Spectra were collected from 4000 to 400 cm⁻¹.

Gel fraction

The gel fractions of the x-Car-SEBS membranes were measured by immersing each membrane in chloroform at room temperature for 48 hours. Then, the membrane was dried in a 40°C oven for 24 hours. Subsequently, the gel fraction was determined using the following equation:

Gel fraction (%) =
$$\frac{W_a}{W_0} \times 100$$

where W_0 is the weight of the dried membrane before the chloroform treatment, and W_a is the weight of the dried membrane after the chloroform treatment.

Water uptake (WU) and swelling ratio (SR)

The water uptake (WU, %) and swelling ratio (SR, %) of each membrane were calculated by soaking the circular membranes in water at 20, 40, 60, and 80°C. The membranes in their OH⁻ form were immersed in DI water for at least 24 hours, the surface of the membrane was wiped dry with a tissue, the sample was quickly weighed (W_{wet}), and the length (L_{wet}) and thickness (T_{wet}) were quickly measured. The membrane was dried under vacuum for 24 hours, and the weight (W_{dry}), length (L_{dry}), and thickness (T_{dry}) of the dry membrane were also recorded. The following equations were used to determine the WU (%) and SR (%):

$$WU (\%) = \frac{W_{wet} - W_{dry}}{W_{dry}} \times 100$$
$$SR (\%) = \frac{L_{wet} - L_{dry}}{L_{dry}} \times 100 \text{ or } \frac{T_{wet} - T_{dry}}{T_{dry}} \times 100$$

Ion exchange capacity (IEC)

The experimental ion exchange capacity (IEC) values of each membrane in OH⁻ form were determined by acid–base back titration. The samples in OH⁻ form were washed with DI water several times and immersed in 10 mL of 0.01 M HCl standard aqueous solution for at least 24 hours to neutralize the OH⁻ ions. Then, the membranes were removed and dried at 40°C in a vacuum oven for 24 h. The residual HCl solution was titrated with a 0.01 M NaOH standard aqueous solution using a phenolphthalein indicator. The following equation was used to determine the experimental IEC (meq g⁻¹):

$$IEC (meq g^{-1}) = \frac{(V_0 \times C_0 - V_{NaOH} \times C_{NaOH})}{W_{dry}}$$

where V_0 and C_0 are the volume and concentration of the HCl standard aqueous solution, respectively, V_{NaOH} and C_{NaOH} are the volume and concentration of NaOH standard aqueous solution used in the back titration, respectively, and W_{dry} is the weight of the membrane after drying in the oven at 40°C for 24 h.

Mechanical properties and thermal stability

A benchtop tensile tester (EZ-TEST E2-L, Shimadzu, Kyoto, Japan) was used to measure the mechanical properties of the membrane in its OH⁻ form at a crosshead speed of 10 mm min⁻¹ at 25°C under 50% relative humidity (RH). The cross-sectional area of the sample in its initial state was used to determine the engineering stress. The initial slope of the stress–strain curve was used to calculate Young's modulus (*E*). For this test, samples of each membrane were prepared in dumbbell shapes of 40 × 10 mm total area and 20 × 10 mm test area.

The thermal stability of the membranes was investigated by thermogravimetric analysis (TGA) using a Scinco TGA N-1000 instrument (Seoul, Korea). The TGA was operated at a heating rate of 10°C min⁻¹ from 30–800 °C under a nitrogen atmosphere.

The glass transition temperature (T_g) of each membrane was measured by differential scanning calorimetry (DSC) using a PerkinElmer DSC 4000 (Waltham, MA, USA). Samples were prepared in aluminum pans and measured from -40 to 200°C for two cycles with heating and cooling rates of 10°C min⁻¹. T_g was determined from the second heating cycle.

Morphological analysis

The morphology of each membrane was analyzed using a field emission transmission electron microscope (FE-TEM, Talos F200X, Thermo Fisher Scientific, Waltham, MA, USA) to image at an accelerating voltage of 200 kV. The samples were prepared as follows: 3–4 drops of 1 wt.% polymer solution in CHCl₃ were placed on a copper grid to make a thin homogeneous film. The grid was dried at 40°C for 12 hours, then treated with TMA solution at 45°C for 12 more hours. After TMA treatment, the grid was washed with DI water to remove excess TMA and dried in a 40°C oven.

The microphase separation of the membranes was observed using atomic force microscopy (AFM, MULTIMODE-8-AM, Bruker, Billerica, MA, USA). The AFM samples were equilibrated to 50% RH for at least 24 hours before imaging, and the AFM analysis of each sample was performed under the same conditions to maintain uniformity. The AFM phase images are provided as recorded without further image processing.

Hydroxide ion conductivity

The hydroxide ion conductivity (σ) of each membrane was measured by a two-probe impedance spectroscopy method using an AC impedance analyzer (SP-200, Bio-Logic SAS, Claix, France). The electrode systems were connected at frequencies from 100 mHz to 2 MHz. Rectangular samples were prepared with the dimensions 1×4 cm. The hydroxide ion conductivity was measured using the resistance (R) in DI water from 20°C to 80°C. The hydroxide ion conductivity was calculated using the following equation:

$$\sigma = \frac{L}{R \times A}$$

where L is the distance between the reference electrodes, and A is the cross-sectional area of the membrane.

The normalized conductivity of each membrane was calculated using the IEC and hydroxide ion conductivity at each temperature, as follows:

Normalized conductivity = σ / IEC.

Alkaline stability

Each membrane in its OH⁻ form was soaked in 2 M KOH solution at 80°C for 840 hours in order to evaluate its chemical stability by measuring the changes in conductivity and IEC. Before the measurements, the membrane was soaked in freshly prepared 1M KOH solution at 60°C for at least 24 hours to completely exchange the counterion from Br⁻ to OH⁻. After this period, the hydroxide conductivity of each membrane was measured in DI water at 20°C, and the IEC was measured by the back-titration method

Fabrication of membrane electrode assembly (MEA) and single-cell measurements in AEMWE

To fabricate an MEA for an AEMWE single-cell measurement, the catalyst ink was prepared by ultra-sonication of IrO_2 (Alfa Aesar, Haverhill, MA, USA) or Pt/C (Pt/C, 60 wt.%, Boyazenergy, Seoul, Republic of Korea) with the catalyst, isopropyl alcohol, and an ionomer (FAA-3-SOLUT-10, Fumatech Co., Germany). Then, the catalyst ink was coated onto the surface of the prepared membranes using an air spray gun. The catalyst was loaded on the anode with a coverage of 2.0 mg_{Ir} cm⁻² on the anode and 0.4 mg_{Pt} cm⁻² on the cathode. The prepared MEA was pre-treated in 1 M KOH solution for 24 hours and washed with DI water. The MEA was sandwiched between the gas diffusion layer (SIGRACET 39BB), the porous transport layer (porous Nickel alloy, Boyazenergy, Seoul, Republic of Korea), and the gasket, and the active area of the unit cell was 5 cm². For the AEMWE single-cell performance test, the cell temperature was maintained at 70°C, and 1 M KOH was fed into the cell anode and cathode at 10 mL min⁻¹.



Scheme S1 Synthetic route to crosslinked dimethylamine-functionalized polycarbazole-SEBS



Fig. S1 ¹H NMR spectra of (a) polycarbazole 4 and (b) DMA-carbazole 2



Fig. S2 ¹H NMR spectra of (a) pristine SEBS, (b) bromohexanoyl SEBS, and (c) bromohexyl SEBS **3**



Fig. S3 Photographs of x-Car-SEBS membranes with different degrees of crosslinking

Fig. S4 FTIR spectra of (a) precursor polymers, (b) x-Car-SEBS membranes with different

degrees of crosslinking, and (c) bromohexyl SEBS and QA-SEBS polymers

Fig. S5 FTIR spectra of (a) 30x-Car-SEBS, (b) 40x-Car-SEBS, and (c) 50x-Car-SEBS before (black) and after (red) alkaline treatment

Fig. S6 (a) IEC values and (b) IR spectra of 40x-Car-SEBS before and after durability test

Fig. S7 (a) Durability test of 40x-Car–SEBS measured in 1 M KOH at 70°C with a current density of 0.5 A cm⁻², (b) AEMWE performance of 40x-Car–SEBS before and after the durability test, and (c) electrochemical impedance spectroscopy (EIS) measurements of the MEAs before and after the durability test at 0.5 A cm⁻²

 Table S1 Gel fractions of x-Car-SEBS membranes

	Weight remaining (%)
30x-Car-SEBS	97.8
40x-Car-SEBS	98.6
50x-Car-SEBS	98.7

Table S2. Water uptake (WU), length swelling ratio (LSR), and thickness swelling ratio (TSR)

of x-Car-SEBS membranes with different degrees of crosslinking and QA-SEBS at each

temperature

	Water uptake (%)				Length swelling ratio (%)				Thickness swelling ratio (%)			
	20°C	40°C	60°C	80°C	20°C	40°C	60°C	80°C	20°C	40°C	60°C	80°C
30x-												
Car— SEBS	65.7	76.9	81.0	83.9	20.9	22.7	26.2	28.6	30.2	32.4	35.0	38.5
40x- Car— SEBS	58.5	64.3	68.8	70.8	18.2	20.5	22.5	23.6	27.3	29.5	30.6	34.2
50x- Car– SEBS	25.9	32.3	40.0	50.0	11.4	12.1	13.6	14.5	20.0	23.3	25.7	29.5
QA– SEBS	222.5	236.0	259.6	273.0	25.0	30.0	36.4	50.0	74.6	78.3	83.9	90.0

	IEC (meq	Ну	droxide cond	Normalized conductivity		
	$g^{-1})^{a}$	20°C	40°C	60°C	80°C	$(mS cm^{-1}) b$
30x-Car– SEBS	$\begin{array}{c} 1.82 \pm \\ 0.02 \end{array}$	$\begin{array}{c} 61.40 \pm \\ 0.02 \end{array}$	$\begin{array}{c} 93.01 \pm \\ 0.02 \end{array}$	$\begin{array}{c} 120.59 \pm \\ 0.02 \end{array}$	$\begin{array}{c} 146.92 \pm \\ 0.59 \end{array}$	80.73
40x-Car– SEBS	$\begin{array}{c} 1.70 \pm \\ 0.03 \end{array}$	$\begin{array}{c} 64.50 \pm \\ 0.01 \end{array}$	$\begin{array}{c} 98.00 \pm \\ 0.03 \end{array}$	$\begin{array}{c} 125.26 \pm \\ 0.13 \end{array}$	$\begin{array}{c}153.16\pm\\0.42\end{array}$	90.09
50x-Car– SEBS	$\begin{array}{c} 1.61 \pm \\ 0.01 \end{array}$	$\begin{array}{c} 54.54 \pm \\ 0.12 \end{array}$	$\begin{array}{c} 75.46 \pm \\ 0.06 \end{array}$	$\begin{array}{c} 102.10 \pm \\ 0.13 \end{array}$	$\begin{array}{c} 128.23 \pm \\ 0.65 \end{array}$	79.65
QA SEBS	$\begin{array}{c} 2.30 \pm \\ 0.02 \end{array}$	$\begin{array}{c} 41.08 \pm \\ 0.05 \end{array}$	$\begin{array}{c} 55.83 \pm \\ 0.10 \end{array}$	$\begin{array}{c} 69.49 \pm \\ 0.22 \end{array}$	$\begin{array}{c} 81.06 \pm \\ 0.30 \end{array}$	35.24

Table S3. Hydroxide conductivity and normalized conductivity of x-Car–SEBS membranes with different crosslinking degrees and QA-SEBS

^a Experimental IEC value ^b measured at 80°C

Table S4 Mechanical properties of x-Car-SEBS membranes with different degrees of

crosslinking

		Br- form		OH- form			
	Tensile Strength (MPa)	Elongation at break (%)	Young's Modulus (MPa)	Tensile Strength (MPa)	Elongation at break (%)	Young's Modulus (MPa)	
30x-Car- SEBS	20.10	86.13	320.47	17.18	136.06	147.34	
40x-Car- SEBS	23.50	70.59	382.20	22.32	138.25	226.23	
50x-Car- SEBS	26.41	57.12	496.67	21.38	109.57	245.39	

AEM	Anion	Polymer type	Tensile strength (MPa)	Elongation at break (%)	Ref
30x-Car-SEBS	OH-	S-S crosslinked ^a	17	136	-
40x-Car-SEBS	OH-	S-S crosslinked ^a	22	138	-
50x-Car-SEBS	OH-	S-S crosslinked ^a	21	110	-
РРО-О-27	Br-	Spacer	43	3	17
PPO-O-40	Br-	Spacer	49	5	17
PPO-C-40	Br-	Spacer	70	7	17
PFPB-QA	OH-	Spacer	28	34	23
PcPBI-Nb-2.33	OH-	Spacer	35	15	50
PcPBI-Nb-2.86	OH-	Spacer	26	30	50
PFTP-0	I-	Main chain	71	46	46
PFTP-13	I-	Main chain	71	25	46
pTP-C1	OH-	Main chain	42	13	21
pTP-OEG4	OH-	Main chain	60	31	21
QMter-co-Mpi-100%	OH-	Main chain	11	24	47
PTP-90	OH-	Main chain	29	7	48
PTP-83	OH-	Main chain	83	15	22
PQP-100	OH-	Main chain	84	16	22
x-PFTP	I-	M-M crosslinked ^b	72	41	51
xTQA50-PPO-SEBS	OH-	M-S crosslinked °	34.3	92	38
50x-PmTP-SEBS	OH-	M-S crosslinked ^c	28.5	165	39

Table S5 Mechanical properties of spacer-type, backbone-type, crosslinked-type AEMs, and x-Car-SEBS membranes

^a Spacer-spacer crosslinked
 ^b Main chain-main chain crosslinked
 ^c Main chain-spacer crosslinked

	Before alkaline treatment	After alkaline treatment
30x-Car-SEBS	1.82 ± 0.02	1.81 ± 0.02
40x-Car-SEBS	1.70 ± 0.03	1.69 ± 0.01
50x-Car-SEBS	1.61 ± 0.01	1.61 ± 0.02

Table S6 IEC value of x-Car-SEBS membranes before and after alkaline treatment

			Conditions		Remaining conductivity	
AEMs	Polymer type	Conc. (M)	Conc. (M) Temp. (°C) Time (h)		(%)	Ref.
30x-Car-SEBS	S–S crosslinked ^a	2	80	840	> 99	-
40x-Car-SEBS	S–S crosslinked ^a	2	80	840	> 99	-
50x-Car-SEBS	S–S crosslinked ^a	2	80	840	> 99	-
QPC-TMA	Spacer	1	80	1000	> 99	24
PPO-O-40	Spacer	5	60	864	47	17
PFTPA-81	Spacer	1	80	1000	98	45
PBPA-4-QA	Spacer	5	80	1000	54	16
PBPA-5-QA	Spacer	5	80	1000	65	16
PBPA-6-QA	Spacer	5	80	1000	79	16
PcPBI-Nb-2.33	Spacer	2	80	2000	72	50
LSCPi	Spacer	1	80	560	98	56
HQA-C0-SEBS	Spacer	1	60	270	64	29
HQA-C12-SEBS	Spacer	1	60	270	92	29
HQA-F1-SEBS	Spacer	1	80	500	93	36
HQA-F5-SEBS	Spacer	1	80	500	97	36
SEBSC6-Mpy	Spacer	1	80	600	87	57
PAP-TP-85	Main chain	1	100	2000	96	15
PAP-BP-60	Main chain	1	100	2000	90	15
PFTP-13	Main chain	5	80	2000	80	46
PDTP-25	Main chain	5	80	1536	73	58
PDTP-25	Main chain	1	80	1536	90	58
PTP-C1	Main chain	1	80	672	70	21
PTP-OEG4	Main chain	1	80	672	94	21
PFPB-QA	Main chain	1	80	720	97	23
QMter-co-Mpi-100%	Main chain	1	60	1000	80	47
PTP-85	Main chain	1	80	960	64	48

Table S7 Comparison of alkaline stability of representative AEMs

PTP-90	Main chain	1	80	960	38	48
PTP-83	Main chain	1	80	1344	pprox 70	22
PQP-100	Main chain	1	80	1344	pprox 75	22
xTQA50-PPO-SEBS	M–S crosslinked ^b	1	80	500	94	38
50x-PmTP—SEBS	M–S crosslinked ^b	2	80	600	> 99	39
FAA-3-50	Commercial	1	80	300	pprox 10	24

^a Spacer–spacer crosslinked ^b Main chain–spacer crosslinked

AFM	Polymer type	Polymer type Electrolyte		Cata	alyst	Cell	Current density Ref
ALW	i orymer type	Licentolyte	Ionomer	Anode	Cathode	(oC)	(A cm-2)
30x-Car-SEBS	S-S crosslinked ^a	1 M KOH	FAA-3- SOLUT-10	IrO2	Pt/C	70	1.02 @ 1.8 V -
40x-Car-SEBS	S-S crosslinked ^a	1 M KOH	FAA-3- SOLUT-10	IrO2	Pt/C	70	1.25 @ 1.8 V -
50x-Car-SEBS	S-S crosslinked ^a	1 M KOH	FAA-3- SOLUT-10	IrO2	Pt/C	70	0.98 @ 1.8 V
PFPB-QA	Spacer	1 M KOH	FAA-3- SOLUT-10	IrO2	Pt/C	70	1.19 @ 2.0 V 23
PFPB-QA	Spacer	1 M KOH	6-QA-PPO	IrO2	Pt/C	70	1.53 @ 2.0 V 23
QPC-TMA	Spacer	1 M KOH	QPC-TMA	IrO2	Pt/C	70	3.50 @ 1.9 V 24
PcPBI-Nb-2.33	Spacer	1 M KOH	PcPBI-Nb- 2.33	IrO2	Pt/C	60	0.27 @ 2.1 V 50
SCPi	Spacer	DI water	SCPi	IrO2	Pt/C	50	0.20 @ 1.8 V 56
LSCPi	Spacer	DI water	LSCPi	IrO2	Pt/C	50	0.30 @ 1.8 V 56
QMter-co-Mpi	Main chain	1 M KOH	QMter-co-Mpi	IrO2	Pt/C	50	0.20 @ 1.8 V 47
PFTP-8	Main chain	1 M KOH	PFBP-14	IrO2	Pt/C	60	3.50 @ 2.0 V 51
PTP-75	Main chain	1 M NaOH	A-Radel	IrO2	Pt/C	55	0.77 @ 2.2 V 48
PTP-90	Main chain	1 M NaOH	A-Radel	IrO2	Pt/C	55	0.91 @ 2.2 V 48
PBP-67	Main chain	1 M NaOH	PBP-67	IrO2	Pt/C	85	^{1.10} @ ^{2.0} V 22
PTP-83	Main chain	1 M NaOH	PBP-67	IrO2	Pt/C	85	1.25 @ 2.0 V 22
PQP-100	Main chain	1 M NaOH	PBP-67	IrO2	Pt/C	85	1.54 @ 2.0 V 22
PcPBI-Nb-C2	M-M crosslinked ^b	1 M KOH	PcPBI-Nb-C2	IrO2	Pt/C	60	0.37 @ 2.1 V 50
PBI1-PVBC1-	M-M	1 M KOH	Homemade	Ir	Pt/C	60	0.70 @ 2.0 57

Table S8 Comparison of the AEMWE single-cell performance with representative AEMs

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	NMPD/OH	crosslinked ^b		black			V
	SEBS-P2O6	M-M crosslinked ^b 1 M KOH	Homemade	Ir black	Pt/C	60	0.68 @ 2.0 V 28
	x-PFTP	M-M crosslinked ^b 1 M KOH	PFBP-14	IrO2	Pt/C	60	2.40 @ 2.0 V 51
	xTQA50-PPO- SEBS	M-S crosslinked ^c 1 M KOH	FAA-3- SOLUT-10	IrO2	Pt/C	70	0.71 @ 1.8 V -
	50x-PmTP-SEBS	M-S crosslinked ^c 1 M KOH	FAA-3- SOLUT-10	IrO2	Pt/C	70	0.84 @ 1.8 V -
	FAA-3-50	Commercial 1 M KOH	FAA-3- SOLUT-10	IrO2	Pt/C	70	0.64 @ 1.8 V -
	PTFE-Sustainion	Commercial 1 M KOH	PFBP-14	IrO2	Pt/C	60	^{1.80} @ ^{2.0} V 51

^a Spacer-spacer crosslinked
 ^b Main chain-main chain crosslinked
 ^c Main chain-spacer crosslinked