Supporting Information

Continuously-tunable fluorescent polypeptides through polymer-assisted assembly strategy

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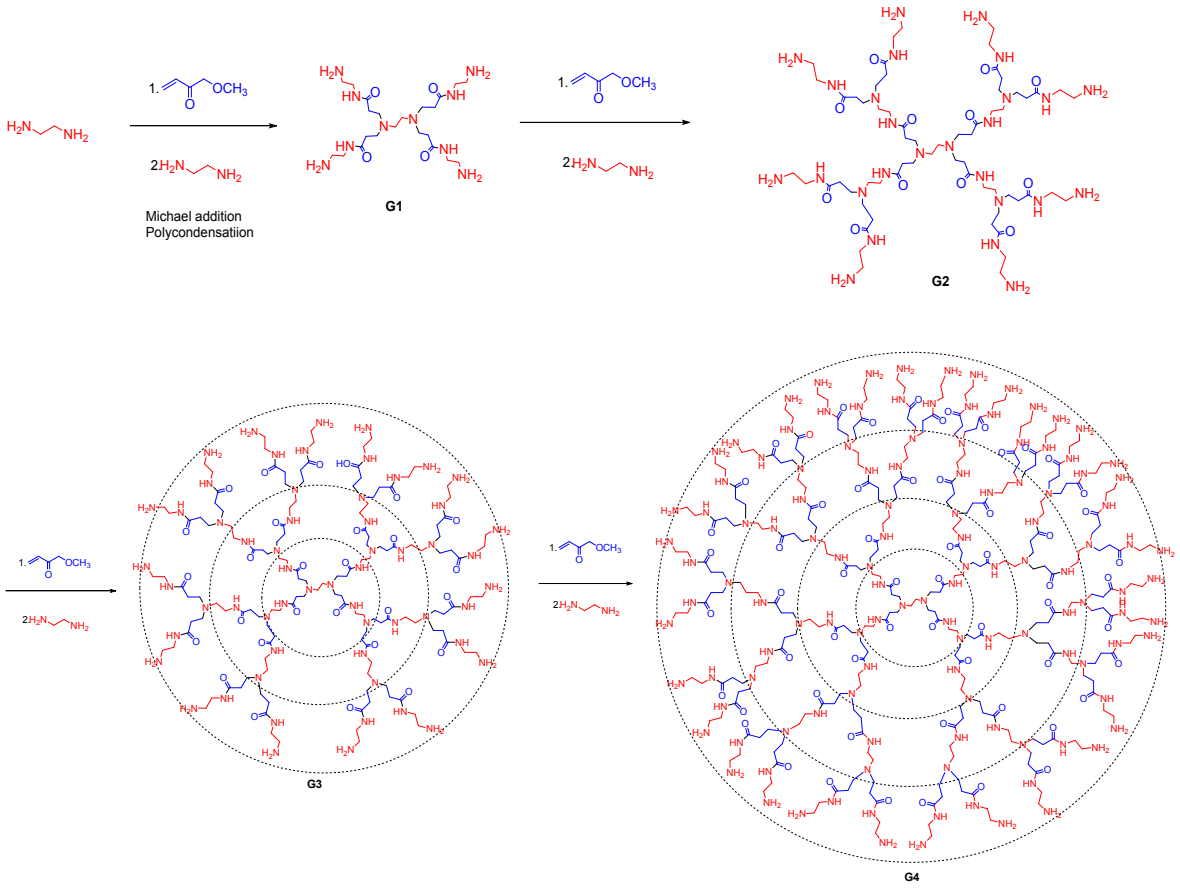
Materials. Ethanediamine, methyl acrylate, L-glutamic acid, benzyl alcohol, pyridine, triphosgene, benzophenone, and aminobenzophenone were purchased from Sigma-Aldrich (China) and used without further purification unless otherwise indicated. Tetrahydrofuran (THF) was distilled from sodium benzophenone ketyl immediately prior to use.

Synthesis.



Scheme S1. Synthetic route for detrimer amidoamine initiators.

Synthesis of detrimer amidoamine initiator. The synthesis protocol for amidoamine initiator was adapted from previous literature procedures.[1] A typical run was shown as follows. A methanol solution (10 mL) of ethylenediamine (0.6 g, 10 mmol) was added dropwise into a methyl acrylate (5.16 g, 60 mmol) in methanol (20 mL) at 0 °C. The resulting reaction mixture was allowed to warm to room temperature and stirred for 2

days under N_2. Then the volatiles were removed under reduced pressure using a rotary evaporator and then in a vacuum at 40 °C to give 3.8 g product (**1**) as slightly yellow oil (yield: 94%). ^{1}H NMR (300 MHz, $CDCl_3$, 298 K): δ 2.42 (m, 12 H, NCH_2), 2.72 (m, 8H, CH_2COOCH$_3$), 3.65 (m, 12H, COOCH_3). The ^{1}H NMR spectrum agreed with the literature data.[1]

A methanol solution (3 mL) of **1** (0.808 g, **2**mmol) was added dropwise into a round-bottomed flask containing ethylenediamine (1.202 g, 20mmol) and anhydrous methanol (7 mL) at 0 °C using an ice/water bath. The reaction mixture was allowed to warm to room temperature and stirred for 7 days under N_2 until complete disappearance of terminal methyl ester groups of **2**, monitored by ^{1}H NMR. Then the volatiles were removed using a rotary evaporator to get crude product. To the crude product was added 20 mL anhydrous methanol and then removed the solvent using a rotary evaporator. Repeat this cycle three times to remove un-reacted ethylenediamine, and finally residual volatiles were removed in vacuum at 40 °C overnight to give 0.986 g product (**2**) as viscous slightly yellow oil (yield: 95%).

G1: ^{1}H NMR (300 MHz, $CDCl_3$, 298 K): δ 2.34–2.44 (m, 12H, COCH_2 and NCH_2CH_2N), 2.64 (m, 8H, CH_2NH$_2$), 2.82 (m, 8H, NCH_2CH$_2$CO), 3.30 (m, 8H, CONHCH_2), 7.95 (br, 4H, CONH). The signal at 3.65 ppm derived from OCH_3 is neglectable. The ^{1}H NMR spectrum agreed with the literature data.[1]

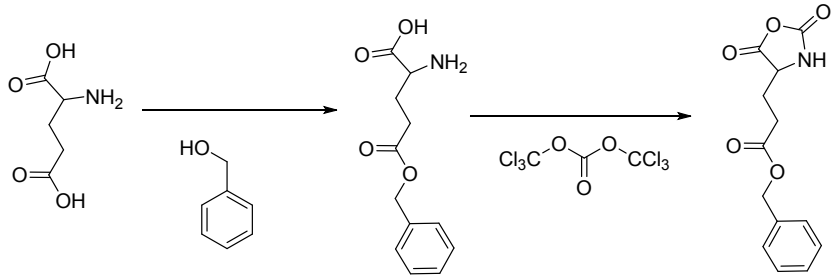
G2: ^{1}H NMR (300 MHz, $CDCl_3$, 298 K): δ 1.70–1.96 (m, 16H, CH$_3$NH_2), 2.28–2.44 (m, 28H, COCH_2 and NCH_2CH_2N), 2.48–2.58 (br, 8H, NCH_2CH$_2$NH), 2.64-2.78 (m, 16H, CH_2NH$_2$), 2.80-2.95 (m, 24H, NCH_2CH$_2$CO), 3.18-3.42 (m, 24H, CONHCH_2),

7.68-8.00 (m, 12H, CON*H*). The signal at 3.65 ppm derived from OCH_3 is neglectable.

G3: ^{1}H NMR (300 MHz, $CDCl_3$, 298 K): δ 1.70–1.96 (m, 32H, CH_3NH_2), 2.28–2.44 (m, 60H, COCH_2 and NCH_2CH_2N), 2.48–2.78 (m, 56H, NCH_2CH$_2$NH and CH_2NH$_2$), 2.80-2.95 (m, 56H, NCH_2CH$_2$CO), 3.18-3.42 (m, 56H, CONHCH_2), 7.57, 8.00 (m, 28H, CON*H*). The signal at 3.65 ppm derived from OCH_3 is neglectable.

G4: ^{1}H NMR (300 MHz, $CDCl_3$, 298 K): δ 1.70–1.96 (m, 64H, CH_3NH_2), 2.28–2.44 (m, 124H, COCH_2 and NCH_2CH_2N), 2.48–2.58 (br, 56H, NCH_2CH$_2$NH), 2.64-2.78 (m, 64H, CH_2NH$_2$), 2.80-2.95 (m, 120H, NCH_2CH$_2$CO), 3.18-3.42 (m, 120H, CONHCH_2), 7.68-8.10 (m, 60H, CON*H*). The signal at 3.65 ppm derived from OCH_3 is neglectable.

Synthesis of γ-Benzyl-L-glutamate carboxyanhydrides (BLG-NCA).



Scheme S2. Synthetic route of γ-benzyl-L-glutamate carboxyanhydrides (BLG-NCA).

The preparation for BLG-NCA was adapted from literature procedures.[2] A typical run was shown as follows. To 100 mL of ethyl ether was slowly added 15 mL of

concentrated sulfuric acid (98%) in an ice-bath. To the solution was slowly added 220 mL of benzyl alcohol. Ethyl either was removed under reduced pressure at room temperature. The solution was heated to 70 °C. To the solution was added 22 g of L-glutamic acid with stirring. This mixture was held at 70 °C with stirring until all glutamic acid was dissolved (ca. 1.5 hours). The reaction mixture was cooled to 30 – 40 °C, and then added to a solution of 33 mL pyridine in 220 mL 95% ethanol under stirring. Precipitation occurred upon cooling to 20 °C and the precipitation was allowed to continue at 3 °C for 12 h. The precipitate was then collected by filtration, washed with ethanol, then with ethyl ether and air-dried. The product was re-crystallized from 500 mL of 5% ethanol aqueous solution, followed by adding sufficient sodium bicarbonate to keep the pH at 7. After filtration, the solution was cooled as rapidly as possible to 3 °C, and left for 12 h. The precipitate was collected by filtration, washed with water and adjusted to pH 7 with sodium bicarbonate, washed with distilled water, slurried with ethanol, filtered, washed with ethyl ether and air-dried, to yield 16.0 g white plates of γ-benzyl-L-glutamate (yield: 31.1%). ^{1}H NMR (300 MHz, $CDCl_3$, 298K): δ 2.41 (m, 2H, $COCH_2CH_2$), 2.84 (t, 2H, $COCH_2CH_2$), 4.37 (t, 1H, $COCHNH_2$), 5.18 (s, 2H, $COOCH_2Ph$), 7.30–7.39 (m, 5H, $COOCH_2Ph$).

γ-Benzyl-L-glutamate (10 g) was suspended in 150 mL anhydrous THF and then triphosgene (4.5 g) was added under nitrogen. The mixture was stirred at 50 °C under N_2 until it turned into a transparent solution within 3 h. The product was precipitated by pouring the solution into 500 mL hexane, isolated by filtration, and purified by re-

crystallizing three times from the THF/hexane mixed solution. The yield was 49%. ^{1}H NMR (300 MHz, $CDCl_3$, 298K): δ 2.14 (m, 2H, $COCH_2CH_2$), 2.61 (t, 2H, $COCH_2CH_2$), 4.39 (t, 1H, COCHNH), 5.12 (br, 2H, $COOCH_2Ph$), 6.6 (s, 1H, CONH), 7.30–7.38 (m, 5H, $COOCH_2Ph$). The ^{1}H NMR spectrum agreed with the literature data.[2c]

Synthesis of star poly(γ-benzyl-L-glutamate) (PBLG).

Prescribed amount of amidoamine initiator (0.035 mmol) and γ-Benzyl-L-glutamate carboxyanhydrides (BLG-NCA) (1.52 mmol) were dissolved in 50 mL anhydrous THF and the solution was stirred for 72 h at room temperature under N_2. The resulting mixture was added dropwise to ethanol. The precipitate was isolated by filtration and dried under vacuum at 40 °C overnight to yield 4-armed poly(γ-benzyl-L-glutamate) as white solids.

PBLG1 (B_{96}): ^{1}H NMR (300 MHz, $CDCl_3$+15 vol% TFA, 298 K): δ 1.96–2.42 (br, 204H, $COCH_2CH_2$ from PBLG, $COCH_2$ and NCH_2CH_2N from **G1**), 2.42–2.66 (br, 192H, $COCH_2CH_2$ from PBLG), 4.58 (br, 96H, COCHNH), 5.12 (br, 192H, $COOCH_2Ph$), 7.15–7.25 (br, 480H, $COOCH_2Ph$), 8.10 (m, 100H, CONH). FT-IR (KBr): υ (cm^{-1}) 3291 (N-H stretching), 3037 (C-H stretching of phenyl rings), 1735 (C=O stretching), 1653 (C=O stretching of α-helix), 1545 (N-H bending of α-helix).

PBLG2 (B_{192}): ^{1}H NMR (300 MHz, $CDCl_3$+15 vol% TFA, 298 K): δ 1.96–2.27 (br, 384H, $COCH_2CH_2$ from PBLG), 2.27–2.66 (br, 420H, $COCH_2CH_2$ from PBLG, $COCH_2$, NCH_2CH_2N and NCH_2CH_2NH from **G2**), 4.58 (br, 192H, COCHNH), 5.12

(br, 384H, COOCH_2Ph), 7.15–7.25 (br, 960H, COOCH$_2$*Ph*), 8.10 (m, 212H, CON*H*). FT-IR (KBr): υ (cm^{-1}) 3291 (N-H stretching), 3037 (C-H stretching of phenyl rings), 1735 (C=O stretching), 1653 (C=O stretching of α-helix), 1545 (N–H bending of α-helix).

PBLG3 (B_{384}): ^{1}H NMR (300 MHz, $CDCl_3$+15 vol% TFA, 298 K): δ 1.96–2.27 (br, 768H, COCH$_2$CH_2 from PBLG), 2.27–2.66 (br, 852H, COCH_2CH$_2$ from PBLG, COCH_2, NCH_2CH_2N and NCH_2CH$_2$NH from **G3**), 4.58 (br, 384H, COC*H*NH), 5.12 (br, 768H, COOCH_2Ph), 7.15–7.25 (br, 1920H, COOCH$_2$*Ph*), 8.10 (m, 428H, CON*H*). FT-IR (KBr): υ (cm^{-1}) 3291 (N-H stretching), 3037 (C–H stretching of phenyl rings), 1735 (C=O stretching), 1653 (C=O stretching of α-helix), 1545 (N–H bending of α-helix).

PBLG4 (B_{768}): ^{1}H NMR (300 MHz, $CDCl_3$+15 vol% TFA, 298 K): δ 1.96–2.27 (br, 768H, COCH$_2$CH_2 from PBLG), 2.27–2.66 (br, 948H, COCH_2CH$_2$ from PBLG, COCH_2, NCH_2CH_2N and NCH_2CH$_2$NH from **G2**), 4.58 (br, 768H, COC*H*NH), 5.12 (br, 1536H, COOCH_2Ph), 7.15–7.25 (br, 3840H, COOCH$_2$*Ph*), 8.10 (m, 860H, CON*H*). FT-IR (KBr): υ (cm^{-1}) 3291 (N–H stretching), 3037 (C–H stretching of phenyl rings), 1735 (C=O stretching), 1653 (C=O stretching of α-helix), 1545 (N–H bending of α-helix).

The ^{1}H NMR spectra of PBLG agreed with the literature data.[3] The number of repeating units, DP, of PBLG synthesized was calculated from ^{1}H NMR using DP = $18I_c/(I_e+I_f - 2I_c)$, I_c, I_e and I_e are the integration of peak at 5.12 ppm and 2.41-2.69 ppm and 1.96-2.41 ppm, respectively. The molecular weight of 8-armed PBLG

synthesized was calculated using M_n=516+DP×219. The values of the number of repeating unit DP and M_n were tabulated in Table S1.

Table S1 Molecular weight, molecular weight distribution, and repeating units of 8-armed star PBLG.

entry	[a]M_n (kg/mol)	[b]M_n (kg/mol)	[c]M_n (kg/mol)	[d]PDI	[e]DP	$8B_n$
PBLG1	25.1	22.5	23.4	1.17	96	$8B_{12}$
PBLG2	47.2	43.5	44.2	1.20	192	$8B_{24}$
PBLG3	91.5	85.5	86.9	1.18	384	$8B_{48}$
PBLG4	180.0	169.6	172.5	1.18	768	$8B_{96}$

[a]Calculated. [b]Determined by ^{1}H NMR. [c]Determined by GPC calibrated with PEO standard. [d]PDI denoted molecular weight distribution of PBLG synthesized. [e]DP denoted the number of repeating units of PBLG synthesized. DP was calculated from ^{1}H NMR using $DP = 18I_c/(I_e+I_f - 2I_c)$, I_c, I_e and I_e are the integration of peak at 5.12 ppm and 2.41-2.69 ppm and 1.96-2.41 ppm, respectively.

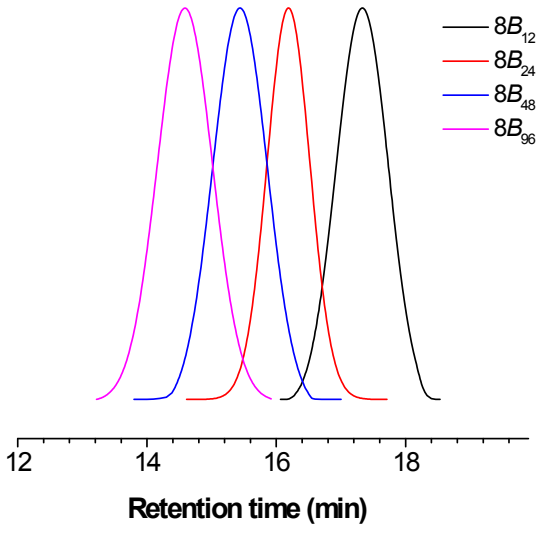


Fig. S1 GPC curves of PBLG with various molecular weights.

Synthesis of 1-(4-aminophenyl)-1,2,2-triphenylethene (1).

A three-necked flask equipped with a magnetic stirrer was charged with zinc powder (7.85 g, 120 mmol) and 150 mL THF under N_2 atmosphere. The mixture was cooled to 0 °C, and $TiCl_4$ (6.5 mL, 60mmol) was slowly added with the temperature below 10 °C. The suspending mixture was warmed to room temperature and stirred for 0.5 h, then heat to reflux for 2.5 h. The mixture was again cooled to 0 °C, charged with pyridine (2.5 mL, 30 mmol) and stirred for 10 min. 2.62 g of benzophenone (14.4 mmol) and 2.36 g of 4-aminobenzophenone (12 mmol) were added slowly. After addition, the reaction mixture was heated at reflux until the carbonyl compounds were consumed (monitored by TLC). The reaction was quenched with 10% K_2CO_3 aqueous solution, and filtered to remove solid. The product was taken up with CH_2Cl_2. The organic layer was collected and concentrated. The crude material was purified by flash chromatography to give the desired products. Yield 82%. ^{1}H NMR (400 MHz, $CDCl_3$), δ (TMS, ppm): 3.40–3.64 (br, 2H, NH_2), 6.36–6.40 (d, 2H, Ar–H), 6.76–6.82 (d, 2H, Ar–H), 6.94–7.11 (m, 15H, Ar–H).

Synthesis of tetraphenylethene decorated PBLG (PBLG-TPE).

Prescribed amount of PBLG, compound **1** and catalytical amount of 2-hydropyridine were dissolved in 50 mL of anhydrous THF. The mixture was stirred at 40 °C for 72 h in dark under N_2. The product was precipitated by adding the reaction mixture dropwise to 500 mL of cold hexane with stirring, filtrated, and washed with hexane (100 mL× 3). The solid was dried in vacuum at 40 °C overnight to yield tetraphenylethene decorated PBLG (PBLG-TPE).

***B*$_{96}$-TPE2:** ^{1}H NMR (300 MHz, $CDCl_3$+15 vol% TFA, 298 K): δ 1.96–2.42 (br, 204H, $COCH_2C\mathit{H}_2$ from PBLG, $COC\mathit{H}_2$ and $NC\mathit{H}_2C\mathit{H}_2N$ from **G1**), 2.42–2.66 (br, 192H, $COC\mathit{H}_2CH_2$ from PBLG), 4.58 (br, 96H, COC*H*NH), 5.12 (br, 188H, $COOC\mathit{H}_2Ph$), 6.90–7.10 (br, 36H, Ar–H), 7.15–7.25 (br, 480H, $COOCH_2\mathit{Ph}$), 8.10 (m, 100H, CON*H*). FT-IR (KBr): υ (cm^{-1}) 3291 (N–H stretching), 3037 (C–H stretching of phenyl rings), 1735 (C=O stretching), 1653 (C=O stretching of α-helix), 1545 (N–H bending of α-helix). FT-IR spectra of PBLG-TPE were shown in Fig. 1.

***B*$_{192}$-TPE2**: ^{1}H NMR (300 MHz, $CDCl_3$+15 vol% TFA, 298 K): δ 1.96–2.27 (br, 384H, $COCH_2C\mathit{H}_2$ from PBLG), 2.27–2.66 (br, 420H, $COC\mathit{H}_2CH_2$ from PBLG, $COC\mathit{H}_2$, $NC\mathit{H}_2C\mathit{H}_2N$ and $NC\mathit{H}_2CH_2NH$ from **G2**), 4.58 (br, 192H, COC*H*NH), 5.12 (br, 376H, $COOC\mathit{H}_2Ph$), 6.90–7.10 (br, 72H, Ar–H), 7.15–7.25 (br, 960H, $COOCH_2\mathit{Ph}$), 8.10 (m, 212H, CON*H*). FT-IR (KBr): υ (cm^{-1}) 3291 (N–H stretching), 3037 (C–H stretching of phenyl rings), 1735 (C=O stretching), 1653 (C=O stretching of α-helix), 1545 (N–H bending of α-helix).

***B*$_{384}$-TPE2**: ^{1}H NMR (300 MHz, $CDCl_3$+15 vol% TFA, 298 K): δ 1.96–2.27 (br, 768H, $COCH_2C\mathit{H}_2$ from PBLG), 2.27–2.66 (br, 852H, $COC\mathit{H}_2CH_2$ from PBLG, $COC\mathit{H}_2$, $NC\mathit{H}_2C\mathit{H}_2N$ and $NC\mathit{H}_2CH_2NH$ from **G3**), 4.58 (br, 384H, COC*H*NH), 5.12 (br, 753H, $COOC\mathit{H}_2Ph$), 6.90–7.10 (br, 146H, Ar–H), 7.15–7.25 (br, 1920H, $COOCH_2\mathit{Ph}$), 8.10 (m, 428H, CON*H*). FT-IR (KBr): υ (cm^{-1}) 3291 (N–H stretching), 3037 (C–H stretching of phenyl rings), 1735 (C=O stretching), 1653 (C=O stretching of α-helix), 1545 (N–H bending of α-helix).

***B*$_{768}$-TPE2**: ^{1}H NMR (300 MHz, $CDCl_3$+15 vol% TFA, 298 K): δ 1.96–2.27 (br,

768H, $COCH_2CH_2$ from PBLG), 2.27–2.66 (br, 948H, $COCH_2CH_2$ from PBLG, $COCH_2$, NCH_2CH_2N and NCH_2CH_2NH from **G2**), 4.58 (br, 768H, COCHNH), 5.12 (br, 1506H, $COOCH_2Ph$), 6.90–7.10 (br, 292H, Ar–H), 7.15–7.25 (br, 3840H, $COOCH_2Ph$), 8.10 (m, 860H, CONH). FT-IR (KBr): υ (cm^{-1}) 3291 (N–H stretching), 3037 (C–H stretching of phenyl rings), 1735 (C=O stretching), 1653 (C=O stretching of α-helix), 1545 (N–H bending of α-helix).

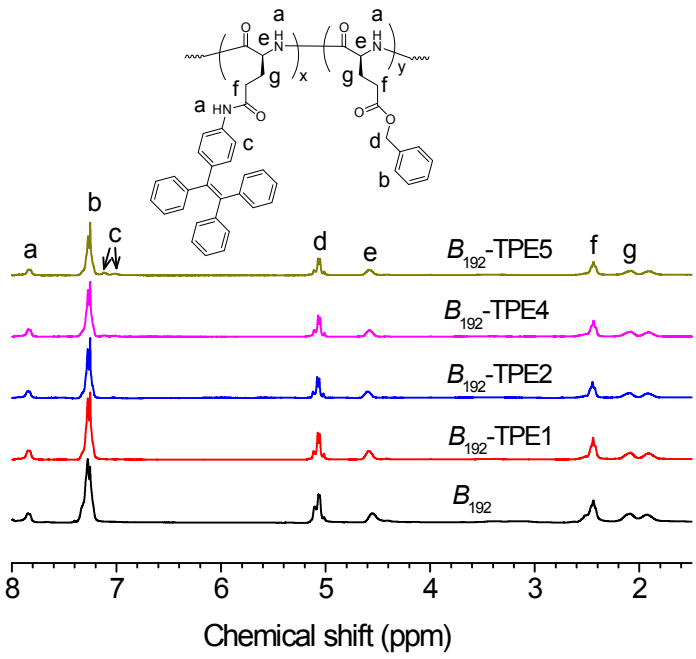
***B*$_{192}$-TPE1**: ^{1}H NMR (300 MHz, $CDCl_3$+15 vol% TFA, 298 K): δ 1.96–2.27 (br, 384H, $COCH_2CH_2$ from PBLG), 2.27–2.66 (br, 420H, $COCH_2CH_2$ from PBLG, $COCH_2$, NCH_2CH_2N and NCH_2CH_2NH from **G2**), 4.58 (br, 192H, COCHNH), 5.12 (br, 380H, $COOCH_2Ph$), 6.90–7.10 (br, 36H, Ar–H), 7.15–7.25 (br, 960H, $COOCH_2Ph$), 8.10 (m, 212H, CONH). FT-IR (KBr): υ (cm^{-1}) 3291 (N–H stretching), 3037 (C–H stretching of phenyl rings), 1735 (C=O stretching), 1653 (C=O stretching of α-helix), 1545 (N–H bending of α-helix).

***B*$_{192}$-TPE4**: ^{1}H NMR (300 MHz, $CDCl_3$+15 vol% TFA, 298 K): δ 1.96–2.27 (br, 384H, $COCH_2CH_2$ from PBLG), 2.27–2.66 (br, 420H, $COCH_2CH_2$ from PBLG, $COCH_2$, NCH_2CH_2N and NCH_2CH_2NH from **G2**), 4.58 (br, 192H, COCHNH), 5.12 (br, 369H, $COOCH_2Ph$), 6.90–7.10 (br, 146H, Ar–H), 7.15–7.25 (br, 960H, $COOCH_2Ph$), 8.10 (m, 212H, CONH). FT-IR (KBr): υ (cm^{-1}) 3291 (N–H stretching), 3037 (C–H stretching of phenyl rings), 1735 (C=O stretching), 1653 (C=O stretching of α-helix), 1545 (N–H bending of α-helix).

***B*$_{192}$-TPE5**: ^{1}H NMR (300 MHz, $CDCl_3$+15 vol% TFA, 298 K): δ 1.96–2.27 (br, 384H, $COCH_2CH_2$ from PBLG), 2.27–2.66 (br, 420H, $COCH_2CH_2$ from PBLG,

COCH_2, NCH_2CH_2N and NCH_2CH$_2$NH from **G2**), 4.58 (br, 192H, COC*H*NH), 5.12 (br, 365H, COOCH_2Ph), 6.90–7.10 (br, 182H, Ar–H), 7.15–7.25 (br, 960H, COOCH$_2$*Ph*), 8.10 (m, 212H, CON*H*). FT-IR (KBr): υ (cm^{-1}) 3291 (N–H stretching), 3037 (C–H stretching of phenyl rings), 1735 (C=O stretching), 1653 (C=O stretching of α-helix), 1545 (N–H bending of α-helix).

Molar percentage of TPE in PBLG-TPE was determined by ^{1}H NMR using m_{TPE}=1-I_d/2I_e, where I_d and I_e were the integration of peak at 5.12 and 4.58 ppm, respectively.



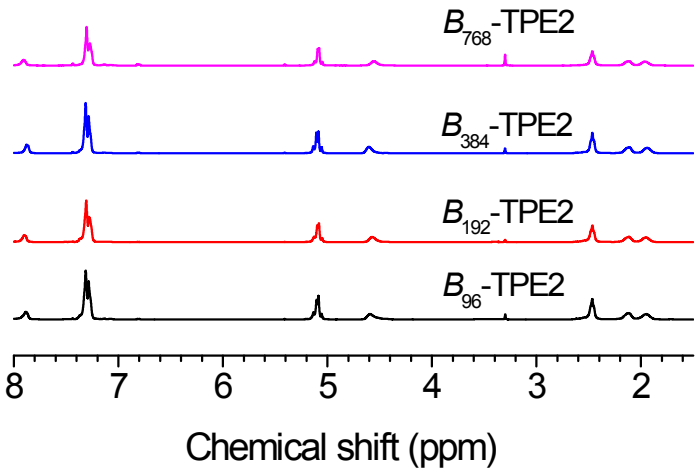


Fig. S2 ^{1}H NMR spectra of 8-armed star PBLG-TPE.

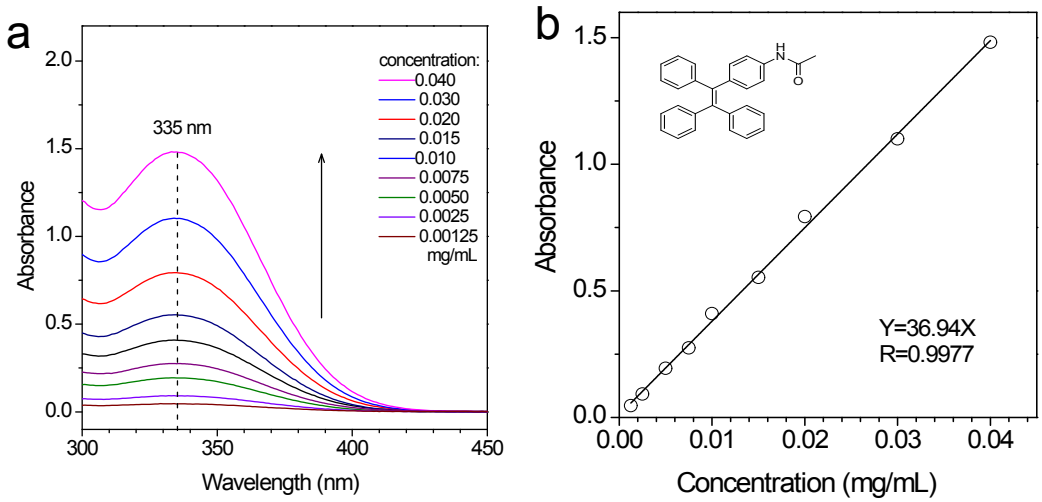


Fig. S3 (a) UV-vis spectra of compound **2**/THF solution at various concentrations and (b) the plots of absorbance (335 nm) against concentration of compound **2**.

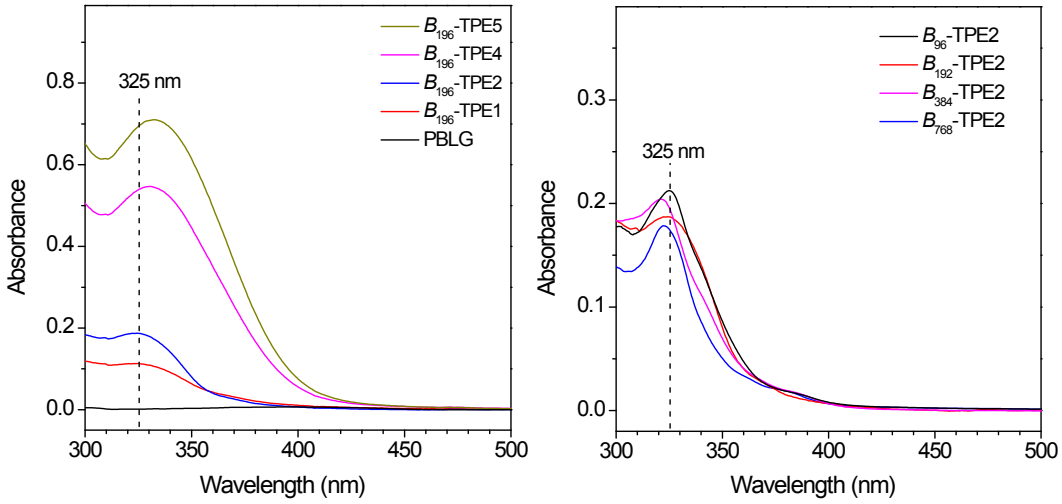


Fig. S4 UV-vis spectra of PBLG-TPE with various TPE percentages (a) and with various molecular weights (b) in THF. Concentration of PBLG-TPE was 0.2 mg/mL.

Calculation of radiative and non-radiative decay rate constants.

Fluorescence typically follows first-order kinetics:

$$[S] = [S]_0 e^{-t/\tau} \quad \text{(S1)}$$

[S] is the concentration of exited state molecules at time *t*, $[S]_0$ is the initial concentration and τ is the fluorescence lifetime.

Decay rate (k) is the inverse of lifetime, consisting of radiative and non-radiative decay rate constants:

$$k = k_{rad} + k_{nrad} \quad \text{(S2)}$$

where k_{rad} is the radiative decay rate constant and k_{nrad} is the nonradiative decay rate constant. The quantum yield (QE) is defined as the fraction of emission process in which emission of light is involved:

$$QE = \frac{k_{rad}}{k_{rad} + k_{nrad}} \quad \text{(S3)}$$

The values of radiative and non-radiative rate constants of TPE and TPE-CDs were

tabulated in Table 1.

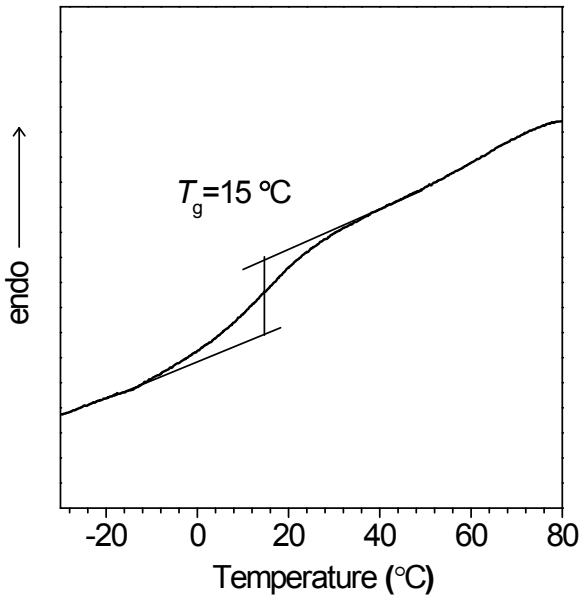


Fig. S5 DSC curve for B_{192}-TPE2.

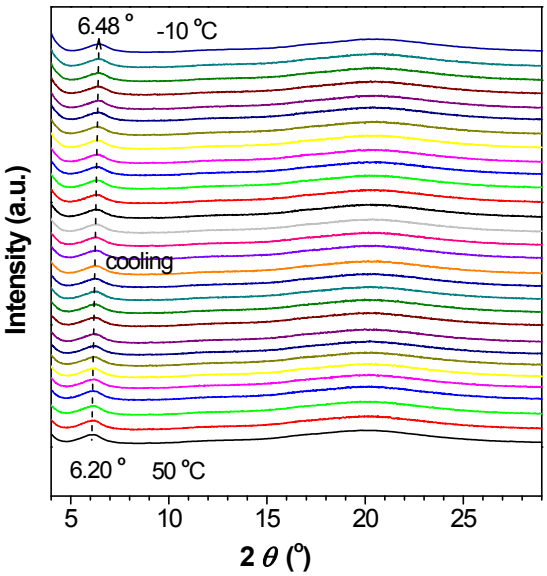


Fig. S6 XRD spectra for B_{192}-TPE2 at various temperatures.

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