Supporting Information for

A Facile and Versatile Platform for Preparing Uniform π -Conjugated Nanofibers of Controlled length and Varying Shells

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SUPPORTING EXPERIMENTAL DETAILS

Materials

2,2'-Azobis(isobutyronitrile) (AIBN, Aldrich, 98%) was recrystallized from anhydrous ethanol twice. Copper(I) chloride (CuCl, Aldrich, 99%) was purified by stirring overnight over CH₃COOH at room temperature, followed by washing the solid with ethanol, diethyl ether and acetone prior to drying in vacuo at 40°C overnight. Tris[(1-benzyl-1H-1,2,3-triazol-4-yl)methyl]amine (TBTA, Aladdin, 97%), silver hexafluorophosphate (AgPF₆, Aldrich, 99.99%), tetraphenylporphyrin tetrasulfonic acid (TPPS, Aladdin, >85.0%), 4-cyano-4-(phenylcarbonothioylthio)pentanoic acid (Macklin, >97%), N-ethyldiisopropylamine (Aldrich, 99%), benzylamine (Aldrich, 95%) and N,N-dimethylethylenediamine (Aladdin, 98%) were used as received. Ethanol (≥99.8%, Aladdin) was used as received for self-assembly experiments. All organic solvents such as dichloromethane (DCM), N,N-dimethyformamide (DMF), trimethylamine (TEA) and tetrahydrofuran (THF) were distilled prior to use. Other reagents not specially mentioned were purchased from Aldrich and used as received without further purification. Alkyne-terminated OPV₅ was prepared according to a previous report.¹ Pentafluorophenyl methacrylate (PFMA) and 4-cyano-4-((thiobenzoyl)sulfanyl)pentanoic acid were prepared according to previous reports.^{2,3}

Instrumentation

¹H NMR (400 MHz) analyses were performed on a JEOL JNM-ECZ400 spectrometer at 25°C in CD₂Cl₂, tetramethylsilane (TMS) was used as internal standard. ¹⁹F NMR analyses were performed on a JEOL JNM-ECZ400 spectrometer at 25°C in THF. MALDI-TOF-MS analyses were measured by MALDI TOF 7090 mass spectrometer, α -cyano-4-hydroxycinnamic acid (CHCA) was used as matrix. Relative molecular weights and molecular weight distributions were measured by conventional GPC using a system equipped with a Waters 1515 Isocratic HPLC pump, a Waters 2414 refractive index detector, a Waters 2487 dual λ absorbance detector and a set of Waters Styragel columns (HR3 (500-30,000), HR4 (5,000-600,000) and HR5 (50,0004,000,000), 7.8×300 mm, particle size: 5 μ m). GPC measurements were carried out at 35°C using THF or DMF (using HR4 column with THF as eluent containing 2.5 g/L tetra-n-butylammonium bromide (TBAB) for OPV₅-*b*-P18C6AM₆₆-*co*-P18C6AM₆₆ and OPV₅-*b*-PDMAEAM₆₆) with a flow rate of 1.0 mL/min. The system was calibrated with linear polystyrene standards.

Transmission electron microscopy (TEM)

TEM images were obtained by a JEOL JEM-2100 instrument operated at 80 kV. A drop of micellar solution (10 μ L) was placed on a Formvar and carbon-coated copper grid for 30 s and then a filter paper touched the edge of drop to absorb most of liquid on the grid. The grid was allowed to dry at room temperature. For each sample, length distribution of micelles was determined by tracing more than 100 individual micelles, and width distribution was determined by making measurements at least 100 different positions on several micelles and analysis using the ImageJ software program from National Institutes of Health. Values of number-average length (L_n), weight-average length (L_w), number-average width (W_n) and weight-average width (W_w), of micelles were calculated as follows:

$$L_{n} = \frac{\sum_{i=1}^{N} N_{i}L_{i}}{\sum_{i=1}^{N} N_{i}}$$

$$L_{n} = \frac{\sum_{i=1}^{N} N_{i}L_{i}}{\sum_{i=1}^{N} N_{i}L_{i}}$$

$$L_{w} = \frac{\sum_{i=1}^{N} N_{i}W_{i}}{\sum_{i=1}^{N} N_{i}W_{i}}$$

$$W_{n} = \frac{\sum_{i=1}^{N} N_{i}}{\sum_{i=1}^{N} N_{i}}$$

$$W_{\mathbf{w}} = \frac{\sum_{i=1}^{N} N_i W_i^2}{\sum_{i=1}^{N} N_i W_i}$$

where N_i is the number of micelles of length L_i and width W_i , N is the number of calculated micelles in each sample. The distribution of micellar length and width is characterized by both L_w/L_n and W_w/W_n respectively, and the standard deviation of length and width distribution σ .

UV/vis and fluorescence spectroscopy

UV/vis absorption spectra were recorded on a Hitachi U-2910 spectrophotometer. Fluorescence spectra were measured by using a Hitachi F-2700 fluorescence spectrophotometer with a 10 nm band width. UV/vis and fluorescence measurements were performed at room temperature for ethanol or THF solutions of OPV_5 -*b*-P18C6AM₆₆-*co*-P18C6AM₆₆ and OPV_5 -*b*-PDMAEAM₆₆ (0.05 mg/mL).

Monomer and Polymer Synthesis



Scheme S1. Synthesis of PPFMA.

Synthesis of PPFMA

Into a 50 mL Schlenk flask pre-equipped with a stirring bar (flame-dried under vacuum prior to use and then kept under N_2), PFMA monomer (3.05 g, 12.1 mmol), 4- cyano-4-((thiobenzoyl)sulfanyl)pentanoic acid (85 mg, 0.24 mmol), AIBN (13 mg, 0.08 mmol), and 7.5 mL of redistilled dioxane were added. The reaction mixture was then subjected to three cycles of freezing-pumping-thawing and purging with N_2 to

remove oxygen. The Schlenk flask was heated in a constant-temperature oil bath at 70°C under a nitrogen atmosphere for 8 hours before quenched with liquid nitrogen. The resulting mixture was precipitated three times with a mixture of *n*-hexane and diethyl ether (v/v = 3/1), dried to obtain a pale pink solid, and further *in vacuo* dried to yield 2.63 g of product. The molar ratio of CTA to monomer was controlled at 1:50. In Figure S1, where a prominent characteristic peak of methylene group in $CH_2(CH_3)C(CO_2)CS_2$ appears at around 2.45 ppm. ¹⁹F NMR (376 MHz): δ (ppm): -150.64 (2F), -156.59 (1F), -161.80 (2F).

GPC: $M_n^{\text{GPC}} = 12,000 \text{ g/mol}, M_w / M_n = 1.13.$

Synthesis of P18C6AM₁₈-co-POEGAM₄₂



Scheme S2. Synthesis of polymers containing DMA and 18C6 units by post-polymerization functionalization.

PPFMA ($M_n^{NMR} = 12,000 \text{ g/mol}, 300 \text{ mg}, 0.025 \text{ mmol}$) and 2,3,5,6,8,9,11,12,14,15decahydrobenzo[b][1,4,7,10,13,16]hexaoxacyclooctadecin-18-amine (682 mg, 2.08 mmol) were dissolved in 15 mL of dry THF under N₂. Next, *N*-ethyldiisopropylamine (0.314 mL, 1.84 mmol) was added via a syringe and the reaction mixture was stirred at 50°C under N₂ for 48 h. The mixture was precipitated in *n*-hexane three times until all free pentafluorophenol and amines were entirely removed. The product of PPFMA-*co*-P18C6AM (brown liquid), was obtained by drying *in vacuo* overnight.

¹⁹F NMR (before purification, 376 MHz): δ (ppm): -152.52 (2F), -159.80 (1F), -164.65

(2F).

PPFMA-*co*-P18C6AM (150mg, 0.012 mmol) and 2,5,8,11,14-pentaoxahexadecan-16-amine (0.310 mL, 1.26 mmol) were dissolved in 15 mL of dry THF under N₂. Next, *N*-ethyldiisopropylamine (0.157 mL, 0.92 mmol) was added via a syringe and the reaction mixture was stirred at 50°C under N₂ for 24 h. The complete substitution of pentafluorophenyl ester with amines was also confirmed by the total disappearance of typical signals attributed to pentafluorophenyl of polymers in Figure 1. The mixture was precipitated in *n*-hexane and diethyl ether until all free pentafluorophenol were entirely removed. The final product of P18C6AM-*co*-POEGAM (dark brown liquid) was obtained. The purified polymer was subjected to GPC (Figure S2) and ¹H NMR (Figure 1) analysis.

GPC: $M_n^{GPC} = 33,000 \text{ g/mol}, M_w / M_n = 1.15.$

Synthesis of PDMAEAM

PPFMA (300 mg, 0.025 mmol) and *N*,*N*-dimethylethylenediamine (0.275 mL, 3.12 mmol) were dissolved in 15 mL of dry THF under N₂. Next, *N*-ethyldiisopropylamine (0.314 mL, 1.84 mmol) was added via a syringe and the reaction mixture was stirred at 50°C under N₂ for 24 h. The complete substitution of pentafluorophenyl ester with amine was also confirmed by the total disappearance of typical signals attributed to pentafluorophenyl of polymers in Figure 6. The mixture was precipitated in *n*-hexane and diethyl ether until all free pentafluorophenol were entirely removed. The final product of PDMAEAM (white solid) was obtained at 45°C overnight. The purified polymer was subjected to ¹H NMR (Figure S4) analysis.

Synthesis of Alkyne-terminated OPV₅

Alkyne-terminated OPV_5 was prepared by Seigrist polycondensation followed by chain end modification. The details about the synthesis of alkyne-terminated OPV_5 were described in our previous report.¹ The product was subjected to ¹H NMR (**Figure S5**), GPC analysis (**Figure S7**), and MALDI-TOF-MS (**Figure S6**).

GPC: $M_n^{GPC} = 2,100 \text{ g/mol}, M_w/M_n = 1.05.$

¹H NMR (400 MHz, CD₂Cl₂): δ (ppm): 7.55-7.34 (m, 8H,ArC*H*=), 7.26-7.03 (m, 8H, Ar*H*), 6.94 (s, 1H, Ar*H*), 6.71 (s, 1H, Ar*H*), 4.60 (s, 2H, ArC*H*₂), 4.20 (d, J = 2.4 Hz, 2H, -OC*H*₂C≡CH), 4.10- 3.87 (m, 20H, ArOC*H*₂-), 2.43(t, J = 2.4 Hz, 1H, -OCH₂C≡C*H*), 1.91-1.73 (m, 20H, ArOCH₂C*H*₂), 1.60-1.45 (m,20H, OCH₂CH₂C*H*₂), 1.42-1.13 (m, 40H, *CH*₂*CH*₂CH3), 1.01-0.74 (t, 30H, *CH*₃).

MALDI-TOF-MS: calculated 1570.4 for C₁₀₃H₁₅₆O₁₁, found 1570.6 [M]⁺.



Synthesis of OPV₅-b-P18C6AM₁₈-co-POEGAM₄₂

Scheme S3. Synthesis of OPV₅-*b*-P18C6AM₁₈-*co*-POEGAM₄₂ by CuAAC reaction.

 OPV_5 -*b*-P18C6AM₁₈-*co*-POEGAM₄₂ copolymers were prepared by Cu-catalyzed alkyne-azide cycloaddition (CuAAC) reaction (Scheme S3) according to our previous work.¹ In a typical procedure, alkyne-terminated OPV₅ (20 mg, 0.011 mmol),

P18C6AM₁₈-co-POEGAM₄₂ (120 mg, 0.017 mmol), CuCl (3mg, 0.03 mmol) and TBTA (15 mg, 0.03 mmol) were dissolved in anhydrous THF (15 ml) under N₂. The flask was degassed by three freeze-pump-thaw cycles followed by immersing the flask into an oil bath set at 50°C for 48 h.

The crude product was purified by silica column chromatography (eluent: $CH_2Cl_2/methanol = 10/1$, $CH_2Cl_2/methanol/triethylamine = 10/1/1$) to remove the unreacted alkyne-terminated OPV₅ and catalyst. After removal of unreacted OPV₅ and P18C6AM-*co*-POEGAM by repeated precipitation in *n*-hexane and dialysis against ethanol (molecular weight cut-off: 50000 g/mol), finally, the product was dried *in vacuo* overnight to afford OPV₅-*b*-P18C6AM₁₈-*co*-POEGAM₄₂ as an orange solid (10 mg). The purified sample was subjected to ¹H NMR and GPC analyses (Figures 1 and S7).

Synthesis of OPV₅-*b*-PDMAEMAM₆₆



Scheme S4. Synthesis of OPV₅-*b*-PDMAEMAM₆₆ by CuAAC reaction.

Alkyne-terminated OPV₅ (20 mg, 0.011 mmol), PDMAEMAM₆₆ (150 mg, 0.016 mmol), CuCl (3mg, 0.03 mmol) and TBTA (15 mg, 0.03 mmol) were dissolved in anhydrous THF (15 ml) under N₂. The flask was degassed by three freeze-pump-thaw cycles followed by immersing the flask into an oil bath set at 50°C for 48 h. An alumina

column was chosen to remove Cu-based salts. The crude product was purified by silica column chromatography (eluent: $CH_2Cl_2/methanol = 10/1$, $CH_2Cl_2/methanol/$ triethylamine = 10/1/1) to remove the unreacted alkyne-terminated OPV₅ and catalyst. After removal of unreacted OPV₅ and PDMAEMAM₆₆ by repeated precipitation in *n*hexane and dialysis against ethanol (molecular weight cut-off: 50000 g/mol), the product was dried *in vacuo* overnight to afford OPV₅-*b*-PDMAEMAM₆₆ as an orange solid (21 mg). The purified sample was subjected to ¹H NMR and GPC analyses (Figures 6 and S7).

Self-assembly Experiments

Self-assembly of OPV₅-*b*-P18C6AM₁₈-*co*-POEGAM₄₂ and OPV₅-*b*-PDMAEMAM₆₆ in ethanol

Typically, suspensions of OPV_5 -*b*-P18C6AM₁₈-*co*-POEGAM₄₂ and OPV_5 -*b*-PDMAEAM₆₆ (0.05 mg/mL in ethanol) were heated at 80°C for 30 min, followed by aging at room temperature (25°C) for 24 h. A drop of solution was placed on a Formvar and carbon-coated copper grid and examined by TEM (Figures 2 and 7).

Preparation of seed micelles of OPV₅-*b*-P18C6AM₁₈-*co*-POEGAM₄₂ and OPV₅-*b*-PDMAEAM₆₆

Seed micelles were prepared by sonicating (SONICS VC 750 ultrasonic processor, 30% power) long cylindrical micelles (0.05 mg/mL) formed by OPV₅-*b*-P18C6AM₁₈*co*-POEGAM₄₂ and OPV₅-*b*-PDMAEAM₆₆ at 0°C for 30 min. After aging at room temperature (25°C), a drop of solution was placed on a Formvar and carbon-coated copper grid and examined by TEM (Figures 3 and 7).

Self-seeding of seed micelles of OPV₅-*b*-P18C6AM₁₈-*co*-POEGAM₄₂ and OPV₅-*b*-PDMAEMAM₆₆

Self-seeding of OPV₅-*b*-P18C6AM₁₈-*co*-POEGAM₄₂ and OPV₅-*b*-PDMAEMAM₆₆ was conducted by thermal annealing at different annealing temperatures in ethanol. Aliquots of seed micellar solution (0.05 mg/mL) with or without the addition of metal ions were placed in several vials (2 mL/vial) followed by putting the vials into water baths set with different temperatures. After heating for 1 h, the vials were removed from the bath and then cooled and aged at 25°C for 24h. Finally, a drop of each solution was placed on a Formvar and carbon-coated copper grid and examined by TEM (Figures 3-4 and S8-10).

Surface functionalization of ribbon-like micelles of OPV₅-*b*-PDMAEMAM₆₆ with Ag nanoparticles

An aqueous solution of AgPF₆ (5 mmol/L, 8 μ L) was added into 1 mL of as-prepared long fiber-like micelles of OPV₅-*b*-PDMAEMAM₆₆ (0.05 mg/mL) in ethanol. The mixture was incubated at room temperature for 1 h. Subsequently, an aqueous solution of NaBH(CO₂CH₃)₃ (5 mmol/L, 8 μ L) was added, followed by aging room temperature for 24 h and observed by TEM (Figure S15).

Surface functionalization of ribbon-like micelles of OPV₅-*b*-PDMAEMAM₆₆ with phosphotungstic acid

An aqueous solution of phosphotungstic acid (0.3 mmol/L, 20 μ L) was added slowly into 0.5 mL of micellar solution of OPV₅-*b*-PDMAEMAM₆₆ (0.05 mg/mL in ethanol), respectively. The mixture was incubated and aged at room temperature for 24 h. A TEM image is shown in Figures 8C-E. SUPPORTING FTGURES



Figure S1. ¹H NMR spectrum of PPFMA in CD₂Cl₂.



Figure S2. GPC curve of PPFMA in DMF.



8 7 6 5 4 3 2 1 (δ (ppm)

Figure S4. ¹H NMR spectrum of PDMAEAM₆₆ in CDCl₃.

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Figure S5. ¹H NMR spectrum of alkyne-terminated OPV₅ in CD₂Cl₂.



Figure S6. MALDI-TOF-MS spectrum of alkyne-terminated OPV₅.



Figure S7. GPC curves of alkyne-terminated OPV_5 , OPV_5 -*b*-P18C6AM₁₈-*co*-POEGAM₄₂ and OPV_5 -*b*-PDMAEAM₆₆ using THF containing 2.5 g/L tetra-*n*-butyl-ammonium bromide (TBAB) as eluent.



Figure S8. TEM images and length distribution histograms of (A) seed micelles and fiber-like micelles of OPV_5 -*b*-P18C6AM₁₈-*co*-POEGAM₄₂ obtained by annealing the seeds (0.05 mg/mL in ethanol) at (B) 45°C, (C) 50°C, (D) 55°C, (E) 60°C and (F) 65°C for 30 min, followed by cooling to 25°C and then aged for 24 h in self-seeding experiments.



Figure S9. TEM images and length distribution histograms of fiber-like micelles of OPV_5 -*b*-P18C6AM₁₈-*co*-POEGAM₄₂ with the addition of CH₃COONa obtained by annealing the seeds (0.05 mg/mL in ethanol) at (A) 40°C, (B) 45°C, (C) 50°C, (D) 55°C, (E) 60°C and (F) 65°C for 30 min, followed by cooling to 25°C and then aged for 24 h in self-seeding experiments.



Figure S10. TEM images and length distribution histograms of fiber-like micelles of OPV_5 -*b*-P18C6AM₁₈-*co*-POEGAM₄₂ with the addition of CH₃COOK obtained by the seeds (0.05 mg/mL in ethanol) at annealing (A) 45°C, (B) 50°C, (C) 55°C, (D) 60°C, (E) 65°C and (F) 70°C for 30 min, followed by cooling to 25°C and then aged for 24 h in self-seeding experiments.



Figure S11. ¹H NMR spectra highlighting the oxymethylene hydrogens of P18C6AM*co*-POEGAM with the addition of different amounts of guest metal ions of (A) Na⁺ and (B) K⁺.



Figure S12. (A) Normalized UV/vis absorption spectrum of OPV_5 -*b*-P18C6AM₁₈-*co*-POEGAM₄₂ in ethanol (0.05 mg/mL) over the aging process at 25°C after heating at 80°C for 30 min. (E) Normalized UV/vis absorption spectrum of OPV_5 -*b*-P18C6AM₁₈-*co*-POEGAM₄₂ with the addition of CH₃COOK in ethanol (0.05 mg/mL) over the aging process at 25°C after heating at 80°C for 30 min.



Figure S13. TEM images of fiber-like micelles of OPV_5 -*b*-P18C6AM₁₈-*co*-POEGAM₄₂ formed with (A) S-PMP and (B) D-Leu.



Figure S14. (A) UV/vis absorption and (B) fluorescence spectra of OPV_5 -*b*-PDMAEAM₆₆ in ethanol (0.05 mg/mL) and in THF (0.05 mg/mL).



Figure S15. TEM images and length distribution histograms of fiber-like micelles of OPV_5 -*b*-PDMAEAM₆₆ obtained by annealing the seeds (0.05 mg/mL in ethanol) at (A) 40°C, (B) 45°C, (C) 50°C, (D) 55°C, (E) 60°C and (F) 65°C for 30 min, followed by cooling to 25°C and then aged for 24 h in self-seeding experiments.



Figure S16. TEM images of fiber-like micelles of OPV₅-*b*-PDMAEAM₆₆ coated with Ag NPs.



Figure S17. (A) Fluorescence and (B) UV/vis absorption spectra of OPV_5 -*b*-PDMAEAM₆₆ (0.05 mg/mL) and TPPS (0.13 mg/mL) in ethanol/DMSO (v/v=30/1).

SUPPORTING TABLES

Table S1. Characteristics of seed micelles and elongated ribbon-like micelles of OPV_5 *b*-P18C6AM₁₈-*co*-POEGAM₄₂ obtained by self-seeding approach in ethanol (0.05 mg/mL)^a

T (°C)	$L_{n}(nm)$	$L_{\rm w}({\rm nm})$	$L_{\rm w}/L_{\rm n}^{\rm b}$	$\sigma^{b}\left(nm\right)$	σ/L_n^b
seed	55	62	1.13	20	0.37
45	176	198	1.13	64	0.36
50	321	348	1.08	94	0.29
55	838	1007	1.20	380	0.45
60	1899	2260	1.19	840	0.44
65	2387	2858	1.20	1090	0.46

^a The mean length of micelles was calculated from measurements of over 100 individual micelles in multiple TEM images.

T (°C)	L_{n} (nm)	$L_{\rm w}$ (nm)	$L_{\rm w}/L_{\rm n}^{\rm b}$	$\sigma^{b}(nm)$	σ/L_n^{b}
40	87	100	1.15	33	0.38
45	111	123	1.11	37	0.33
50	133	147	1.11	44	0.34
55	307	374	1.22	145	0.47
60	830	986	1.19	363	0.44
65	1624	1908	1.18	687	0.42

Table S2. Characteristics of seed micelles and elongated ribbon-like micelles of OPV_5 *b*-P18C6AM₁₈-*co*-POEGAM₄₂ with the addition of CH₃COONa (molar ratio of 18C6 unit to Na⁺ of 1:1) obtained by self-seeding approach in ethanol (0.05 mg/mL)^a

^a The mean length of micelles was calculated from measurements of over 100 individual micelles in multiple TEM images.

T (°C)	L_{n} (nm)	$L_{\rm w}$ (nm)	$L_{\rm w}/L_{\rm n}^{\rm b}$	$\sigma^{b}\left(nm\right)$	σ/L_n^{b}	
45	55	62	1.13	20	0.37	
50	176	198	1.13	64	0.36	
55	321	348	1.08	94	0.29	
60	838	1007	1.20	380	0.45	
65	1899	2260	1.19	840	0.44	
70	2387	2858	1.20	1090	0.46	

Table S3. Characteristics of seed micelles and elongated ribbon-like micelles of OPV_5 *b*-P18C6AM₁₈-*co*-POEGAM₄₂ with the addition of CH₃COOK (molar ratio of 18C6 unit to K⁺ of 1:1) obtained by self-seeding approach in ethanol (0.05 mg/mL)^a

^a The mean length of micelles was calculated from measurements of over 100 individual micelles in multiple TEM images.

T (°C)	L_{n} (nm)	$L_{\rm w}$ (nm)	$L_{\rm w}/L_{\rm n}{}^{\rm b}$	$\sigma^{b}\left(nm\right)$	σ/L_n^{b}	
seed	60	77	1.29	33	0.55	
40	109	126	1.16	44	0.41	
45	145	175	1.21	67	0.46	
50	194	233	1.20	89	0.46	
55	359	433	1.21	165	0.46	
60	1237	1543	1.25	638	0.52	
65	1982	2389	1.21	942	0.48	

Table S4. Characteristics of seed micelles and elongated ribbon-like micelles of OPV_5 -*b*-PDMAEMAM₆₆ obtained by self-seeding approach in ethanol (0.05 mg/mL)^a

^a The mean length of micelles was calculated from measurements of over 100 individual micelles in multiple TEM images.

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