Chiral Helical Disubstituted polyacetylenes Construct Optically Active Particles through Precipitation Polymerization

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Preparation of monomer and crosslinker

Figure S1. The preparation process of (+)-M1, (−)-M1 and crosslinker.
**Figure S2.** FT-IR spectra of (+)-M1 and (+)-CCPMs (in KBr tablet).

In FT-IR spectrum of (+)-M1 and (+)-CCPMs, 2223 cm$^{-1}$ for (–C≡C); 1702 cm$^{-1}$, 1290 cm$^{-1}$ and 1189 cm$^{-1}$ for (–CO–O–), 765 cm$^{-1}$ and 690 cm$^{-1}$ for benzene ring.

**Figure S3.** $^1$H NMR (A) and $^{13}$C NMR (B) spectrum of (+)-M1 in CDCl$_3$.

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ ppm, 7.34-7.66 (m, 5H, Ar-H), $\delta$ 5.04 (t, 1H, CH), $\delta$ 2.0-2.4 (m, 2H, CH$_2$), $\delta$ 1.78 (t, 1H, CH), $\delta$ 1.35-1.72 (m, 2H, CH$_2$), $\delta$ 1.09-1.28 (m, 2H, CH$_2$), $\delta$ 0.93 (s, 3H, CH$_3$), $\delta$ 0.90 (s, 6H, CH$_3$).

$^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ ppm, 154.8, 133.2, 130.1, 128.2, 119.5, 85.7, 82.2, 80.8, 48.9, 47.5, 44.5, 36.2, 27.9, 27.4, 19.6, 13.2.
Figure S4. FT-IR spectrum of the crosslinker (in KBr tablet).

In FT-IR spectrum of the cross-linker, 2223 cm\(^{-1}\) for (–C≡C); 1702 cm\(^{-1}\), 1290 cm\(^{-1}\) and 1189 cm\(^{-1}\) for (–CO–O–), 765 cm\(^{-1}\) and 690 cm\(^{-1}\) for benzene ring.

Figure S5. \(^{1}\)H NMR spectrum of crosslinker in CDCl\(_3\).

\(^{1}\)H NMR (400 MHz, CDCl\(_3\)): \(\delta\) ppm, 7.33-7.66 (m, 10H, Ar-H), \(\delta\) 4.24 (t, 4H, CH\(_2\)), \(\delta\) 1.64-1.78 (t, 4H, CH\(_2\)), \(\delta\) 1.43-1.54 (t, 4H, CH\(_2\)).

\(^{13}\)C NMR (100 MHz, CDCl\(_3\)): \(\delta\) ppm, 154.7, 133.1, 130.2, 128.3, 119.6, 85.7, 82.2, 64.1, 28.4, 25.5.
Figure S6. GPC chromatogram of (+)-P1 calibrated with polystyrenes, using DMF as eluent, at room temperature, polymer concentration 1 mg/ml.
Figure S7. SEM images of (+)-CCPMs prepared in dioxane/ butyl acetate solvent mixture with varied ratio: A, 1/9; B, 2/8; C, 3/7; D, 4/6; E, 5/5; F, 6/4; (v/v) WCl₆, 10 mg; Ph₄Sn, 10.7 mg; (+)-M1 amount, 50 mg; crosslinker amount, 10 mg; polymerization at 80 °C for 24 h.
**Figure S8.** SEM image of polymer particles prepared with (+)-M1 in the absence of crosslinker; (+)-M1 50 mg, dioxane/DMF = 9.85/0.15 (v/v), WCl$_6$/Ph$_4$Sn = 10/10.7 in mg.

![SEM image](image)

**Figure S9.** CD and UV-vis absorption spectra of (+)- and (-)-CCPMs mixture with equal amount, measured by dispersing microspheres in dioxane solvent.

![CD and UV-vis spectra](image)
**Figure S10.** SEM images of NPs prepared by WCl<sub>6</sub>-Ph<sub>4</sub>Sn with different amount: A, 10/11; B, 20/21.5; C, 30/33; D, 40/43 in mg. (dioxane/butyl acetate, 2/8, v/v; (+)-M1 amount, 0.14 ml; polymerization at 80 °C for 24 h)

**Figure S11.** FT-IR spectrum of M2 and corresponding NPs (with KBr as tablet).

In FT-IR spectrum of M2 and NPs, 2237 cm<sup>-1</sup> for (–C≡C); 1646 cm<sup>-1</sup> for (C=C).
Figure S12. GPC chromatogram of P2 calibrated with polystyrenes, using DMF as eluent, at room temperature, polymer concentration 1mg/ml.

Figure S13. CD and UV-vis absorption spectra of M2 and P2, measured by dissolving M2 and P2 in DMF solvent (c= 10⁻⁴ M, by monomer units).