Supporting Information

Chiral, fluorescent microparticles constructed by optically active helical substituted polyacetylene: preparation and enantioselective recognition ability

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Figure S1. NMR spectra of monomer. A, 1H NMR (measured in DMSO-d6 at room temperature); B, 13C NMR (measured in CDCl3 at room temperature).

1H NMR (400 MHz, DMSO-d6): δ8.89 (d, 1H, Ar–H), 8.37 (d, 1H, Ar–H), 8.27–7.97 (m, 2H, Ar–H), 7.88–7.47 (m, 2H, Ar–H), 5.50–4.06 (s, 1H, N–H), 4.02–3.48 (m, 2H, CH2N), 3.35–2.99 (m, 6H, N(CH3)2), 2.92 (s, 1H, HC≡C), 2.74 (t, 1H, HC), 1.37–0.92 (ddt, 3H, CH3).

13C NMR (100 MHz, CDCl3): δ170.9, 133.8, 130.9, 129.9, 129.1, 128.8, 123.3, 118.4, 115.5, 79.0, 71.5, 52.9, 45.8, 29.1, 18.9, 8.6.
Figure S2. SEM image of D-CFMPs prepared in the solvent mixture of CHCl₃/ n-heptane: 3/5 (in ml).

Figure S3. Typical ¹H NMR (A) and ¹³C NMR (B) spectra of the chiral fluorescent substituted polyacetylene microparticles (measured in CDCl₃ at room temperature). A new sharp signal of Ha appeared around 6.5 ppm (¹H NMR). According to the ratio of this signal’s intensity to that of Hb, the cis content of the polymer chains constructing the chiral fluorescent substituted polyacetylene was almost 100%.

¹³C NMR (100 MHz, CDCl₃): δ92.4, 92.2, 91.8, 77.3, 77.0, 76.7, 76.5, 76.2, 71.9, 71.6, 71.3, 69.1, 68.7, 68.4, 62.9, 62.5, 62.2, 45.9, 45.4, 40.4, 8.6, 8.1.

Elemental analysis: Anal. Calcd for C₁₈H₂₁N₃O₃S: C, 60.15; H, 5.89; N, 11.69; S, 8.92. Found: C, 60.06; H, 5.86; N, 11.73; S, 8.95.
**Figure S4.** TGA curve of the chiral fluorescent microparticles (at a rate of 10 °C /min in N\textsubscript{2}). The polymer chain began to disintegrate at about 160 °C. Approx. 16 wt% of the original mass remained up to 1000 °C.

**Figure S5.** DSC thermogram of the chiral fluorescent microparticles measured at a heating rate of 10 °C /min from 40 to 200 °C in N\textsubscript{2}. Tg is determined around 90 °C.

**Figure S6.** CD (A) and UV-vis (B) spectra of L-CFMPs dispersed in water with different ratio of L-alanine/D-alanine: 0/5; 1/4; 2/3; 2.5/2.5; 3/2; 4/1; 5/0; 0/0 (mol/mol). The variation of the change in spectra are considered being due to experimental error.
Figure S7. CD (A) and UV-vis (B) spectra of D-CFMPs dispersed in water with different ratio of L-/D-alanine: 0/5; 1/4; 2/3; 2.5/2.5; 3/2; 4/1; 5/0; 0/0 (mol/mol).

Figure S8. CD (A,C) and UV-vis (B,D) spectra of L-CFMPs dissolved in CHCl$_3$ with different amount of R-PEA and S-PEA: 0; 0.25; 0.5; 0.75; and 1 ml. The CD and UV spectra of R-PEA and S-PEA were measured qualitatively in CHCl$_3$ to illustrate that the CD signal at around 280 nm was caused by R(S)-PEA.
**Figure S9.** CD (A,C) and UV-vis (B,D) spectra of D-CFMPs dissolved in CHCl₃ with different amount of R-PEA and S-PEA: 0; 0.25; 0.5; 0.75; and 1 ml. The CD and UV spectra of R-PEA and S-PEA were measured qualitatively in CHCl₃ to illustrate that the CD signal at around 280 nm was caused by R(S)-PEA.

**Figure S10.** Fluorescence emission spectra of chiral fluorescent microparticles (D-CFMPs) dissolved in CHCl₃ with different amount of R-PEA and S-PEA: 0; 0.25; 0.5; 0.75; and 1 ml.