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, <sup>1</sup>H NMR (measured in DMSO-d6 at room temperature); B, <sup>13</sup>C NMR (measured in CDCl<sub>3</sub> at room temperature). <sup>1</sup>H 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, CH<sub>2</sub>N), 3.35−2.99 (m, 6H, N(CH<sub>3</sub>)<sub>2</sub> ), 2.92 (s, 1H, HC≡C), 2.74 (t, 1H, HC), 1.37−0.92 (dtd, 3H, CH<sub>3</sub>). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ170.9, 133.8, 130.9, 129.9, 129.8, 129.5, 128.8, 123.3,

<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): 8170.9, 133.8, 130.9, 129.9, 129.8, 129.5, 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  $CHCl_3/n$ -heptane: 3/5 (in ml).



**Figure S3**. Typical <sup>1</sup>H NMR (A) and <sup>13</sup>C NMR (B) spectra of the chiral fluorescent substituted polyacetylene microparticles (measured in CDCl<sub>3</sub> at room temperature). A new sharp signal of Ha appeared around 6.5 ppm (<sup>1</sup>H 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%. <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>):  $\delta$ 92.4, 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 C<sub>18</sub>H<sub>21</sub>N<sub>3</sub>O<sub>3</sub>S: 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  $N_2$ ). 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  $^{\circ}$ C /min from 40 to 200  $^{\circ}$ C in N<sub>2</sub>. Tg is determined around 90  $^{\circ}$ 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<sub>3</sub> 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<sub>3</sub> 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 CHCl<sub>3</sub> 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<sub>3</sub> 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 CHCl<sub>3</sub> with different amount of R-PEA and S-PEA: 0; 0.25; 0.5; 0.75; and 1 ml.