S1 of S3

Supplementary Materials: Hierarchical Self-Assembly of Amino Acid Derivatives into Enzyme-Responsive Luminescent Gel

Yibao Li, Yu Peng, Wei Liu, Yulan Fan, Yongquan Wu, Xun Li and Xiaolin Fan

1. Synthesis and Characterization of Amino Acid Derivatives (NPPD)

An amount of 0.392 g (1.0 mmol) perylene-3,4,9,10-tetracarboxylic dianhydride, 0.330 g (2.0 mmol) L-phenylalanine and 2.0 g imidazole were heated at 90 °C for 40 min under nitrogen atmosphere. When the imidazole was melting, the temperature was raised to 120 °C for 6 h. Then 25 mL of CH₃CH₂OH was poured into the round-bottom flask, refluxed for 6 h at 90 °C and cooled down to room temperature, then let stand for 12 h. An amount of 1.0 moL/L HCl was added dropwise untill the mixture into was acidi. The mixture was kept at an ambient temperature overnight to let it precipitate out. The precipitate was filtered and washed with H₂O. The product was dried at room temperature to obtain mulberry powder (yield: 0.665 g, 97%). The structure and purity of the product were confirmed by ¹H NMR, FT-IR and MS.



Figure S1. Synthesis route of amino acid derivatives.

IR (KBr): 1252.4, 1396.4, 1501.2 cm⁻¹ (C=C), 1344.9, 1432.5 cm⁻¹ (-CH₂-), 1656.6, 1697.0, 1737.9 cm⁻¹ (C=O), 2934.8 cm⁻¹ (C-H), 3439.3 cm⁻¹ (O-H); 1H NMR (DMSO, 400 MHz, ppm): 8.31–8.45 (q, 8H), 7.13–7.23 (m, 8H), 7.04–7.08 (t, 2H), 5.92–5.95 (q, 2H), 3.59–3.64 (q, 2H), 3.40–3.44 (t, 2H), 3.32 (s, 2H); MALDI-TOF-MS: calcd for C₄₂H₂₈N₂O₈, 686.1.

2. Photos of Gels



Figure S2. Optical photograph of NPPD/ α -CD/RF = 1/1/32hydrogel.



3. Photos of One Components Solution with Different Mole

Figure S3. Optical images of compounds in a mixture solution ($V_{THF}/V_{H2O} = 4/6$) under ultrasound at room temperature with different mole, from left to right: (**a**) α -CD; (**b**) RF; (**c**) NPPD.

4. Photos of One and Two Components Solution with Same Mole



Figure S4. Optical images of compounds in a mixture solution under ultrasound at room temperature, from left to right, (a) NPPD solution; (b) RF solution; (c) α -CD solution; (d) solution of α -CD/RF = 1/2, (e) solution of NPPD/RF = 1/2, (f) solution of NPPD/ α -CD = 1/1.

5. FT-IR Data



Figure S5. FT-IR spectra diluted with KBr for a NPPD/ α -CD /RF = 1/1/2 xerogel (blue), NPPD (green), RF (black), α -CD (red).

6. CLSM Images of Gels and Hydrogel after α-Amylase Processing



Figure S6. (a) CLSM images of NPPD/ α -CD /RF (1/1/2) hydrogel ([GP] = 10⁻³ M); (b) for NPPD/RF/ α -CD hydrogel in a bright filed image; (c) CLSM images of hydrogel after α -amylase processing; (d) for hydrogel after α -amylase processing in a bright filed image.