Helical periodic mesoporous 1,4-phenylene-silica nanorods with chiral crystalline walls

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General methods

Transmission electron microscopy (TEM) images were obtained using an FEI Tecnai G220 instrument, and field emission scanning electron microscopy (FESEM) images with a Hitachi S-4800 instrument. High resolution transmission electron microscopy (HRTEM) images were taken using FEI Tecnai G2 F20S-TWIN instrument. Before taking FESEM images, the sample surfaces were coated with 10 nm of platinum. Specific surface areas and pore-size distributions were determined by the Brunauer-Emmett-Teller (BET) and Barrett-Joyner-Halenda (BJH) methods, from N₂ adsorption isotherms measured with a Micromeritics ASAP 2020M+C instrument. The SAXRD and wide-angle X-ray diffraction (WAXRD) patterns were recorded with an X' Pert-Pro MPD X-ray diffractometer. Circular dichroism (CD) and ultraviolet-visible (UV-vis) spectra were obtained using an AVIV 410 spectrometer.

Characterization of L-**18Ala11NBr**: FT-IR (KBr): 3293 cm⁻¹ (v_{N-H} , amide A), 1636 cm⁻¹ ($v_{C=O}$, amide I), 1560 cm⁻¹ (δ_{N-H} , amide II). ¹H-NMR (400 MHz, CDCl₃, TMS, 25 °C): $\delta = 0.88$ (t, J = 6.6 Hz, 3H; CH₂CH₂CH₂3), 1.24-1.32 (m, 33H; alkyl), 1.40 (t, J = 6.4 Hz, 14H; alkyl), 1.48-1.54 (m, 2H; CH₂CH₂CH₂), 1.58-1.66 (m, 2H; CH₂CH₂CH₂), 1.76 (s, 9H; N(CH₂CH₃)₃), 2.26 (m, 2H; CH₂CONH), 3.14-3.31 (m, 4H; (CH₂)₃NCH₂, CONHCH₂), 3.51 (m, 6H; N(CH₂CH₃)₃), 4.44 (m, 1H; NHCHCONH), 6.87 (d, J = 7.6 Hz, 1H; CONHCH). 6.92 (t, J = 5.6 Hz, 1H; CONHCH₂). C₃₈H₇₈BrN₃O₂ (Mw: 688.95). Elemental analysis calcd (%): C, 66.25; H, 11.41; N, 6.10. Found: C, 63.78; H, 10.94; N, 5.68.

Characterization of D-**18Ala11Et₃NBr**: FT-IR (KBr): 3291 cm⁻¹ (v_{N-H}, amide A), 1635 cm⁻¹ (v_{C=O}, amide I), 1560 cm⁻¹ (δ_{N-H} , amide II). ¹H-NMR (400 MHz, CDCl₃-*d*, TMS, 25 °C): $\delta = 0.88$ (*t*, *J* = 6.8 Hz, 3H; CH₂CH₂C<u>H₃</u>), 1.24-1.32 (*m*, 33H; alkyl), 1.40 (*t*, *J* = 6.6 Hz, 14H; alkyl), 1.48-1.54 (*m*, 2H; CH₂C<u>H₂CH₂</u>), 1.59-1.64 (*m*, 2H; CH₂C<u>H₂CH₂</u>), 1.74 (*s*, 9H; N(CH₂C<u>H₃</u>)₃), 2.26 (*m*, 2H; C<u>H₂CONH</u>), 3.15-3.32 (*m*, 4H; (CH₂)₃NC<u>H₂</u>, CONHC<u>H₂</u>), 3.51 (*m*, 6H; N(C<u>H₂CH₃</u>)₃), 4.47 (*m*, 1H; NHC<u>H</u>CONH), 6.85 (*d*, *J* = 7.6 Hz, 1H; CON<u>H</u>CH). 6.90 (*t*, *J* = 5.2 Hz, 1H; CON<u>H</u>CH₂). C₃₈H₇₈BrN₃O₂ (*M*w: 688.95). Elemental analysis calcd (%): C, 66.25; H, 11.41; N, 6.10. Found: C, 62.65; H, 11.66; N, 5.69.

Typical synthetic procedure for the mesoporous 1,4-phenylene silicas: L-18Ala11NEt₃Br (100 mg, 0.15 mmol) was dissolved in 100.0 mL of deionized water together with 0.70 mL of aqueous NaOH (2.0 M) solution. Then 0.25 mL (0.61 mmol) of bis(triethoxysilyl)benzene (BTEB) was added to the mixture with stirring at 80 1C. The reaction mixture was kept at 80 1C for 2.0 h, then L-18Ala11NEt₃Br was removed by extraction with a mixture of HCl aq. and methanol for 24 h, and dried in air at room temperature.



Figure S1. CD spectra of viscous aqueous solutions of L- and D-18Ala11NEt₃Br (10.0 mg mL⁻¹).



Figure S2. FESEM images of the 1,4-phenylene-silicas prepared using D-18Ala11Et₃NBr.



Figure S3. HRTEM images of the 1,4-phenylene-silicas prepared using D-18Ala11Et₃NBr.



Figure S4. FESEM images of the reaction mixture taken after different reaction times. (a) 40 s; (b) 2.0 min; (c) 6.0 min; (d)1.0 h.