

Supplementary information for

Tuning the diameters of self-assembled nanotubes by modification of an alkyl size in a series of diamides

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S1. Compounds BHPB-n

Hexyle 3,5-dihydroxybenzoate (A6) : same protocol as A5 : white solid (yield 63 %). M. p : 55.7 °C. ¹H NMR (400 MHz, CDCl₃) : δ [ppm] 7.08 (d, 2 H, J = 2.3 Hz, C2-H, C6-H), 6.93 (s large, 2 H, O-H), 6.61 (t, 2H, J = 2.2 Hz, C4-H), 4.29 (t, 2 H, J = 6.8 Hz, COOCH₂), 1.74 (m, 2 H, COOCH₂CH₂), 1.33 (m, 6 H, COOCH₂CH₂(CH₂)₃), 0.89 (t, 3 H, J = 6.5 Hz, CH₃) ; ¹³C NMR (100 MHz, CDCl₃) : δ [ppm] 167.7 (COO), 157.0 (C3,C5), 131.8 (C1), 109.0 (C2, C6), 107.8 (C4), 66.0 (COOCH₂), 31.3 (COO(CH₂)₃CH₂), 28.6 (COO(CH₂)₂CH₂), 25.5 (COOCH₂CH₂), 22.4 (COO(CH₂)₄CH₂), 13.8 (CH₃) ; FTIR (ATR-diamond) ν_{max} : 3313, 2953, 2930, 2859, 1719, 1687, 1680, 1599 cm⁻¹; HRMS (FAB+) m/z 239.1279 (MH⁺, calcd for C₁₃H₁₈O₄ : 239.1283).

Anal. Found C, 65.06 ; H, 7.48. Calcd for C₁₃H₁₈O₄ : C, 65.53; H, 7.61.

Heptyl 3,5-dihydroxybenzoate (A7) : same protocol as A5 white solid (yield 79 %). M. p. 56.2 °C. ¹H NMR (400 MHz, CDCl₃) : δ [ppm] 7.03 (d, 2 H, J = 1.8 Hz, C2-H, C6-H), 6.92 (s large, 2 H, O-H), 6.61 (t, 1 H, J = 2.0 Hz, C4-H), 4.30 (t, 2 H, J = 6.6 Hz, COOCH₂), 1.74 (m, 2 H, COOCH₂CH₂), 1.30 (m, 8 H, COOCH₂CH₂(CH₂)₄), 0.88 (t, 3 H, J = 6.5 Hz, CH₃) ; ¹³C NMR (100 MHz, CDCl₃) : δ [ppm] 167.9 (COO), 156.8 (C3,C5), 131.8 (C1), 108.8 (C2, C6), 107.3 (C4), 66.3 (COOCH₂), 31.8 (COO(CH₂)₄CH₂), 29.0 (COO(CH₂)₃CH₂), 28.5 (COOCH₂CH₂), 26.0 (COO(CH₂)₂CH₂), 22.6 (COO(CH₂)₅CH₂), 14.1 (CH₃). FTIR (ATR-diamond) ν_{max} : 3374, 2956, 2932, 2858, 1722, 1692, 1684, 1604 cm⁻¹. HRMS (FAB+) m/z 253.1417 (MH⁺, calcd for C₁₄H₂₀O₄ : 253.1434).

Anal. Found : C, 66.83 ; H, 8.01. Calcd for C₁₄H₂₀O₄ : C, 66.65 ; H, 7.99.

Octyl 3,5-dihydroxybenzoate (A8) : same protocol as A5 white solid (yield 85 %). M. p. 65.9 °C. ¹H NMR (400 MHz, CDCl₃) : δ [ppm] 7.08 (d, 2 H, J = 2.1 Hz, C2-H, C6-H), 6.94 (s large, 2 H, O-H), 6.61 (t, 1 H, J = 2.1 Hz, C4-H), 4.27 (t, 2 H, J = 6.5 Hz, COOCH₂), 1.72 (m, 2 H, COOCH₂CH₂), 1.33 (m, 10 H, COOCH₂CH₂(CH₂)₅), 0.88 (t, 3 H, J = 6.1 Hz, CH₃) ; ¹³C NMR (100 MHz, CDCl₃) : δ [ppm] 167.9 (COO), 156.7 (C3,C5), 131.8 (C1), 108.5 (C2, C6), 106.9 (C4), 66.3 (COOCH₂), 31.8 (COO(CH₂)₅CH₂), 29.3 (COO(CH₂)₃CH₂), 29.2 (COO(CH₂)₄CH₂), 28.6 (COOCH₂CH₂), 26.0 (COO(CH₂)₂CH₂), 22.6 (COO(CH₂)₆CH₂), 14.1 (CH₃). FTIR (KBr) ν_{max} : 3374, 2955, 2925, 2854, 1721, 1691, 1680, 1604 cm⁻¹. MS (FAB+) m/z 267.1 (MH⁺, calcd for C₁₅H₂₂O₄ : 266.1).

Anal. Found : C, 67.78 ; H, 8.32. Calcd for C₁₅H₂₂O₄ : C, 67.64 ; H, 8.33.

Nonyl 3,5-dihydroxybenzoate (A9) : same protocol as for A5 : white solid (yield 77 %). M. p. 67.2 °C. ¹H NMR (400 MHz, CDCl₃) : δ [ppm] 7.38 (s large, 2 H, O-H), 7.04 (d, 2 H, J = 2.1 Hz, C2-H, C6-H), 6.61 (t, 1 H, J = 2.1 Hz, C4-H), 4.28 (t, 2 H, J = 6.7 Hz, COOCH₂), 1.73 (m, 2 H, COOCH₂CH₂), 1.33 (m, 12 H, COOCH₂CH₂(CH₂)₆), 0.88 (t, 3 H, J = 6.6 Hz, CH₃) ; ¹³C NMR (100 MHz, CDCl₃) : δ [ppm] 167.9 (COO), 156.8 (C3,C5), 131.9 (C1), 108.6 (C2, C6), 107.0 (C4), 66.2 (COOCH₂), 31.9 (COO(CH₂)₆CH₂), 29.5 (COO(CH₂)₅CH₂), 29.3 (COO(CH₂)₃CH₂), 29.3 (COO(CH₂)₄CH₂), 28.6 (COOCH₂CH₂), 26.0 (COO(CH₂)₂CH₂), 22.6 (COO(CH₂)₇CH₂), 14.1 (CH₃). FTIR (ATR-diamond) ν_{max} : 3396, 3361, 2955, 2923, 2854, 1689, 1608, 1448 cm⁻¹. HRMS (FAB+) m/z 281.1738 (MH⁺, calcd for C₁₆H₂₄O₄ : 281.1747).

Anal. Found : C, 68.72 ; H, 8.87. Calcd for C₁₆H₂₄O₄ : C, 68.54 ; H, 8.63.

Undecyl 3,5-dihydroxybenzoate (A11) : same protocol as A5, but the the crude was purified by recrystallisation in cyclohexane to afford A11 as a white solid (yield 73 %). M. p. 69.4°C. ¹H NMR (400 MHz, CDCl₃) : δ [ppm] 7.09 (d, 2 H, J = 2.2 Hz, C2-H, C6-H), 6.57 (t, 1 H, J = 2.2 Hz, C4-H), 5.45 (s large, 2 H, O-H), 4.25 (t, 2 H, J = 6.7 Hz, COOCH₂), 1.70 (m, 2 H, COOCH₂CH₂), 1.38 (m, 16 H, CH₂)₈CH₃), 0.86 (t, 3 H, J = 6.7 Hz, CH₃) ; ¹³C NMR (100 MHz, CDCl₃) : δ [ppm] 167.8 (COO), 156.8 (C3,C5), 131.9 (C1), 108.6 (C2, C6), 106.9 (C4), 66.3 (COOCH₂), 31.9 (COO(CH₂)₈CH₂), 29.7 (COO(CH₂)₃CH₂), 29.7 (COO(CH₂)₄CH₂), 29.6 (COO(CH₂)₅CH₂), 29.4 (COO(CH₂)₆CH₂), 29.4 (COO(CH₂)₇CH₂), 28.6 (COOCH₂CH₂), 26.1 (COO(CH₂)₂CH₂), 22.7 (COO(CH₂)₉CH₂), 14.1 (CH₃). FTIR (ATR-diamond) ν_{max} : 3465, 3308, 2919, 2849, 1698, 1684, 1600 cm⁻¹. HRMS (FAB+) m/z 309.2054 (MH⁺, calc for C₁₈H₂₈O₄ : 309.2060).

Anal. Found : C, 70.13 ; H, 9.38. Calcd for C₁₈H₂₈O₄ : C, 70.10 ; H, 9.15.

Dodecyl 3,5-dihydroxybenzoate (A12): same protocol as A5, but the the crude was purified by recrystallization in cyclohexane to afford A12 as a white solid (yield 73 %). M. p. 61.3°C. ^1H NMR (400 MHz, CDCl_3) : δ [ppm] 7.12 (d, 2 H, J = 2.3 Hz, C2-H, C6-H), 6.57 (t, 1 H, J = 2.3 Hz, C4-H), 5.33 (s large, 2 H, OH), 4.29 (t, 2 H, J = 6.7 Hz, COOCH_2), 1.72 (m, 2 H, $\text{COOCH}_2\text{CH}_2$), 1.38 (m, 18 H, $(\text{CH}_2)_9\text{CH}_3$), 0.88 (t, 3 H, J = 6.8 Hz, CH₃). ^{13}C NMR (100 MHz, CDCl_3) : δ [ppm] 167.8 (COO), 156.8 (C3,C5), 131.9 (C1), 108.9 (C2, C6), 107.5 (C4), 66.2 (COOCH_2), 31.9 ($\text{COO}(\text{CH}_2)_9\text{CH}_2$), 29.7 (COO(CH₂)₃CH₂), 29.7 (COO(CH₂)₄CH₂), 29.6 (COO(CH₂)₅CH₂), 29.4 (COO(CH₂)₆CH₂), 29.4 (COO(CH₂)₇CH₂), 29.3 (COO(CH₂)₈CH₂), 28.6 (COOCH₂CH₂), 26.0 (COO(CH₂)₂CH₂), 22.7 (COO(CH₂)₁₀CH₂), 14.1 (CH₃). FTIR (KBr) ν_{max} : 3379, 2955, 2918, 2850, 1722, 1692, 1681, 1604 cm⁻¹. MS (FAB⁺) m/z 323.2 (MH⁺, calcd $\text{C}_{19}\text{H}_{30}\text{O}_4$: 322.2). Anal. Found : C, 70.88 ; H, 9.51. Calcd for $\text{C}_{19}\text{H}_{30}\text{O}_4$: C, 70.77 ; H 9.38.

Tridecyl 3,5-dihydroxybenzoate (A13): same protocol as A5, but the the crude was purified by chromatography iPOH/ CH_2Cl_2 : 3/97 as eluent to yield 13 as a white solid (yield 75 %). M. p. 70.8 °C. ^1H NMR (400 MHz, CDCl_3) : δ [ppm] 7.12 (d, 2 H, J = 2.3 Hz, C2-H, C6-H), 6.59 (t, 1 H, J = 2.0 Hz, C4-H), 5.62 (s large, 2 H, OH), 4.29 (t, 2 H, J = 6.6 Hz, COOCH_2), 1.74 (m, 2 H, $\text{COOCH}_2\text{CH}_2$), 1.38 (m, 20 H, $(\text{CH}_2)_{10}\text{CH}_3$), 0.88 (t, 3 H, J = 6.8 Hz, CH₃). ^{13}C NMR (100 MHz, CDCl_3) : δ [ppm] 167.8 (COO), 157.0 (C3,C5), 131.9 (C1), 109.0 (C2, C6), 107.7 (C4), 66.1 (COOCH_2), 31.9 (COO(CH₂)₁₀CH₂), 29.7 (COO(CH₂)₃CH₂), 29.7 (COO(CH₂)₄CH₂), 29.7 (COO(CH₂)₅CH₂), 29.7 (COO(CH₂)₆CH₂), 29.6 (COO(CH₂)₇CH₂), 29.4 (COO(CH₂)₈CH₂), 29.3 (COO(CH₂)₉CH₂), 28.7 (COOCH₂CH₂), 26.0 (COO(CH₂)₂CH₂), 22.7 (COO(CH₂)₁₁CH₂), 14.1 (CH₃). FTIR (KBr) ν_{max} : 3379, 2955, 2918, 2850, 1722, 1692, 1681, 1604 cm⁻¹. HRMS (FAB⁺) m/z 337.2376 (MH⁺, calcd for $\text{C}_{20}\text{H}_{32}\text{O}_4$: 337.2373). Anal. Found C, 71.77 ; H, 9.80. Calcd for $\text{C}_{20}\text{H}_{32}\text{O}_4$: C, 71.39 ; H, 9.59.

Tetradecyl 3,5-dihydroxybenzoate (A14): same protocol as A5, but the the crude was purified by chromatography with EtOAc/C₆H₁₂ : 20/80 as eluent to yield 13 as a white solid (yield 39 %). M. p. 70.3-70.8 °C. ^1H NMR (400 MHz, CDCl_3) : δ [ppm] 7.12 (d, 2 H, J = 2.1 Hz, 2H, C2-H, C6-H), 6.58 (t, 1 H, J = 2.3 Hz, C4-H), 5.30 (s, 2H, OH), 4.29 (t, 2H, J = 6.7 Hz, COOCH_2), 1.72 (m, 2H, $\text{COOCH}_2\text{CH}_2$), 1.38 (m, 22H, CH₂), 0.88 (t, 3H, J = 6.7 Hz, CH₃). ^{13}C NMR (100 MHz, CDCl_3) : δ [ppm] 167.5 (COO), 156.9 (C3, C5), 132.1 (C1), 108.9 (C2, C6), 107.6 (C4), 66.1 (OCH₂), 31.9, 29.7, 29.6 29.5, 29.4, 29.3, 28.6, 26.1 (COOCH₂CH₂CH₂), 22.7 (CH₂CH₃), 14.1 (CH₃). HRMS (FAB⁺) m/z 351.25404 (MH⁺, calcd for $\text{C}_{21}\text{H}_{34}\text{O}_4$: 351.25354).

Pentadecyl 3,5-dihydroxybenzoate (A15): same protocol as A5, but the the crude was purified by chromatography (CH₃OH/ CH_2Cl_2 : 1/99 to 5/95 gradient eluent) to yield 15 as a white solid (yield 83.2 %). M. p. 88.2°C. ^1H NMR (400 MHz, (CD₃)₂CO) : δ [ppm] 7.70 (s, 2 H, O-H), 6.19 (d, 2 H, J = 2.2 Hz, C2-H, C6-H), 5.75 (t, 1 H, J = 2.3 Hz, C4-H), 3.42 (t, 2 H, J = 6.58 Hz, COOCH₂), 1.22 (m, 2 H, $\text{COOCH}_2\text{CH}_2$), 0.91 (m, 2 H, $\text{COOCH}_2\text{CH}_2\text{CH}_2$), 0.45 (m, 22 H, $\text{COOCH}_2\text{CH}_2$ (CH₂)₁₁ CH₃), 0.05 (t, 3 H, J = 6.74 Hz, CH₃); ^{13}C NMR (100 MHz, (CD₃)₂CO) : δ [ppm] 166.0 (COO), 158.8 (C3,C5), 132.9 (C1), 108.0 (C2, C6), 107.8 (C4), 64.7 (COOCH₂), 32.0 (CH₃CH₂CH₂-), 30.4 (COOCH₂CH₂), 29.7-28.8 (COOCH₂CH₂CH₂ (CH₂)₁₀-), 26.1 (COO(CH₂)₂CH₂), 22.7 (-CH₂-CH₃), 13.7 (CH₃); HRMS (FAB⁺) m/z 371.2750 (MLi⁺, calcd for $\text{C}_{22}\text{H}_{36}\text{O}_4$: 371.2769). Anal. Found : C, 72.31; H, 10.07; O, 17.82; calcd for $\text{C}_{22}\text{H}_{36}\text{O}_4$: C, 72.49; H, 9.95; O, 17.56.

Hexadecyl 3,5-dihydroxybenzoate (A16): same protocol as A5 but the crude was prurified by recrystallization in cyclohexane to afford A16 as a white solid (yield 64 %). M. p. 81.2°C. ^1H NMR (400 MHz, CDCl_3) : δ [ppm] 7.12 (d, 2 H, J = 2.2 Hz, C2-H, C6-H), 6.59 (t, 1 H, J = 2.0 Hz, C4-H), 5.32 (s large, 2 H, OH), 4.29 (t, 2 H, J = 6.6 Hz, COOCH_2), 1.74 (m, 2 H, $\text{COOCH}_2\text{CH}_2$), 1.36 (m, 26 H, $(\text{CH}_2)_{13}\text{CH}_3$), 0.89 (t, 3 H, J = 6.8 Hz, CH₃). ^{13}C NMR (100 MHz, CDCl_3) : δ [ppm] 167.0 (COO), 157.0 (C3,C5), 132.3 (C1), 109.1 (C2, C6), 107.6 (C4), 65.8 (COOCH₂), 31.9 (COO(CH₂)₁₃CH₂), 29.7 (COO(CH₂)₃CH₂), 29.7 (COO(CH₂)₄CH₂), 29.7 (COO(CH₂)₅CH₂), 29.7 (COO(CH₂)₆CH₂), 29.6 (COO(CH₂)₇CH₂), 29.4 (COO(CH₂)₈CH₂), 29.3 (COO(CH₂)₉CH₂), 29.3 (COO(CH₂)₁₀CH₂), 29.3 (COO(CH₂)₁₁CH₂), 29.3 (COO(CH₂)₁₂CH₂), 28.6 (COOCH₂CH₂), 26.0 (COO(CH₂)₂CH₂), 22.7 (COO(CH₂)₁₄CH₂), 14.1 (CH₃). FTIR (KBr) ν_{max} : 3378, 2955, 2917, 2850, 1719, 1697, 1675, 1594 cm⁻¹. HRMS (FAB⁺) m/z 401.2669 (MNa⁺, calcd for $\text{C}_{23}\text{H}_{38}\text{O}_4$: 401.2662).

Heptyl 3,5-bis-(5-hexylcarbamoyl-pentoxy)-benzoate (BHPB-7): same protocol as BHPB5. White solid (yield 67 %). M. p. : 82.1°C. ^1H NMR (400 MHz, CDCl_3) : δ [ppm] 7.14 (d, 2 H, J = 2.3 Hz, C2-H, C6-H), 6.60 (t, 1 H, J = 2.3 Hz, C4-H), 5.40 (s broad, 2 H, NH), 4.28 (t, 2 H, J = 6.8 Hz, COOCH_2), 3.97 (t, 4 H, J = 6.3 Hz, ArOCH₂), 3.24 (q, 4 H, J = 6.6 Hz, CH₂NHCO), 2.19 (t, 4 H, J = 7.3 Hz, CH₂CONH), 1.76 (m, 10 H, $\text{COOCH}_2\text{CH}_2$, ArOCH₂CH₂, ArOCH₂CH₂CH₂), 1.55-1.29 (m, 28 H, CH₂), 0.88 (t, 9 H, J = 6.6 Hz, CH₃); ^{13}C NMR (100 MHz, CDCl_3) : δ [ppm] 172.7 (CONH), 166.5 (COO), 160.0 (C3,C5), 132.3 (C1), 107.7 (C2, C6), 106.3 (C4), 68.0 (ArOCH₂), 65.3 (COOCH₂), 39.5 (CH₂NH), 36.6 (CH₂CONH), 31.7 (COO(CH₂)₄CH₂), 31.5 (CONH(CH₂)₃CH₂), 29.6 (CONHCH₂CH₂), 28.9 (COO(CH₂)₃CH₂), 28.9 (ArOCH₂CH₂), 28.7 (COOCH₂CH₂), 26.6 (CONH(CH₂)₂CH₂), 26.0 (COO(CH₂)₂CH₂), 25.8 (CH₂CH₂CONH), 25.5 (CH₂(CH₂)₂CONH), 22.6 (COO(CH₂)₅CH₂), 22.5 (CONH(CH₂)₄CH₂), 14.1 (COO(CH₂)₆CH₃), 14.0 (CONH(CH₂)₅CH₃); FTIR (ATR) ν_{max} : 3303, 2930, 2858, 1723, 1642, 1600, 1554, 1465, 1348, 130, 1234, 1176 cm⁻¹. HRMS (FAB⁺) m/z 647.4975 (MH⁺, calcd for C₃₈H₆₆N₂O₆ : 647.4994). Anal. Found : C, 70.61 ; H, 10.47 ; N, 4.38. Calcd for C₃₈H₆₆N₂O₆ : C, 70.55 ; H, 10.28 ; N, 4.33.

3,5-bis-(5-hexylcarbamoyl-pentoxo)-benzoic acid octyl ester (BHPB-8) : same protocol as BHPB5; white solid (yield 77 %). M. p. 79.6°C. ^1H NMR (400 MHz, CDCl_3) : δ [ppm] 7.14 (d, 2 H, $J = 2.3$ Hz, C2-H, C6-H), 6.60 (t, 1 H, $J = 2.3$ Hz, C4-H), 5.42 (s broad, 2 H, NH), 4.28 (t, 2 H, $J = 6.5$ Hz, COOCH_2), 3.96 (t, 4 H, $J = 6.5$ Hz, ArOCH₂), 3.24 (q, 4 H, $J = 6.2$ Hz, CH_2NHCO), 2.19 (t, 4 H, $J = 7.4$ Hz, CH_2CONH), 1.76 (m, 10 H, $\text{COOCH}_2\text{CH}_2$, ArOCH₂CH₂, ArOCH₂CH₂CH₂), 1.56-1.29 (m, 30 H, CH₂), 0.88 (t, 9 H, $J = 6.6$ Hz, CH₃); ^{13}C NMR (100 MHz, CDCl_3) : δ [ppm] 172.9 (CONH), 166.5 (COO), 160.0 (C3,C5), 132.2 (C1), 107.6 (C2, C6), 106.2 (C4), 67.9 (ArOCH₂), 65.3 (COOCH₂), 39.5 (CH₂NH), 36.6 (CH₂CONH), 31.7 (COO(CH₂)₅CH₂), 31.4 (CONH(CH₂)₃CH₂), 29.5 (CONHCH₂CH₂), 29.2 (COO(CH₂)₃CH₂), 29.1 (COO(CH₂)₄CH₂), 28.8 (ArOCH₂CH₂), 28.6 (COOCH₂CH₂), 26.5 (CONH(CH₂)₂CH₂), 25.9 (COO(CH₂)₂CH₂), 25.7 (CH₂CH₂CONH), 25.4 (CH₂(CH₂)₂CONH), 22.6 (COO(CH₂)₆CH₂), 22.5 (CONH(CH₂)₄CH₂), 14.1 (COO(CH₂)₇CH₃), 14.0 (CONH(CH₂)₅CH₃); IR (solid, KBr) ν_{max} : 3302, 2929, 2857, 1720, 1642, 1599, 1553.1, 1465.0, 1348, 1300, 1230, 1176 cm⁻¹; MS (FAB+) found m/z 661.4 (MH⁺, calcd for C₃₉H₆₈N₂O₆ : 661.5).

Anal. Found : C, 70.84 ; H 10.58 ; N, 4.13. Calcd for C₃₉H₆₈N₂O₆ : C, 70.87 ; H 10.37 ; N, 4.24.

3,5-bis-(5-hexylcarbamoyl-pentoxo)-benzoic acid nonyl ester (BHPB-9) : same protocol as BHPB5; white solid (yield 84 %). M. p. 87.9°C; ^1H NMR (400 MHz, CDCl_3) : δ [ppm] 7.14 (d, 2 H, $J = 2.2$ Hz, C2-H, C6-H), 6.60 (t, 1 H, $J = 2.2$ Hz, C4-H), 5.47 (s broad, 2 H, NH), 4.28 (t, 2 H, $J = 6.7$ Hz, COOCH_2), 3.96 (t, 4 H, $J = 6.4$ Hz, ArOCH₂), 3.23 (q, 4 H, $J = 6.7$ Hz, CH_2NHCO), 2.19 (t, 4 H, $J = 7.5$ Hz, CH_2CONH), 1.76 (m, 10 H, $\text{COOCH}_2\text{CH}_2$, ArOCH₂CH₂, ArOCH₂CH₂CH₂), 1.56-1.29 (m, 32 H, CH₂), 0.87 (t, 9 H, $J = 6.7$ Hz, CH₃); ^{13}C NMR (100 MHz, CDCl_3) : δ [ppm] 172.7 (CONH), 166.5 (COO), 160.0 (C3,C5), 132.3 (C1), 107.7 (C2, C6), 106.2 (C4), 68.0 (ArOCH₂), 65.3 (COOCH₂), 39.5 (CH₂NH), 36.7 (CH₂CONH), 31.9 (COO(CH₂)₆CH₂), 31.5 (CONH(CH₂)₃CH₂), 29.7 (CONHCH₂CH₂), 29.5 (COO(CH₂)₅CH₂), 29.3 (COO(CH₂)₃CH₂), 29.2 (COO(CH₂)₄CH₂), 28.8 (ArOCH₂CH₂), 28.6 (COOCH₂CH₂), 26.6 (CONH(CH₂)₂CH₂), 25.9 (COO(CH₂)₂CH₂), 25.7 (CH₂CH₂CONH), 25.4 (CH₂(CH₂)₂CONH), 22.6 (COO(CH₂)₇CH₂), 22.5 (CONH(CH₂)₄CH₂), 14.1 (COO(CH₂)₈CH₃), 14.0 (CONH(CH₂)₅CH₃); ATR-IR (solid) ν_{max} : 3299, 3087, 2855, 1716, 1641, 1598, 1547, 1464, 1346, 1299, 1232, 1172 cm⁻¹. HRMS (FAB+) m/z 675.5283 (MH⁺, calcd for C₄₀H₇₀N₂O₆ : 675.5307).

Anal. Found : C, 71.40 ; H 10.76 ; N, 4.16. Calcd for C₄₀H₇₀N₂O₆ : C, 71.18 ; H 10.45 ; N, 4.15.

3,5-bis-(5-hexylcarbamoyl-pentoxo)-benzoate acid undecyl ester (BHPB11) : same protocol as BHPB5; white solid (yield 86 %). M. p. 94.8°C. ^1H NMR (400 MHz, CDCl_3) : δ [ppm] 7.14 (d, 2 H, $J = 2.1$ Hz, C2-H, C6-H), 6.60 (t, 1 H, $J = 2.1$ Hz, C4-H), 5.42 (s broad, 2 H, NH), 4.28 (t, 2 H, $J = 6.8$ Hz, COOCH_2), 3.97 (t, 4 H, $J = 6.3$ Hz, ArOCH₂), 3.24 (q, 4 H, $J = 6.7$ Hz, CH_2NHCO), 2.19 (t, 4 H, $J = 7.1$ Hz, CH_2CONH), 1.76 (m, 10 H, $\text{COOCH}_2\text{CH}_2$, ArOCH₂CH₂, ArOCH₂CH₂CH₂), 1.53-1.26 (m, 36 H, CH₂), 0.87 (t, 9 H, $J = 6.1$ Hz, CH₃); ^{13}C NMR (100 MHz, CDCl_3) : δ [ppm] 172.7 (CONH), 166.5 (COO), 160.0 (C3,C5), 132.3 (C1), 107.7 (C2, C6), 106.2 (C4), 68.0 (ArOCH₂), 65.3 (COOCH₂), 39.5 (CH₂NH), 36.7 (CH₂CONH), 31.9 (COO(CH₂)₈CH₂), 31.5 (CONH(CH₂)₃CH₂), 31.5 (CONHCH₂CH₂), 29.6 (COO(CH₂)₃CH₂), 29.6 (COO(CH₂)₄CH₂), 29.5 (COO(CH₂)₅CH₂), 29.3 (ArOCH₂CH₂), 29.3 (COO(CH₂)₆CH₂), 28.9 (COO(CH₂)₇CH₂), 28.7 (COOCH₂CH₂), 26.6 (CONH(CH₂)₂CH₂), 26.0 (COO(CH₂)₂CH₂), 25.8 (CH₂CH₂CONH), 25.5 (CH₂(CH₂)₂CONH), 22.7 (COO(CH₂)₉CH₂), 22.5 (CONH(CH₂)₄CH₂), 14.1 (COO(CH₂)₁₀CH₃), 14.0 (CONH(CH₂)₅CH₃). ATR-IR (solid) : ν_{max} : 3287, 3093, 2952, 2919, 2852, 1713, 1643, 1600, 1551, 1466, 1446 1321, 1301, 1239, 1166 cm⁻¹. HRMS (FAB+) m/z 703.5595 (MH⁺, calcd for C₄₂H₇₄N₂O₆ : 703.5620). Anal. Found : C, 71.78 ; H, 10.89 ; N, 4.03. Calcd for C₄₂H₇₄N₂O₆ : C, 71.75 ; H, 10.61 ; N, 3.98.

3,5-bis-(5-hexylcarbamoyl-pentoxo)-benzoic acid dodecyl ester (BHPB12) : white solid (yield 79 %). M. p. 90.4°C. ^1H NMR (400 MHz, CDCl_3) : δ [ppm] 7.14 (d, 2 H, $J = 2.3$ Hz, C2-H, C6-H), 6.60 (t, 1 H, $J = 2.3$ Hz, C4-H), 5.44 (s broad, 2 H, NH), 4.28 (t, 2 H, $J = 6.8$ Hz, COOCH_2), 3.97 (t, 4 H, $J = 6.3$ Hz, ArOCH₂), 3.24 (q, 4 H, $J = 6.7$ Hz, CH_2NHCO), 2.19 (t, 4 H, $J = 7.4$ Hz, CH_2CONH), 1.76 (m, 10 H, $\text{COOCH}_2\text{CH}_2$, ArOCH₂CH₂, ArOCH₂CH₂CH₂), 1.53-1.26 (m, 38 H, CH₂), 0.87 (t, 9 H, $J = 6.8$ Hz, CH₃); ^{13}C NMR (100 MHz, CDCl_3) : δ [ppm] 172.8 (CONH), 166.5 (COO), 160.0 (C3,C5), 132.3 (C1), 107.7 (C2, C6), 106.2 (C4), 67.9 (ArOCH₂), 65.3 (COOCH₂), 39.5 (CH₂NH), 36.7 (CH₂CONH), 31.9 (COO(CH₂)₈CH₂), 31.5 (CONH(CH₂)₃CH₂), 29.6 (COO(CH₂)₃CH₂), 29.6 (COO(CH₂)₄CH₂), 29.5 (COO(CH₂)₅CH₂), 29.3 (ArOCH₂CH₂), 29.3 (COO(CH₂)₆CH₂), 28.9 (COO(CH₂)₇CH₂), 28.7 (COOCH₂CH₂), 26.6 (CONH(CH₂)₂CH₂), 26.0 (COO(CH₂)₂CH₂), 25.8 (CH₂CH₂CONH), 25.5 (CH₂(CH₂)₂CONH), 22.7 (COO(CH₂)₁₀CH₂), 22.5 (CONH(CH₂)₄CH₂), 14.1 (COO(CH₂)₁₁CH₃), 14.0 (CONH(CH₂)₅CH₃). ATR-IR (solid) ν_{max} : 3302, 2924, 2852, 1725, 1641, 1600, 1548, 1466, 1347, 1301, 1240, 1175 cm⁻¹. MS (FAB⁺) m/z 717.5 (MH⁺, calcd for C₄₃H₇₆N₂O₆ : 717.6). Anal. Found : C, 72.08 ; H, 10.91 ; N, 3.91. Calcd for C₄₃H₇₆N₂O₆ : C, 72.02 ; H, 10.68 ; N, 3.91.

3,5-bis-(5-hexylcarbamoyl-pentoxo)-benzoic acid tridecyl ester (BHPB13) : white solid (yield 74 %). M. p. 95.4°C. ^1H NMR (400 MHz, CDCl_3) : δ [ppm] 7.14 (d, 2 H, $J = 2.3$ Hz, C2-H, C6-H), 6.60 (t, 1 H, $J = 2.3$ Hz, C4-H), 5.43 (s broad, 2 H, NH), 4.27 (t, 2 H, $J = 6.7$ Hz, COOCH_2), 3.97 (t, 4 H, $J = 6.3$ Hz, ArOCH₂), 3.24 (q, 4 H, $J = 6.7$ Hz, CH_2NHCO), 2.19 (t, 4 H, $J = 7.6$ Hz, CH_2CONH), 1.78 (m, 10 H, $\text{COOCH}_2\text{CH}_2$, ArOCH₂CH₂, ArOCH₂CH₂CH₂), 1.53-1.25 (m, 40 H, CH₂), 0.87 (t, 9 H, $J = 6.7$ Hz, CH₃); ^{13}C NMR (100 MHz, CDCl_3) : δ [ppm] 172.7 (CONH), 166.5 (COO), 160.0 (C3,C5), 132.3 (C1), 107.7 (C2, C6), 106.2 (C4), 68.0 (ArOCH₂), 65.3 (COOCH₂), 39.5 (CH₂NH), 36.7 (CH₂CONH), 31.9 (COO(CH₂)₁₀CH₂), 31.5 (CONH(CH₂)₃CH₂), 31.5 (CONHCH₂CH₂), 29.7 (COO(CH₂)₃CH₂), 29.6 (COO(CH₂)₄CH₂), 29.6 (COO(CH₂)₅CH₂), 29.6 (COO(CH₂)₆CH₂), 29.5 (COO(CH₂)₇CH₂), 29.3 (ArOCH₂CH₂), 29.3 (COO(CH₂)₈CH₂), 28.9 (COO(CH₂)₉CH₂), 28.7 (COOCH₂CH₂), 26.6 (CONH(CH₂)₂CH₂), 26.0 (COO(CH₂)₂CH₂), 25.7 (CH₂CH₂CONH), 25.5 (CH₂(CH₂)₂CONH), 22.7 (COO(CH₂)₁₀CH₂), 22.5 (CONH(CH₂)₄CH₂), 14.1 (COO(CH₂)₁₁CH₃), 14.0 (CONH(CH₂)₅CH₃). ATR-IR (solid) ν_{max} : 3302, 2924, 2852, 1725, 1641, 1600, 1548, 1466, 1347, 1301, 1240, 1175 cm⁻¹. MS (FAB⁺) m/z 717.5 (MH⁺, calcd for C₄₃H₇₆N₂O₆ : 717.6). Anal. Found : C, 72.02 ; H, 10.68 ; N, 3.91.

(COO(CH₂)₉CH₂), 28.7 (COOCH₂CH₂), 26.6 (CONH(CH₂)₂CH₂), 26.0 (COO(CH₂)₂CH₂), 25.7 (CH₂CH₂CONH), 25.5 (CH₂(CH₂)₂CONH), 22.7 (COO(CH₂)₁₁CH₂), 22.5 (CONH(CH₂)₄CH₂), 14.1 (COO(CH₂)₁₂CH₃), 14.0 (CONH(CH₂)₅CH₃). FTIR (ATR-diamond) ν_{max} : 3302, 2922, 2852, 1725, 1641, 1599, 1546, 1467, 1347, 1301, 1240, 1176 cm⁻¹. HRMS (FAB⁺) m/z 731.5911 (MH⁺, calcd for C₄₄H₇₈N₂O₆ : 731.5933).

Anal. Found : C, 72.00 ; H 10.91 ; N, 3.83. Calcd for C₄₄H₇₈N₂O₆ : C, 72.28 ; H 10.75 ; N, 3.89.

3,5-bis-(5-hexylcarbamoyl-pentoxo)-benzoic acid tetradecyl ester (BHPB14) : same protocol as BHPB5 but the crude is purified by recrystallization in EtOAc to afford BHPB14 as a white solid (yield 43%). M. p. 103.7-103.8°C. ¹H NMR (400 MHz, CDCl₃) : δ [ppm] 7.14 (d, 2H, J = 2.2 Hz, C2-H, C6-H), 6.60 (t, 1H, J = 2.2 Hz, C4-H), 5.45 (s, 2H, NH), 4.28 (t, 2H, J = 6.7 Hz, COOCH₂), 3.97 (t, 4H, J = 6.2 Hz, ArOCH₂), 3.25 (q, 4H, J = 6.7 Hz, CH₂NHCO), 2.19 (t, 4H, J = 7.4 Hz, NHCOCH₂), 1.77 (m, 6H, COOCH₂CH₂, ArOCH₂CH₂), 1.53-1.27 (m, 46, CH₂), 0.88 (t, 9H, J = 6.4 Hz, CH₃) ; ¹³C NMR (100 MHz, CDCl₃) : δ [ppm] 172.7 (CONH), 166.5 (COO), 160.0 (C3, C5), 132.3 (C1), 107.7 (C2, C6), 106.2 (C4), 67.9 (ArOCH₂), 65.3 (COOCH₂), 39.5 (CH₂NH), 36.7 (CH₂CONH), 31.9, 31.5, 31.5, 29.7, 29.7, 29.6, 29.6, 29.6, 29.6, 29.3, 29.3, 28.9, 28.7, 26.6, 26.0, 25.7, 25.5, 22.7, 22.5 (CONHCH₂CH₂), 14.1 (ester CH₃), 14.0 (CH₃) ; FTIR (KBr) ν_{max} 3299, 2918, 2852, 1712, 1640, 1611, 1550, 1446, 1328, 1235, 1166, 762 cm⁻¹. HRMS (FAB⁺) m/z 745.60924 (MH⁺, calcd for C₄₅H₈₀N₂O₄ 745.60946).

Anal. Found : C, 72.13 ; H, 11.07 ; N, 3.71. Calcd for C₄₅H₈₀N₂O₆ : C, 72.54 ; H, 10.82 ; N, 3.76.

3,5-bis-(5-hexylcarbamoyl-pentoxo)-benzoic acid pentadecyl ester (BHPB15) : same protocol as BHPB5 but the crude is purified by recrystallization in CH₃CN to afford BHPB15 as a white solid (yield 95 %). M. p. 95.4°C. ¹H NMR (400 MHz, CDCl₃) : δ [ppm] 7.14 (d, 2H, J = 2.3 Hz, C2-H, C6-H), 6.60 (t, 1H, J = 2.3 Hz, C4-H), 5.43 (s broad, 2H, NH), 4.27 (t, 2H, J = 6.7 Hz, COOCH₂), 3.97 (t, 4H, J = 6.3 Hz, ArOCH₂), 3.24 (q, 4H, J = 6.7 Hz, CH₂NHCO), 2.19 (t, 4H, J = 7.6 Hz, CH₂CONH), 1.78 (m, 10H, COOCH₂CH₂, ArOCH₂CH₂, ArOCH₂CH₂CH₂), 1.53-1.25 (m, 40H, CH₂), 0.87 (t, 9H, J = 6.7 Hz, CH₃) ; ¹³C NMR (100 MHz, CDCl₃) : δ [ppm] 172.7 (CONH), 166.5 (COO), 160.0 (C3,C5), 132.3 (C1), 107.7 (C2, C6), 106.2 (C4), 68.0 (ArOCH₂), 65.3 (COOCH₂), 39.5 (CH₂NH), 36.7 (CH₂CONH), 31.9 (COO(CH₂)₁₀CH₂), 31.5 (CONH(CH₂)₃CH₂), 31.5 (CONHCH₂CH₂), 29.7 (COO(CH₂)₃CH₂), 29.6 (COO(CH₂)₄CH₂), 29.6 (COO(CH₂)₅CH₂), 29.6 (COO(CH₂)₆CH₂), 29.5 (COO(CH₂)₇CH₂), 29.3 (ArOCH₂CH₂), 29.3 (COO(CH₂)₈CH₂), 28.9 (COO(CH₂)₉CH₂), 28.7 (COOCH₂CH₂), 26.6 (CONH(CH₂)₂CH₂), 26.0 (COO(CH₂)₂CH₂), 25.7 (CH₂CH₂CONH), 25.5 (CH₂(CH₂)₂CONH), 22.7 (COO(CH₂)₁₁CH₂), 22.5 (CONH(CH₂)₄CH₂), 14.1 (COO(CH₂)₁₂CH₃), 14.0 (CONH(CH₂)₅CH₃) ; ATR-IR (solid) ν_{max} : 3302, 2922, 2852, 1725, 1641, 1599, 1546, 1467, 1347, 1301, 1240, 1176 cm⁻¹ ; HRMS (FAB⁺) m/z 731.5911 (MH⁺, calcd for C₄₄H₇₈N₂O₆ : 731.5933).

Anal. Found : C, 72.00 ; H, 10.91 ; N, 3.83. Calcd for C₄₄H₇₈N₂O₆ : C, 72.28 ; H, 10.75 ; N, 3.89.

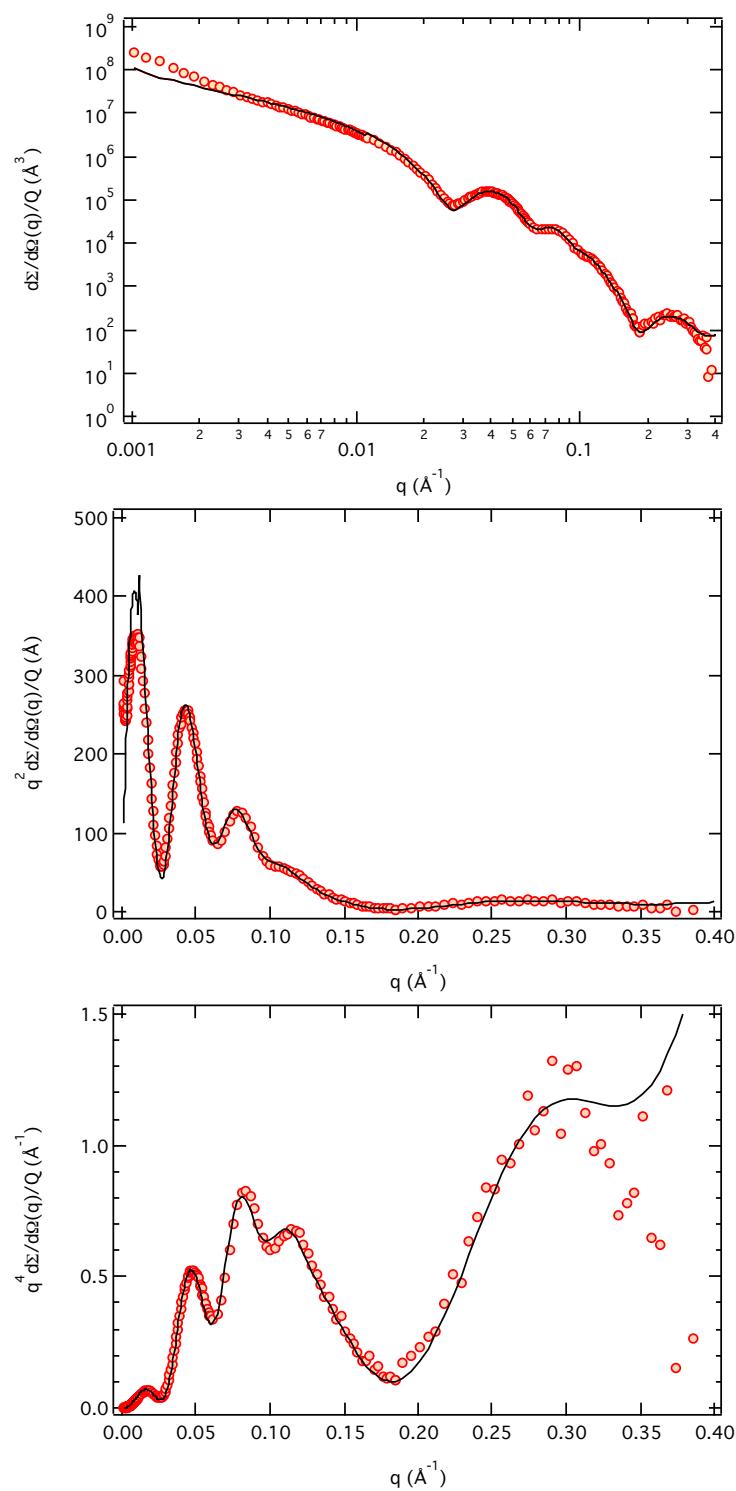
3,5-bis-(5-hexylcarbamoyl-pentoxo)-benzoic acid hexadecyl ester (BHPB16) : white solid (yield 79 %). M. p. 101.6 °C. ¹H NMR (400 MHz, CDCl₃) : δ [ppm] 7.14 (d, 2H, J = 2.3 Hz, C2-H, C6-H), 6.60 (t, 1H, J = 2.3 Hz, C4-H), 5.43 (s broad, 2H, NH), 4.27 (t, 2H, J = 6.7 Hz, COOCH₂), 3.97 (t, 4H, J = 6.3 Hz, ArOCH₂), 3.24 (q, 4H, J = 6.7 Hz, CH₂NHCO), 2.19 (t, 4H, J = 7.6 Hz, CH₂CONH), 1.78 (m, 10H, COOCH₂CH₂, ArOCH₂CH₂, ArOCH₂CH₂CH₂), 1.53-1.25 (m, 46H, CH₂), 0.87 (t, 9H, J = 6.7 Hz, CH₃) ; ¹³C NMR (100 MHz, CDCl₃) : δ [ppm] 172.7 (CONH), 166.5 (COO), 160.0 (C3,C5), 132.3 (C1), 107.7 (C2, C6), 106.2 (C4), 68.0 (ArOCH₂), 65.3 (COOCH₂), 39.5 (CH₂NH), 36.7 (CH₂CONH), 31.9 (COO(CH₂)₁₃CH₂), 31.5 (CONH(CH₂)₃CH₂), 31.5 (CONHCH₂CH₂), 29.7 (COO(CH₂)₃CH₂), 29.6 (COO(CH₂)₄CH₂), 29.6 (COO(CH₂)₅CH₂), 29.6 (COO(CH₂)₆CH₂), 29.5 (COO(CH₂)₇CH₂), 29.3 (ArOCH₂CH₂), 29.3 (COO(CH₂)₈CH₂), 29.2 (COO(CH₂)₉CH₂), 29.2 (COO(CH₂)₁₀CH₂), 29.0 (COO(CH₂)₁₁CH₂), 28.9 (COO(CH₂)₁₂CH₂), 28.7 (COOCH₂CH₂), 26.6 (CONH(CH₂)₂CH₂), 26.0 (COO(CH₂)₂CH₂), 25.7 (CH₂CH₂CONH), 25.5 (CH₂(CH₂)₂CONH), 22.7 (COO(CH₂)₁₄CH₂), 22.5 (CONH(CH₂)₄CH₂), 14.1 (COO(CH₂)₁₅CH₃), 14.0 (CONH(CH₂)₅CH₃) ; FTIR (ATR-diamond) ν_{max} : 3301, 3066, 2955, 2920, 2851, 1717, 1638, 1598, 1541, 1465, 1345, 1300, 1240, 1172 cm⁻¹ ; HRMS (FAB⁺) m/z 773.6395 (MH⁺, calcd for C₄₇H₈₅N₂O₆ : 773.6402).

Anal. Found : C, 72.92 ; H, 10.95 ; N, 3.65. Calcd for C₄₇H₈₄N₂O₆ : C, 73.01 ; H, 10.95 ; N, 3.62.

S2. Best fits performed on BHPB-n in log-log, Kratky and Porod representations

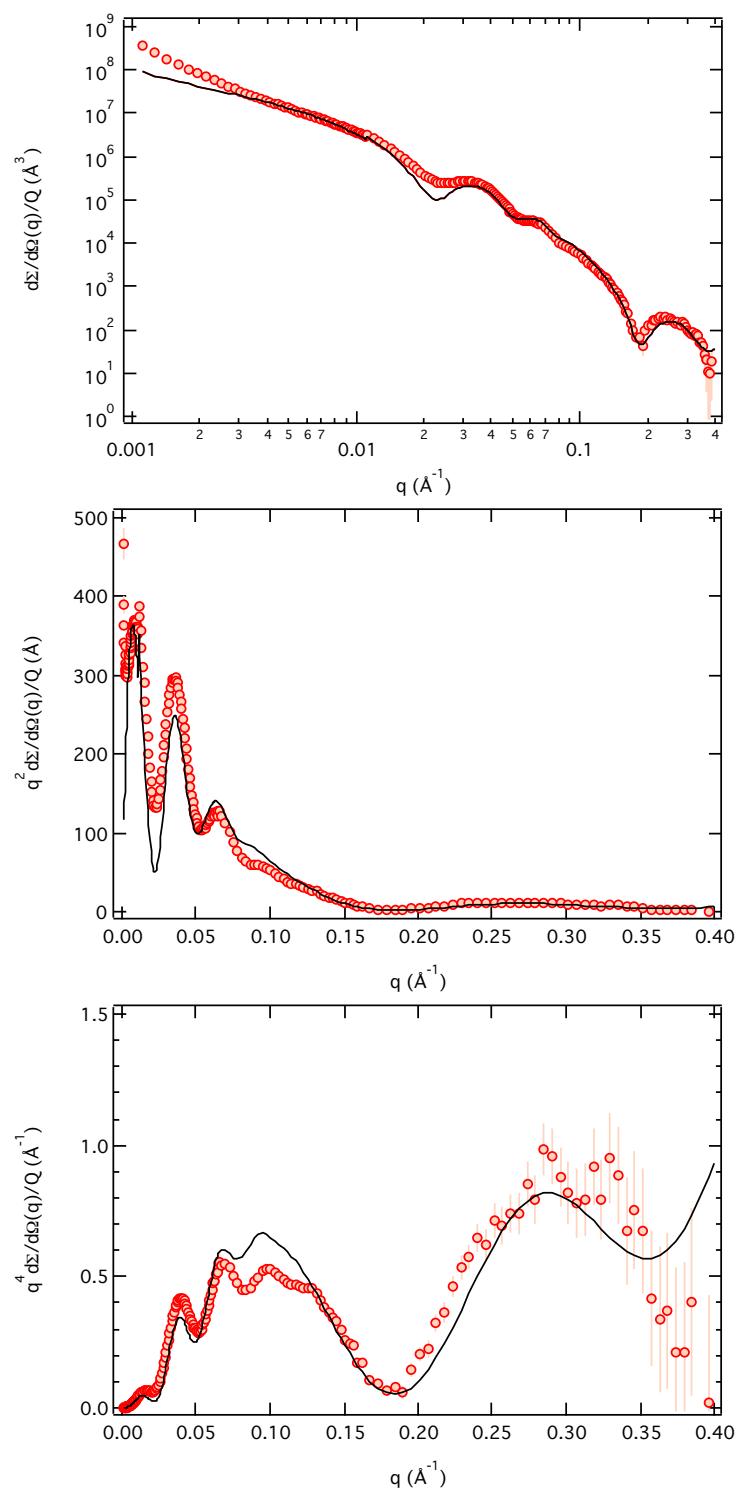
\bar{r}_e is the average value of the Gaussian distribution of the external radius (Å) and Δr_{ext} its standard deviation (Å). e is the thickness of the wall (Å), and $\Delta r_{ext}/\bar{r}_{ext}$ the polydispersity index. f_{Cluster} is the fraction of nanotubes forming clusters. Each sample for which a fraction of $d\Sigma_{\text{BHPB-11}}/d\Omega/Q$ has been removed is indicated with an asterisk *. $f_{\text{BHPB-11}}$ is the corresponding removed fraction. $scale$ is the scale factor and bkg the background (Å³).

BHPB-8



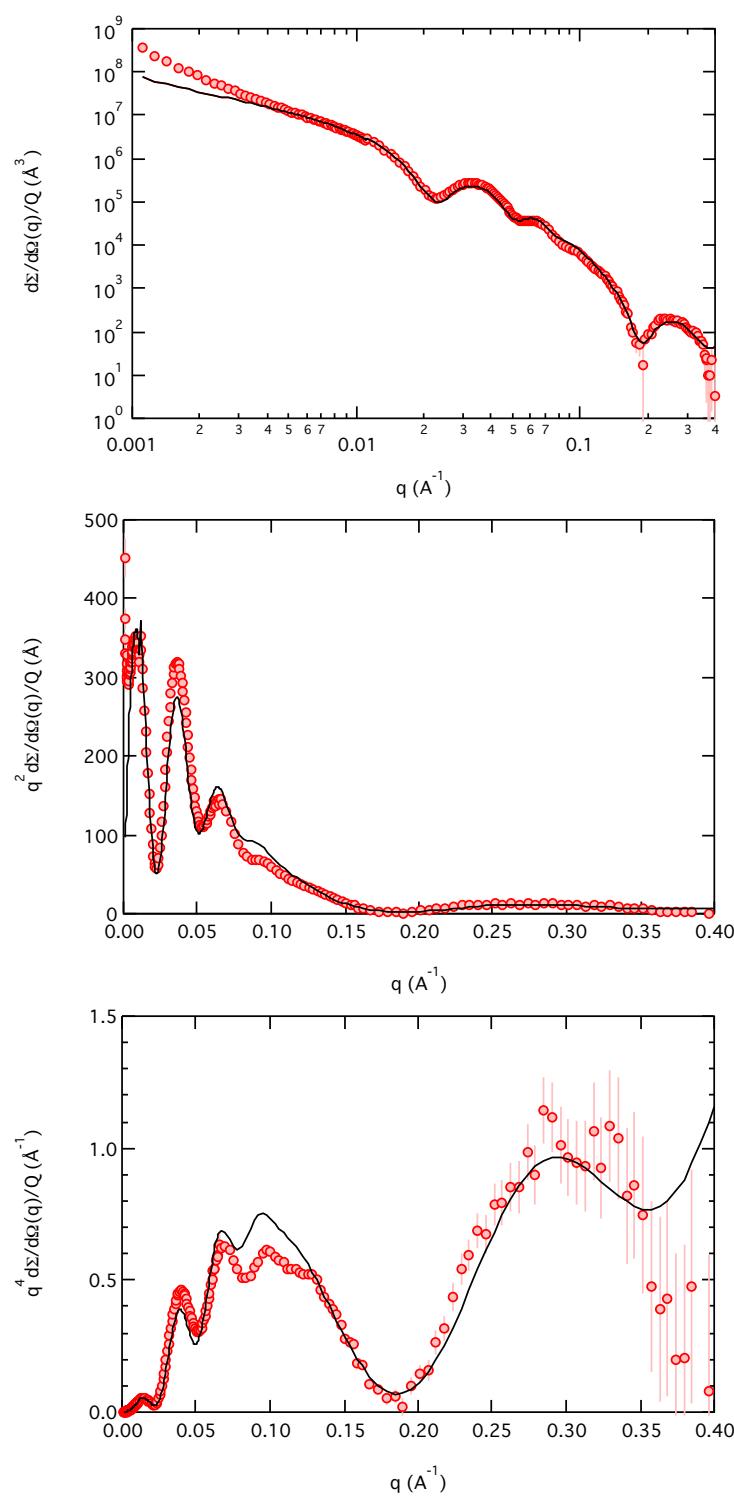
$\overline{r_{ext}} = 105.8 \pm 1 \text{ Å}$, $\Delta r_{ext} = 10.0 \pm 0.5 \text{ Å}$, $e = 34 \pm 1 \text{ Å}$, $\Delta r_{ext}/\overline{r_{ext}} = 0.094$, $f_{Cluster} = 0.4$, $f_{BHPB-II} = 0$, $scale = 0.96$, $bkg = 70 \text{ Å}^3$.

BHPB-9



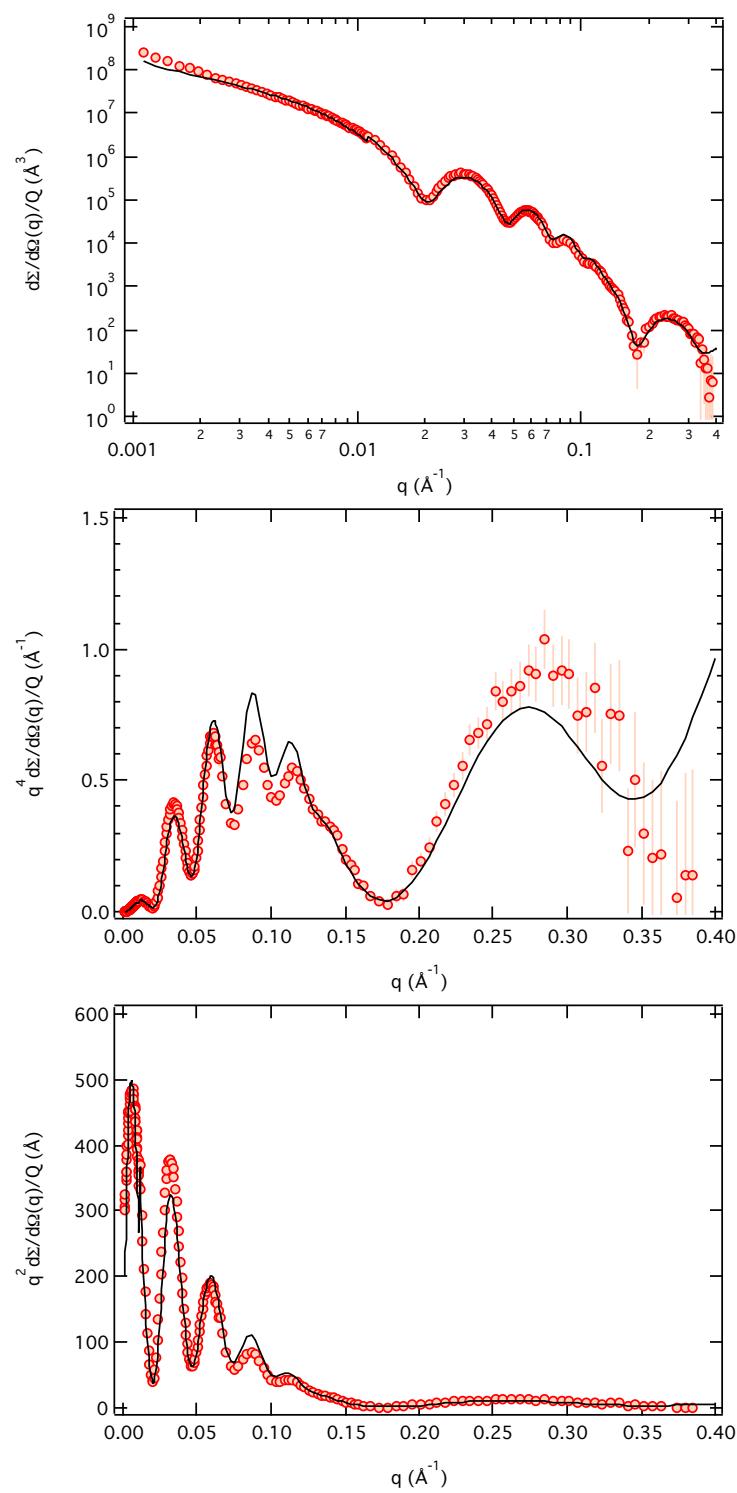
$$\overline{r_{ext}} = 125.0 \pm 1 \text{ Å}, \Delta r_{ext} = 13.5 \pm 0.5 \text{ Å}, e = 34 \pm 1 \text{ Å}, \Delta r_{ext}/\overline{r_{ext}} = 0.108, f_{Cluster} = 0.3, f_{BHPB-II} = 0, scale = 0.88, bkg = 30 \text{ Å}^3.$$

BHPB-9 *



$\overline{r_{ext}} = 125.0 \pm 1 \text{ \AA}$, $\Delta r_{ext} = 12.5 \pm 0.5 \text{ \AA}$, $e = 33.5 \pm 1 \text{ \AA}$, $\Delta r_{ext}/\overline{r_{ext}} = 0.100$, $f_{Cluster} = 0$, $f_{BHPB-II} = 0.19$, $scale = 0.95$, $bkg = 40 \text{ \AA}^3$.

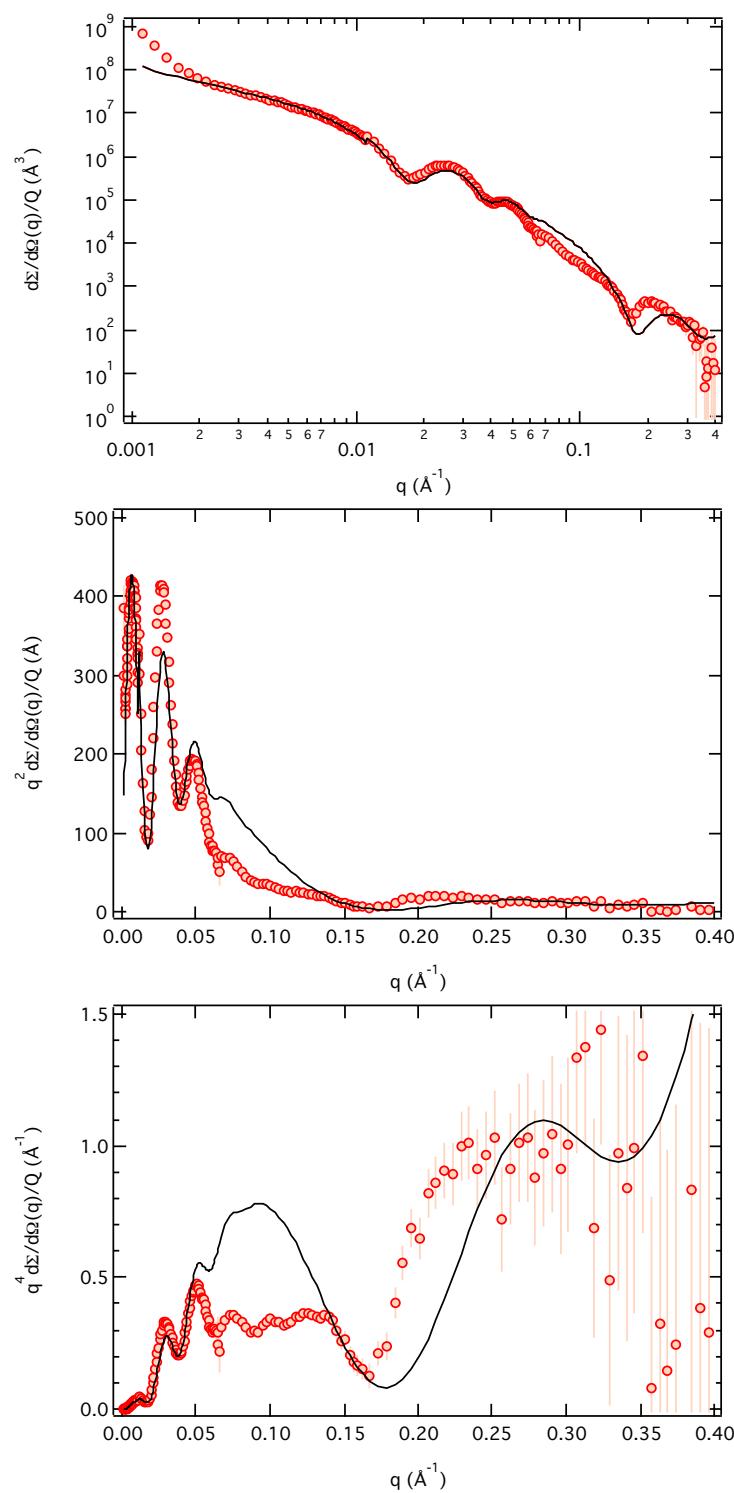
BHPB-10



$\overline{r_{ext}} = 135.4 \pm 1 \text{ \AA}$, $\Delta r_{ext} = 6.8 \pm 0.5 \text{ \AA}$, $e = 35.5 \pm 1 \text{ \AA}$, $\Delta r_{ext}/\overline{r_{ext}} = 0.050$, $f_{Cluster} = 0.8$, $f_{BHPB-11} = 0$, $scale = 0.96$, $bkg = 25$

\AA^3 .

BHP-12 *



$\overline{r_{ext}} = 160.3 \pm 2 \text{ Å}$, $\Delta r_{ext} = 16.3 \pm 1 \text{ Å}$, $e = 35 \pm 2 \text{ Å}$, $\Delta r_{ext}/\overline{r_{ext}} = 0.101$, $f_{Cluster} = 0$, $f_{BHPB-II} = 0.64$, $scale = 1.06$, $bkg = 60 \text{ Å}^3$.