Supporting Information

Preparation and Temperature-controlled Morphology of Helical Microrods Composed of Supramolecular α-Cyclodextrin Assemblies

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1. Rheological properties of organogels formed by mixing a HFIP solution of α -CD and 2-pentanol



Figure S1. Plots of the storage modulus (G') and the loss modulus (G'') of organogels, which were formed by stirring a mixture of an α -CD/HFIP solution and 2-pentanol for 3 h, followed by allowing to stand for 72 h, against angular frequency (stress: 10 Pa).

2. 2-Pentanol/HFIP molar ratio in the α-CD/HFIP/2-pentanol gel and the solution (supernatant) produced from the gel after about 10 months of storage



Figure S2. Molar ratio of 2-pentanol to HFIP (a) in the gel formed by stirring a mixture of α -CD/HFIP solution (0.5 mL) [25 mM] and 2-pentanol (2.5 mL) for 3 h, followed by standing for 72 h, and (b) in the solution (supernatant) produced from the gel after standing for about 10 months.



Figure S3. ¹H NMR spectrum (solvent: DMSO- d_6) of the gel formed by stirring a mixture of α -CD/HFIP solution (0.5 mL) [25 mM] and 2-pentanol (2.5 mL) for 3 h, followed by standing for 72 h.



Figure S4. ¹H NMR spectrum (solvent: DMSO- d_6) of the solution (supernatant) produced from the α -CD/HFIP/2-pentanol gel after standing for about 10 months.

3. SEM images of precipitates obtained by allowing the α -CD/HFIP/2-pentanol gel to stand under high humidity conditions



Figure S5. SEM images of precipitates obtained by allowing the α -CD/HFIP/2-pentanol gel [an α -CD/HFIP solution [25 mM]:2-pentanol = 1:5 (v/v)] to stand at 20 °C under high humidity conditions for (a) 6 h, (b) 12 h, (c) 36 h, (d) 48 h and (e) 120 h.



Figure S6. SEM images of precipitates obtained by allowing the α -CD/HFIP/2-pentanol gel [an α -CD/HFIP solution [25 mM]:2-pentanol = 1:5 (v/v)] to stand at 30 °C under high humidity conditions for (a) 6 h, (b) 12 h, (c) 36 h, (d) 48 h and (e) 120 h.



Figure S7. SEM images of precipitates obtained by allowing the α -CD/HFIP/2-pentanol gel [an α -CD/HFIP solution [25 mM]:2-pentanol = 1:5 (v/v)] to stand at 40 °C under high humidity conditions for (a) 6 h, (b) 12 h, (c) 36 h, (d) 48 h and (e) 120 h.



Figure S8. SEM images of precipitates obtained by allowing the α -CD/HFIP/2-pentanol gel [an α -CD/HFIP solution [25 mM]:2-pentanol = 1:5 (v/v)] to stand at 50 °C under high humidity conditions for (a) 6 h, (b) 12 h, (c) 36 h, (d) 48 h and (e) 120 h.



Figure S9. SEM images of precipitates obtained by allowing the α -CD/HFIP/2-pentanol gel [an α -CD/HFIP solution [25 mM]:2-pentanol = 1:5 (v/v)] to stand at 60 °C under high humidity conditions for (a) 6 h, (b) 12 h, (c) 36 h, (d) 48 h and (e) 120 h.

4. XRD patterns of precipitates obtained by allowing the α -CD/HFIP/2-pentanol gel to stand under high humidity conditions



Figure S10. XRD patterns of precipitates obtained by allowing the α -CD/HFIP/2-pentanol gel [an α -CD/HFIP solution [25 mM]:2-pentanol = 1:5 (v/v)] to stand at 20 °C under high humidity conditions for (a) 6 h, (b) 12 h, (c) 36 h, (d) 48 h and (e) 120 h.



Figure S11. XRD patterns of precipitates obtained by allowing the α -CD/HFIP/2-pentanol gel [an α -CD/HFIP solution [25 mM]:2-pentanol = 1:5 (v/v)] to stand at 30 °C under high humidity conditions for (a) 6 h, (b) 12 h, (c) 36 h, (d) 48 h and (e) 120 h.



Figure S12. XRD patterns of precipitates obtained by allowing the α -CD/HFIP/2-pentanol gel [an α -CD/HFIP solution [25 mM]:2-pentanol = 1:5 (v/v)] to stand at 40 °C under high humidity conditions for (a) 6 h, (b) 12 h, (c) 36 h, (d) 48 h and (e) 120 h.



Figure S13. XRD patterns of precipitates obtained by allowing the α -CD/HFIP/2-pentanol gel [an α -CD/HFIP solution [25 mM]:2-pentanol = 1:5 (v/v)] to stand at 50 °C under high humidity conditions for (a) 6 h, (b) 12 h, (c) 36 h, (d) 48 h and (e) 120 h.



Figure S14. XRD patterns of precipitates obtained by allowing the α -CD/HFIP/2-pentanol gel [an α -CD/HFIP solution [25 mM]:2-pentanol = 1:5 (v/v)] to stand at 60 °C under high humidity conditions for (a) 6 h, (b) 12 h, (c) 36 h, (d) 48 h and (e) 120 h.

5. Molar ratios of H_2O and 2-pentanol to α -CD contained in microrods



Figure S15. ¹H NMR spectrum (solvent: DMSO- d_6) of microrods obtained by allowing the α -CD/HFIP/2-pentanol gel to stand for 120 h at 20 °C under high humidity conditions.



Figure S16. ¹H NMR spectrum (solvent: DMSO- d_6) of microrods obtained by allowing the α -CD/HFIP/2-pentanol gel to stand for 120 h at 30 °C under high humidity conditions.



Figure S17. ¹H NMR spectrum (solvent: DMSO- d_6) of microrods obtained by allowing the α -CD/HFIP/2-pentanol gel to stand for 120 h at 40 °C under high humidity conditions.



Figure S18. ¹H NMR spectrum (solvent: DMSO- d_6) of microrods obtained by allowing the α -CD/HFIP/2-pentanol gel to stand for 120 h at 50 °C under high humidity conditions.



Figure S19. ¹H NMR spectrum (solvent: DMSO- d_6) of microrods obtained by allowing the α -CD/HFIP/2-pentanol gel to stand for 120 h at 60 °C under high humidity conditions.



Figure S20. ¹H NMR spectrum (solvent: DMSO- d_6) of the solid obtained after drying the α -CD/HFIP/2-pentanol gel for 24 h *in vacuo*.