Electronic Supplementary Material (ESI) for Chemical Communications.

Supporting Information

Supramolecular Organogel Formation through Three-dimensional α-Cyclodextrin Nanostructures: Solvent Chirality-selective Organogel Formation

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Contents

1. Experimental procedures

SEM measurements were performed with a JSM-6701F instrument (JEOL Ltd., Japan). X-ray diffraction patterns of powder samples were obtained at room temperature on a Rigaku RINT InPlane/ultraX18SAXS-IP diffractometer using monochromatic Cu-Kα radiation generated at 40 kV, 200 mA. The scan rate was $2\theta = 1°$ min^{-1} between $2\theta = 5°$ and 40°.

2. Preparation procedure of organogel

α-CD (12 mg), which was dried at 80 °C for 12 h *in vacuo* before use, was dissolved in HFIP (0.50 mL) to prepare an α-CD/HFIP solution (24 mg/mL). This solution was added dropwise to a poor solvent (2.5 mL) stirred at 500 rpm using a syringe, and the mixture was stirred for 3 h and allowed to stand for 3 days. The organogel formation was confirmed by rheology measurements.

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

The oscillatory shear measurements were carried out using a stress-controlled rheometer (HAAKE Rheostress RS 1) with a parallel plate-type geometry (plate diameter 20 mm, plate height 1 mm). The storage modulus G' and the loss modulus G'' were measured at a stress of 1 Pa as a function of the angular frequency from 0.1 to 3 rad s^{-1} at 20 °C.

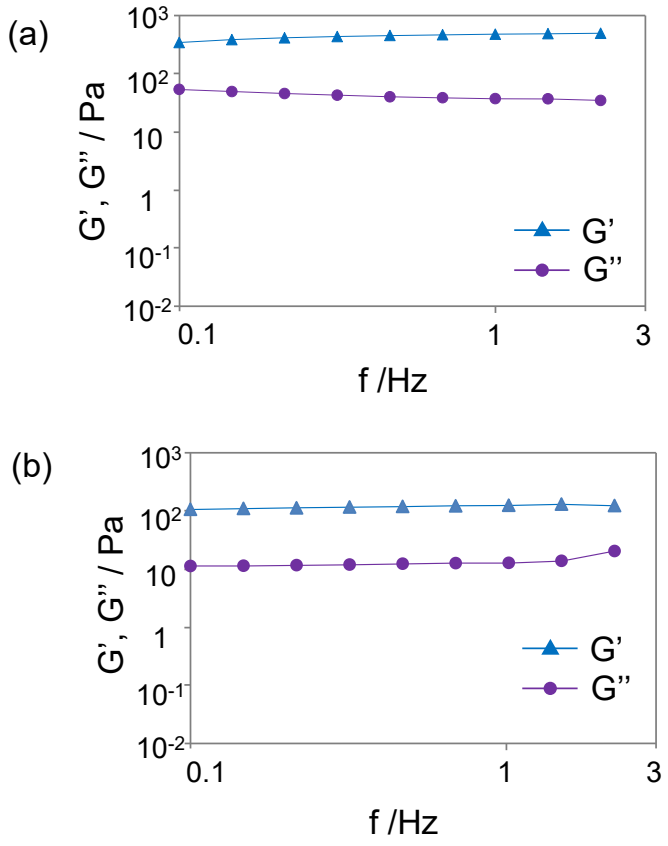

Figure S1. Plots of the storage modulus (G') and the loss modulus (G'') of organogels, which were formed by stirring a mixture of an α-CD/HFIP solution and a poor solvent for 3 h and then allowing to stand for 72 h, against angular frequency (stress: 1 Pa). Poor solvent: (a) 1-butanol and (b) 2-butanol.

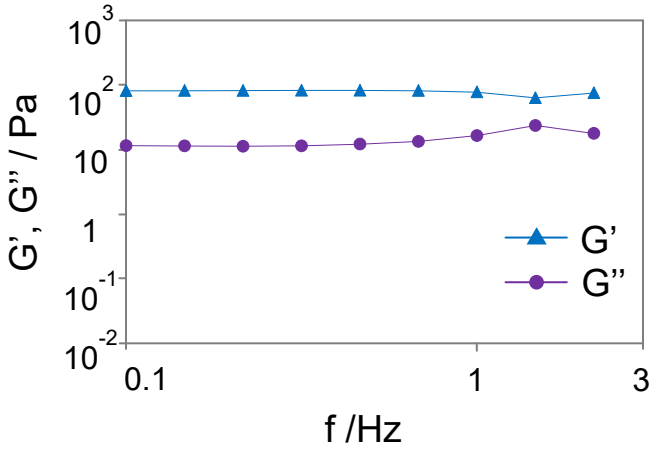

Figure S2. Plots of the storage modulus (G') and the loss modulus (G'') of the organogel, which was formed by stirring a mixture of an α-CD/HFIP solution and (*S*)-2-butanol for 3 h and then allowing to stand for 72 h, against angular frequency (stress: 1 Pa).

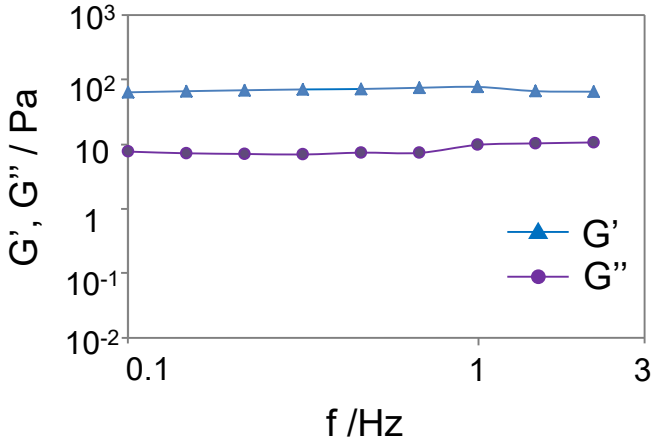

Figure S3. Plots of the storage modulus (G') and the loss modulus (G'') of the organogel, which was formed by stirring a mixture of an α-CD/HFIP solution and 2-butanol with a 3:1 *R*/*S* ratio for 3 h and then allowing to stand for 72 h, against angular frequency (stress: 1 Pa).

4. Schematic illustration of diffraction planes of α-CD assemblies

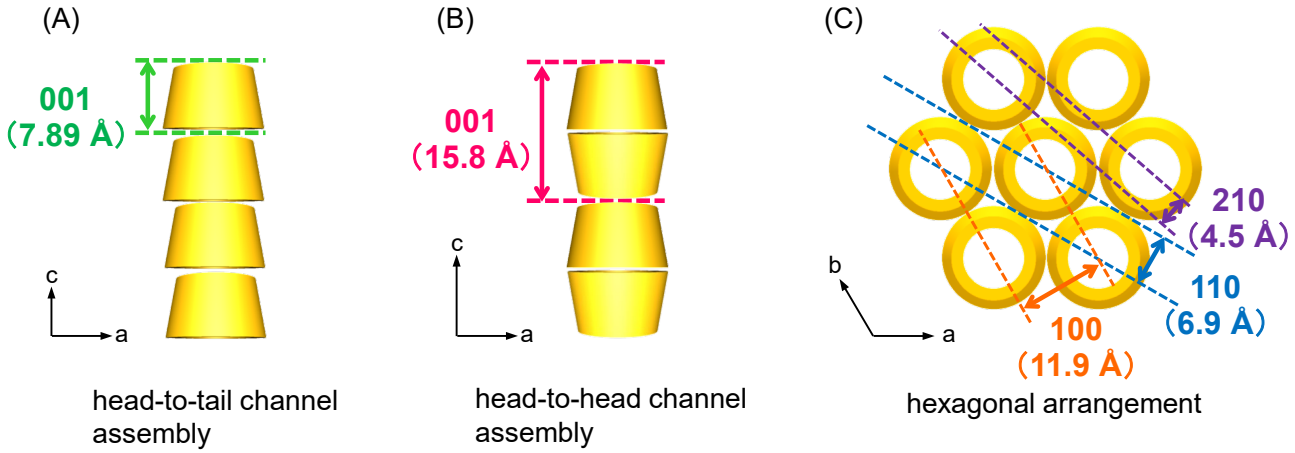

Figure S4. Schematic illustration of diffraction planes of (A) head-to-tail and (B) head-to-head α-CD channel assemblies and (C) hexagonal arrangement of α-CD molecules.

5. Photographs of mixtures of an α-CD/HFIP solution and 2-butanol with different *R*/*S* ratios

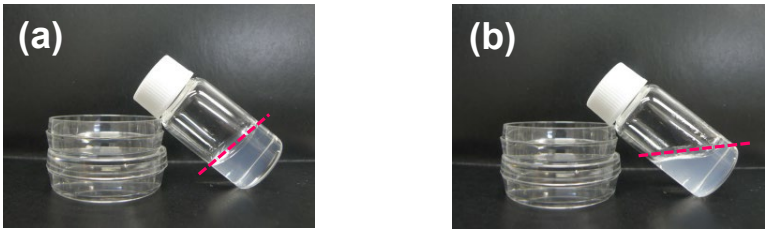

Figure S5. Photographs of mixtures of an α-CD/HFIP solution (0.5 mL) [25 mM] and 2-butanol with different *R*/*S* ratios (2.5 mL) after stirring for 3 h and then allowing them to stand for 54 h. *R*/*S* ratio: (a) 3:1 and (b) 9:1.

6. SEM images of structures obtained by drying gels and suspensions composed of mixtures of an α-CD/HFIP solution and 2-butanol with different *R*/*S* ratios

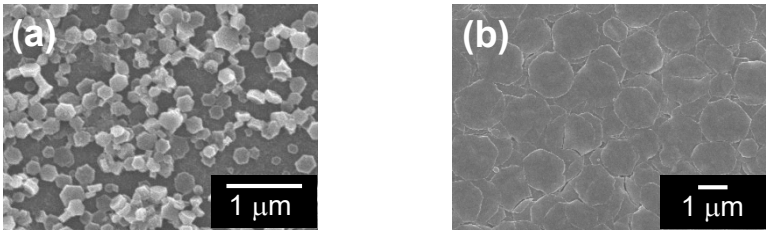

Figure S6. SEM images of structures obtained by drying gels and suspensions composed of mixtures of an α-CD/HFIP solution (0.5 mL) [25 mM] and 2-butanol with different *R*/*S* ratios (2.5 mL). *R*/*S* ratio: (a) 3:1 and (b) 9:1.

7. XRD patterns of structures obtained by drying gels and suspensions which are composed of mixtures of an α-CD/HFIP solution and 2-butanol with different *R*/*S* ratios

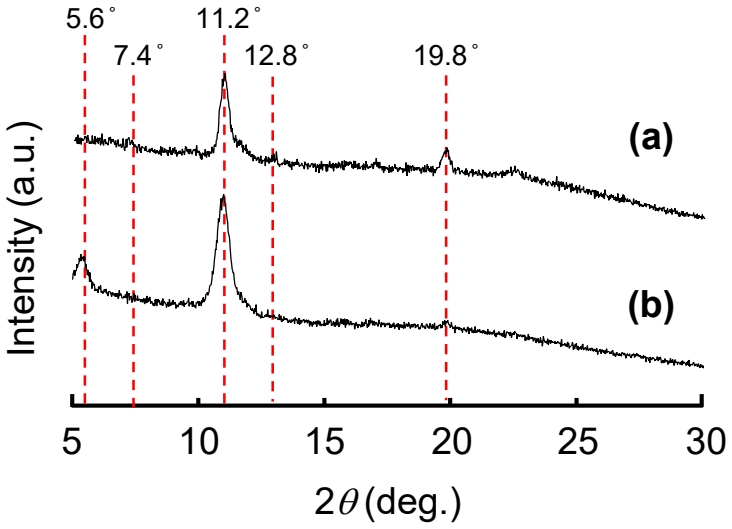

Figure S7. XRD patterns of structures obtained by drying gels and suspensions which are composed of mixtures of an α-CD/HFIP solution (0.5 mL) [25 mM] and 2-butanol with different *R*/*S* ratios (2.5 mL). *R*/*S* ratio: (a) 3:1 and (b) 9:1.

8. ^{1}H NMR analysis of solids obtained after drying a (*S*)-2-butanol gel and a non-gelated (*R*)-2-butanol suspension

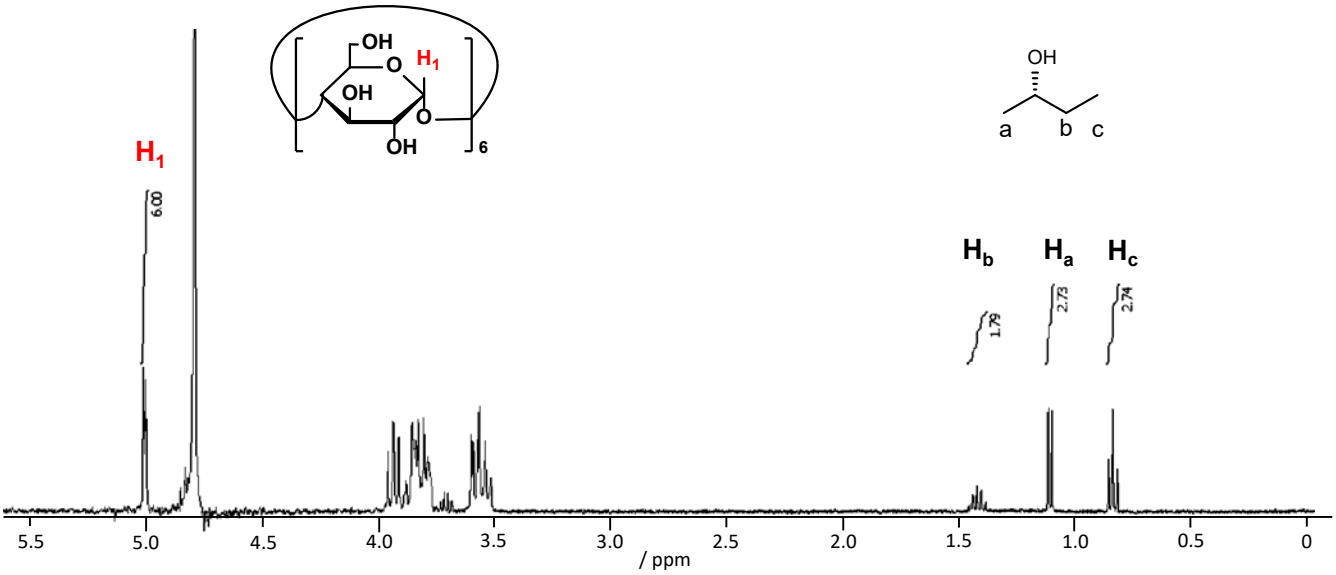

Figure S8. ^{1}H NMR spectrum (D_2O) of solid obtained after drying a (*S*)-2-butanol gel for 24 h at 70 °C *in vacuo*.

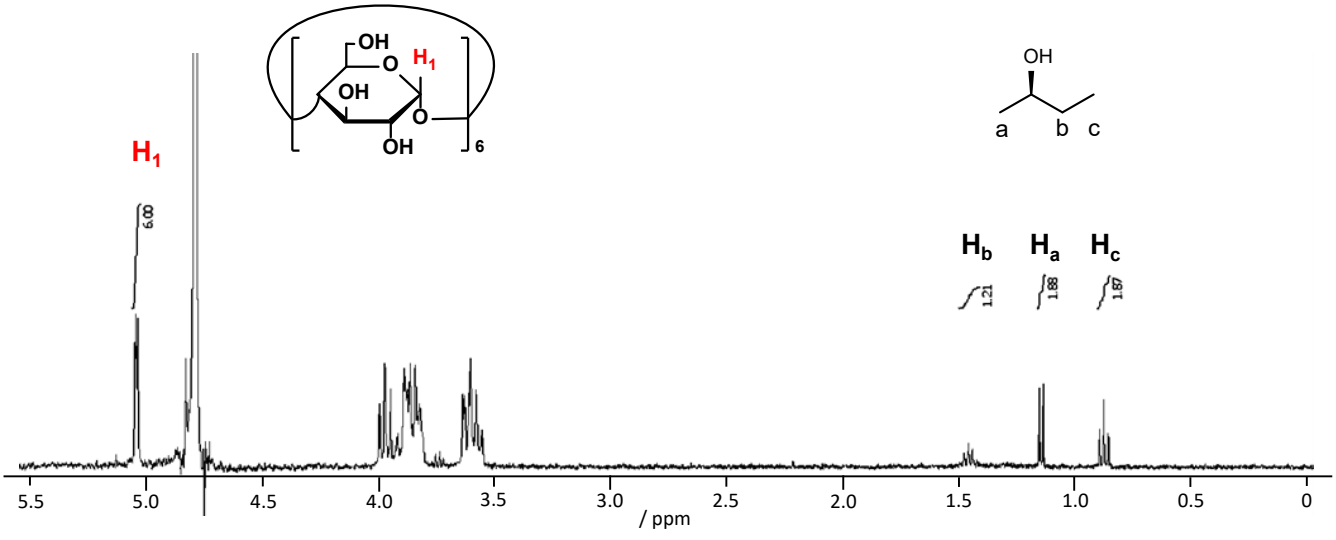

Figure S9. ^{1}H NMR spectrum (D_2O) of solid obtained after drying a non-gelated (*R*)-2-butanol suspension for 24 h at 70 °C *in vacuo*.

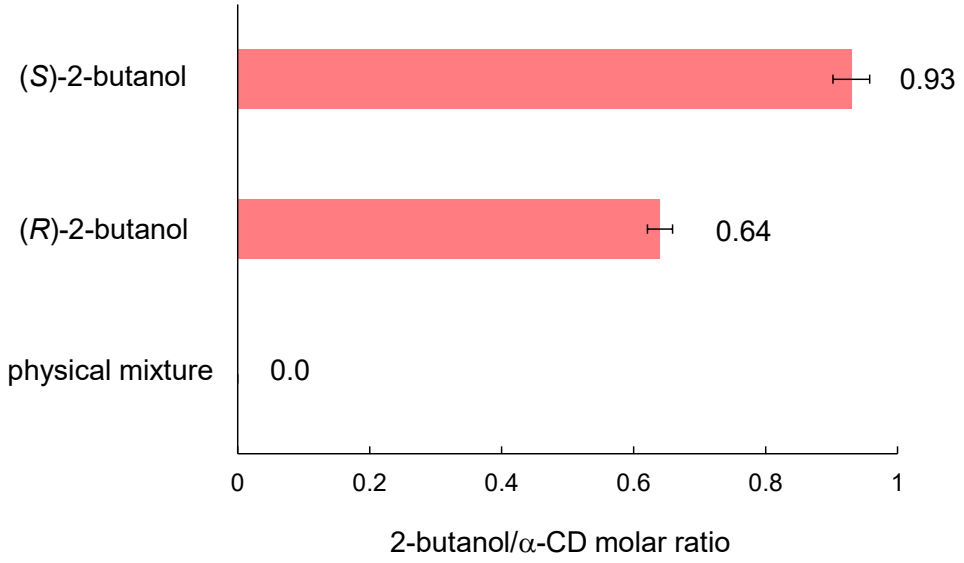

Figure S10. Molar ratio of chiral 2-butanol and α-CD (estimated by ^{1}H NMR) contained in solids obtained after drying a (*S*)-2-butanol gel, a non-gelated (*R*)-2-butanol suspension and a physical mixture of α-CD and (*S*)-2-butanol for 24 h at 70 °C *in vacuo*.

9. ^{1}H NMR analysis of solid obtained by drying a 1-butanol gel

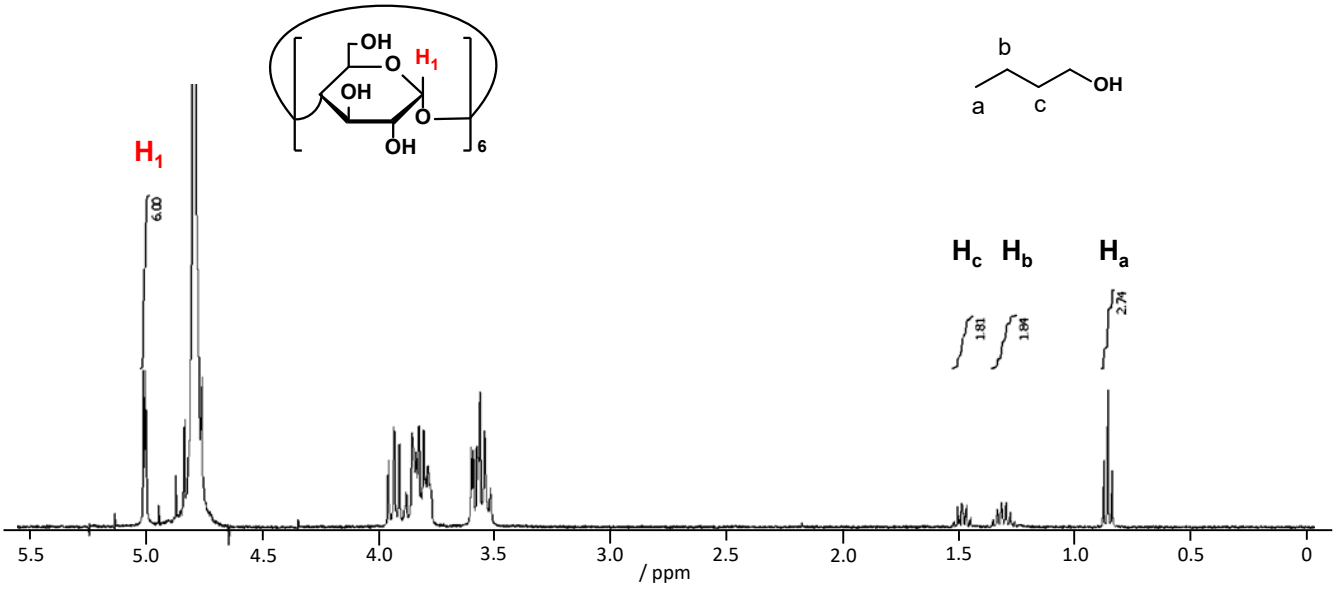

Figure S11. ^{1}H NMR spectrum (D_2O) of solid obtained after drying a 1-butanol gel for 24 h at 70 °C *in vacuo*.