

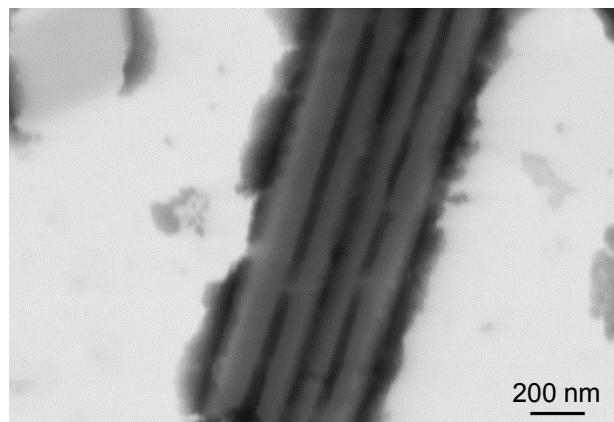
## **Electronic Supplemental Information (ESI)**

Time-Controllable Roll-Up Onset of Polythiophene Sheets into Nanotubes That Exhibit Circularly Polarized Luminescence

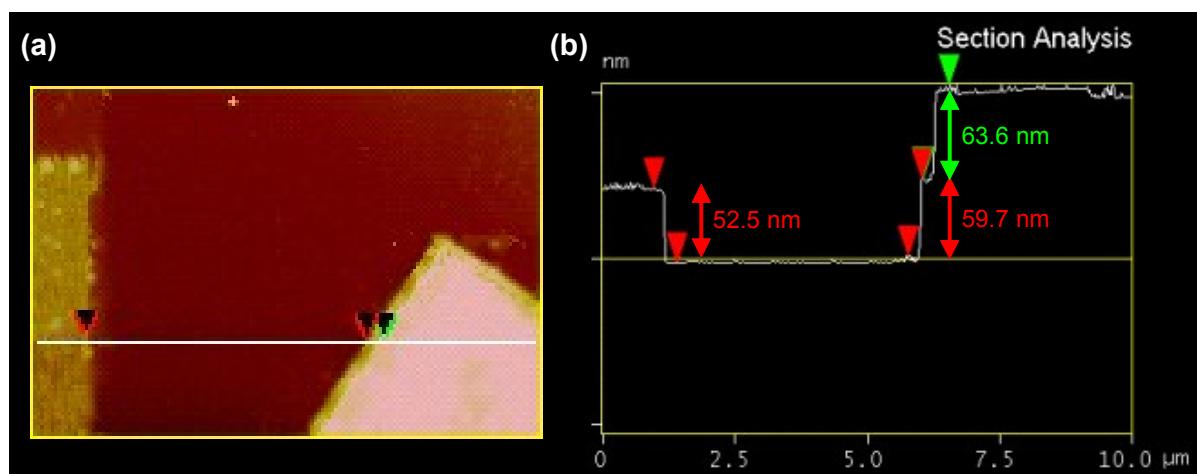
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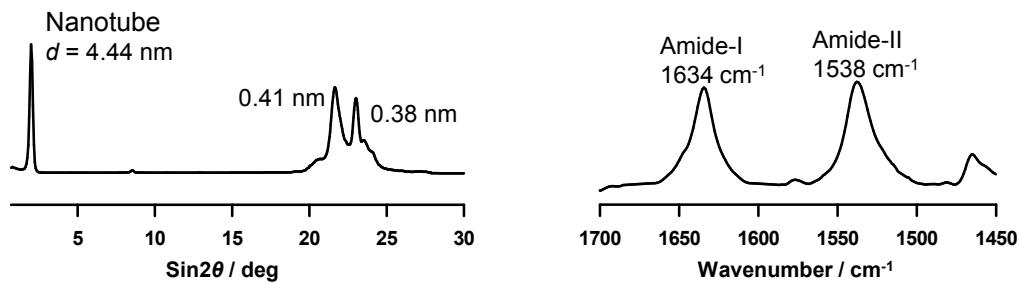


**Fig. S1** Scanning transmission electron microscopy image of the PTB-GlcOle-nanotubes. The nanotube channels were visualized by means of negative staining with 2 wt % phosphotungstate.

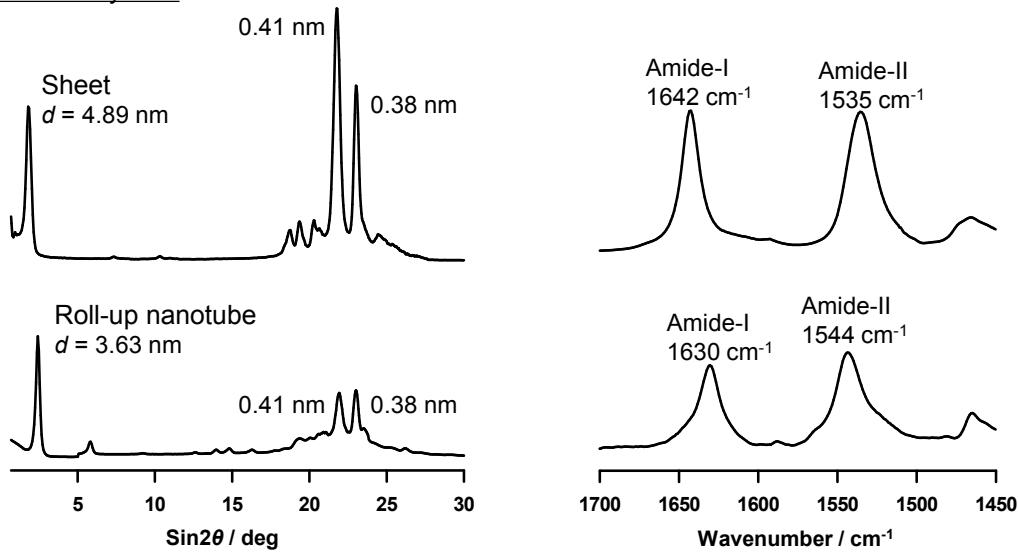


**Fig. S2** (a) Tapping mode atomic force microscopy image and (b) height profile of the PTB-GlcSte-sheets.

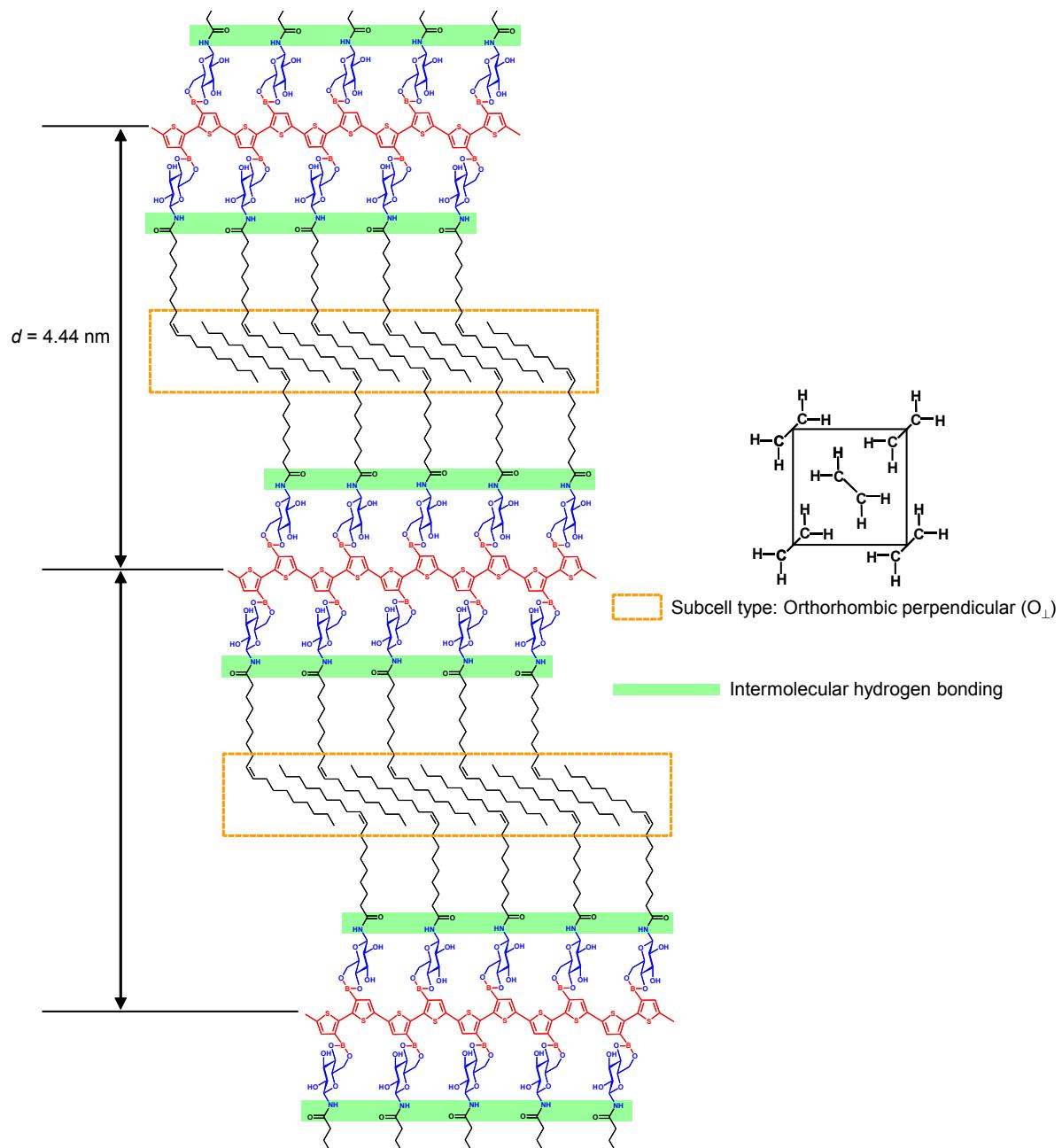
PTB-GlcOle system



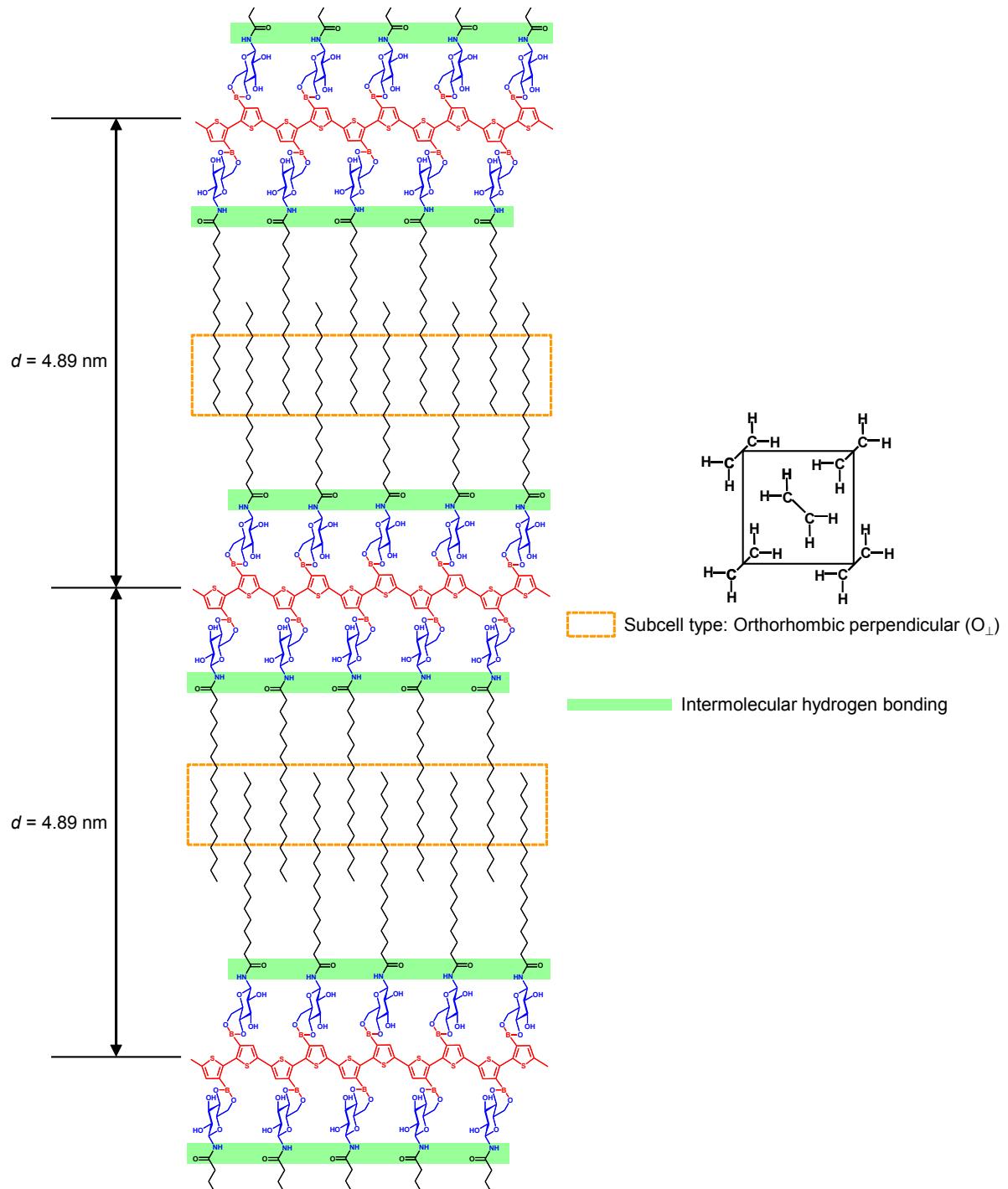
PTB-GlcSte system



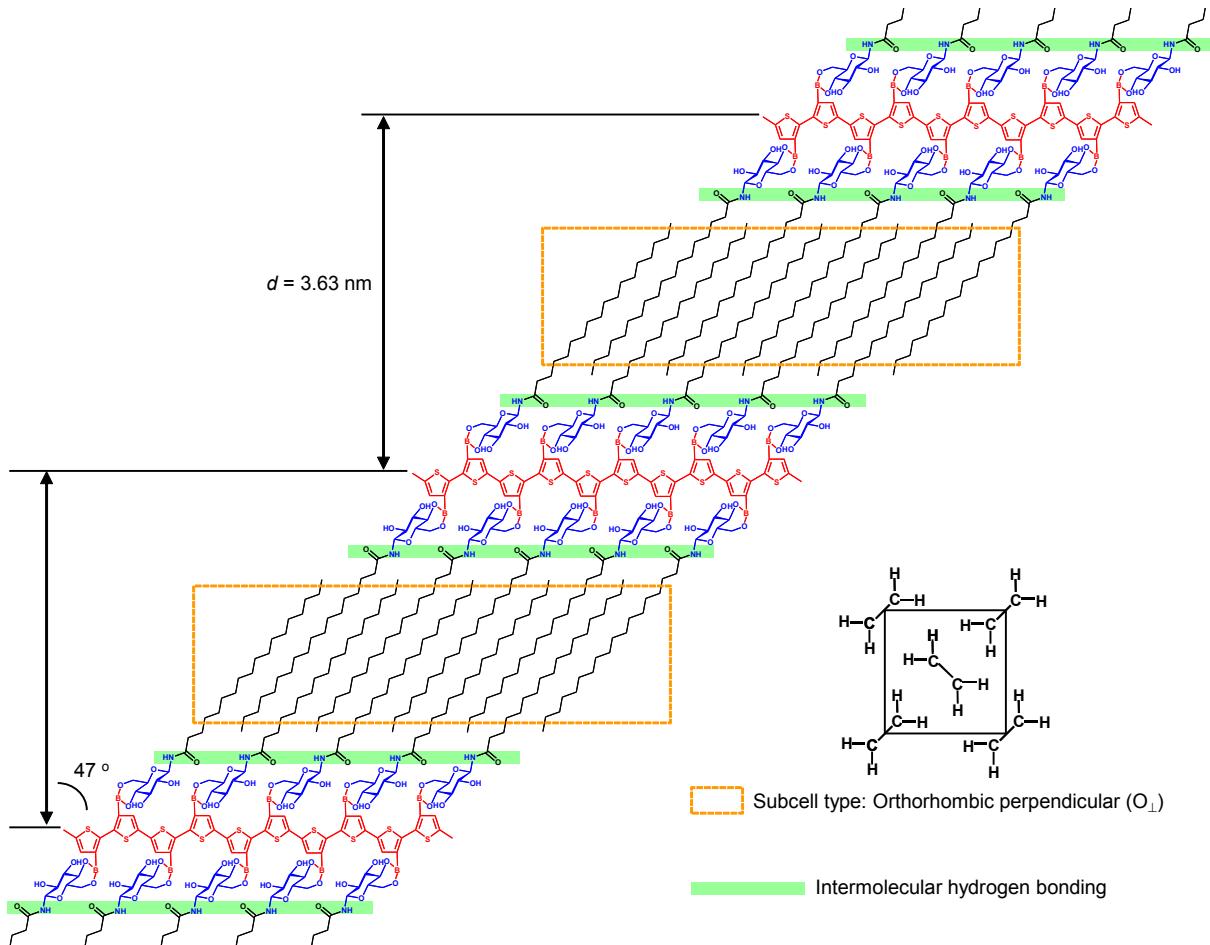
**Fig. S3** Powder X-ray diffraction patterns (left panels) and Fourier transform infrared spectra (right panels) showing the amide-I and -II bands of the indicated supramolecular structures.



**Fig. S4** Schematic representation of the bilayer structures of the PTB-GlcOle-nanotube.

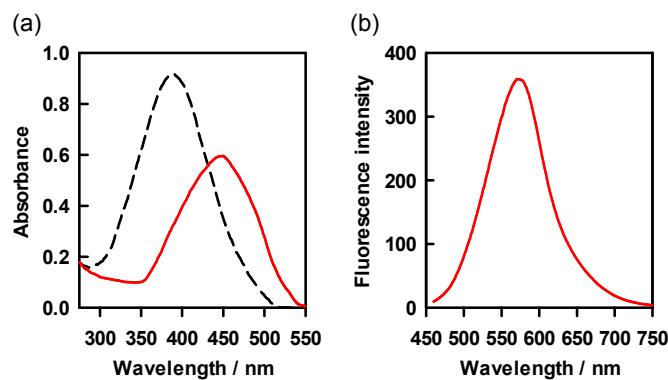


**Fig. S5** Schematic representation of the bilayer structures of the PTB-GlcSte-sheet.

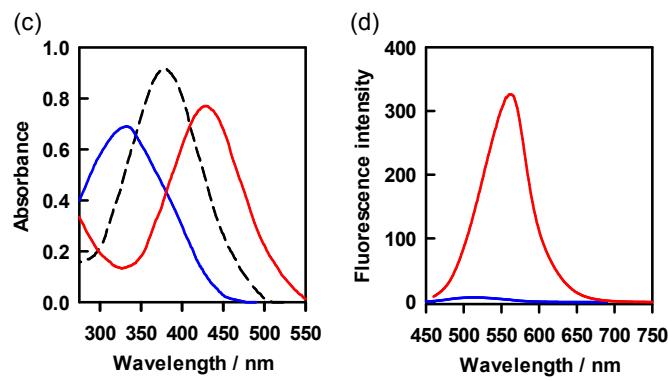


**Fig. S6** Schematic representation of the bilayer structures of the PTB-GlcSte-nanotube.

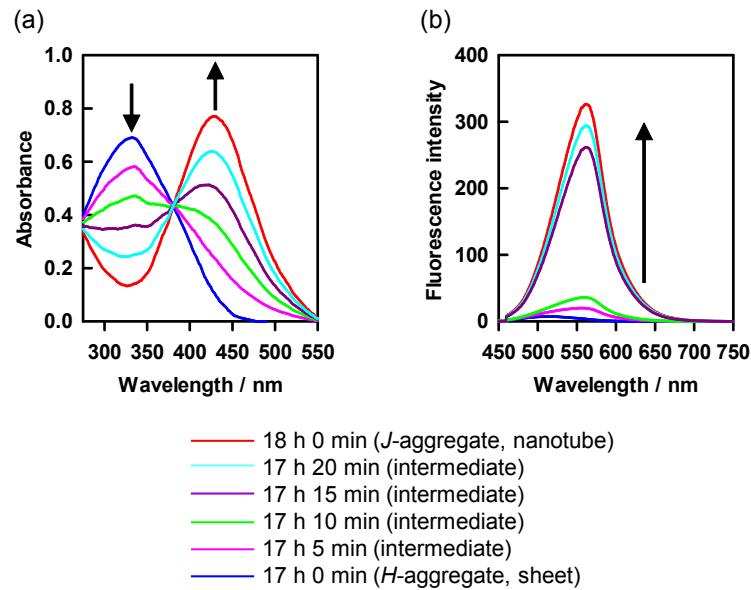
PTB-GlcOle system



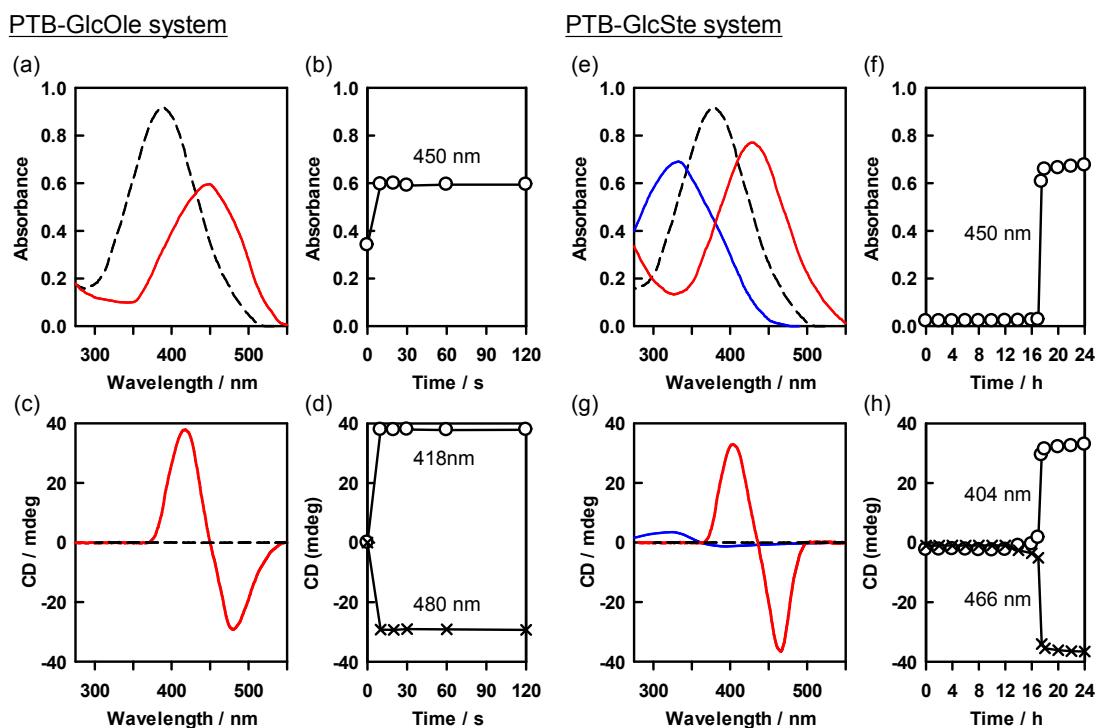
PTB-GlcSte system



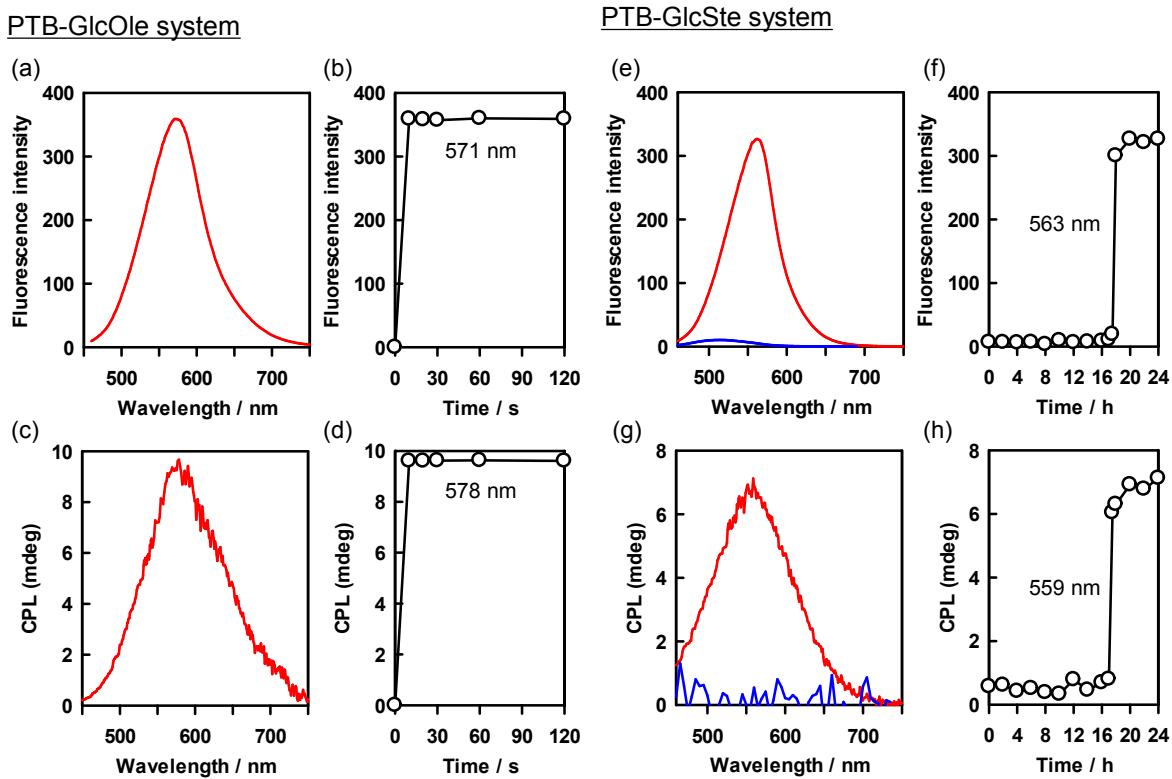
**Fig. S7** (a) Absorption spectra of the PTB-GlcOle-nanotubes dispersed in ethanol at 25 °C (red) and free PTB-GlcOle molecules dissolved in ethanol at 70 °C (black): [PTB-GlcOle-nanotube] = [free PTB-GlcOle molecule] =  $1.0 \times 10^{-5}$  M. (b) Fluorescence spectrum of the PTB-GlcOle-nanotubes dispersed in ethanol at 25 °C: [PTB-GlcOle-nanotube] =  $1.0 \times 10^{-6}$  M. The excitation wavelength was 450 nm. (c) Absorption spectra of the PTB-GlcSte-sheets dispersed in ethanol at 25 °C (blue), PTB-GlcSte-nanotubes dispersed in ethanol at 25 °C (red), and free PTB-GlcSte molecules dissolved in ethanol at 70 °C (black): [PTB-GlcSte-sheet] = [PTB-GlcSte-nanotube] = [free PTB-GlcSte molecule] =  $1.0 \times 10^{-5}$  M. (d) Fluorescence spectra of the PTB-GlcSte-sheets (blue) and PTB-GlcSte-nanotubes (red) dispersed in ethanol at 25 °C: [PTB-GlcSte-sheet] = [PTB-GlcSte-nanotube] =  $1.0 \times 10^{-6}$  M. The excitation wavelengths were 332 nm for the PTB-GlcSte-sheets and 430 nm for the PTB-GlcSte-nanotubes.



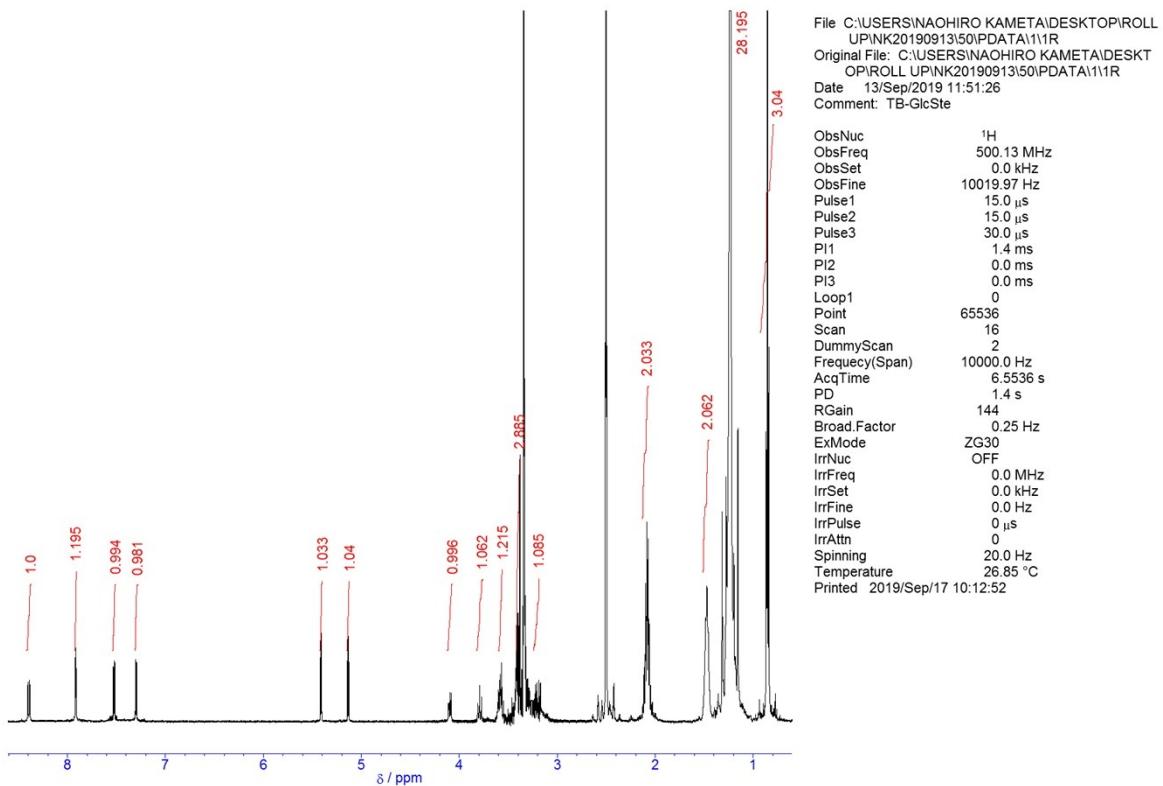
**Fig. S8** (a) Time-dependent absorption spectra of the roll-up process at 25 °C. (b) Time-dependent fluorescence spectra of the roll-up process at 25 °C.



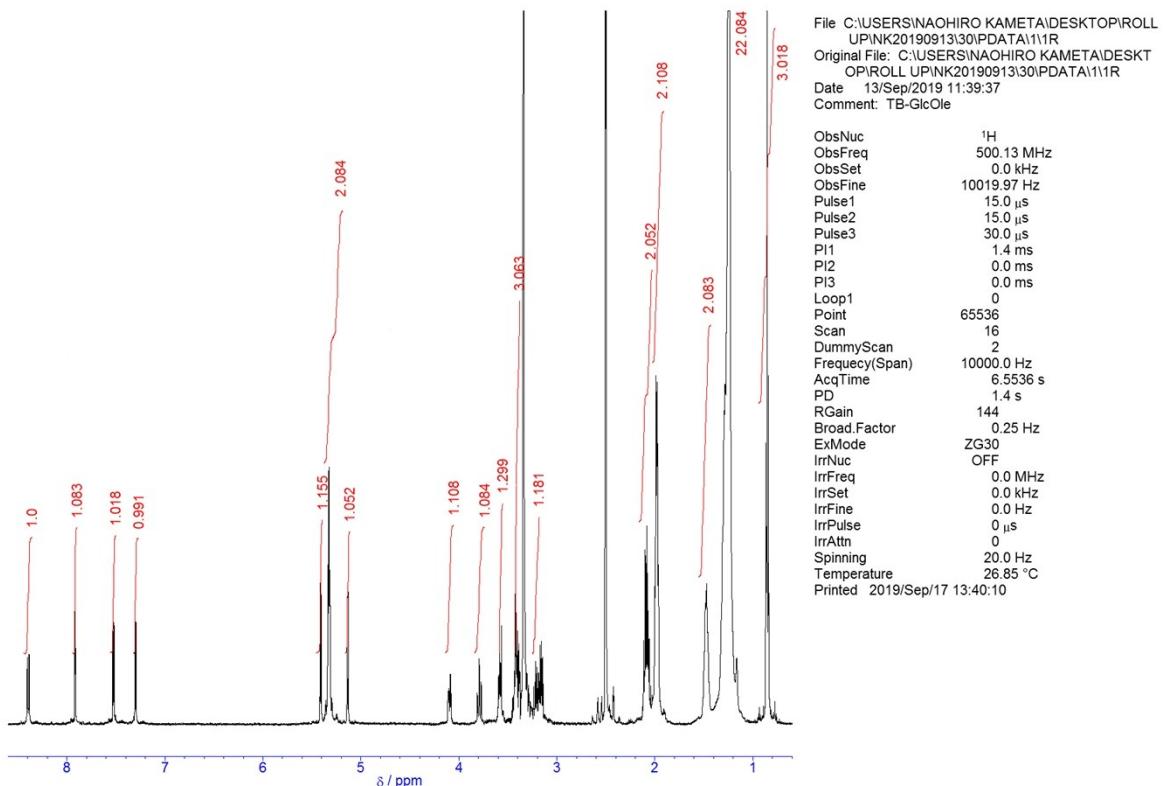
**Fig. S9** (a) Absorption spectra of the PTB-GlcOle-nanotubes dispersed in ethanol at 25 °C (red) and free PTB-GlcOle molecules dissolved in ethanol at 70 °C (black): [PTB-GlcOle-nanotube] = [free PTB-GlcOle molecule] =  $1.0 \times 10^{-5}$  M. (b) Time course of the direct formation of the PTB-GlcOle-nanotubes by self-assembly of the PTB-GlcOle, as indicated by the change in the absorbance at 450 nm. (c) Circular dichroism spectra of the PTB-GlcOle-nanotubes dispersed in ethanol at 25 °C (red) and free PTB-GlcOle molecules dissolved in ethanol at 70 °C (black): [PTB-GlcOle-nanotube] = [free PTB-GlcOle molecule] =  $1.0 \times 10^{-5}$  M. (d) Time course of direct formation of the PTB-GlcOle-nanotubes by self-assembly of the PTB-GlcOle, as indicated by the change in the circular dichroism intensities at 418 and 480 nm. (e) Absorption spectra of the PTB-GlcSte-sheets dispersed in ethanol at 25 °C (blue), PTB-GlcSte-nanotubes dispersed in ethanol at 25 °C (red), and free PTB-GlcSte molecules dissolved in ethanol at 70 °C (black): [PTB-GlcSte-sheet] = [PTB-GlcSte-nanotube] = [free PTB-GlcSte molecule] =  $1.0 \times 10^{-5}$  M. (f) Time course of the formation of the PTB-GlcSte-nanotubes by the roll-up of the PTB-GlcSte-sheets, as indicated by the change in the absorbance at 450 nm. (g) Circular dichroism spectra of the PTB-GlcSte-sheets dispersed in ethanol at 25 °C (blue), PTB-GlcSte-nanotubes dispersed in ethanol at 25 °C (red), and free PTB-GlcSte molecules dissolved in ethanol at 70 °C (black): [PTB-GlcSte-sheet] = [PTB-GlcSte-nanotube] = [free PTB-GlcSte molecule] =  $1.0 \times 10^{-5}$  M. (h) Time course of the formation of the PTB-GlcSte-nanotubes by roll-up of the PTB-GlcSte-sheets, as indicated by the change in the circular dichroism intensities at 404 and 466 nm.



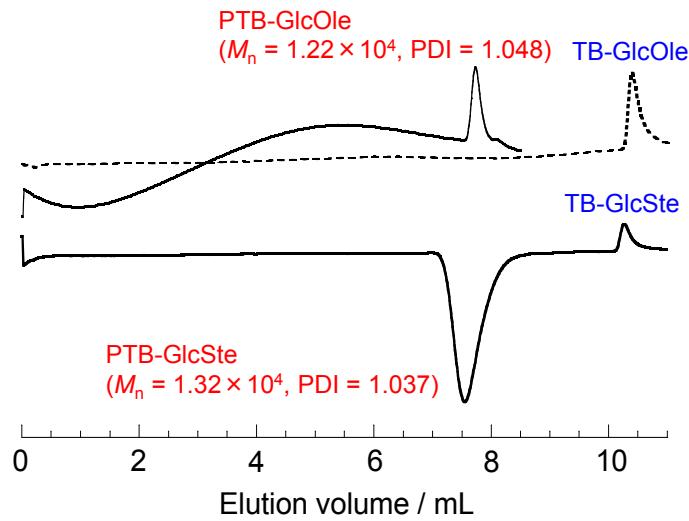
**Fig. S10** (a) Fluorescence spectrum of the PTB-GlcOle-nanotubes dispersed in ethanol at 25 °C: [PTB-GlcOle-nanotube] =  $1.0 \times 10^{-6}$  M. (b) Time course of the direct formation of the PTB-GlcOle-nanotubes by self-assembly of the PTB-GlcOle, as indicated by the change in the fluorescence intensity at 571 nm. (c) Circularly polarized luminescence spectrum of the PTB-GlcOle-nanotubes dispersed in ethanol at 25 °C: [PTB-GlcOle-nanotube] =  $1.0 \times 10^{-6}$  M. (d) Time course of the direct formation of the PTB-GlcOle-nanotubes by self-assembly of the PTB-GlcOle, as indicated by the change in the circularly polarized luminescence intensity at 578 nm. (e) Fluorescence spectra of the PTB-GlcSte-sheets dispersed in ethanol at 25 °C (blue) and PTB-GlcSte-nanotubes dispersed in ethanol at 25 °C (red): [PTB-GlcSte-sheet] = [PTB-GlcSte-nanotube] =  $1.0 \times 10^{-6}$  M. (f) Time course of the formation of the PTB-GlcSte-nanotubes by roll-up of the PTB-GlcSte-sheets, as indicated by the change in the fluorescence intensity at 563 nm. (g) Circularly polarized luminescence spectra of the PTB-GlcSte-sheets dispersed in ethanol at 25 °C (blue) and PTB-GlcSte-nanotubes dispersed in ethanol at 25 °C (red): [PTB-GlcSte-sheet] = [PTB-GlcSte-nanotube] =  $1.0 \times 10^{-6}$  M. (h) Time course of the formation of PTB-GlcSte-nanotubes by roll-up of the PTB-GlcSte-sheets, as indicated by the change in the circularly polarized luminescence intensity at 559 nm.



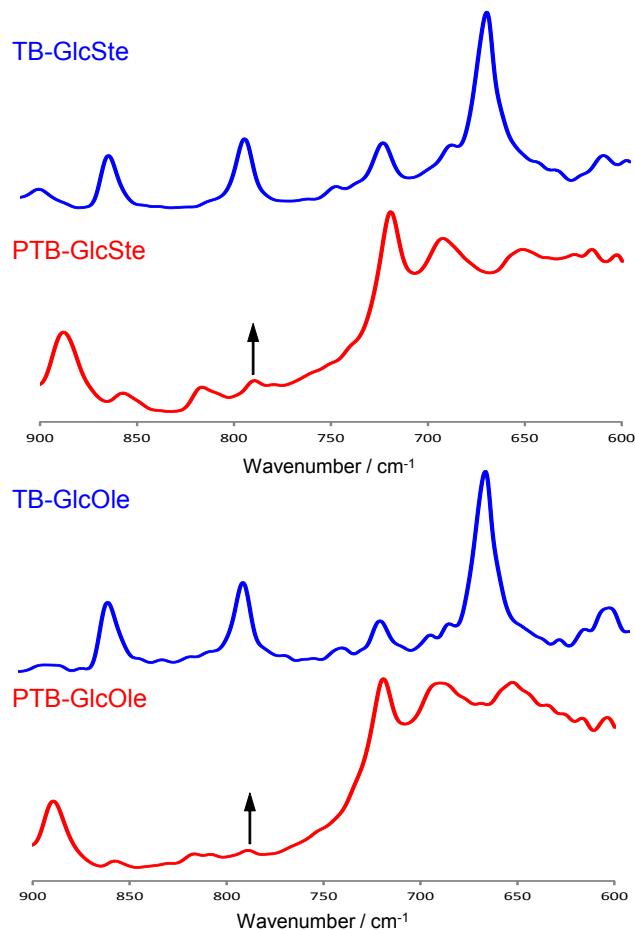
**Fig. S11** <sup>1</sup>H-NMR spectrum of the TB-GlcSte in DMSO-*d*<sub>6</sub>.



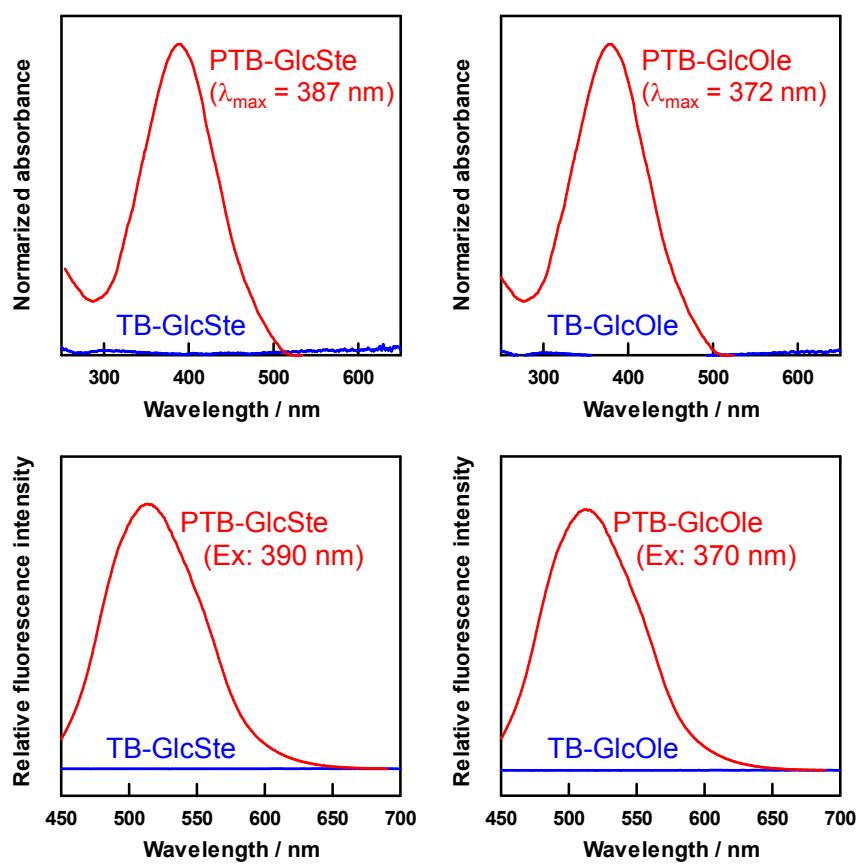
**Fig. S12** <sup>1</sup>H-NMR spectrum of the TB-GlcOle in DMSO-*d*<sub>6</sub>.



**Fig. S13** Size-exclusion chromatograms of the PTB-GlcOle (top) and PTB-GlcSte (bottom). Column: KF-803 column (Shodex), eluent: THF, flow rate:  $1.0 \text{ mL min}^{-1}$ .



**Fig. S14** Fourier transform infrared spectra of the indicated monomers (blue) and polymers (red). The out-of-plane vibration bands of the PTB-GlcSte and PTB-GlcOle at  $788 \text{ cm}^{-1}$  indicate that the polymerization occurred at the 2- and 5-positions of the thiophene moieties.



**Fig. S15** Absorption and fluorescence spectra of the indicated monomers in ethanol at 25 °C (blue) and polymers in ethanol at 70 °C (red).