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Supplementary Information for

Reversible pH-Responsive Switching of Strong Circularly Polarized Luminescence in the Polysaccharide-Templated Co-assemblies

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1. Instruments and materials

Materials

All reagents and solvents were commercially available and used without further purification, unless otherwise noted. Carboxymethyl chitosan (CMCS), methylene blue (MB), and 4,4'-bis(2-sulfostyrylene-based) biphenyl disodium salt (CBS) were purchased from Aladdin Chemistry Co., Ltd. Milli-Q water (18.2 MΩ·cm) was used in all cases.

Characterization

Circular dichroism (CD) and UV–vis spectra were recorded on a JASCO J-1500 spectropolarimeter using quartz cuvettes with an optical path length of 0.1 mm. The CD spectra were recorded over the wavelength range of 200–800 nm with a scanning speed of 1000 nm min^{-1}. Each CD spectrum was obtained by averaging two scans. The corresponding UV–vis absorption spectra were recorded using the same optical path length.

Circular polarized luminescence (CPL) spectra were recorded on a JASCO CPL-300 instrument using quartz cuvettes with an optical path length of 0.1 mm. The CPL spectra were recorded over the wavelength range of 570–900 nm upon excitation at 560 nm. The scanning speed was 500 nm min^{-1}, and the slit width was 3000 μm. Each CPL spectrum was obtained by averaging two scans.

The fluorescence spectra of the suspensions were measured on a Hitachi F-7100 fluorescence spectrophotometer using a Xenon lamp as the excitation light source. The fluorescence measurements were carried out using quartz cuvettes with an optical path length of 10 mm.

2. Methods

Preparation of CMCS aqueous solutions. CMCS powder (20.0 mg) was dissolved in Milli-Q water (10 mL) under stirring to obtain a CMCS aqueous solution ($[CMCS]_{unit}$ = 10 mM). CMCS aqueous solutions with lower concentrations were obtained by diluting the 10 mM CMCS aqueous solution with Milli-Q water. $CMCS_{unit}$ refers to the monosaccharide repeating unit of CMCS rather than the whole polymer chain. The $CMCS_{unit}$ concentration was calculated from the weighed mass of CMCS and the molecular weight of the monosaccharide repeating unit.

Preparation of CMCS aqueous solutions at different pH. For pH-dependent experiments, the pH values of the CMCS aqueous solutions were adjusted to the desired values before the addition of dye solutions. The pH was adjusted using 1 M HCl or NaOH aqueous solutions and measured with a calibrated pH meter (PHS-25, Shanghai INESA Scientific Instrument Co., Ltd., China). The CMCS aqueous solutions were allowed to stand for 10 min before use.

Preparation of dye aqueous solutions. MB powder (64.0 mg) was dissolved in Milli-Q water (10 mL) to obtain a MB aqueous solution (20 mM). CBS aqueous solutions (20 mM) were prepared in the same method.

Preparation of CMCS/dye mixtures. Typically, a certain volume of MB solutions was dropwise added into different concentrations of CMCS aqueous solutions at different $CMCS_{unit}$/MB molar ratios (1:0.2, 1:0.4, 1:0.6, 1:0.8, and 1:1). The obtained CMCS/MB mixtures were allowed to stand for 10 min before CD, fluorescence and CPL measurements. The $CMCS_{unit}$/MB molar ratio represents the stoichiometric ratio between CMCS repeating units and MB molecules.

Reversible pH-responsive experiments. The pH of the CMCS/MB mixtures was alternately adjusted between pH 7 and pH 12 using 1 M HCl or NaOH aqueous solutions. After each pH adjustment, the mixtures were allowed to stand for 10 min before CD and CPL measurements.

3. Supplementary figures

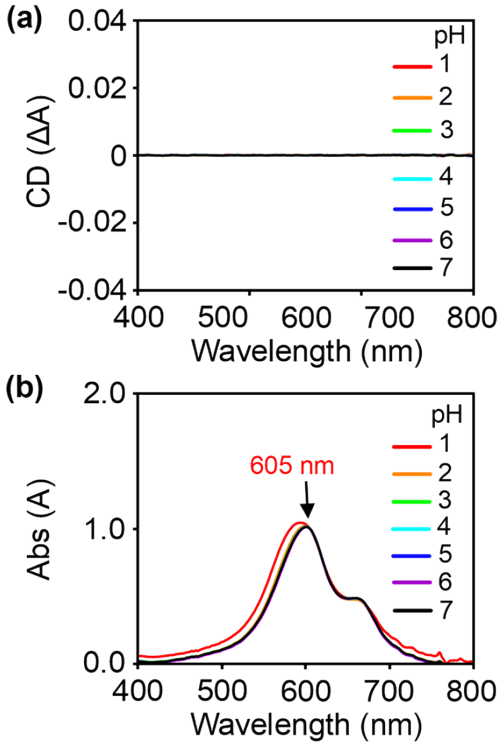

Fig. S1 Normalized CD (a) and UV–vis absorption (b) spectra of MB solution and CMCS/MB system at a 1:0.6 molar ratio and different pH values. $[CMCS]_{unit}$ = 5 mM.

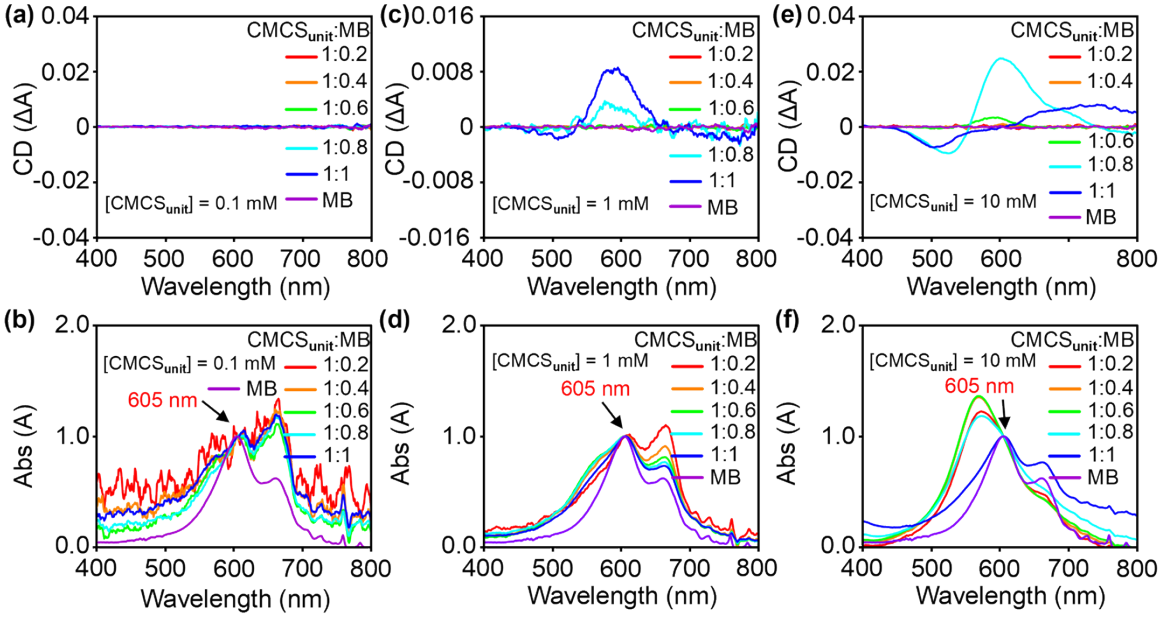

Fig. S2 Normalized CD (a, c, e) and UV–vis absorption (b, d, f) spectra of MB solution and CMCS/MB system at different $CMCS_{unit}$/MB molar ratios with CMCS concentrations of (a, b) 0.1 mM, (c, d) 1 mM, and (e, f) 10 mM.

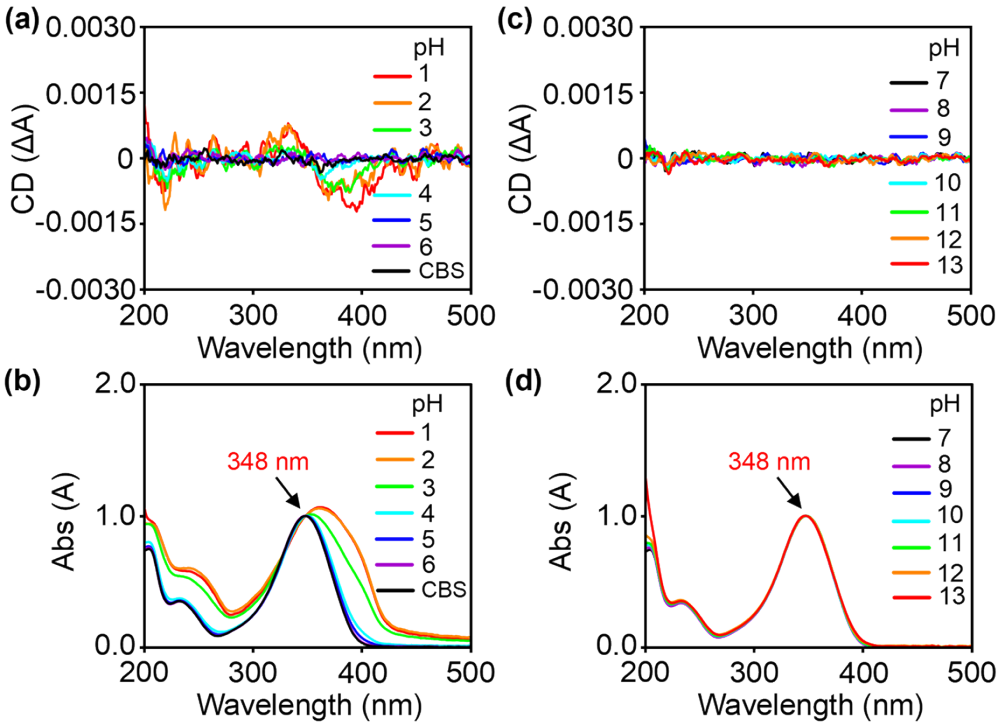

Fig. S3 Normalized CD (a, c) and UV–vis absorption (b, d) spectra of CBS solution and CMCS/CBS system at a 1:0.2 molar ratio and different pH values. $[CMCS]_{unit}$ = 5 mM.

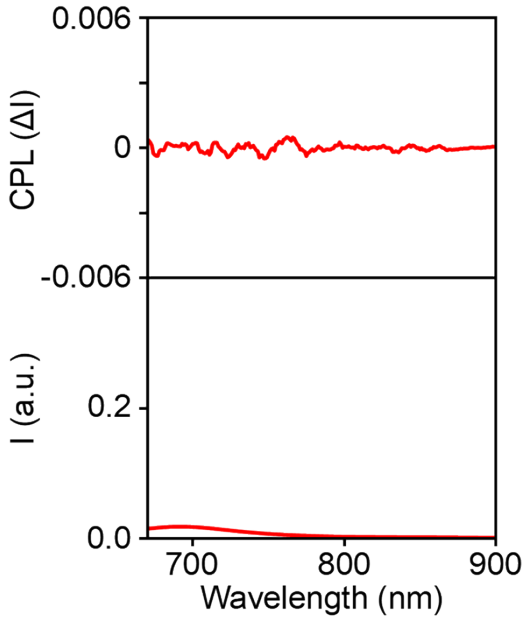

Fig. S4 CPL spectrum of CMCS/MB system at a 1:0.6 molar ratio and pH 12. λ_{ex} = 560 nm, $[CMCS]_{unit}$ = 5 mM.

Fig. S5 Fluorescence spectrum of CMCS/MB system at a 1:0.6 molar ratio and pH 12. λ_{ex} = 560 nm, $[CMCS]_{unit}$ = 5 mM.

Fig. S6 Fluorescence spectrum of CMCS/MB system at a 1:1 molar ratio and pH 12. λ_{ex} = 560 nm, $[CMCS]_{unit}$ = 1 mM.

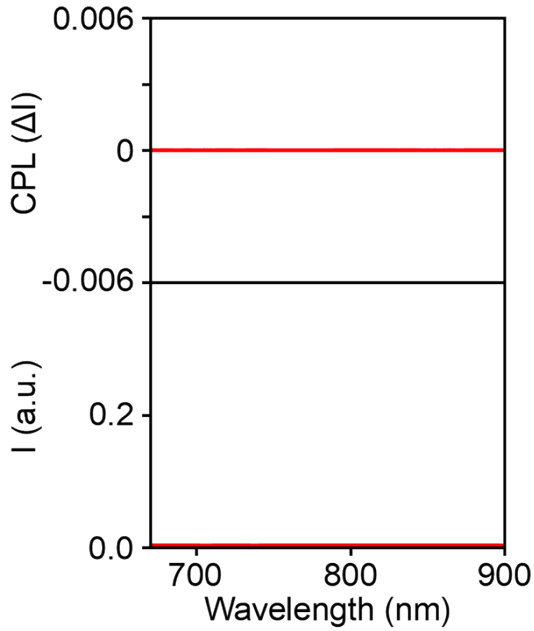

Fig. S7 CPL spectrum of CMCS solution at pH 12. $[CMCS]_{unit}$ = 1 mM, λ_{ex} = 560 nm.

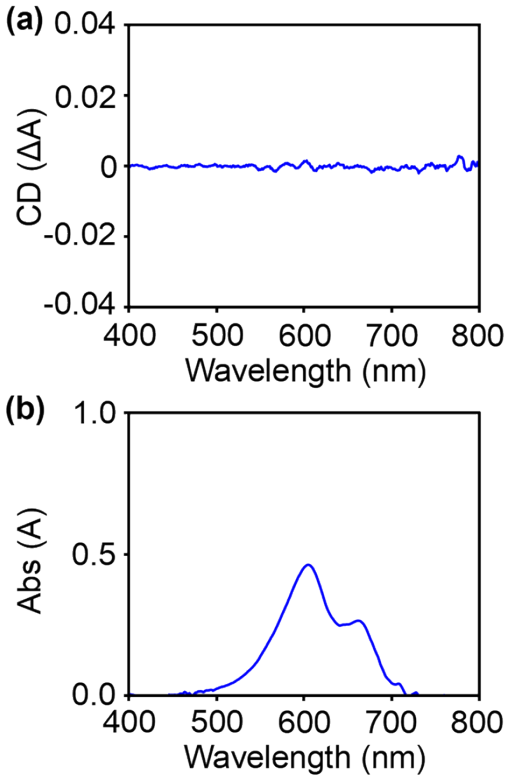

Fig. S8 CD (a) and UV–vis absorption (b) spectra of CMCS/MB system at a 1:1 molar ratio and pH 7. $[CMCS]_{unit}$ = 1 mM, λ_{ex} = 560 nm.

Fig. S9 Reversible switching of the g_{lum} value at 790 nm of the CMCS/MB system upon alternating the solution pH between 7 and 12 over multiple cycles. $[CMCS]_{unit}$ = 1 mM, $CMCS_{unit}$/MB = 1:1.