Supporting Information for

## Phosphatase-Responsive Amphiphilic Calixarene Assembly

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Figure S1. <sup>1</sup>H NMR spectrum (400 MHz,  $D_2O$ , 298.15K) of AC4AH.



**Figure S2.** <sup>13</sup>C NMR spectrum (400 MHz, D<sub>2</sub>O, 298.15K) of AC4AH.

**CAC of AC4AH.** Vibronic band intensities in pyrene monomer fluorescence are a convenient probe to accurately determine CAC values. As shown in Figure S3, the ratio (band III:bandI) increased in the presence of AC4AH, indicating that AC4AHs constitute micellelike aggregates presenting hydrophobic domains formed by alkyl chains that serve as a binding site for pyrene molecules. The CAC is approximated to

be 0.6 mM.<sup>1</sup>



Figure S3. Plots of bands III:I ratio versus [AC4AH] in pyrene fluorescence at 25 °C:

[pyrene] = 0.001 mM, excitation 335 nm.



**Figure S4.** (a) Optical transmittance of AC4AH–ADP complex at different concentrations at 25 °C. Inset: dependence of the optical transmittance at 450 nm on ADP concentration. (b) Optical transmittance of AC4AH–AMP complex. Inset: dependence of the optical transmittance at 450 nm on AMP concentration.



Figure S5. High-resolution TEM images of AC4AH–ATP particles.



**Figure S6.** Fluorescence emission spectra of HPTS with AC4AH–ATP complex in the absence of CIAP (a) and in the presence of denatured CIAP (b) at different time within 30 minutes.

## References

[1] S. Shinkai, S. Mori, H. Koreishi, T. Tsubaki and O. Manabe, J. Am. Chem. Soc., 1986, 108, 2409.