### **Supporting Information**

# Supramolecular Binary Hydrogels from Calixarene and Amino Acids and their Entrapment-Release of Model Dye Molecules

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# a) <sup>1</sup>H-NMR and <sup>13</sup>C-NMR spectra of TPC.









Fig. S2 <sup>13</sup>C-NMR spectra of TPC.

## b) Crystal Structure of TPC.



Fig. S3 Crystal structure of TPC.

#### c) CMC Measurement.



**Fig. S4** (a) CMC determination for TPC using CD spectra.<sup>1</sup> (b) The plot of ellipse degree at 255 nm vs. concentration of TPC, gives a CMC of 1.2 mM at pH 3.0.

### d) Zeta potential Measurement.



Fig. S5 Zeta potential measurement of micellar aggregation of TPC at 5 mM, 25 °C.

## e) TEM and SEM Studies.



Fig. S6 (a) TEM and (b) SEM images for the nanoscale micelles of TPC.

#### f) AFM Studies.



**Fig. S7** (a) and (b) Height profile plot of the nanoscale spheres obtained from the solution of TPC. (c) 3D topographic view of a.



**Fig. S8** (a) and (b) Height profile plot of the gel network obtained from the TPC/arginine LMWH. (c) 3D topographic view of a.



**Fig. S9** (a) and (b) Height profile plot of the gel network obtained from the TPC/histidine LMWH. (c) 3D topographic view of a.



**Fig. S10** (a) and (b) Height profile plot of the gel network obtained from the TPC/lysine LMWH. (c) 3D topographic view of a.

### g) T<sub>gel</sub> Measurements.



Fig. S11 Plot of  $T_{gel}$  versus concentration of TPC in LMWHs based on (a) TPC/arginine, (b) TPC/histidine and (c) TPC/lysine.

#### h) Fluorescence Studies.



**Fig. S12** Fluorescence spectra of ANS in LMWHs through temperature increasing (left) and decreasing (right). (a) and (b): TPC/arginine LMWH; (c) and (d): TPC/histidine LMWH; (e) and (f): TPC/lysine LMWH. Condition: [TPC] = 10 mM, [basic amino acids] = 40 mM in water, [ANS] = 10  $\mu$ M,  $\lambda_{ex}$  = 360 nm.



**Fig. S13** Changes in fluorescence intensity of ANS as a function of cyclical variation of temperature between 25 and 70 °C in (a) TPC/arginine LMWH, (b) TPC/histidine LMWH and (c) TPC/lysine LMWH.

<sup>(1)</sup> S. Bonkoski, J. H. Perrin, J Pharm Sci., 1969, 58, 1428–1429.