

## Supporting Information

# A smart pH-switchable luminescent hydrogel

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## Experimental details

### Materials

Diisopropylethylamine (DIEA), Fmoc-amino acids, and O-(7-Azabenzotriazole-1-yl)-1,1,3,3-tetramethyluronium hexafluorophosphate (HATU) were purchased from BO MAI JIE Technology Co., Ltd (Beijing, China). Other chemical reagents and solvents were purchased from Heowns Biochemical Technology Co., Ltd (Tianjin, China). Carboxylated tetraphenylethylene (TPE-COOH) was synthesized as our previous work.<sup>1</sup>

### Peptides Synthesis

TPE-MAX was synthesized by standard Fmoc solid-phase peptide synthesis (SPPS) using CLEAR Amide Resin and the corresponding N-Fmoc protected amino acids with side chains properly protected. Resins were first treated with 20% piperidine for deprotection of Fmoc group and then, the first amino acid Fmoc-Lys(Boc)-OH was coupled to the free amino group on the resin in anhydrous N, N' – dimethylformamide (DMF) using DIEA and HATU as the coupling reagent. After deprotection of Fmoc group from the first amino acid by 20% piperidine, the following amino acids were coupled as the first one. In the last coupling step, TPE-COOH was used to cap the amine group of the peptide. After the last coupling step, the resins were washed by DMF, followed by dichloromethane (DCM). TPE-MAX was cleaved from the resins by reagent A (containing TFA, 1,2-ethanedithiol, thioanisole and anisole) and the mixture was filtered and poured into ice-cold diethylether. The resulting precipitate was dissolved in TFA, reprecipitated by ice-cold diethylether and the product was followed to be dissolved in acetonitrile for HPLC purification.

### General methods

The purified product was analyzed by High Performance Liquid Chromatography (HPLC) (Waters 2796) and MALDI-TOF-MS using a Microflex LRF System spectrometer (Bruker Daltonics). TPE-MAX was analyzed using RP-HPLC on a C18 column. Elution was monitored with a diode array detector at wavelengths of 330 nm. TPE-MAX was eluted with a linear gradient at the flow rate of 1 ml/min, where solvent A consisted of water and solvent B consisted of acetonitrile. A linear gradient of 5 to 95%B over 45 min was used. TEM of TPE-MAX and TPE-COOH aggregate in solution was performed on a Hitachi HT7700 transmission electron microscope (TEM) with 120 kV acceleration voltage. Rheology test was performed on an HAAKE MARS system.

### Thioflavin-T Fluorescence

Fluorescence spectra of thioflavin-T were measured by a fluorescence spectrophotometer (HITACHI F-4600). Dilute solutions (50  $\mu\text{M}$ ) of TPE-MAX at pH 6.0 or 10.0 was incubated with 1mM thioflavin-T for ten minutes at room temperature and then, the emission spectrum was recorded with the excitation at 440 nm.

## Rheology

Rheology test was performed on an HAAKE MARS system. In this experiment, 20 mm parallel plates were used for the test. First, the 1.0 wt% TPE-MAX solution at pH 6.0 (200  $\mu\text{L}$ ) was added onto the plate and then the pH was adjusted to pH 10.0 using NaOH. After the incubation for ten minutes at room temperature to form the hydrogel, the linear viscoelastic region (LVR) was determined. And then, frequency dependence of dynamic storage modulus ( $G'$ ) and loss modulus ( $G''$ ) for TPE-MAX hydrogel were measured by the mode of dynamic frequency sweep in the region of 0.1-100 rad/s.

## Transmission electron microscope (TEM) and scanning electron microscopy (SEM) characterization

TEM of TPE-MAX and TPE-COOH aggregate in solution was performed on a Hitachi HT7700 transmission electron microscope (TEM) with 120 kV acceleration voltage. TEM samples of the TPE-MAX and TPE-COOH aggregate in solution were dropped on the surface of a carbon-coated copper (purchased from Zhongjingkeyi Technology Co. Ltd., Beijing, China), respectively. The samples were volatilized to dry out at room temperature and then were pre-strained with 1% uranyl acetate.

SEM of TPE-MAX hydrogel was performed on a Hitachi S4800 scanning electron microscopy. SEM samples of the hydrogel were prepared as following: a silicon chip was vertically dipped into the hydrogels for 5 seconds, and then the sample underwent a liquid nitrogen freeze treatment before it was freeze-dried.

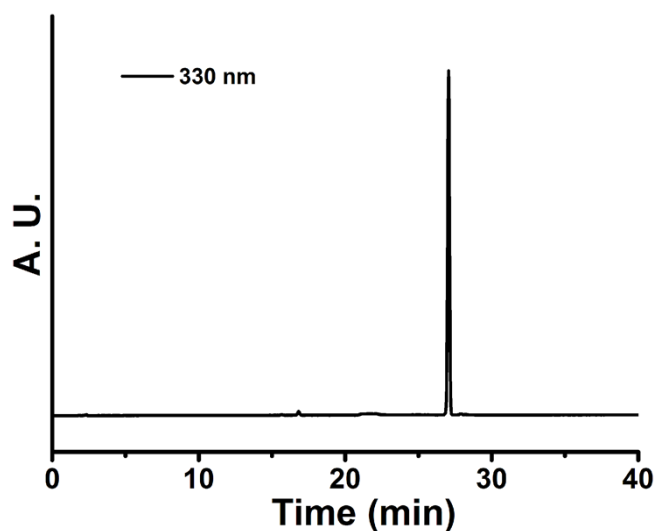
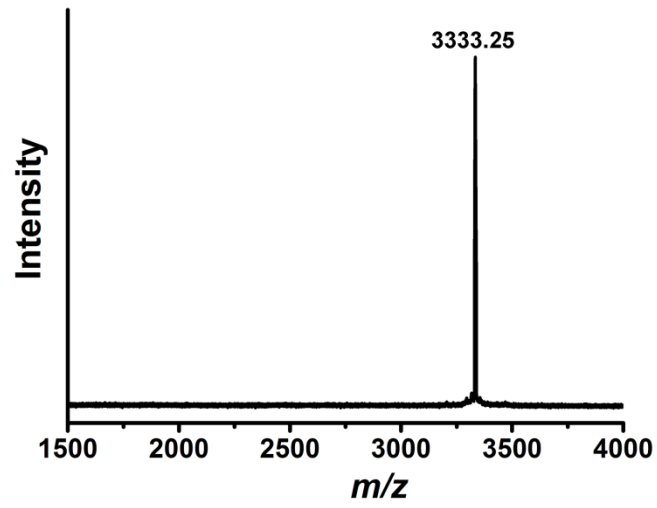
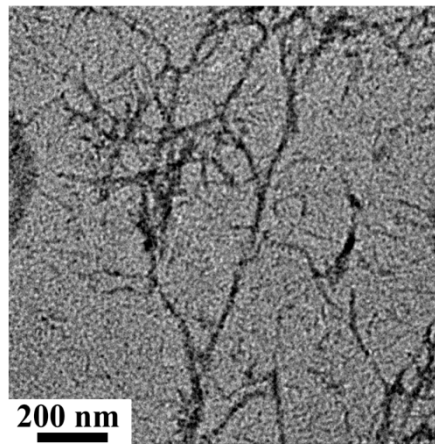


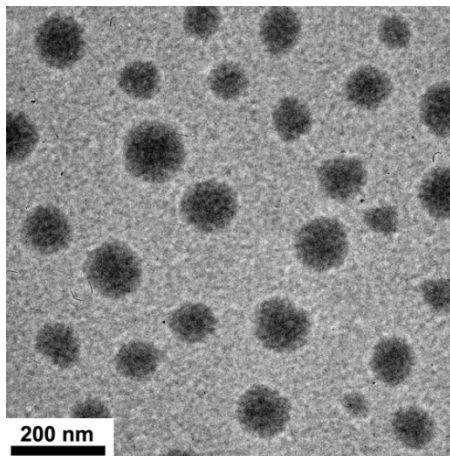
Figure S1. Characterization of TPE-MAX by HPLC



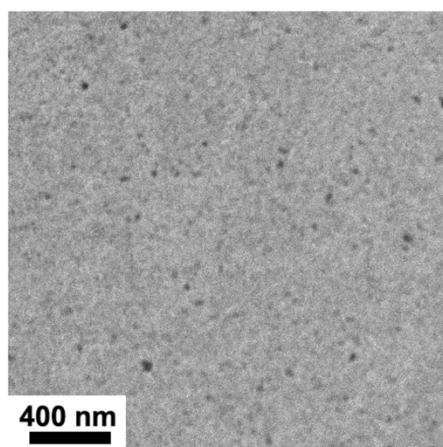
**Figure S2.** Characterization of TPE-MAX by MALDI-TOF-MS



**Figure S3.** TEM image of solution of TPE-MAX at pH 10.0 at the concentration of 50  $\mu$ M



**Figure S4.** TEM images of solution of TPE-COOH at the concentration of 50  $\mu$ M



**Figure S5.** TEM image of solution of TPE-MAX at pH 6.0 at the concentration of 50  $\mu\text{M}$

1. C. Zhang, C. Liu, X. Xue, X. Zhang, S. Huo, Y. Jiang, W. Q. Chen, G. Zou and X. J. Liang, *ACS. Appl. Mater. Interfaces.*, 2014, 6, 757; C. Zhang, S. Jin, S. Li, X. Xue, J. Liu, Y. Huang, Y. Jiang, W. Q. Chen, G. Zou and X. J. Liang, *ACS. Appl. Mater. Interfaces.*, 2014, 6, 5212.