

Electronic Supplementary Material (ESI)

Fabrication of reversible pH-responsive aggregation- induced emission luminogen assisted by a block copolymer *via* a dynamic covalent bond

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Scheme S1. Synthetic Route of TPE-CHO.

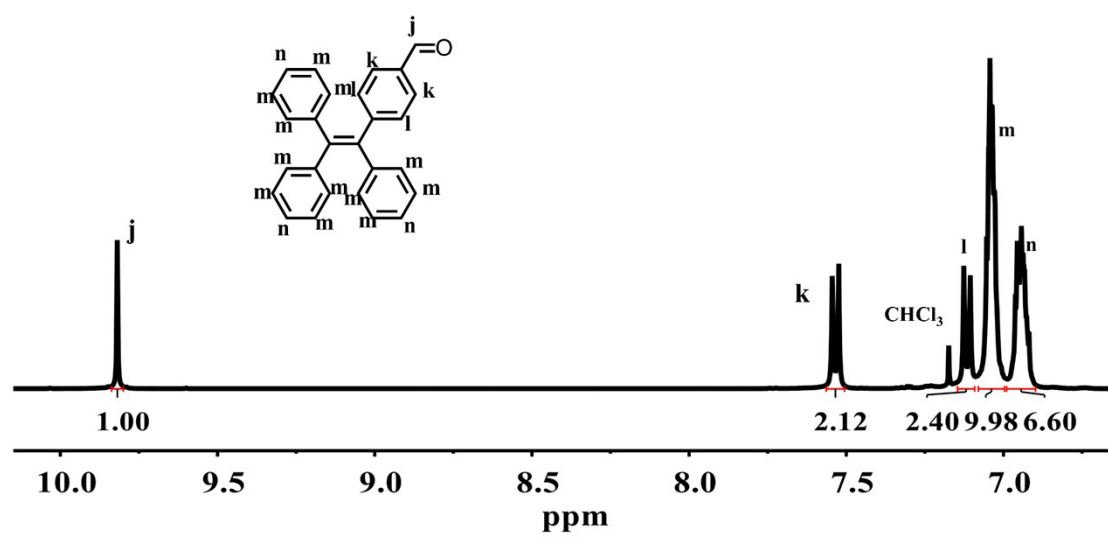
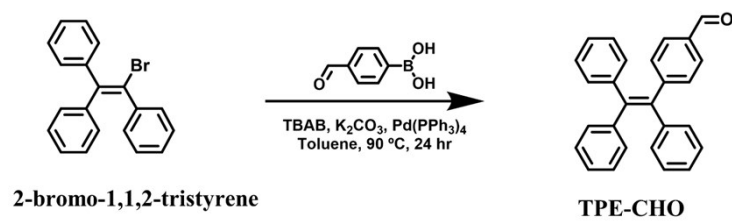


Figure S1. ¹H NMR spectrum for TPE-CHO in CDCl₃.

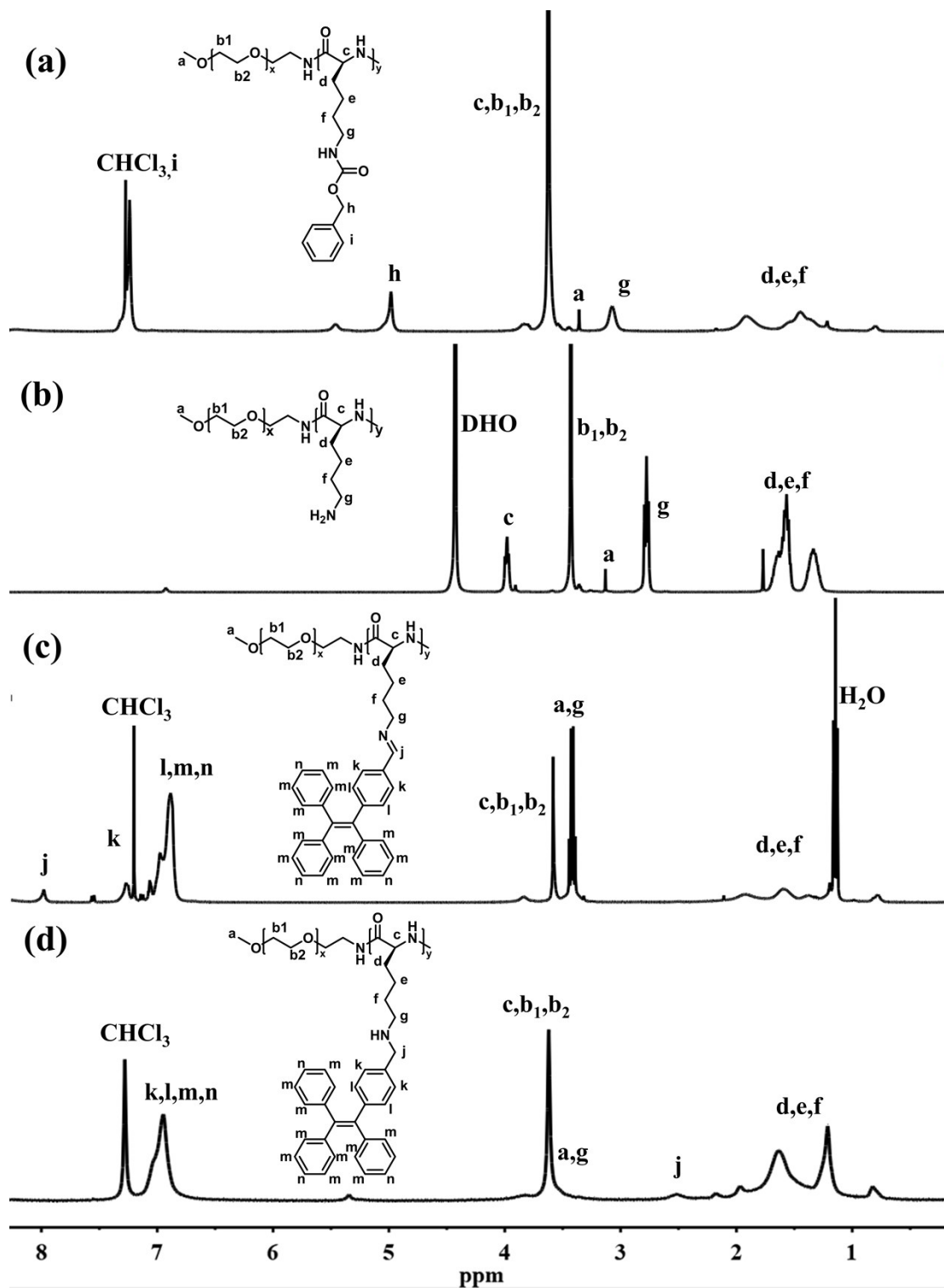


Figure S2. ^1H NMR spectra for (a) PEG_x-b-PZLyS_y in CDCl₃; (b) PEG_x-b-PLyS_y in D₂O; (c) PEG_x-b-P(Lys-TPE)_y in CDCl₃ and (d) H-PEG_x-b-P(Lys-TPE)_y in CDCl₃.

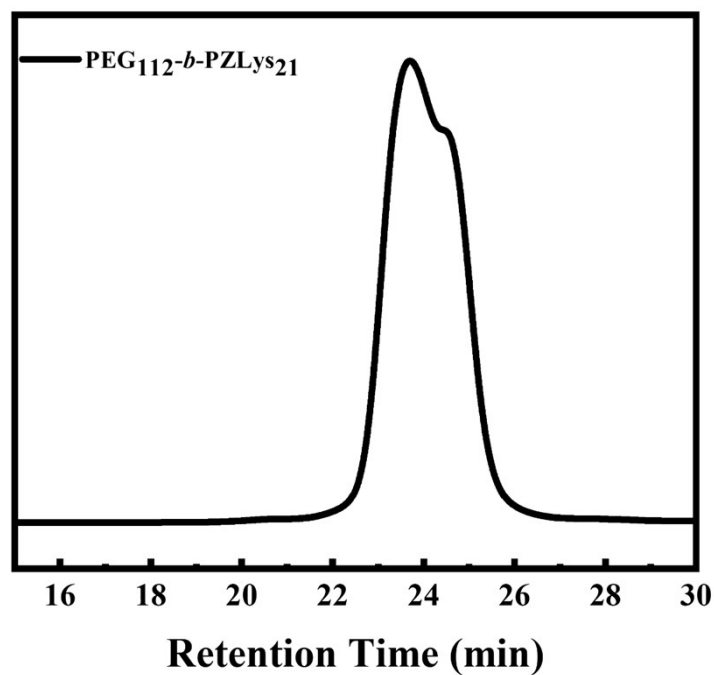


Figure S3. GPC chromatogram of the PEG₁₁₂-*b*-PZLys₂₁.

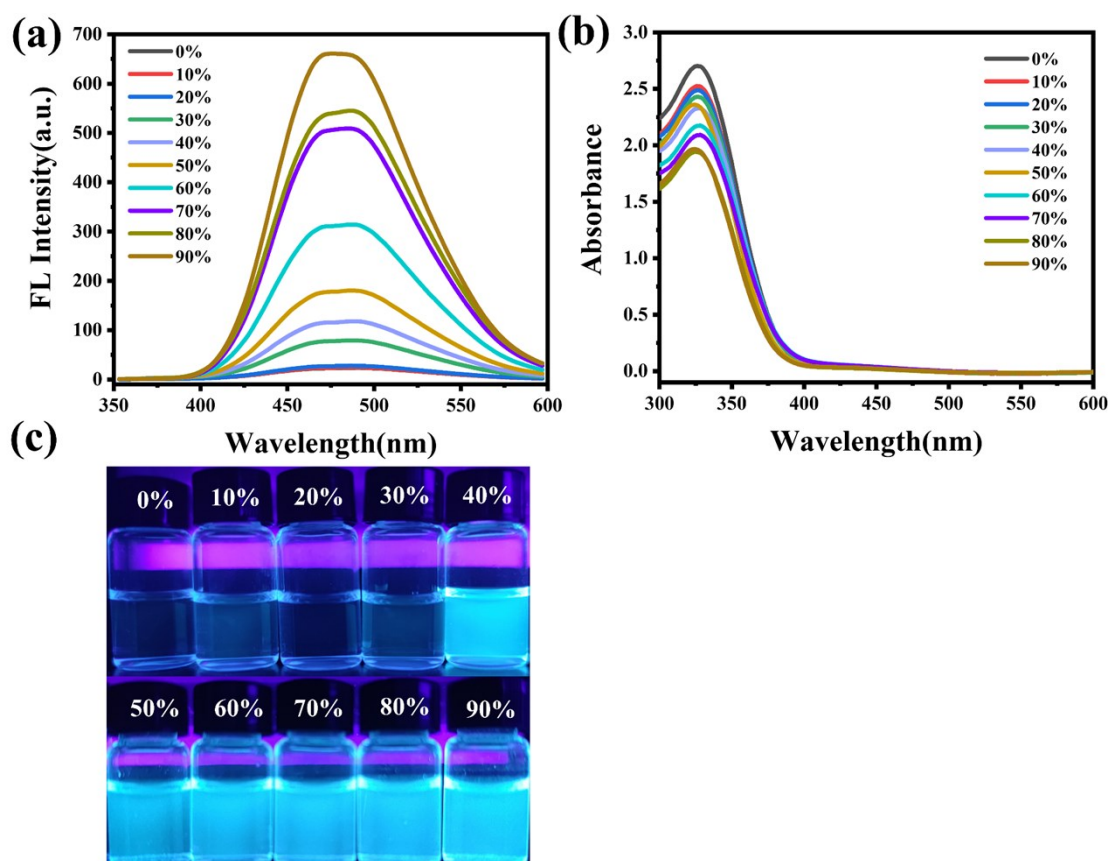


Figure S4. The fluorescence spectra ($\lambda_{\text{ex}} = 330 \text{ nm}$) (a), UV absorption spectra (b) and digital photos (c) under a hand-held UV-lamp ($\lambda_{\text{max}} = 365 \text{ nm}$) for H-PEG₁₁₂-*b*-P(Lys-TPE)₂₁ in H₂O/DMF mixtures at a concentration of 0.1 mg/mL by changing water fraction from 0% to 90%.

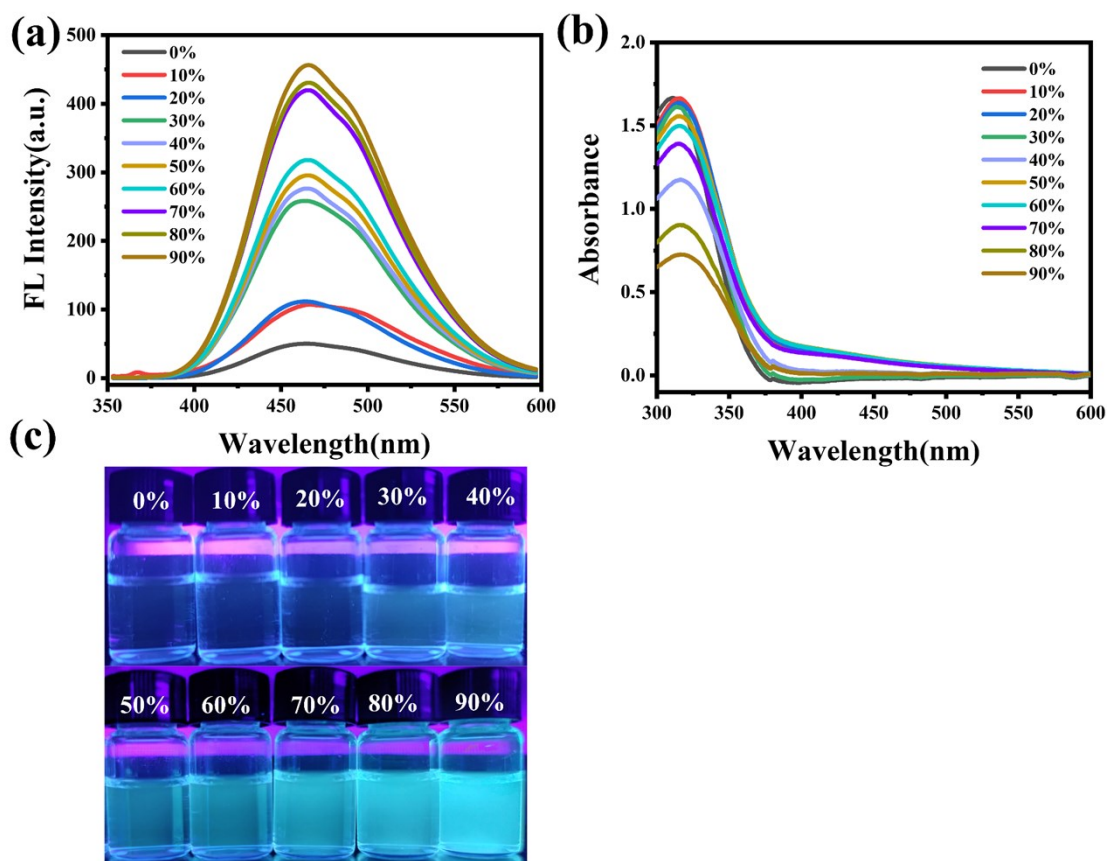


Figure S5. The fluorescence spectra ($\lambda_{\text{ex}} = 330 \text{ nm}$) (a), UV absorption spectra (b) and digital photos (c) under a hand-held UV-lamp ($\lambda_{\text{max}} = 365 \text{ nm}$) for $\text{PEG}_{112}\text{-}b\text{-P(Lys-TPE)}_{21}$ in THF/ H_2O mixtures at a concentration of 0.1 mg/mL by changing water fraction from 0% to 90%.

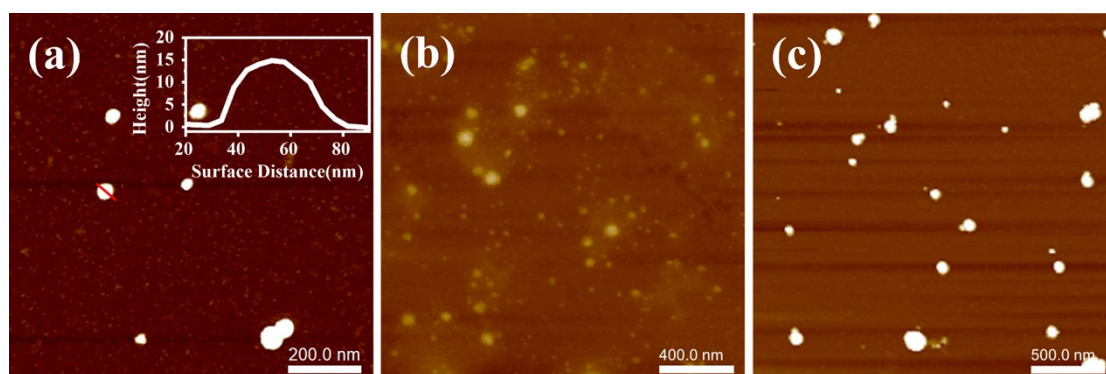


Figure S6. AFM images for $\text{PEG}_{112}\text{-}b\text{-P(Lys-TPE)}_{21}$ (0.1 mg/mL) in water content of 10% (a), 20% (b) and 90% (c) in DMF solution.

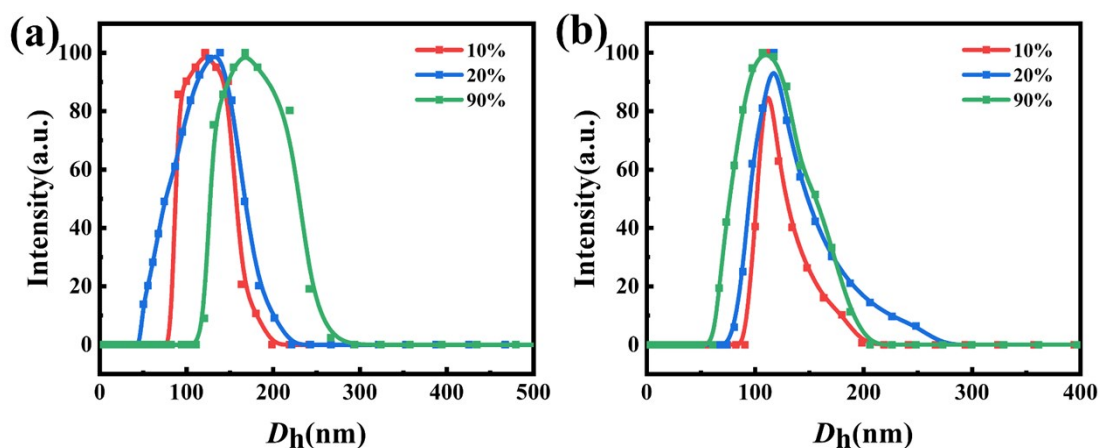


Figure S7. The D_h distributions of self-assembled PEG₁₁₂-*b*-P(Lys-TPE)₂₁ (0.1 mg/mL) in mixed solvents of H₂O/DMF with the volume ratio of water in mixture: 10%, 20% and 90% (a), in mixed solvents of H₂O/THF with the volume ratio of water in mixture: 10%, 20% and 90% (b).

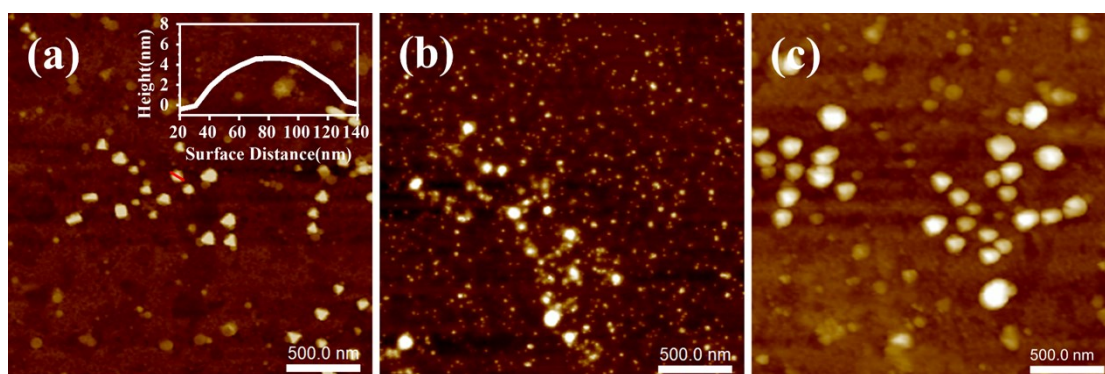


Figure S8. AFM images for PEG₁₁₂-*b*-P(Lys-TPE)₂₁ (0.1 mg/mL) in water content of 10% (a), 20% (b) and 90% (c) in THF solution.

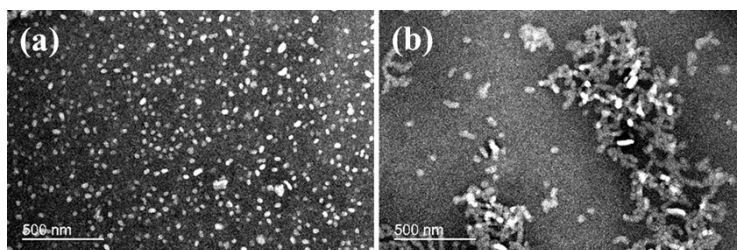


Figure S9. TEM images for H-PEG₁₁₂-*b*-P(Lys-TPE)₂₁ (0.1 mg/mL) in 10% water content (a), in 90% water content (b) DMF/ H₂O mixed solution.

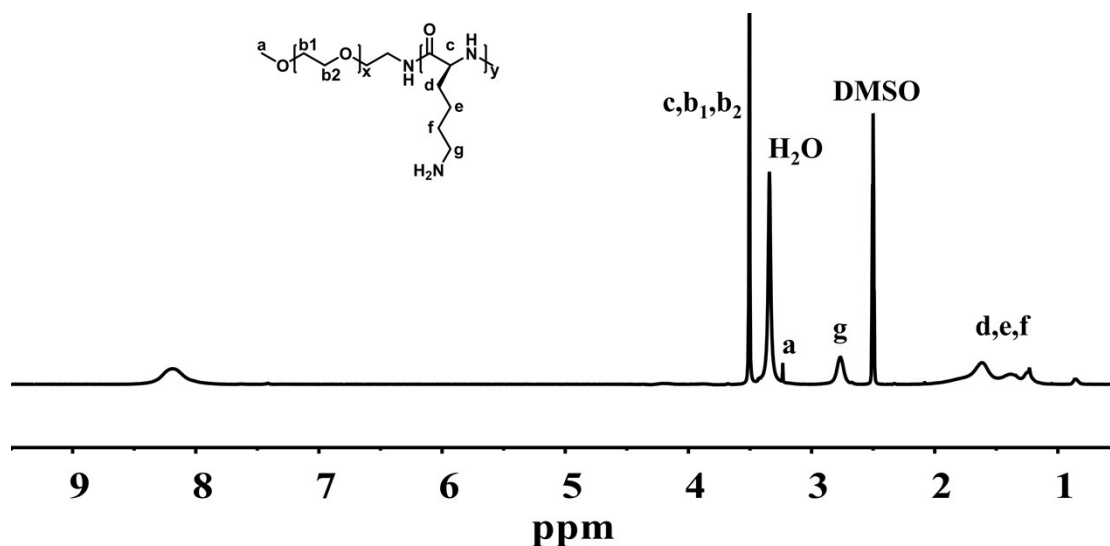


Figure S10. ^1H NMR spectrum of the sample collected from $\text{PEG}_x\text{-}b\text{-P(Lys-TPE)}_y$ solution after adding solution at pH 1.4 in DMSO.

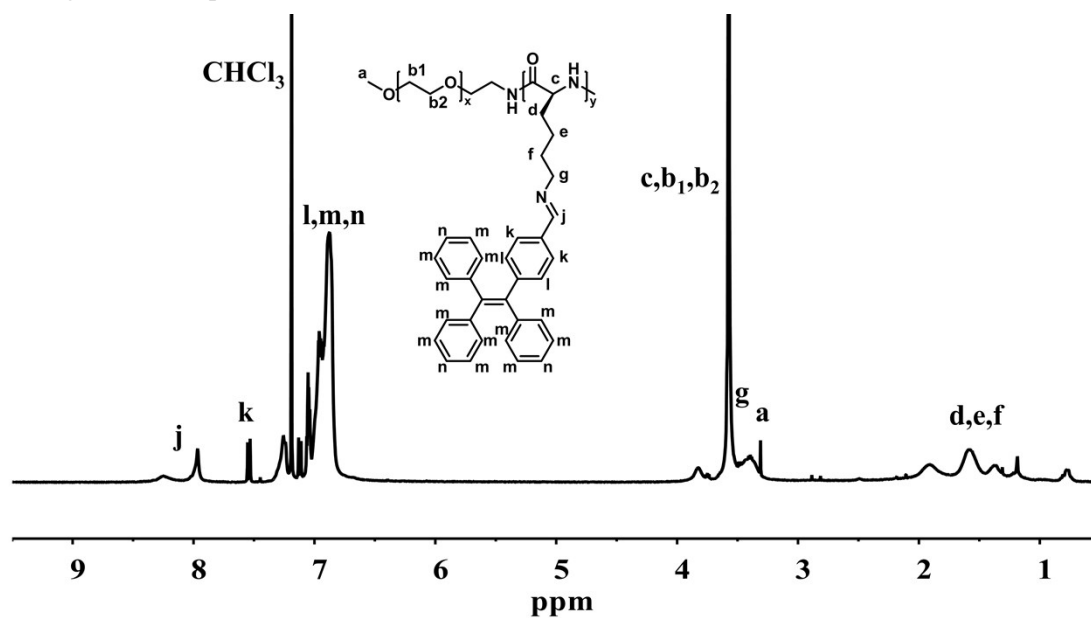


Figure S11. ^1H NMR spectrum of the sample collected from $\text{PEG}_x\text{-}b\text{-P(Lys-TPE)}_y$ solution after sequentially adding solution at pH 1.4 and 12.6 in CDCl_3 .

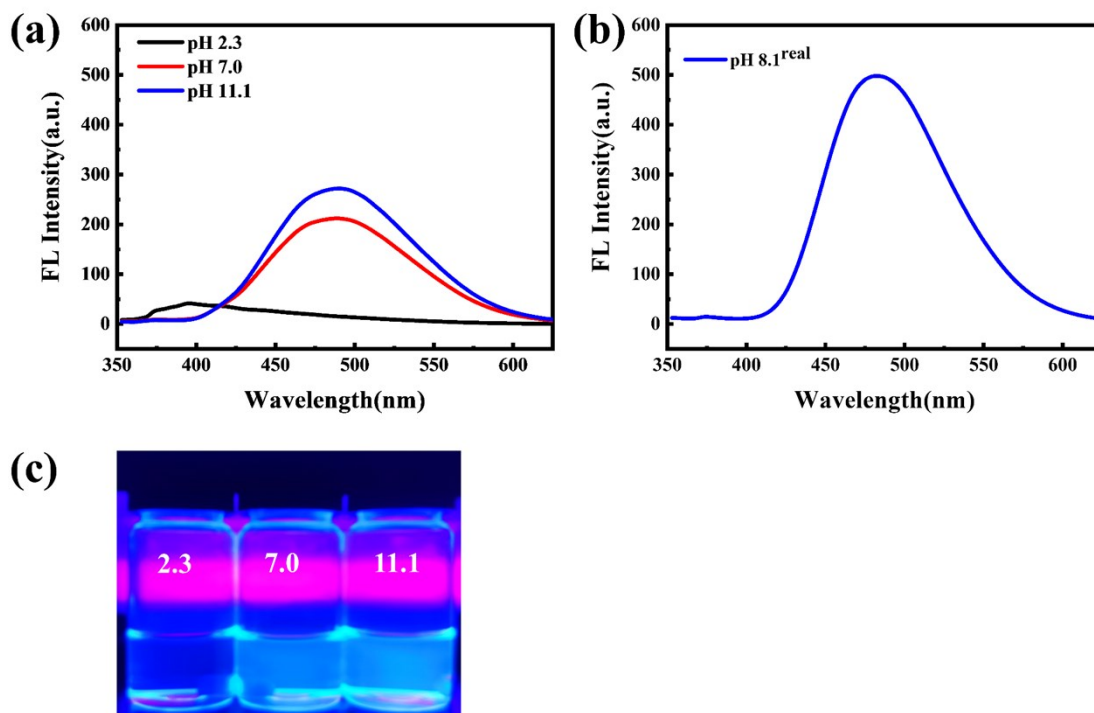


Figure S12. FL spectra ($\lambda_{\text{ex}} = 330 \text{ nm}$) for $\text{PEG}_{112}\text{-}b\text{-P(Lys-TPE)}_{21}$ solutions with addition of aqueous solution at different pH ($v_{\text{THF}}:v_{\text{H}_2\text{O}} = 9:1$, 0.1 mg/mL) (a), the mixture with addition of solution at pH 2.3, followed by adding solution at pH 11.7 with stirring (the solution pH value is determined to be ~ 8.1 after THF evaporation) (b), and digital photos under a hand-held UV-lamp ($\lambda_{\text{max}} = 365 \text{ nm}$) for $\text{PEG}_{112}\text{-}b\text{-P(Lys-TPE)}_{21}$ solutions with addition of aqueous solution at different pHs ($v_{\text{THF}}:v_{\text{H}_2\text{O}} = 9:1$, 0.1 mg/mL) (c).

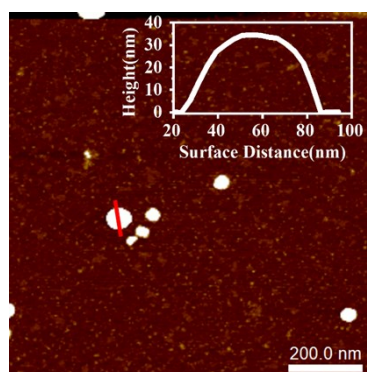


Figure S13. AFM image for $\text{PEG}_{112}\text{-}b\text{-P(Lys-TPE)}_{21}$ in DMF/H₂O mixture solvent ($v:v = 9:1$, 0.1 mg/mL) with addition of solution at pH 1.4, followed by adding solution at pH 12.6 with stirring.

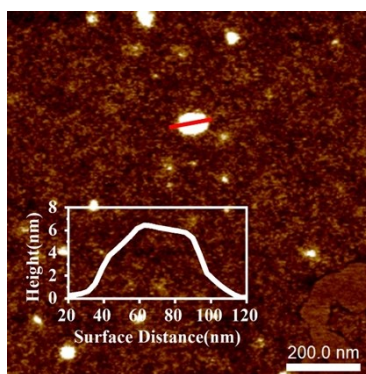


Figure S14. AFM image for PEG₁₁₂-*b*-P(Lys-TPE)₂₁ with addition of aqueous solution at pH 2.3 ($v_{\text{THF}}:v_{\text{H}_2\text{O}} = 9:1$, 0.1 mg/mL) followed by adding solution at pH 11.7 with stirring (the solution pH value is determined to be ~ 8.1 after THF evaporation).

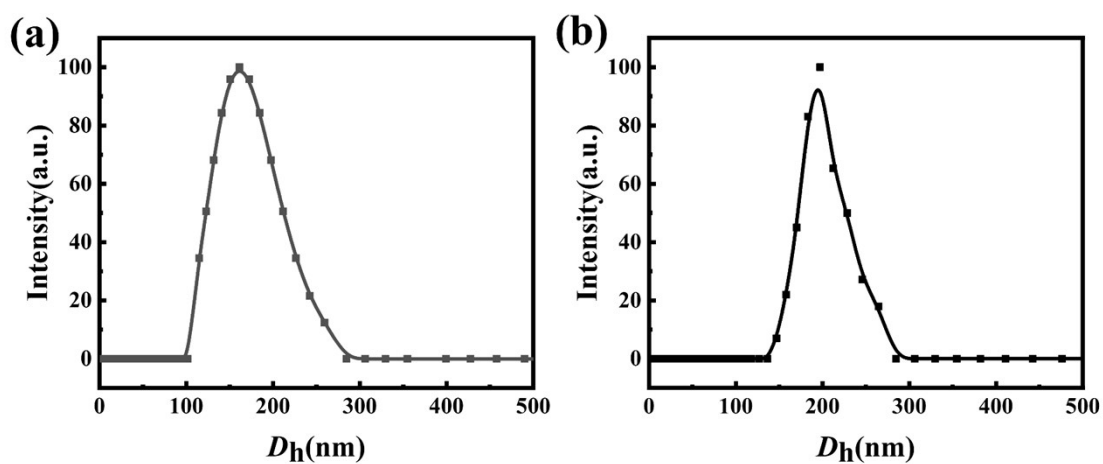


Figure S15. The D_h distributions for PEG₁₁₂-*b*-P(Lys-TPE)₂₁ solution with addition of aqueous solution at pH 1.4 ($v_{\text{DMF}}:v_{\text{H}_2\text{O}} = 9:1$, 0.1 mg/mL), followed by adding solution at pH 12.6 with stirring (a) and PEG₁₁₂-*b*-P(Lys-TPE)₂₁ solution with addition of aqueous solution at pH 2.3 ($v_{\text{THF}}:v_{\text{H}_2\text{O}} = 9:1$, 0.1 mg/mL), followed by adding solution at pH 11.7 with stirring (b) (the solution pH value is determined to be ~ 8.1 after THF evaporation).

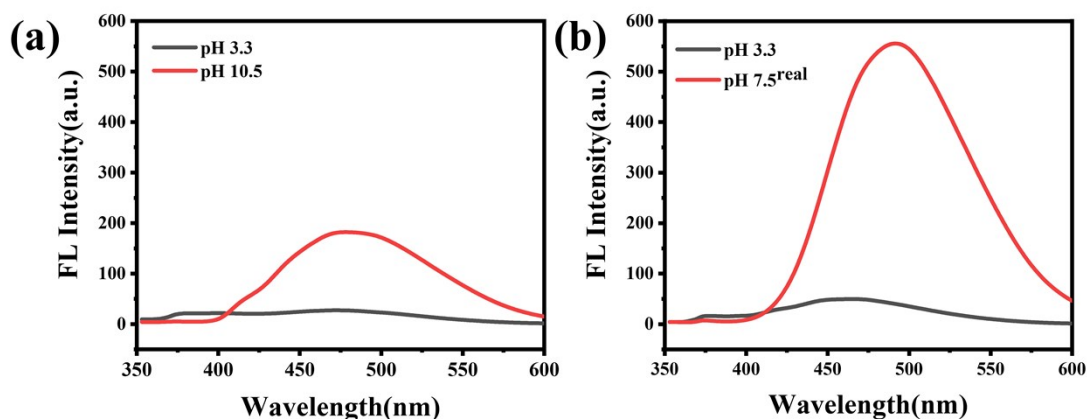


Figure S16. FL spectra ($\lambda_{\text{ex}} = 330 \text{ nm}$) for $\text{PEG}_{112}\text{-}b\text{-P(Lys-TPE)}_{21}$ solutions with addition of solution at pH 3.3, followed by adding solution at pH 10.5 with stirring in DMF/ H_2O mixture ($v : v = 9:1$, 0.1 mg/mL) (a), and with addition of solution at pH 3.3, followed by adding basic solution with stirring in THF/ H_2O mixture ($v : v = 9:1$, 0.1 mg/mL) (the solution pH value is determined to be ~ 7.5 after THF evaporation) (b).

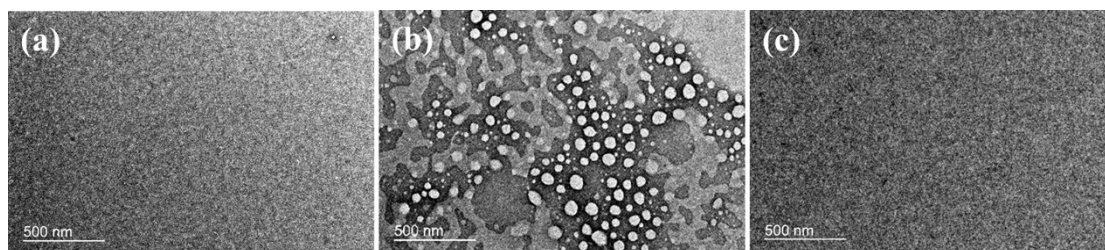


Figure S17. TEM images for $\text{PEG}_{112}\text{-}b\text{-P(Lys-TPE)}_{21}$ (0.1 mg/mL) in aqueous solution at pH of 3.5 (a), 7.0 (b) and 11.4 (c).

Table S1. Molecular parameters of diblock copolymers.

samples	Feed ratio ^a (NCA/initiator)	n^b	M_n^b (kg/mol)	M_n^c (kg/mol)	Dispersity ^c (\mathcal{D})
$\text{PEG}_{112}\text{-}b\text{-PZLyS}_{21}$	20/1	21	10.5	13.5	1.13

^aFeed molar ratio of NCA/initiator. ^bCalculated from the proton integral ratios of allyl group or propargyl group to the phenyl group by ^1H NMR spectra. ^cDetermined from GPC; n represents the average DP of $\text{PEG}_{112}\text{-}b\text{-PZLyS}_{21}$.