

Supplementary Information

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Experimental

50 mg of the mPEG-*b*-PS₂₀ copolymer and 0.25mg of Nile Red was dissolved in 2.0 mL of tetrahydrofuran (THF). The mixture was added to 5.0 mL of deionized water under vigorous stirring at 37 °C. The mixture was then transferred to a dialysis bag (MWCO 3500) and dialyzed for 24 h. After that, the Nile Red loaded nanoparticle solution was concentrated to the concentration of 2.0 mg mL⁻¹.

The H₂O₂-triggered release profiles of Nile Red from the mPEG-*b*-PS₂₀ nanoparticles were studied using a dialysis bag (MWCO = 3500) in different media. Typically, Nile Red-loaded nanoparticle solution (3 mL) in a dialysis bag was suspended in 27 mL PBS solution at different H₂O₂ concentration of 100, 200 and 400mM, and gently shaken at 37 °C in a thermostatic rotary shaker at 100 rpm. The H₂O₂ solution was withdrawn at predetermined time intervals for analysis, and replenished each time with an equal volume of fresh H₂O₂ solution to keep a constant volume of the medium. The content of Nile Red that was released into the H₂O₂ solution was measured by quantifying the absorbance of Nile Red using fluorescence emission spectra. The data were averaged with three independent measurements.

Results and discussion

Table S1 Synthesis of mPEG-*b*-PS copolymer

Sample ^[a]	mPEG/mg	MS/mg	N-435/mg	Methylbenzene/ml	Time/h	T/°C
mPEG ₄₅ - <i>b</i> -PS ₅	400	296	30	3	8	65
mPEG ₄₅ - <i>b</i> -PS ₈	250	296	30	3	8	65
mPEG ₄₅ - <i>b</i> -PS ₁₀	200	296	30	3	8	65
mPEG ₄₅ - <i>b</i> -PS ₁₅	133.3	296	30	3	8	65
mPEG ₄₅ - <i>b</i> -PS ₂₀	100	296	30	3	8	65

mPEG ₄₅ - <i>b</i> -PS ₃₀	66	296	30	3	8	65
mPEG ₄₅ - <i>b</i> -PS ₅₀	40	296	30	3	8	65

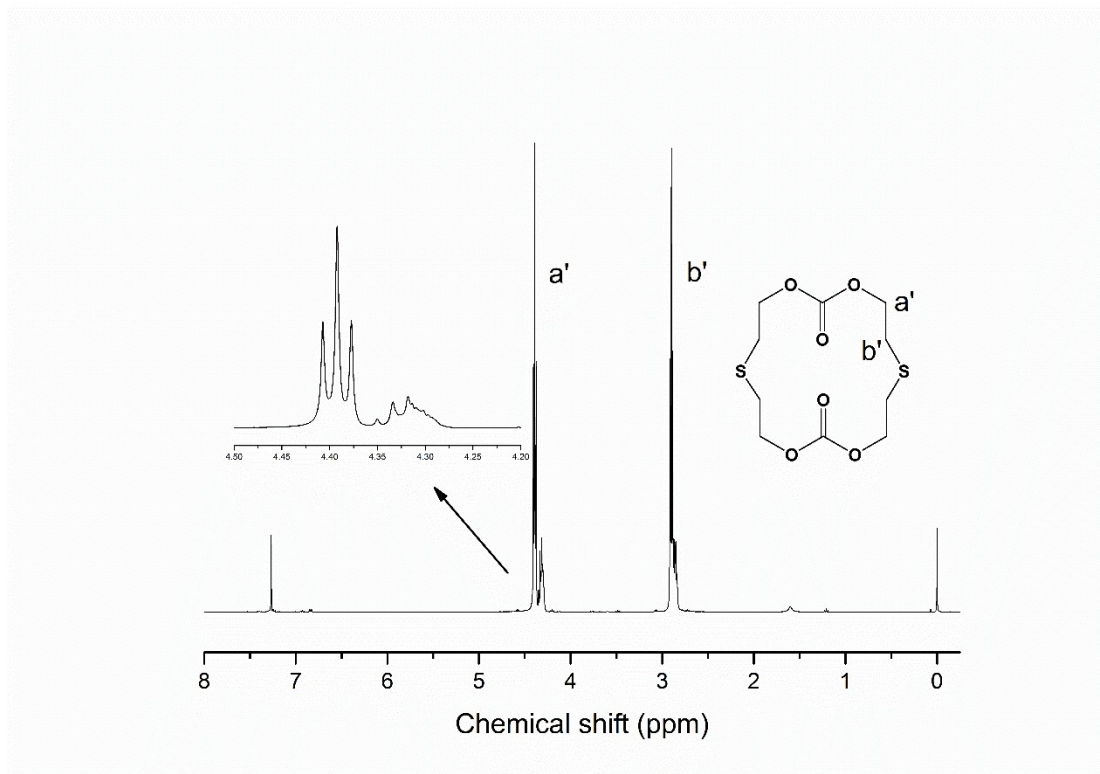


Fig. S1 ¹H NMR spectrum of MS monomer crude product

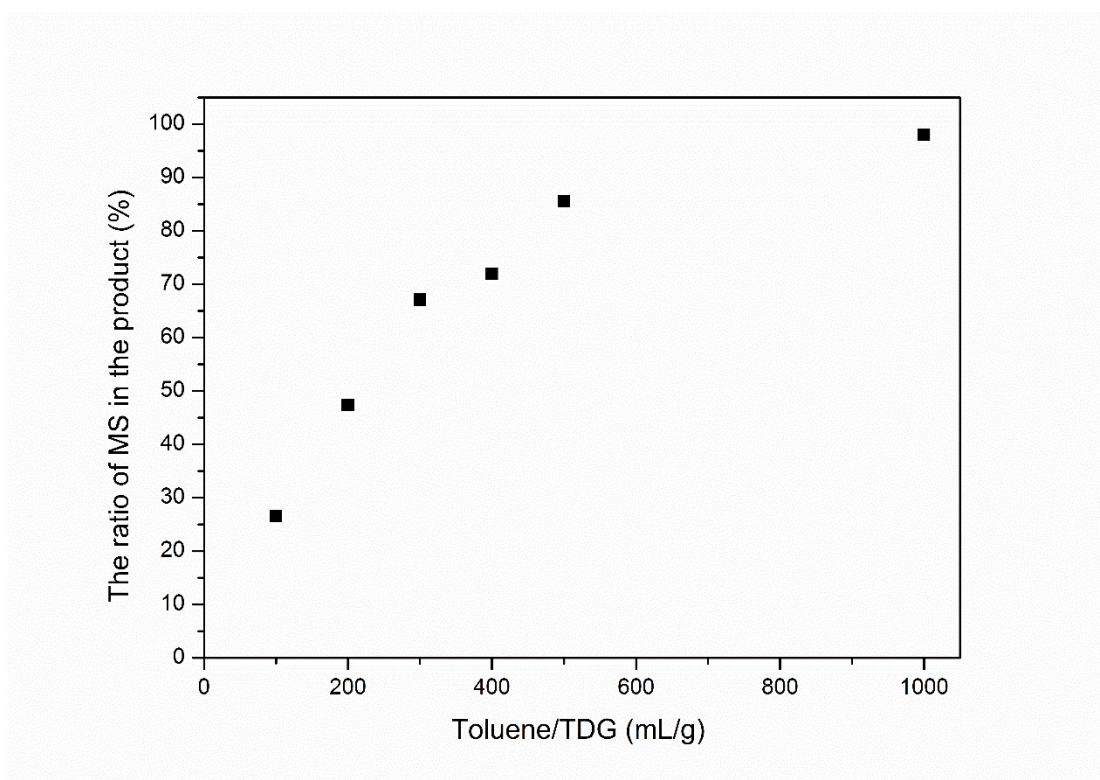


Fig. S2 The ratio of MS monomer in the crude product

FT-IR (ν , cm^{-1}): 1744 cm^{-1} (C=O). ^1H NMR (400MHz, CDCl_3 , δ , ppm): $\delta=4.39$ (t, OCH_2 , 8H), 2.90 (m, $\text{OCH}_2\text{CH}_2\text{S}$, 8H); ^{13}C NMR (500MHz, CDCl_3): $\delta=154.96$ (CO), 67.01 ($\text{OCH}_2\text{CH}_2\text{S}$), 31.31 ($\text{OCH}_2\text{CH}_2\text{S}$).

HRMS (ESI, m/z): M^+ Calculated for $\text{C}_{10}\text{H}_{16}\text{O}_6\text{S}_2\text{Na}$, 319.0286; found, 319.0288.

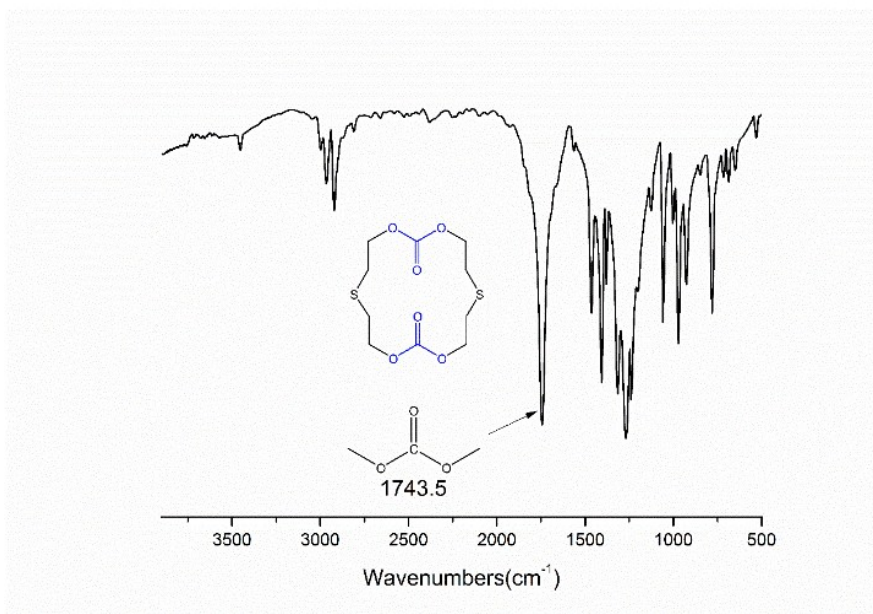


Fig. S3 FT-IR of MS monomer

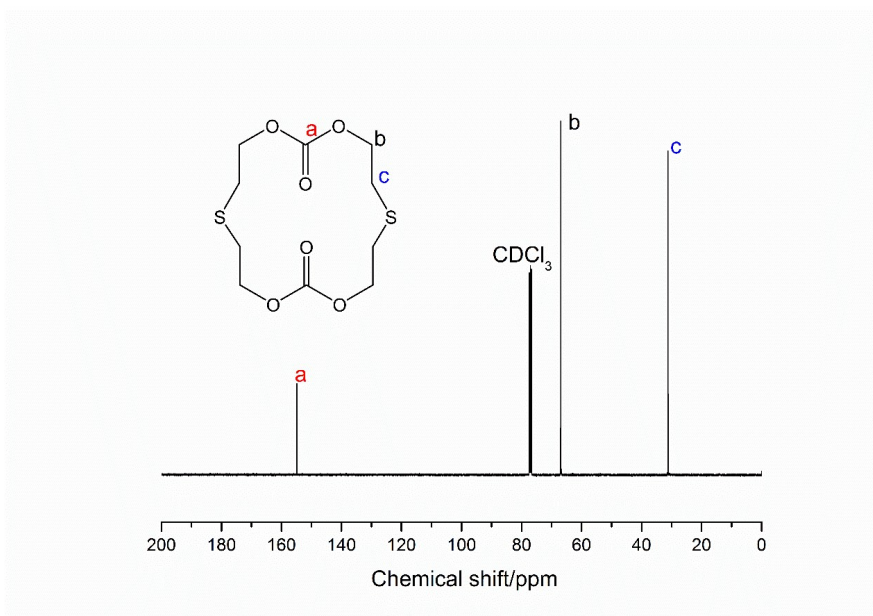


Fig. S4 ^{13}C NMR spectrum of MS monomer

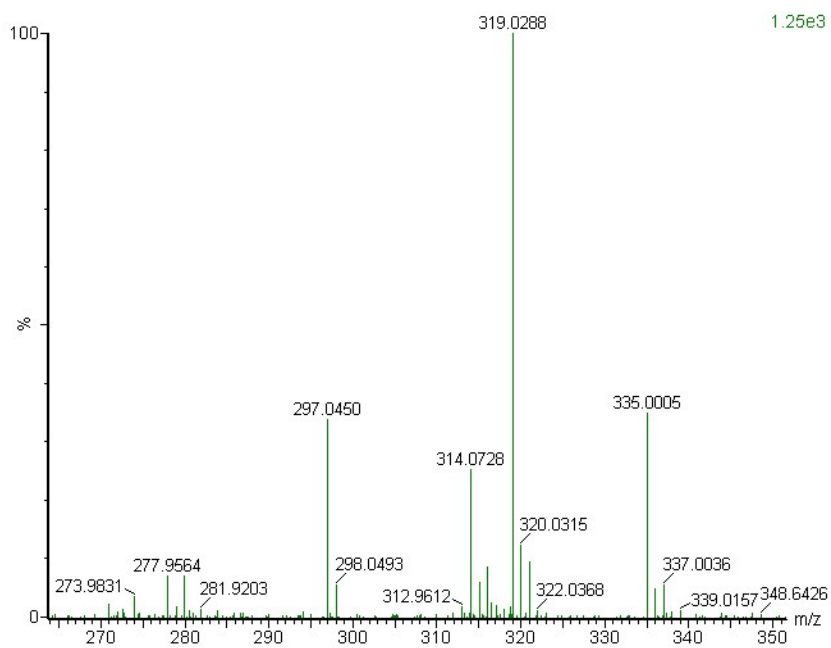
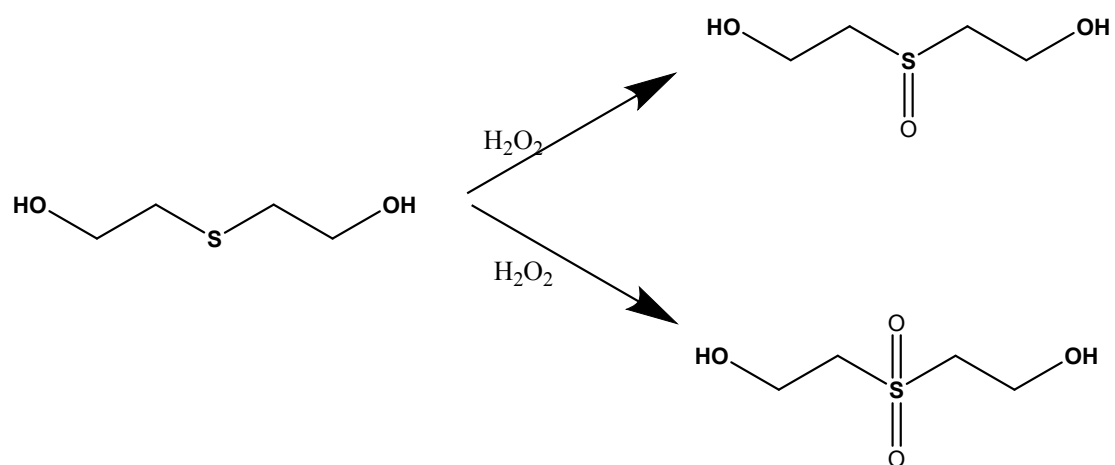


Fig. S5 ESI of MS monomer

The oxidation of TDG



Scheme S1. The oxidation of TDG

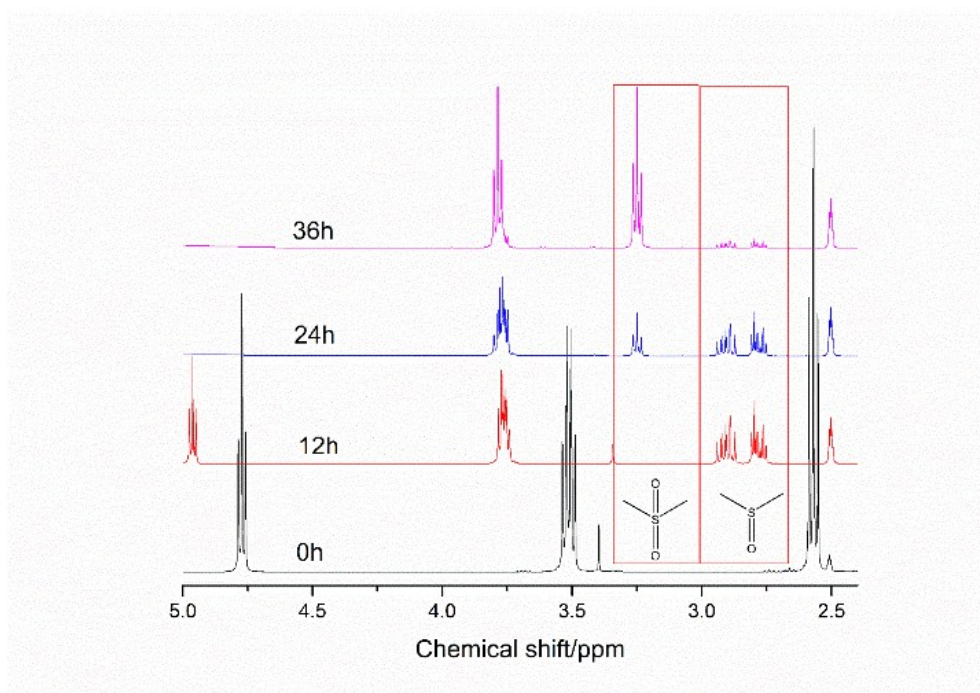


Fig. S6. Selection of time-resolved ^1H NMR spectra of the oxidation of TDG with 200 mM H_2O_2 .

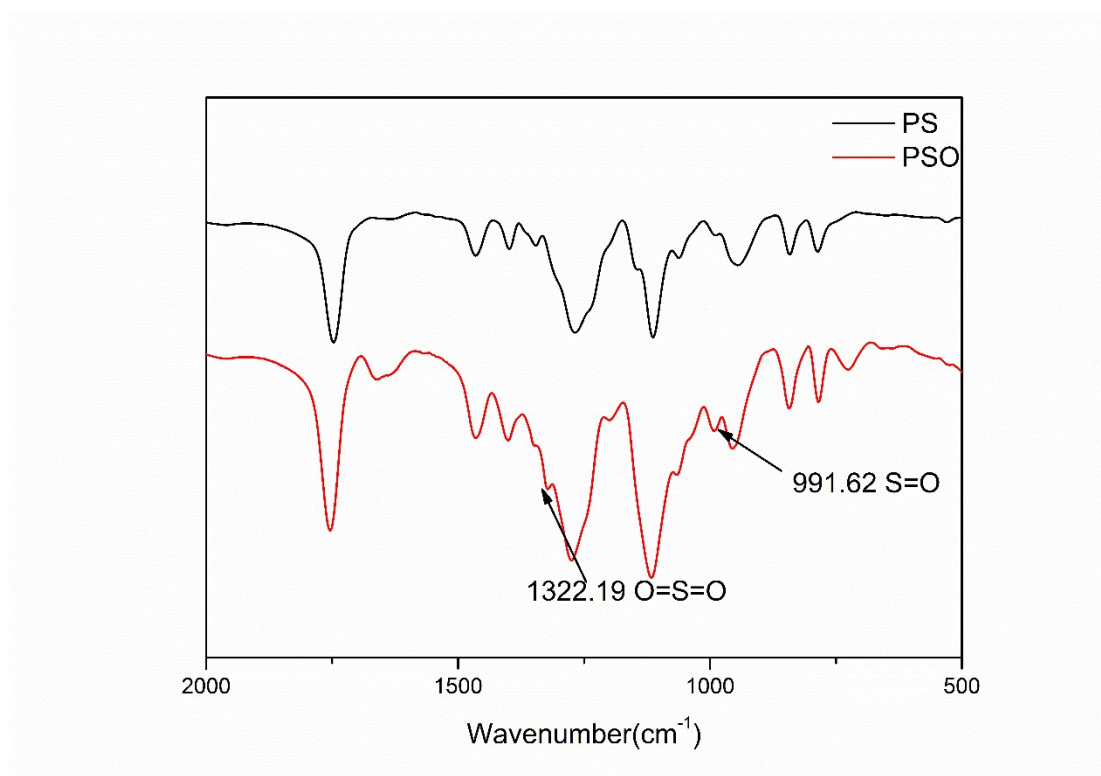


Fig. S7 The FT-IR comparison of mPEG-*b*-PS (PS) and mPEG-*b*-OPS (PSO).

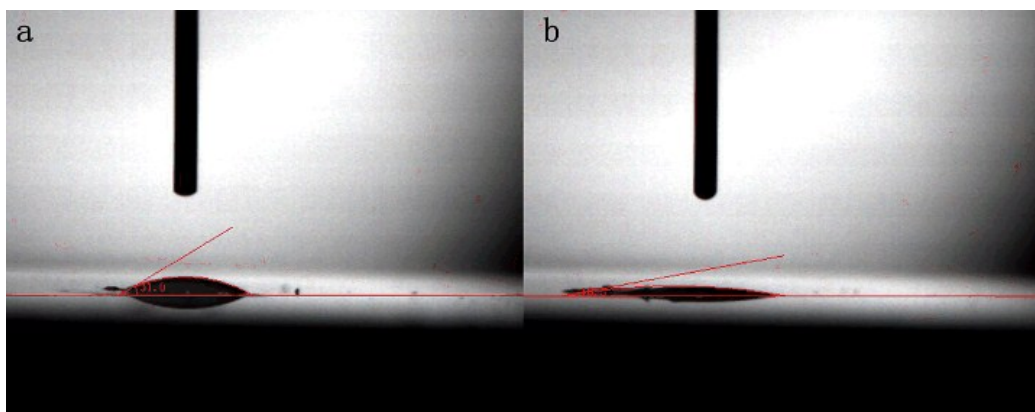


Fig. S8 The contact angle comparison of mPEG-*b*-PS (a) and mPEG-*b*-OPS (b).

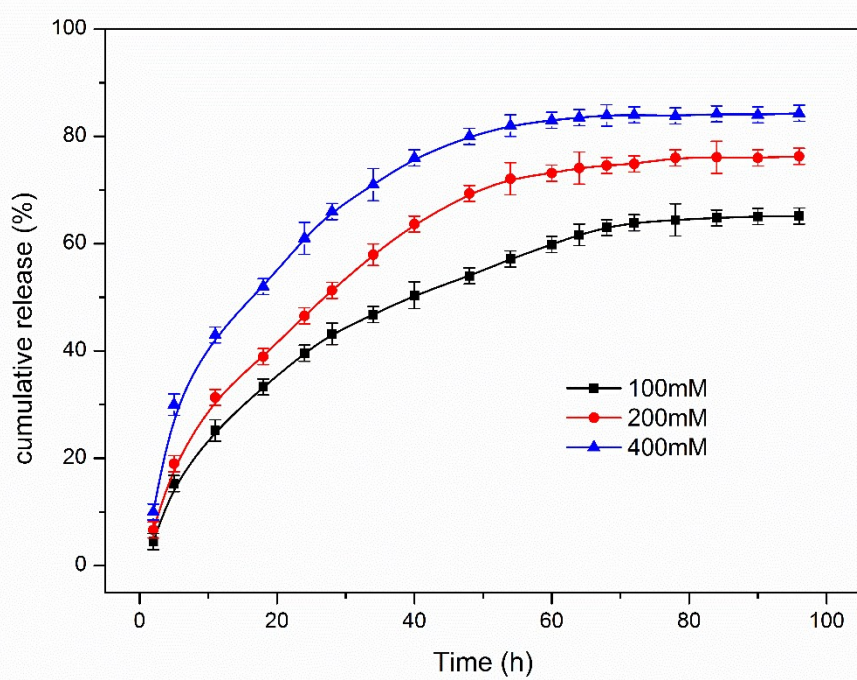


Fig. S9 *In vitro* Nile Red release from mPEG-*b*-PS nanoparticles at different H₂O₂ concentrations at 37 °C.