Supplementary Information

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Experimental

50 mg of the mPEG-b-PS\textsubscript{20} 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\textsuperscript{-1}.

The H\textsubscript{2}O\textsubscript{2}-triggered release profiles of Nile Red from the mPEG-b-PS\textsubscript{20} 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 H\textsubscript{2}O\textsubscript{2} concentration of 100, 200 and 400mM, and gently shaken at 37 °C in a thermostatic rotary shaker at 100 rpm. The H\textsubscript{2}O\textsubscript{2} solution was withdrawn at predetermined time intervals for analysis, and replenished each time with an equal volume of fresh H\textsubscript{2}O\textsubscript{2} solution to keep a constant volume of the medium. The content of Nile Red that was released into the H\textsubscript{2}O\textsubscript{2} 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

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<th>Sample\textsuperscript{[a]}</th>
<th>mPEG/mg</th>
<th>MS/mg</th>
<th>N-435/mg</th>
<th>Methylbenzene/ml</th>
<th>Time/h</th>
<th>T/°C</th>
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Fig. S1 $^1$H NMR spectrum of MS monomer crude product

Fig. S2 The ratio of MS monomer in the crude product
FT-IR (v, cm$^{-1}$): 1744 cm$^{-1}$ (C=O). $^1$H NMR (400MHz, CDCl$_3$, δ, ppm): δ=4.39 (t, OCH$_2$, 8H), 2.90 (m, OCH$_2$CH$_2$S, 8H); $^{13}$C NMR (500MHz, CDCl$_3$): δ=154.96 (CO), 67.01 (OCH$_2$CH$_2$S), 31.31 (OCH$_2$CH$_2$S).

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

Fig. S3 FT-IR of MS monomer

Fig. S4 $^{13}$C NMR spectrum of MS monomer
The oxidation of TDG

Scheme S1. The oxidation of TDG
Fig. S6. Selection of time-resolved $^1$H NMR spectra of the oxidation of TDG with 200 mM H$_2$O$_2$.

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 H$_2$O$_2$ concentrations at 37 °C.