Electronic Supplementary Information

Multi-hierarchical Responsive Polymers: Stepwise Oxidation of a

Selenium- and Tellurium-Containing Block Copolymer with Sensitivity

to both Chemical and Electrochemical Stimuli

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Characterization and control experiment

1. ¹H NMR characterization of the selenium- and tellurium-containing copolymer Se-Te-PEG2000.



Figure S1. ¹H NMR spectrum of Se-Te-PEG2000 (400 MHz, CDCl₃).

 δ (ppm): 4.15 (t, NH₂COO*CH*₂(CH₂)₁₀Se), 4.13 (t, NH₂COO*CH*₂(CH₂)₁₀Te), 3.64 (t, O*CH*₂CH₂ of PEG), 2.62 (t, Te*CH*₂CH₂), 2.54 (t, Se*CH*₂CH₂), 2.19 (SeCH₂CH₂, TeCH₂CH₂), 1.77-1.19 (m, NHCOOCH₂(CH₂)₈CH₂CH₂Se, NHCOOCH₂(CH₂)₈CH₂CH₂Te).

2. GPC characterization of the selenium- and tellurium-containing copolymer.



3. CAC of Se-Te-PEG2000 measured by the concentration-dependent DLS measurement.



Figure S3. CAC measurements by the concentration-dependent DLS.

4. DLS measurements of micelle suspension before and after oxidation by H_2O_2 for 5 h.



Figure S4. DLS plots of micelle suspension before and after oxidation by H_2O_2 of different concentration for 5 h.

5. Cryo-TEM images of micelle suspension after oxidation H₂O₂ of different concentrations and periods.



Figure S5. Cryo-TEM images of the Se-Te-PEG2000 after oxidation by H_2O_2 of different concentration and period. (a) 500 μ M, 12 h. (b) 1 mM, 12 h. (c) 5 mM, 12 h.(d) 10 mM, 12 h. Scale bar: 500 nm.

6. Current-time curves during electrochemical oxidation process of Se-Te-PEG2000.



Figure S6. Current-time curves during electrochemical oxidation of Se-Te-PEG2000. (a) 0.6 V, 24 h. (b) 1.2 V, 24 h.