Electronic Supplementary Material (ESI) for Polymer Chemistry.


# Supporting Information

*Oxidation-responsive micelles by a one-pot polymerization-induced self-assembly approach*

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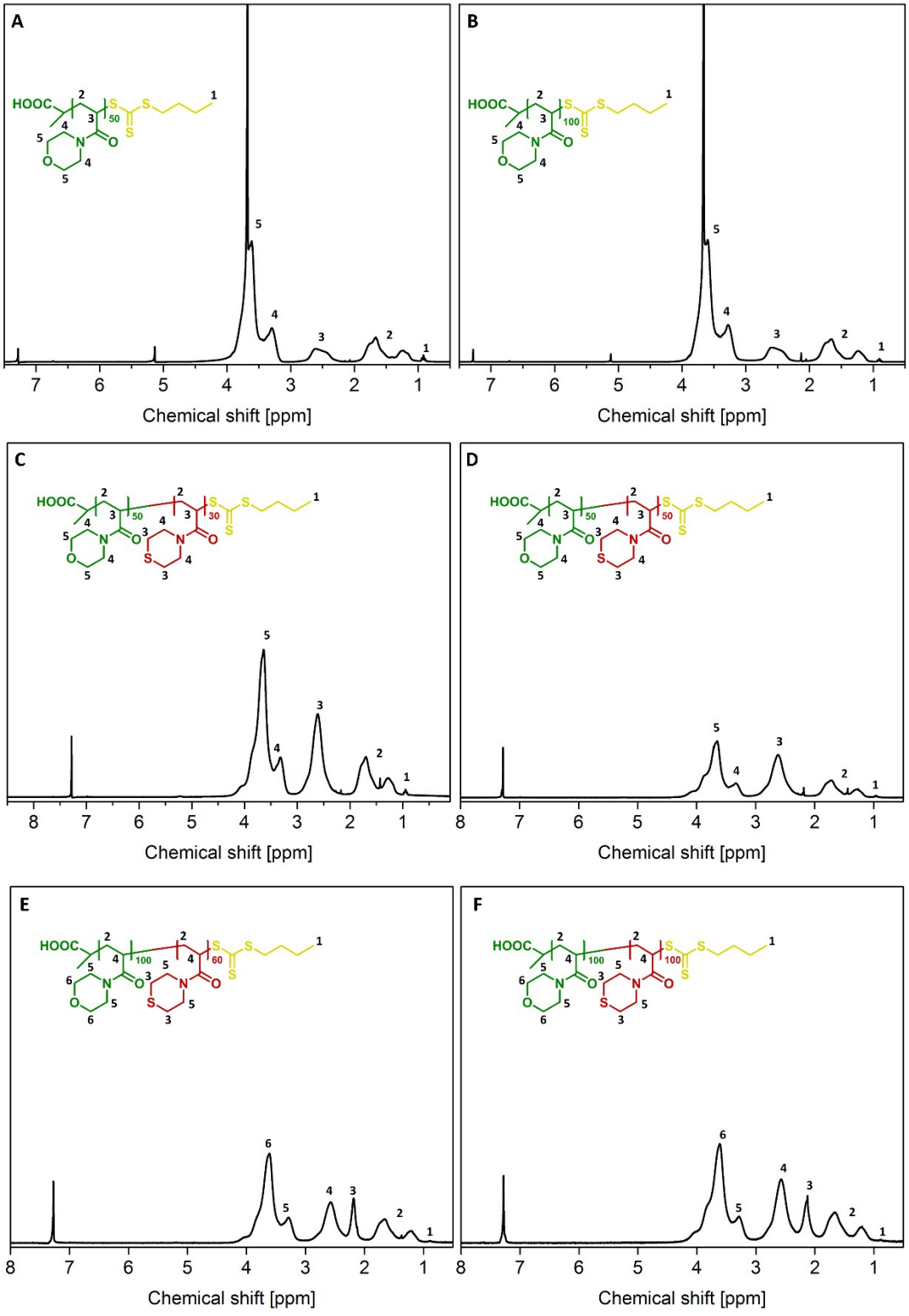


## Results

**Figure S1.** $^{1}$H-NMR (300 MHz, $CDCl_3$) spectra of the synthesized polymers. A) $PNAM_{50}$ (**P1**). B) $PNAM_{100}$ (**P2**). C) $P(NAM_{50}$-*b*-$NAT_{30})$ (**P3**). D) $P(NAM_{50}$-*b*-$NAT_{50})$ (**P4**). E) $P(NAM_{100}$-*b*-$NAT_{60})$ (**P5**). F) $P(NAM_{100}$-*b*-$NAT_{100})$ (**P6**).

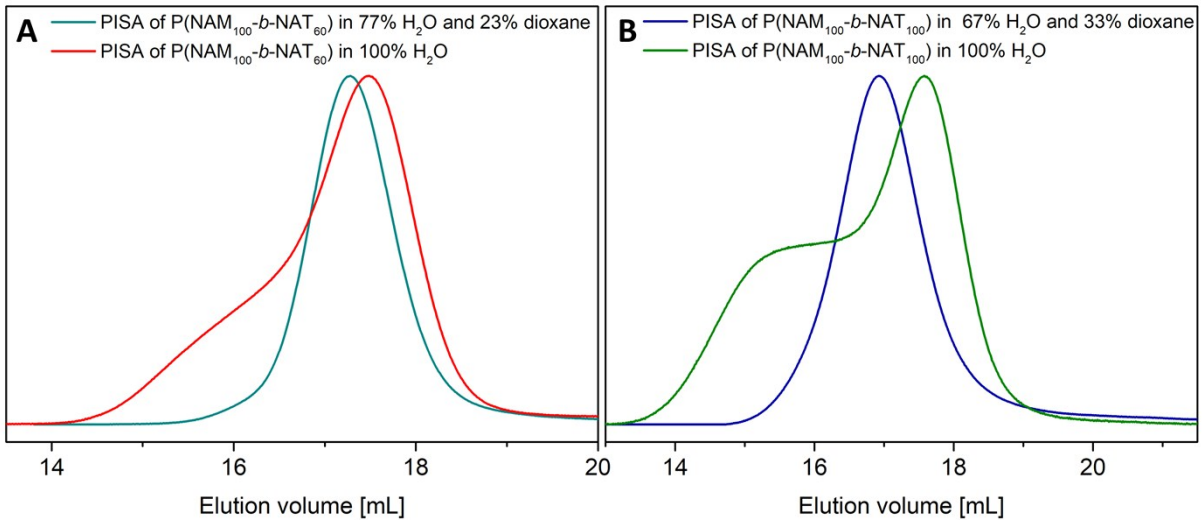


**Figure S2.** SEC traces of the block copolymers **P5** and **P6** polymerized in different solvents. A) PISA of **P5** in 77% $H_2O$ and 23% 1,4-dioxane compared to 100% $H_2O$. B) PISA of **P6** in 66% $H_2O$ and 33% 1,4-dioxane compared to 100% $H_2O$.

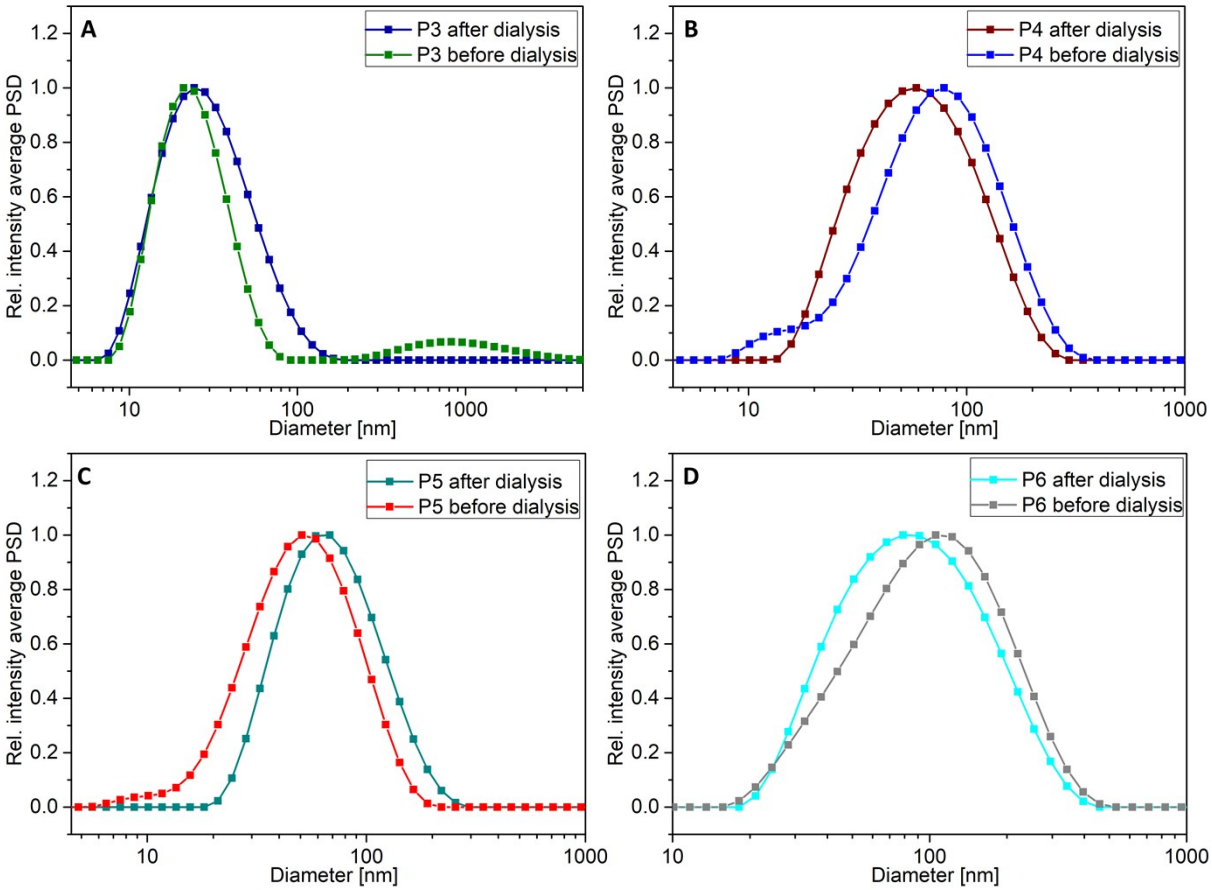

**Figure S3.** DLS analysis (n = 3, c ~ 1 mg $mL^{-1}$) of the micelles **P3** (A), **P4** (B), **P5** (C) and **P6** (D) before and after dialysis.

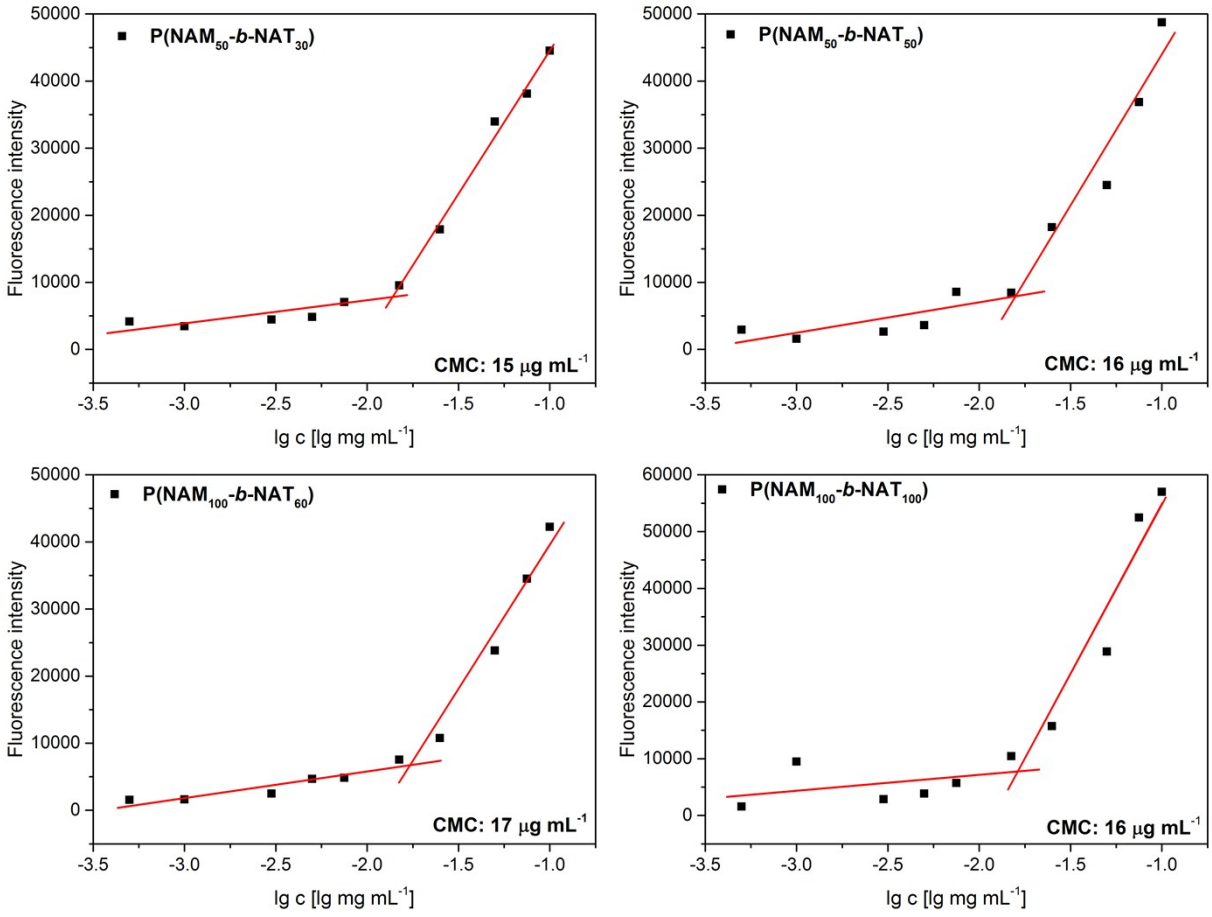


**Figure S4.** CMC of the nanostructures determined by fluorescence quenching of Nile red. The CMC was determined as the point of intersection of the linear plots in the emission intensity *versus* log c spectrum.

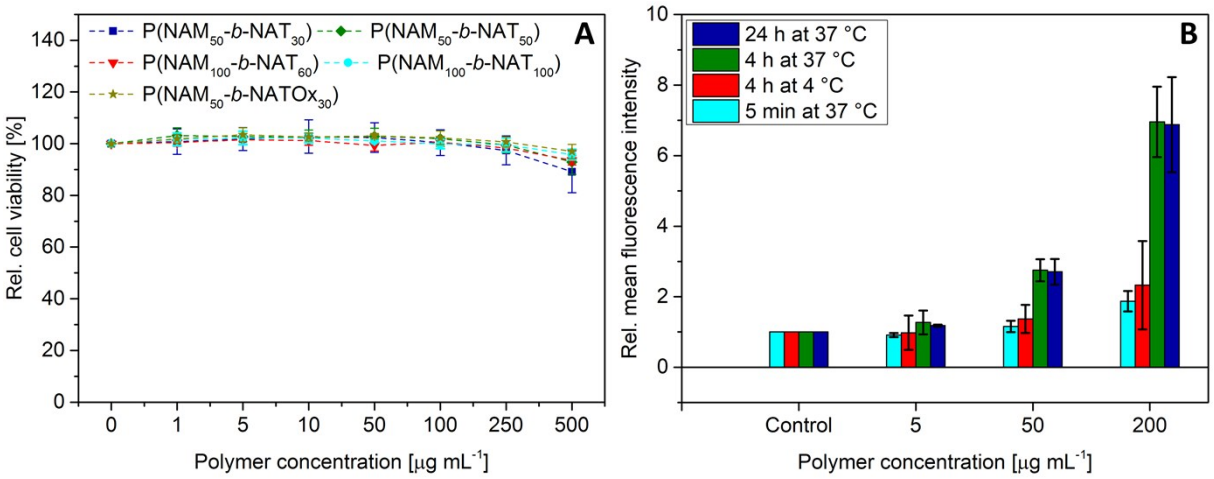


**Figure S5.** Evaluation of the toxicity and uptake behavior of the synthesized micelles. A) Cytotoxicity assay of the polymers **P3**–**P6** and of the oxidized form P($NAM_{50}$-*b*-$NATOx_{30}$). Non-treated cells served as 100% relative viability. L929 cells were treated 24 h with the indicated concentrations of the polymers. Values represent the mean ± S.D. (n=3). B) Uptake study on P($NAM_{50}$-*b*-$NAT_{30}$) (**P3**) with encapsulated Nile red. HEK cells were treated at 4 and 37 °C and at 5 min, 4 and 24 h with the indicated concentrations of the polymer. Values represent the mean ± S.D. (n=3).

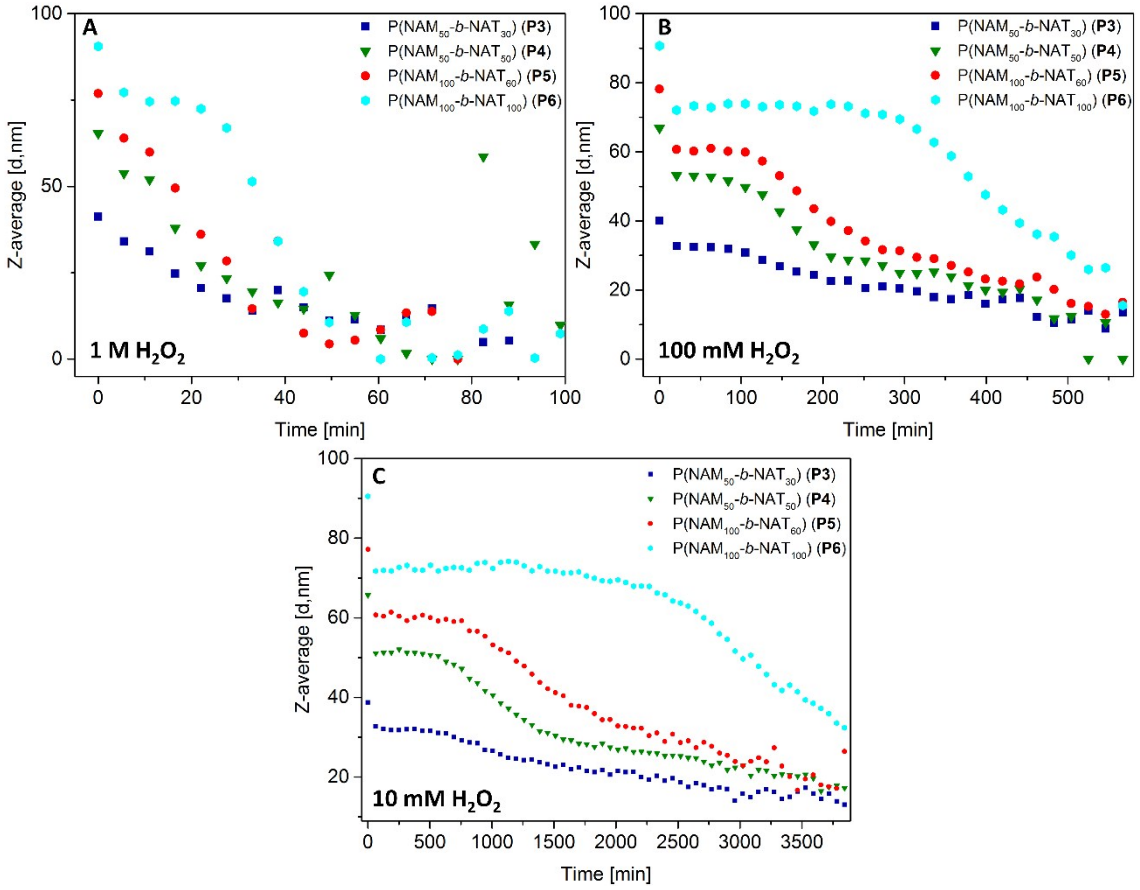


**Figure S6.** Oxidation-induced degradation of the micelles **P3** to **P6** analyzed by DLS (37 °C, in PBS). A) The decrease of the Z-average diameter at 1 M $H_2O_2$. B) The decrease of the Z-average diameter at 100 mM $H_2O_2$. C) The decrease of the Z-average diameter at 10 mM $H_2O_2$.

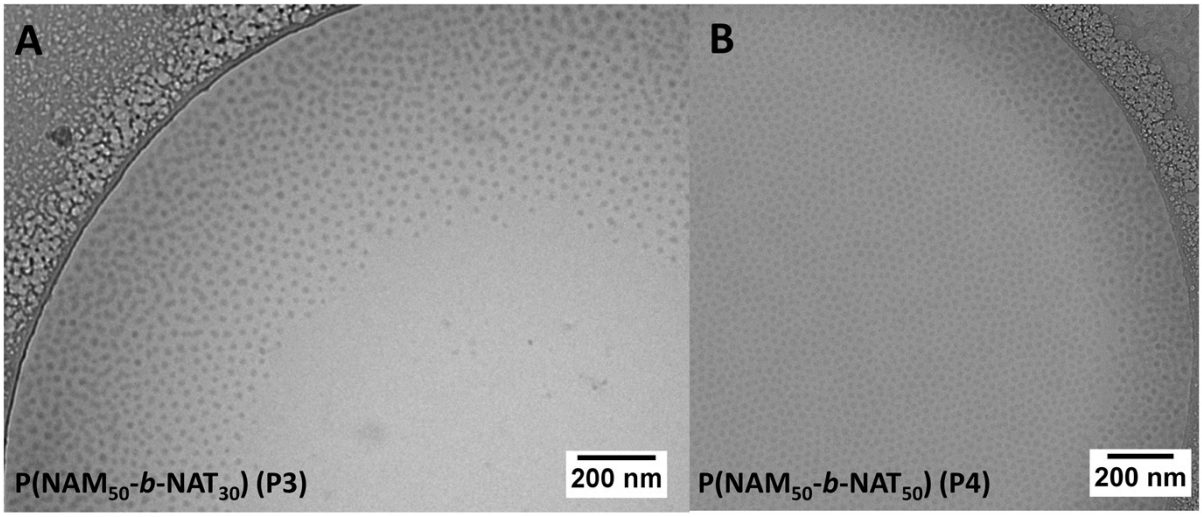


**Figure S7.** Cryo-TEM images of polymers **P3** (A) and **P4** (B).

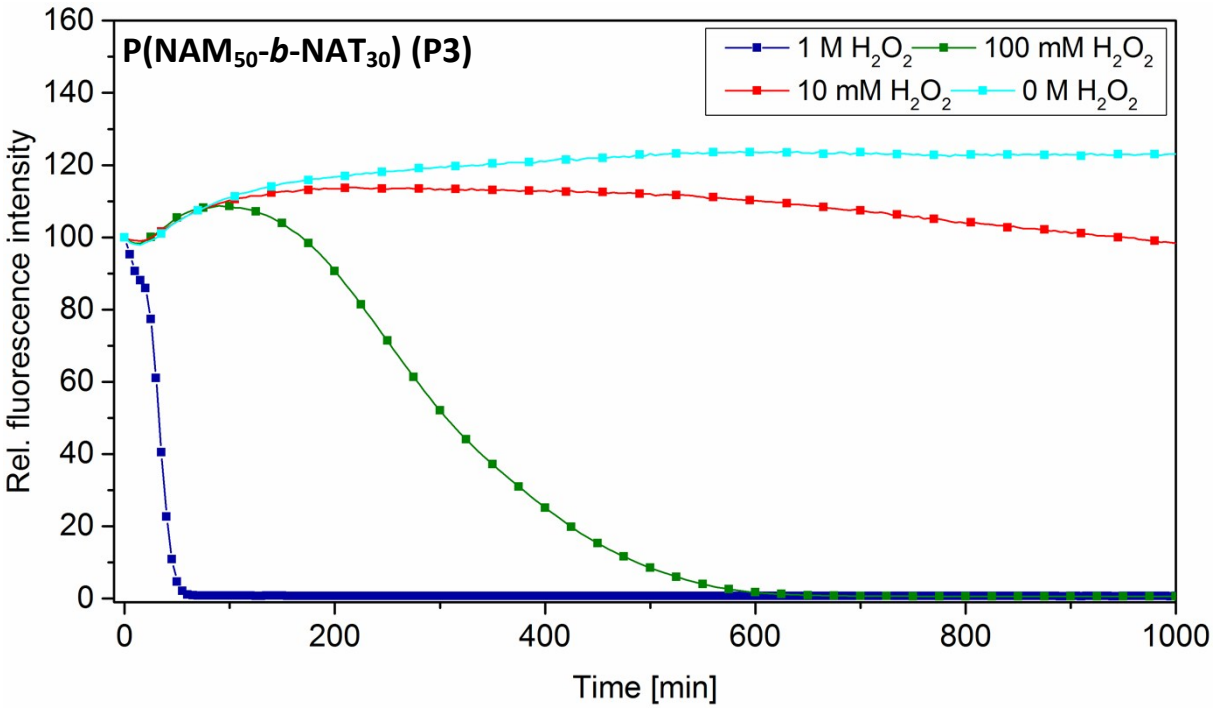


**Figure S8.** Oxidation-induced fluorescence quenching of encapsulated Nile red in the micelle **P3**, which was analyzed by fluorescence spectroscopy (37 °C).

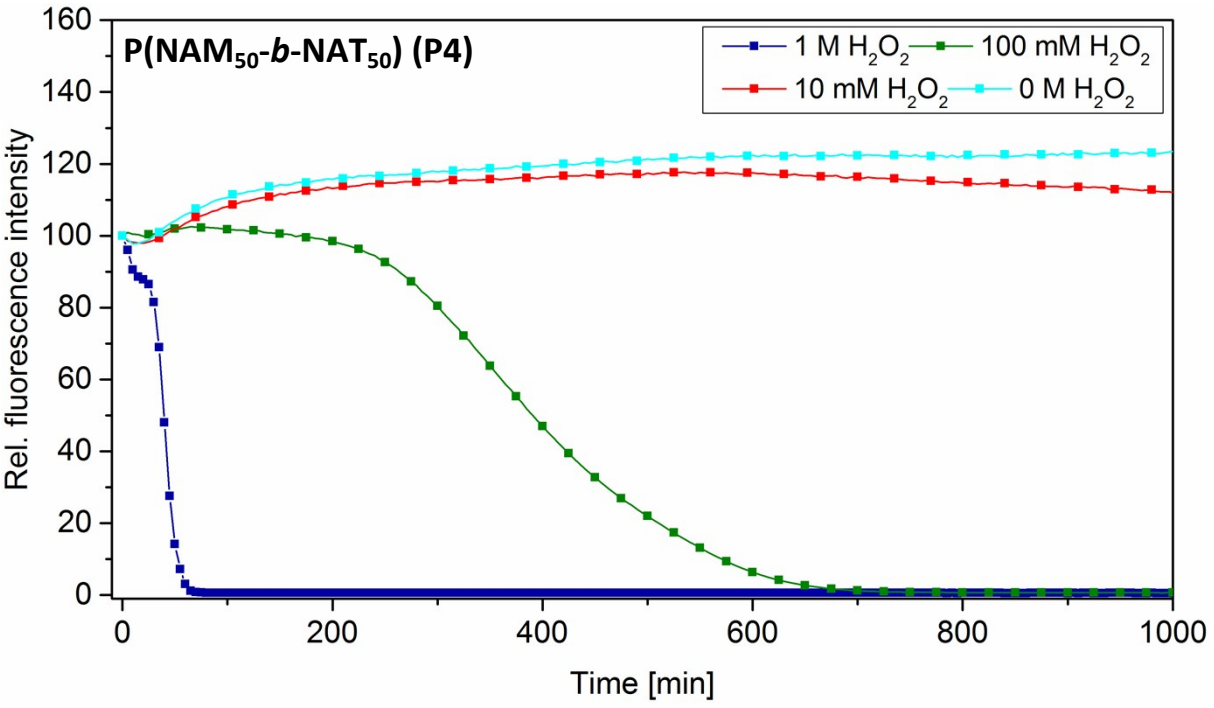


**Figure S9.** Oxidation-induced fluorescence quenching of encapsulated Nile red in the micelle **P4**, which was analyzed by fluorescence spectroscopy (37 °C).

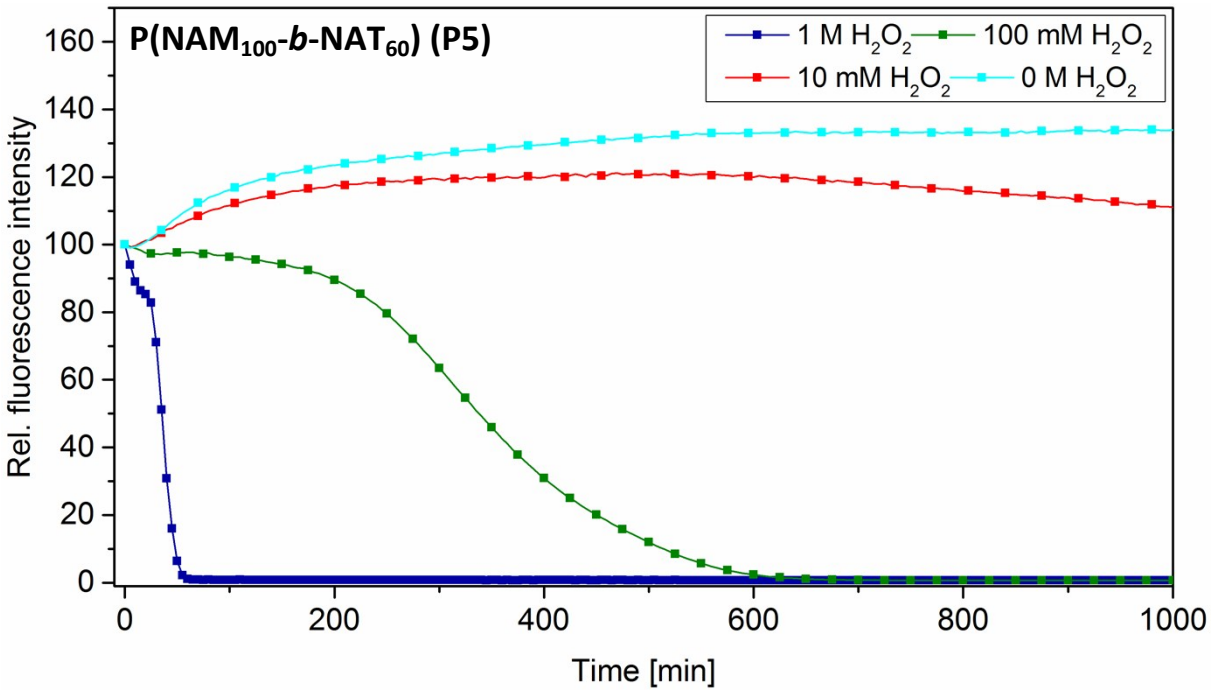


**Figure S10.** Oxidation-induced fluorescence quenching of encapsulated Nile red in the micelle **P5**, which was analyzed by fluorescence spectroscopy (37 °C).

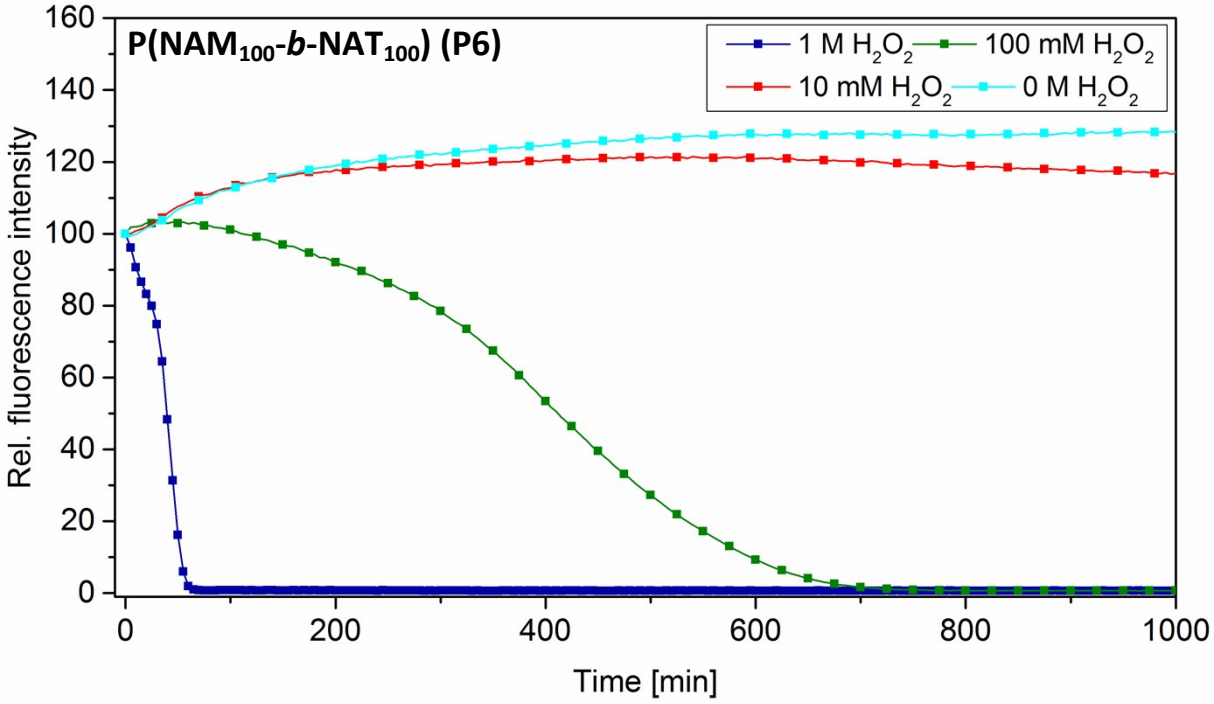


**Figure S11.** Oxidation-induced fluorescence quenching of encapsulated Nile red in the micelle **P6**, which was analyzed by fluorescence spectroscopy (37 °C).