SUPPORTING INFORMATION

Cytocompatible In Situ Cross-Linking of Degradable LbL Films Based on Thiol-Exchange Reaction

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Table S1. Zeta potential measurement of of MSNP@CLM$^{n/m}$.

Sample	Zeta Potential (mV)
MSNP@CLM$^{1/0}$	3.93±0.40
MSNP@CLM$^{1/1}$	-40.03±0.35
MSNP@CLM$^{2/1}$	3.54±0.28
MSNP@CLM$^{2/2}$	-32.78±0.64
MSNP@CLM$^{3/2}$	3.85±0.34
MSNP@CLM$^{3/3}$	-29.62±0.74
MSNP@CLM$^{4/3}$	5.85±0.24
MSNP@CLM$^{4/4}$	-35.77±0.24
MSNP@CLM$^{5/4}$	6.11±0.11
MSNP@CLM$^{5/5}$	-32.41±0.52

Table S2. Concentration of pyridinethione (byproduct) in the supernatant.

Sample	Concentration (M)
MSNP@CLM$^{1/1}$	3.37×10^{-4} M
MSNP@CLM$^{2/1}$	2.04×10^{-4} M
MSNP@CLM$^{2/2}$	1.91×10^{-4} M
MSNP@CLM$^{3/2}$	2.12×10^{-4} M
MSNP@CLM$^{3/3}$	2.66×10^{-4} M
MSNP@CLM$^{4/3}$	2.63×10^{-4} M
MSNP@CLM$^{4/4}$	4.40×10^{-4} M
MSNP@CLM$^{5/4}$	3.27×10^{-4} M
MSNP@CLM$^{5/5}$	4.43×10^{-4} M

Table S3. Dynamic light scattering (DLS) measurement of MSNP@CLM$^{n/n}$.

Sample	Particle size (nm)
MSNP@CLM$^{1/1}$	112.3±1.72
MSNP@CLM$^{2/2}$	151.0±3.00
MSNP@CLM$^{3/3}$	159.6±2.54
MSNP@CLM$^{4/4}$	168.8±4.40
MSNP@CLM$^{5/5}$	185.0±10.00

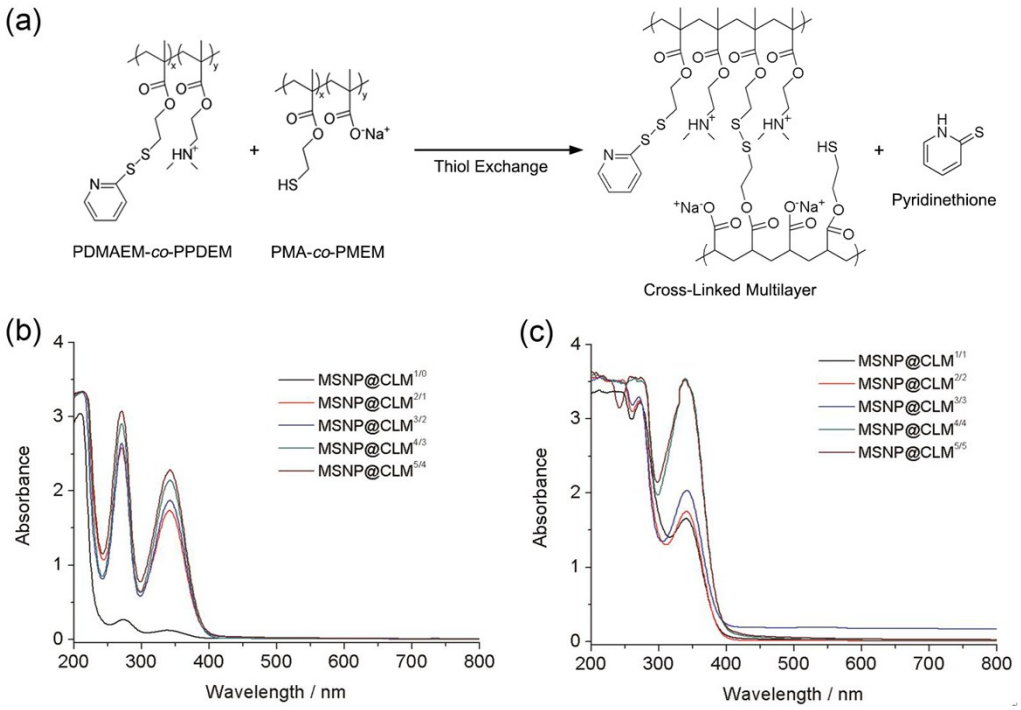

Figure S1. (a) Scheme for *in situ* cross-linking reaction generating pyridinethione as a byproduct. (b and c) UV-Vis spectra for pyridinethione after CLM formation. The absorption at 343 nm was used to measure the amount of pyridinethione. (b) UV-Vis spectra after coating with the disulfide-containing polymer, PDMAEM-*co*-PPDEM. (c) UV-Vis spectra after coating with the thiol-containing polymer, PMA-*co*-PMEM.

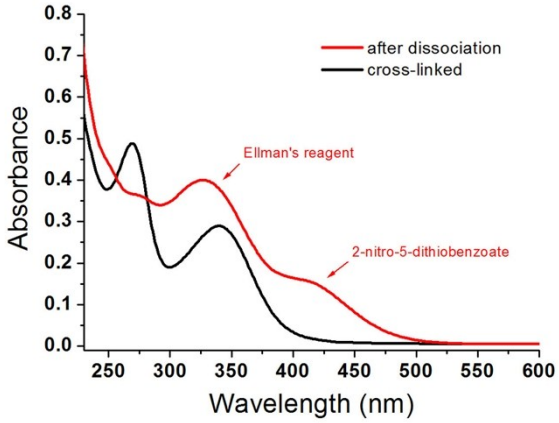

Figure S2. (a) UV-Vis spectra of the phosphate-buffered solutions (pH 7.4) of PDMEAM-*co*-PPDEM (0.1 mg/mL) and PMA-*co*-PMEM (0.1 mg/mL) in the presence of Ellman's reagent (50 mM) before and after 30 min GSH-treatment (1 mM). To remove the remaining GSH, from the solution, the polymer was purified by dialysis before UV-Vis analysis.

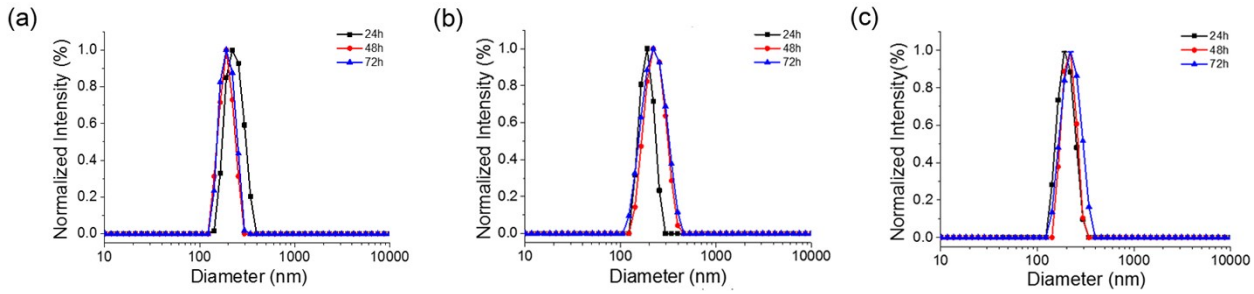

Figure S3. DLS measurements of MSNP@CLM$^{5/5}$ in (a) sodium acetate buffer (pH 5.0), (b) phosphate-buffered saline (pH 7.4), and (c) RPMI 1640 with 10% FBS medium after 24 h, 48 h and 72 h of immersion. The size of MSNP@CLM$^{5/5}$ was not changed during the periods of time, indicating the long-term colloidal stability of the particles in the biocompatible environments.

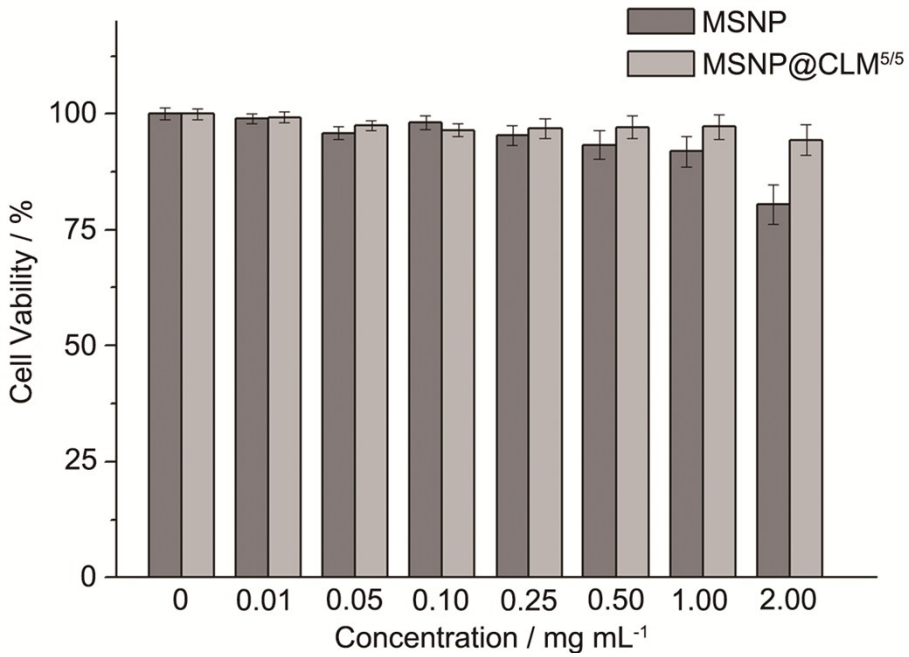

Figure S4. Viability of HeLa Cells with different concentrations of MSNPs and MSNP@CLM$^{5/5}$ after 24-h incubation.

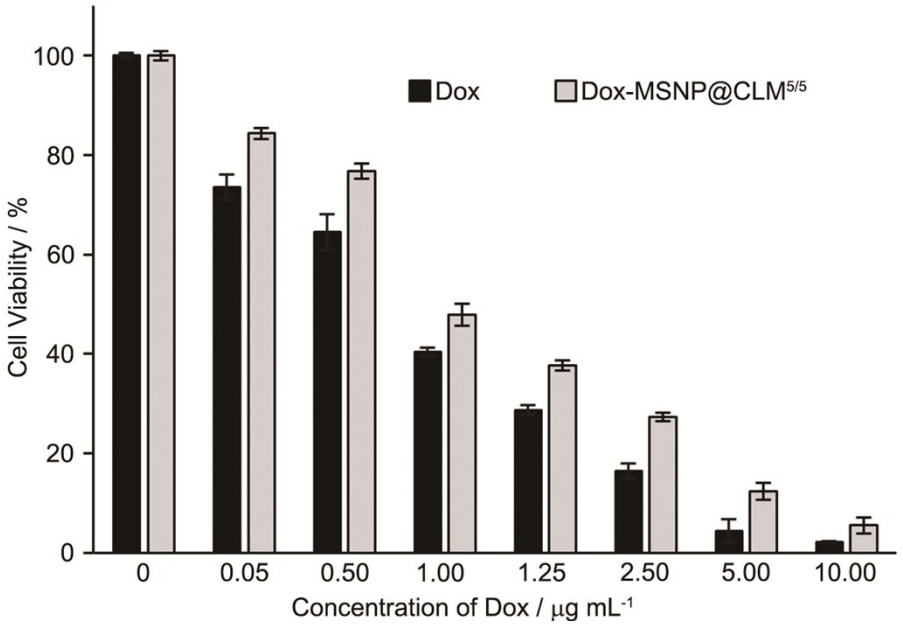

Figure S5. Cell-viability analysis of HeLa cells after 24 h of incubation with Dox-containing MSNP@CLM$^{5/5}$.