## pH Sensitive Polyelectrolyte Complex Micelles for Highly Effective Combination Chemotherapy

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## Methods

## **Physical characterization**

The FT-IR spectrum was recorded using a Thermo Scientific Nicolet Nexus 670 FT-IR Spectrometer and Smart iTR with a diamond window (Thermo Fisher Scientific Inc., Waltham, MA). The crystallinity index of samples was obtained using an X-ray diffractometer (X'Pert PRO MPD diffractometer, Almelo, the Netherlands) with a copper anode (Cu K $\alpha$  radiation) as the source of radiation.

Fluorescence spectrophotometer (Perkin Elmer LS 55, USA) was employed to record the fluorescence spectra of combined free drugs and MTX/DOX-M at an excitation wavelength ( $\lambda_{ex}$ ) of 480 and 610 nm for DOX and MTX, respectively, and with a bandwidth of 5 nm for excitation and emission.



**Figure S1**. Particle size of MTX/DOX-loaded PEC micelles prepared with low molecular weight PEO-*b*-PAA (MW 6700) at R=0.5. Note: at lower feeding ratio (R=0.25), micelles were unstable.



**Figure S2**. Entrapment efficiency of different ratio of MTX and DOX at two charge ratios (R=0.25 and 0.5).



**Figure S3**. Release profiles of MTX-loaded micelles at pH 7.4 and 5.0. The drug-loaded micelles were prepared with PEO-*b*-PAA (MW 6700) at R=0.5.



Figure S4. Stability analysis of MTX/DOX-M in terms of particle size and PDI at 4°C