

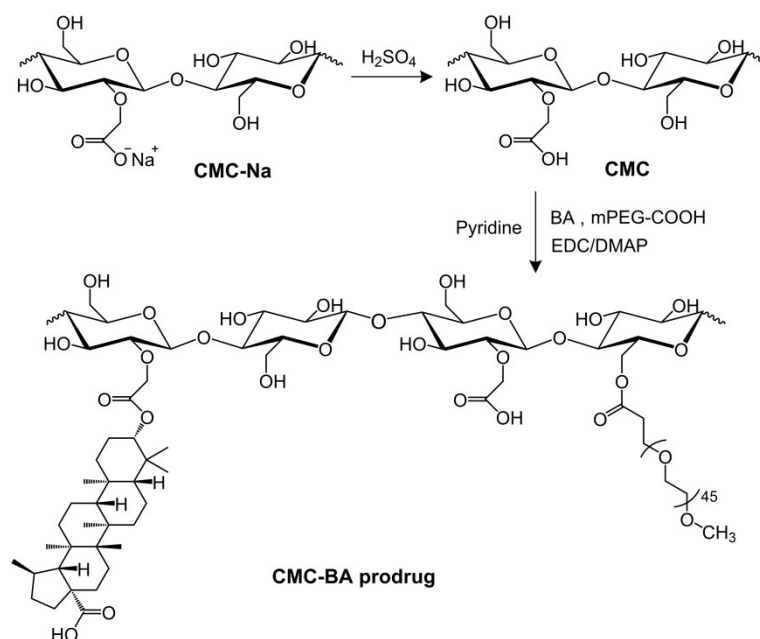
## Supporting Information

### Self-assembled PEG-carboxymethylcellulose nanoparticles/ $\alpha$ -cyclodextrin hydrogels for injectable and thermosensitive drug delivery

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#### Synthesis of CMC-BA Prodrug

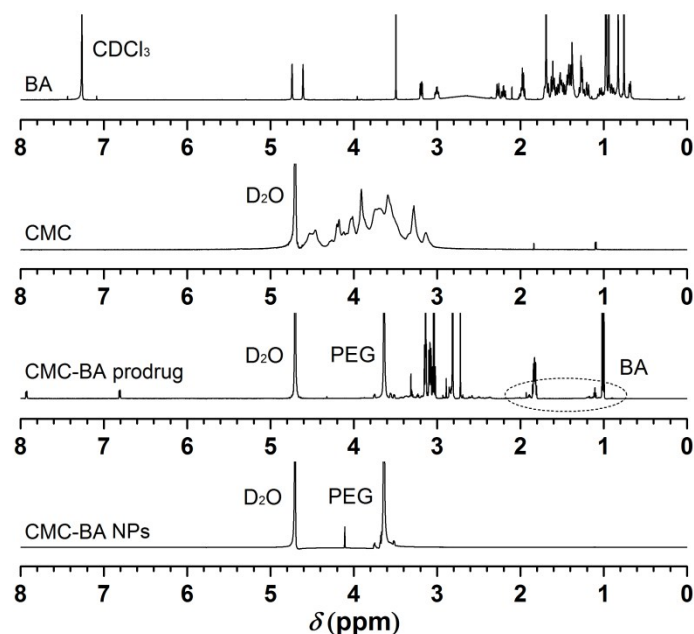


**Fig. S1** Synthesis CMC-BA prodrug. Note that the distribution of BA or F-PEG-COOH substitutions is random: for ease of depiction, monomers are drawn in simplified form.

CMC-COOH (0.20 g) was dissolved in 20 mL of dry pyridine and maintained by gentle heating. BA (0.47 g, 1.00 mmol), M-PEG-COOH (0.03 g, 0.16 mmol), EDC (0.46 g, 2.40 mmol), NHS (0.28 g, 2.40 mmol), and DMAP (0.03 g, 0.24 mmol) were successively added to the mixture, and the reaction was allowed to proceed at 35 °C. After overnight stirring, deionized water was gradually added and the solution was dialyzed against deionized water

for 48 h in a dialysis membrane (MWCO 3.5 kDa). After lyophilization, CMC-BA prodrug was obtained as a white powder.

### <sup>1</sup>H-NMR of CMC-BA Prodrug and NPs



**Fig. S2** <sup>1</sup>H-NMR spectra of BA in CDCl<sub>3</sub>, CMC, CMC-BA prodrug and CMC-BA nanoparticles in D<sub>2</sub>O.

Direct proofs confirming BA and PEG conjugated onto CMC came from Fig. S2. Partial structure of BA can be identified at peaks from 0.8 to 1.4 ppm,<sup>1</sup> the broad peaks of PEG from 3.6 to 3.7 ppm. By contrast, the signals of BA was significantly weakened and only that of PEG was detected in D<sub>2</sub>O, further indicating a core-shell system of the nanoparticles in water.<sup>2,3</sup>

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2. Ding M, Song N, He X, et al. Toward the next-generation nanomedicines: design of multifunctional multiblock polyurethanes for effective cancer treatment. *ACS nano*, 2013, 7(3): 1918-1928.
3. Tai W, Mo R, Lu Y, et al. Folding graft copolymer with pendant drug segments for co-delivery of anticancer drugs. *Biomaterials*, 2014, 35(25): 7194-7203.