Electronic Supplementary Material (ESI) for Polymer Chemistry.

Supplementary Information

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Synthesis of 2,2-bis(hydroxyl methyl)propionate (1)

KOH (9.6g, 171.4 mmol), 2,2-bis(hydroxyl methyl) propionic acid (20g, 149.1 mmol) and anhydrous DMF (110 mL) were added in a round bottom flask. Potassium salt was formed at 100 °C for 1.5 h, then propargyl bromide (19.6g, 164.7 mmol) was introduced dropwise. The reaction mixture was stirred for 72 h at 80 °C, and filtered. After the solvent was evaporated off, the residues were dissolved in 280 mL dichloromethane and washed three times with saturation sodium chloride (3×100 mL), dried over $MgSO_4$, filtered and concentrated to obtain crude product, which was purified by column chromatography (eluent: ethyl acetate). Yield: 11.0 g (42.8 %). ^1H NMR (400 MHz, $CDCl_3$, ppm) δ 4.73 (d, J = 2.5 Hz, 2H, $CHCC\mathbf{H_2}CO$-), 3.89 (d, J = 11.3 Hz, 2H, -$C\mathbf{H_2}OH$), 3.71 (d, J = 11.3 Hz, 2H, -$C\mathbf{H_2}OH$), 2.49 (t, J = 2.4 Hz, 1H, $C\mathbf{H}CCH_2CO$-), 1.09 (s, 3H, $C\mathbf{H_3}CC$-); ^{13}C NMR (400 MHz, $CDCl_3$, ppm) δ 175.1, 75.4, 67.6, 52.6, 49.5, 17.1.

Synthesis of bis-(tosylate ethyl) disulfide（2）

2-hydroxyethyl disulfide (3.08 g, 0.02 mol) and TEA (5.06 g, 0.05 mol) were dissolved in dichloromethane (30 mL) and cooled in an ice bath for 30 min. *p*-toluenesulfonyl chloride (TsCl, 11.45 g, 0.15 mol) was then dissolved in chloroform and this solution was introduced dropwise to the mixture. After 1 h, the reaction was removed from the ice bath and stirred for 24 h at room temperature. The reaction mixture was washed with saturated Na_2CO_3 (3 × 100 mL) and with DI water (50 mL). The washed solution was dried over $MgSO_4$, filtered, and evaporated under vacuum. A white solid was obtained. Yield: 9.0 g (97 %). 1H NMR (400 MHz, $CDCl_3$, ppm) δ 7.80 (d, J = 8.3 Hz, 4H, Ar**H**), 7.36 (d, J = 8.0 Hz, 4H, Ar**H**), 4.20 (d, J = 6.7 Hz, 4H, $(-SCH_2C\mathbf{H_2}PhCH_3)_2$), 2.84 (t, J = 6.7 Hz, 4H, $(-SC\mathbf{H_2}CH_2PhCH_3)_2$), 2.46 (s, 6H, $(-SCH_2CH_2PhC\mathbf{H_3})_2$); ^{13}C NMR (400 MHz, $CDCl_3$, ppm) δ 145.4, 132.7, 130.2, 128.3, 67.6, 37.0, 21.4.

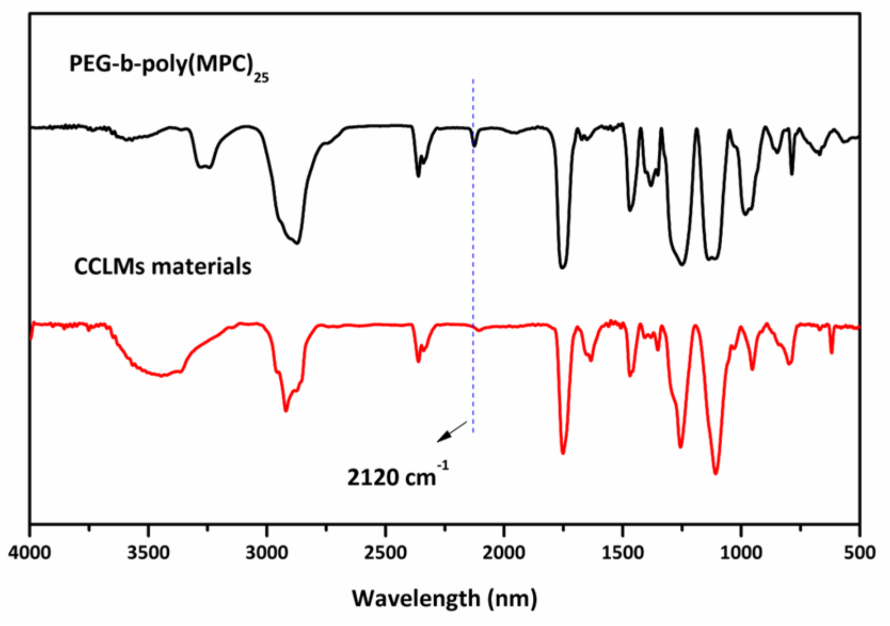


Figure S1. FTIR spectra of PEG-b-poly(MPC) and CCLMs materials. After the crosslinking, the alkynyl band at 2120 cm^{-1} disappeared.

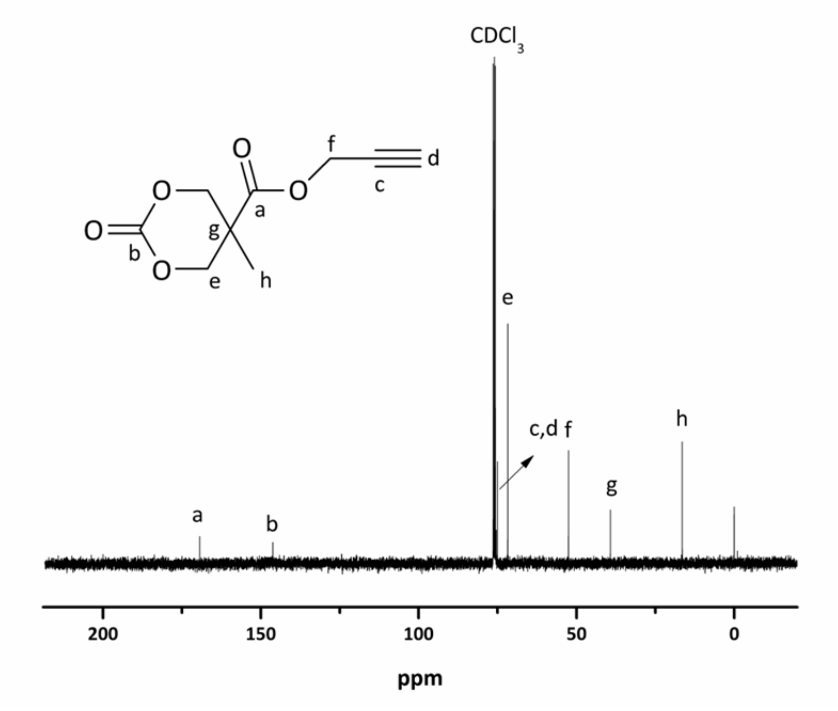


Figure S2. ^{13}C NMR spectra of MPC.

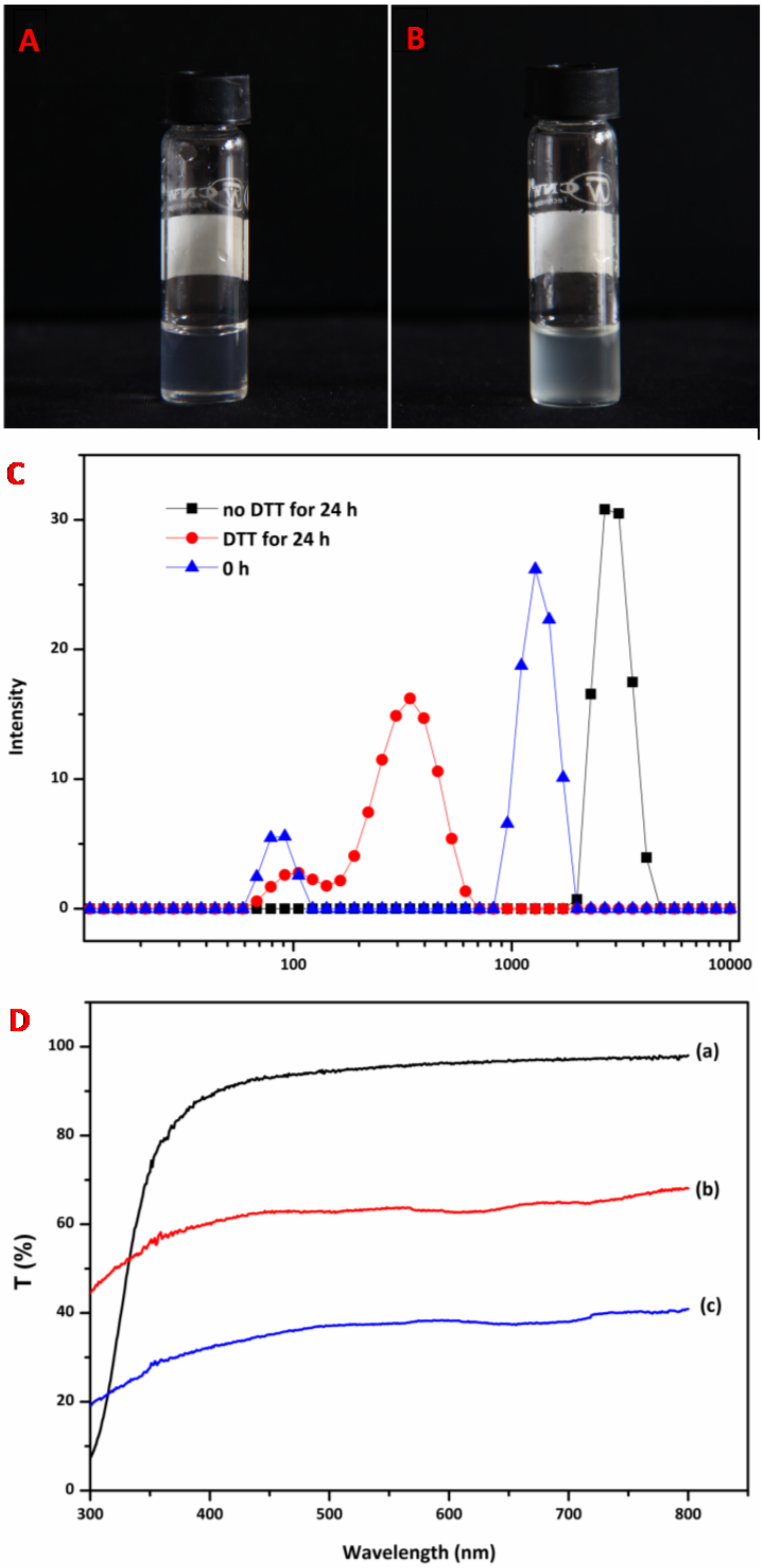


Figure S3. Photographs of lyophilized CCLMs in DMF: (A) treatment with 10 mM DTT for 24 h and (B) without DTT for 24 h at 37 °C; the DLS data of three samples (0.4 mg/mL) treated with DMF (4 mL) under different conditions (C); and the UV-vis spectroscopy of three samples (0.4 mg/mL) treated with DMF (4 mL) for 0 h (a), 24 h (b) and 24 h with 10 mM DTT (c) (D).

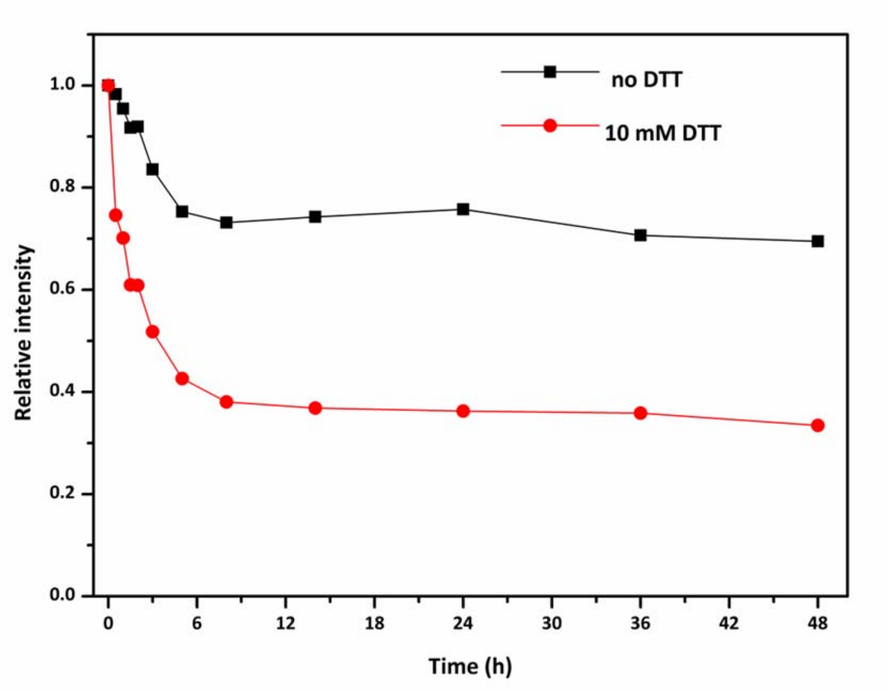
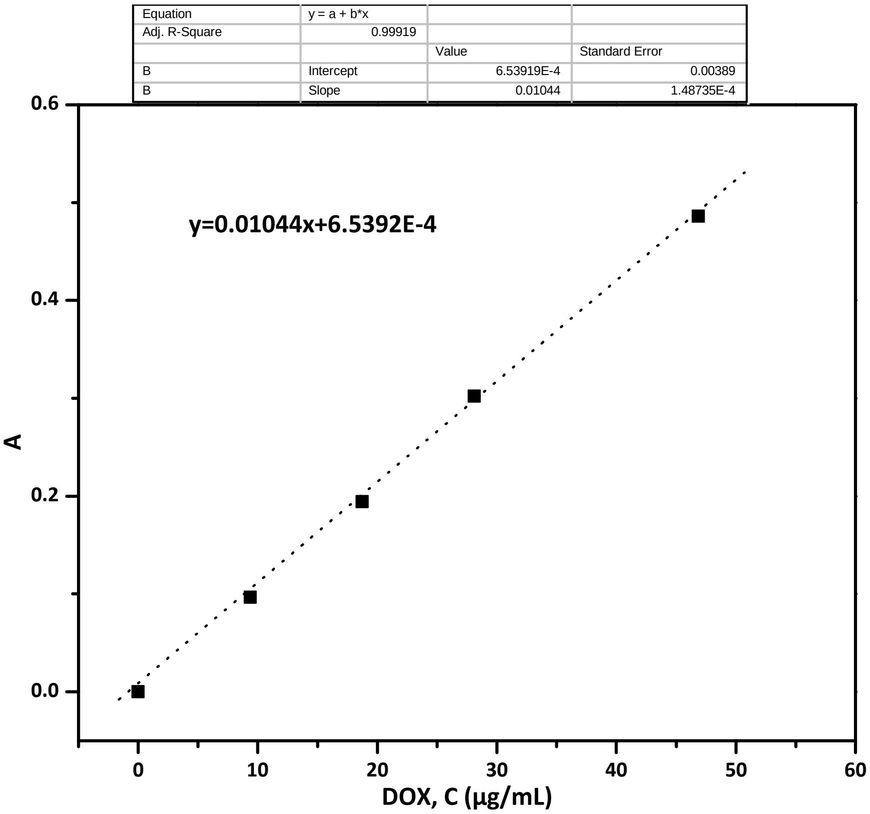


Figure S4. Nile Red release from CCLMs in 20 mM PBS (pH = 7.4) with and without 10 mM DTT.



Equation	y = a + b*x		
Adj. R-Square	0.99919		
		Value	Standard Error
B	Intercept	6.53919E-4	0.00389
B	Slope	0.01044	1.48735E-4

Figure S5 Plot of UV absorbance vs. concentration of DOX in PBS.