

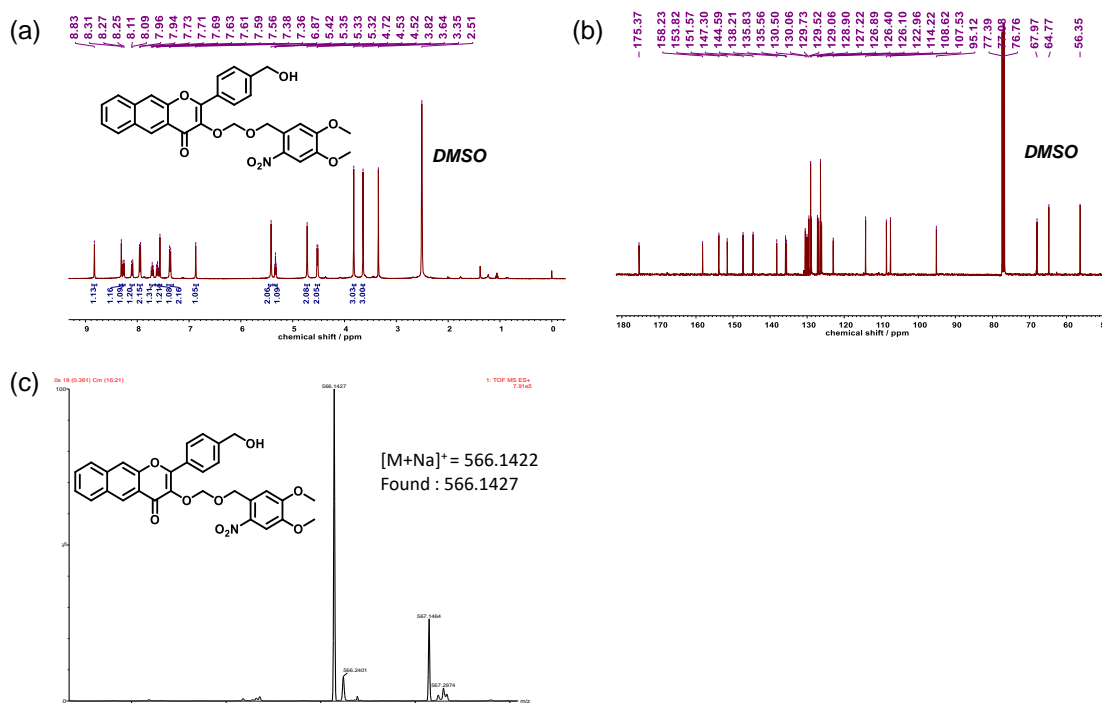
# Photo-responsive micelles with controllable and co-release of carbon monoxide, formaldehyde and doxorubicin

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§ The two contribute equally to the work.



**Figure S1** (a) <sup>1</sup>H NMR spectra recorded in DMSO-d<sub>6</sub> for compound A; (b) <sup>13</sup>C NMR spectra recorded in DMSO-d<sub>6</sub> for compound A; (c) ESI mass spectrum recorded for compound A in acetonitrile.

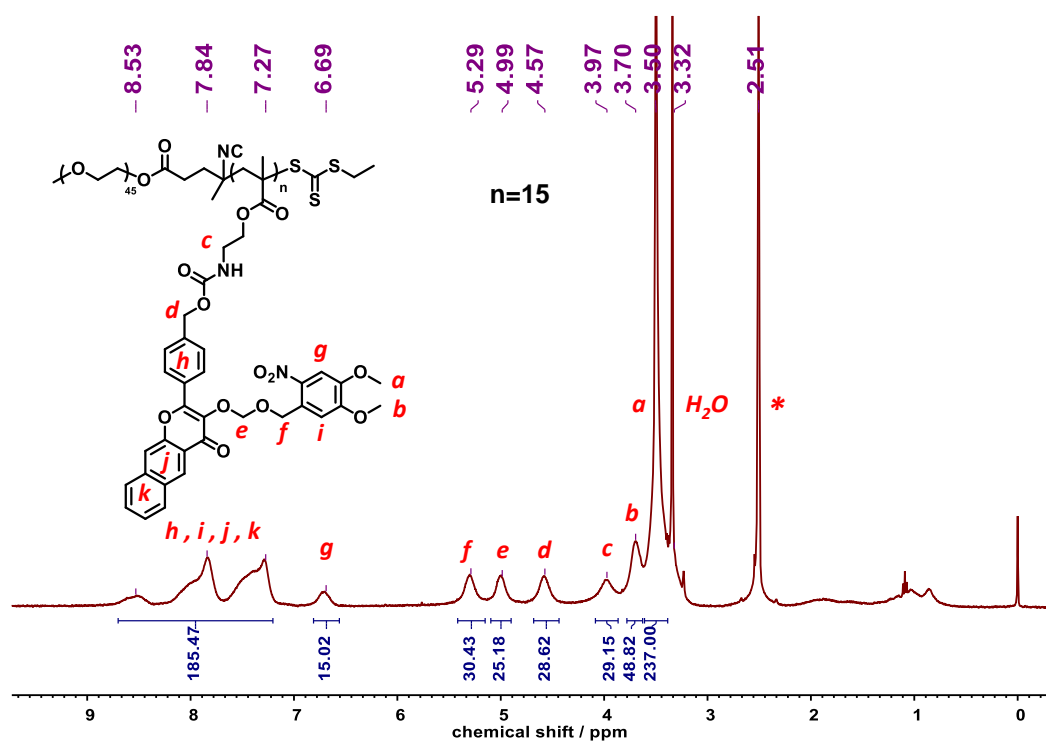


Figure S2 <sup>1</sup>H NMR spectra recorded in DMSO-d<sub>6</sub> for PEO<sub>45</sub>-b-PFFM<sub>15</sub> (PCOFA).

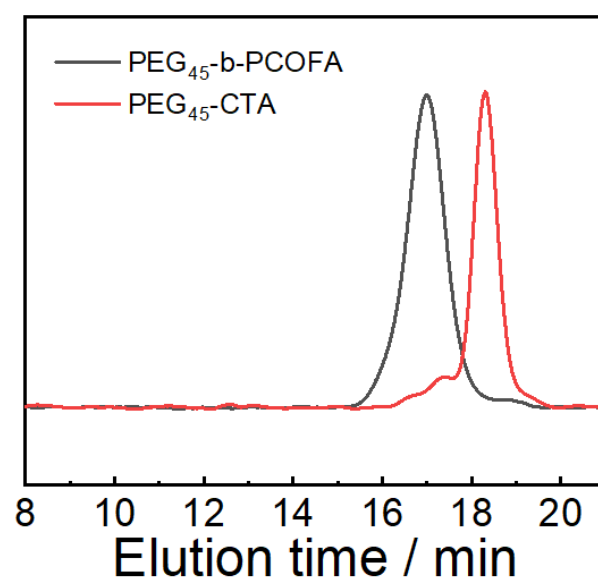
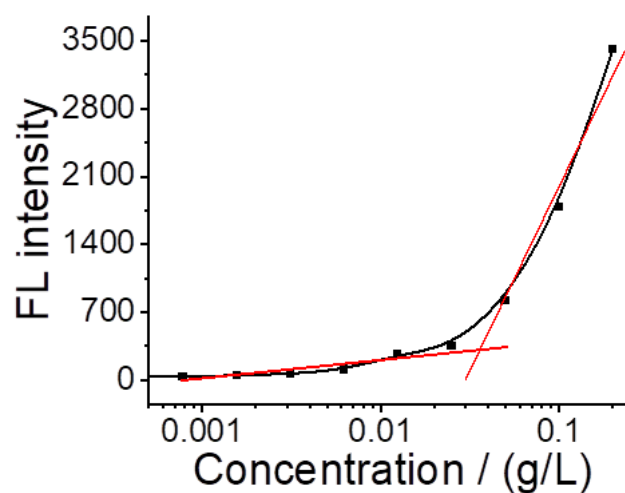
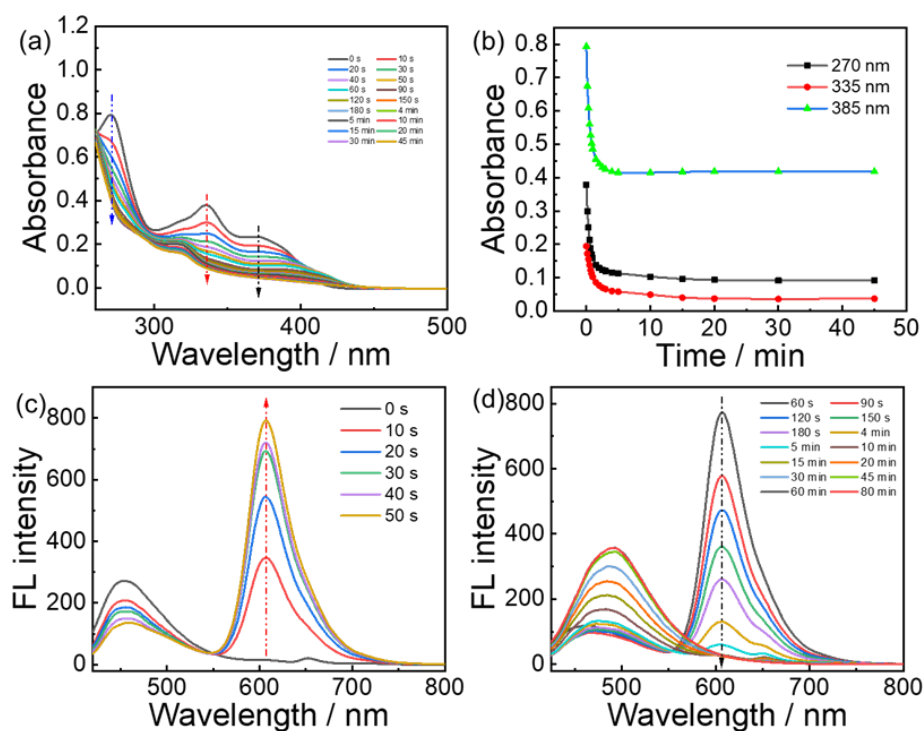


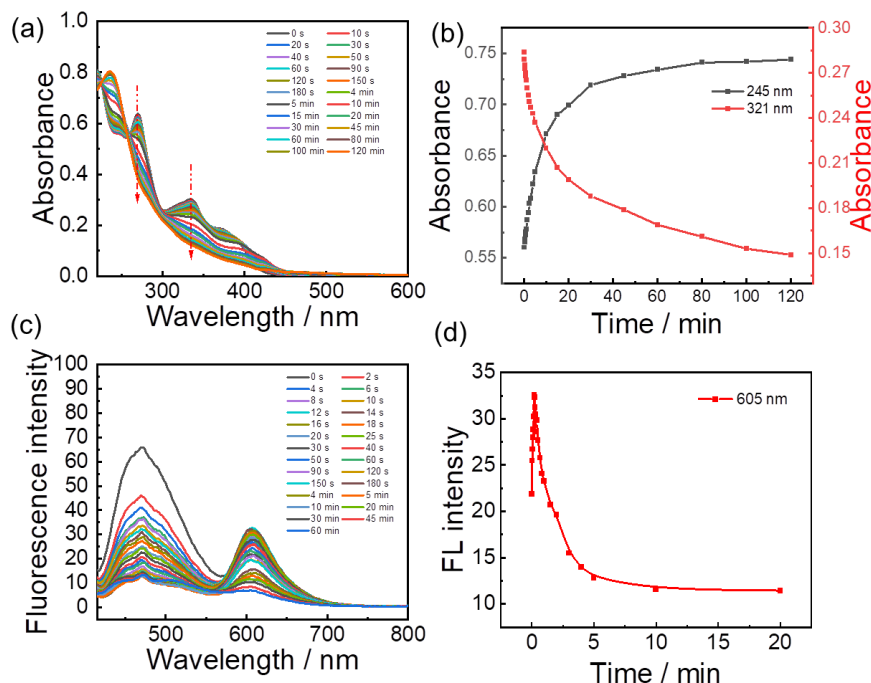
Figure S3 GPC Elution profiles of PEG<sub>45</sub>-based macroinitiator and PCOFA.



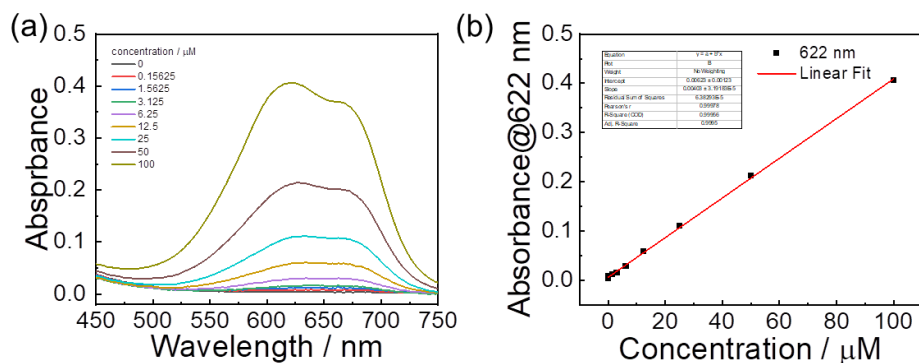
**Figure S4** Concentration-dependent changes of surface tension of aqueous dispersions of PCOFA diblock copolymer.



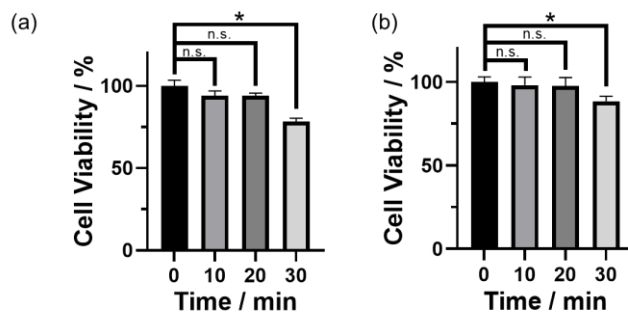
**Figure S5** UV absorption spectra (a) and trend diagram of main characteristic absorption peaks with illumination time (b) of Compound B ( $80 \mu\text{M}$ ,  $\text{DMSO}/\text{H}_2\text{O}$ ,  $v/v=8/2$ ) irradiated by 410 nm LED lamp ( $28 \text{ mW}/\text{cm}^2$ ). The fluorescence emission spectrum of B from 0-50 s (c) and 60s-90s (d) ( $\lambda_{\text{ex}} = 405 \text{ nm}$ ; slit widths:  $E_{\text{x}} = 5.0 \text{ nm}$ ,  $E_{\text{m}} = 5.0 \text{ nm}$ ).



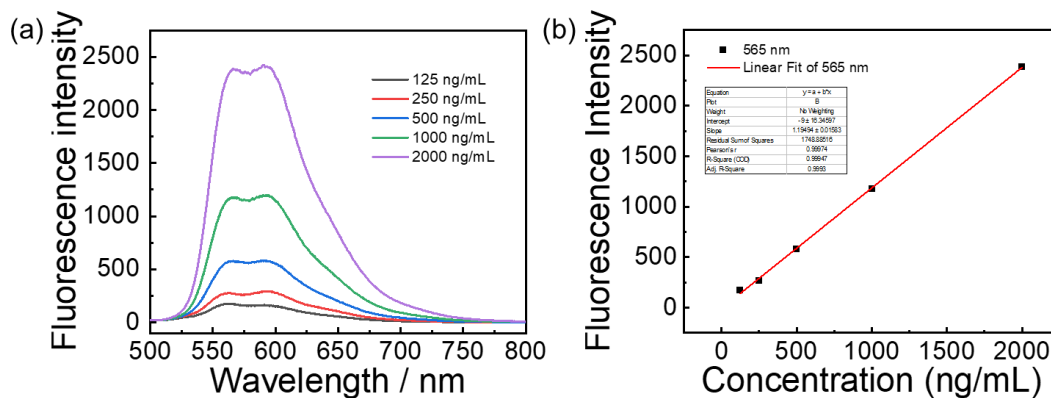
**Figure S6** UV absorption spectra (a) and trend profile of main characteristic absorption peaks with illumination time (b) of PCOFA micelle solution (0.1 g/L) irradiated by 410 nm LED lamp (28 mW/cm<sup>2</sup>). The fluorescence emission spectrum (c) and trend profile of main characteristic absorption peaks with illumination time (d) of PCOFA micelle solution (0.1 g/L) ( $\lambda_{\text{ex}} = 405 \text{ nm}$ ; slit widths:  $E_{\text{x}} = 5.0 \text{ nm}$ ,  $E_{\text{m}} = 5.0 \text{ nm}$ ).



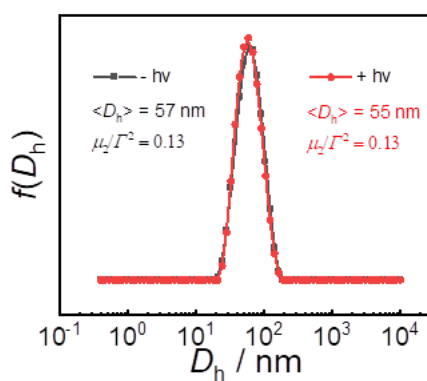
**Figure S7** UV absorption spectra of (a) different concentrations of FA in water and (b) linear fitting standard curve at 622 nm.



**Figure S8** Cell viability of (a) HeLa and (b) L929 under 410 nm irradiation ( $28 \text{ mW/cm}^2$ ) for varying duration after 24 hours incubation. Data are presented as mean  $\pm$  s.d. ( $n=3$ ); n.s., not significant; p values were calculated in comparison with the non-irradiated groups. \* $p < 0.05$ ; \*\* $p < 0.01$ .



**Figure S9** (a) Fluorescent spectra of DOX at different concentrations in DOMSO ( $\lambda_{\text{ex}} = 480 \text{ nm}$ ; slit widths:  $E_{\text{x}} = 10 \text{ nm}$ ,  $E_{\text{m}} = 10 \text{ nm}$ ) (b) Fitting curve of DOX concentration and fluorescence intensity at 565 nm.



**Figure S10** Number-average hydrodynamic distributions of PCOFA micelles without and with light irradiation. In all cases, the irradiation wavelength was 410 nm and the intensity was  $28 \text{ mW/cm}^2$ .