**Supporting information** 

## DOUBLE STIMULI-RESPONSIVE AZOBENZENE CONTAINING POLY(2-OXAZOLINE)S: SYNTHESIS, LIGHT, AND TEMPERATURE-RESPONSIVE BEHAVIOR

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	EtOx	MeOTos	ACN	Terminator (KOH)	Time	M <sub>n</sub> (g/mol)	Đ
PEtOx <sup>6</sup>	8 mL, 79.4 mmol	120 μL, 0.8 mmol	11.8 mL	3.2 mmol	16 min	6000	1.39
PEtOx <sup>15.1</sup>	8 mL, 79.4 mmol	120 μL, 0.8 mmol	11.8 mL	3.2 mmol	19 min	15100	1.11
PEtOx <sup>17.5</sup>	8 mL, 79.4 mmol	81 μL, 0.5 mmol	11.8 mL	2.1 mmol	29 min	17500	1.22

Table S1. Setups of three series of polymers.

**Table S2.** Mn and Đ of hydrolyzed polymers.

Hydrolyzed polymers	Hydrolysis time, min	Mn (g/mol)	Ð	Hydrolysis degree
$P(EtOx_{0.94}-co-PEI_{0.06})^{5.9}$	10	5900	1.35	6%
P(EtOx <sub>0.91</sub> -co-PEI <sub>0.09</sub> ) <sup>6.9</sup>	50	6900	1.30	9%
$P(EtOx_{0.88}-co-PEI_{0.12})^{7}$	100	7000	1.29	12%

$P(EtOx_{0.78}-co-PEI_{0.22})^{7.3}$	200	7300	1.27	22%
$P(EtOx_{0.97}-co-PEI_{0.03})^{15.2}$	50	15200	1.12	3%
P(EtOx <sub>0.93</sub> - <i>co</i> -PEI <sub>0.07</sub> ) <sup>15.4</sup>	100	15400	1.11	7%
$P(EtOx_{0.85}-co-PEI_{0.15})^{14.4}$	200	14400	1.18	15%
$P(EtOx_{0.97}-co-PEI_{0.03})^{21.6}$	50	21600	1.08	3%
$P(EtOx_{0.93}-co-PEI_{0.07})^{18.3}$	100	18300	1.20	7%
$P(EtOx_{0.87}-co-PEI_{0.13})^{19.3}$	200	19300	1.17	13%



**Figure S1.** SEC (DMAc / LiCl) – Elugrams of PEtOx<sup>6</sup> series: initial PEtOx's (black), hydrolyzed polymers  $P(EtOx_x$ -co-PEI<sub>y</sub>) (red), and modified polymers  $P(EtOx_x$ -co-AzBenOx<sub>y</sub>) (blue).



**Figure S2.** SEC (DMAc / LiCl) – Elugrams of  $PEtOx^{15.1}$  series: initial PEtOx's (black), hydrolyzed polymers  $P(EtOx_x$ -co- $PEI_y$ ) (red), and modified polymers  $P(EtOx_x$ -co- $AzBenOx_y$ ) (blue).



**Figure S3.** SEC (DMAc / LiCl) – Elugrams of  $PEtOx^{17.5}$  series: initial PEtOx's (black), hydrolyzed polymers  $P(EtOx_x$ -co- $PEI_y$ ) (red), and modified polymers  $P(EtOx_x$ -co- $AzBenOx_y$ ) (blue).



*Figure S4. UV/Vis spectroscopy results of*  $P(EtOx_{0.91}$ *-co-AzBenOx*<sub>0.09</sub>)<sup>6.4</sup>, 0.02 mg/mL in water at 45°C. Arrows indicate from the beginning to the end of the corresponding light irradiation.



**Figure S5.** UV/Vis spectroscopy results of  $P(EtOx_{0.84}$ -co-AzBen $Ox_{0.16})^{6.1}$ , 0.02 mg/mL in water at room temperature. Arrows indicate from the beginning to the end of the corresponding light irradiation.



*Figure S6.* Autocorrelation functions and respective CONTIN plots for micellar nanoparticles in aqueous solution under different irradiation times (365 nm, LED).