

## SUPPORTING INFORMATION

### An Insight into Aqueous Photoinitiated Polymerization-Induced Self-Assembly (Photo-PISA) for the preparation of Diblock Copolymer Nano-Objects

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## ADDITIONAL RESULTS

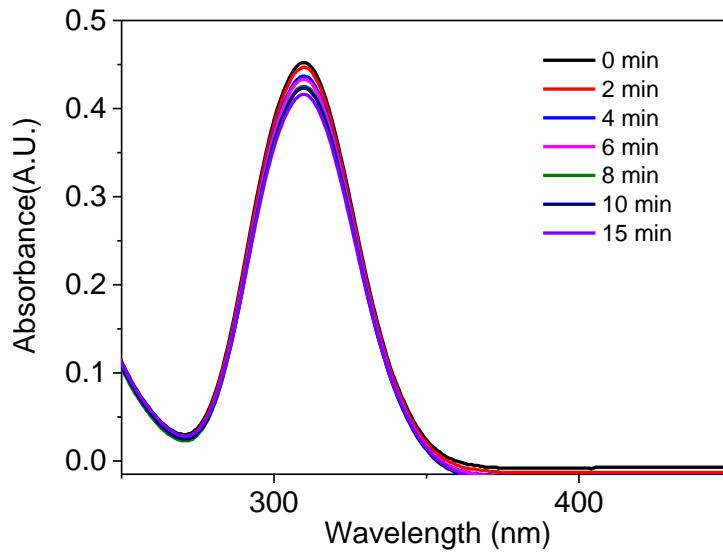


Figure S1. UV-vis absorbance spectra of PGMA<sub>68</sub>-PETTC in an ethanol/water (30/70, w/w) mixture under 405 nm visible light irradiation at different times.

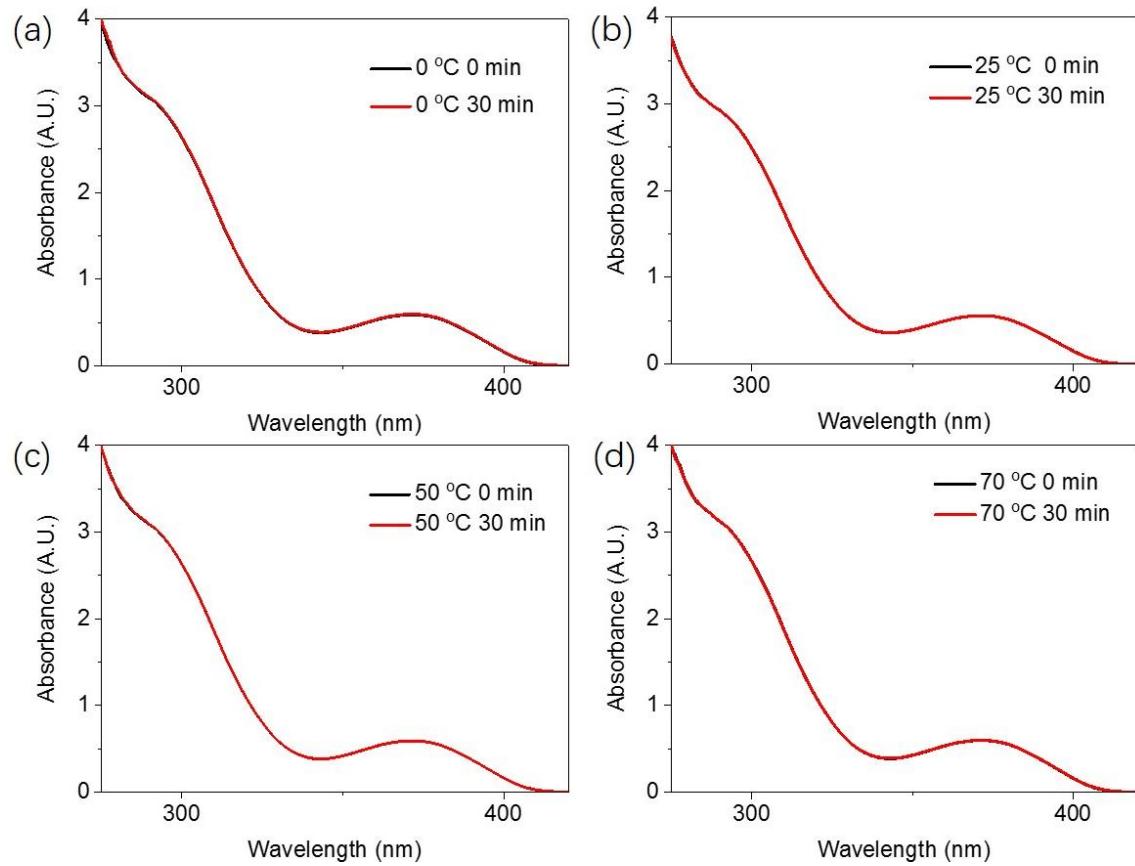


Figure S2. UV-vis absorbance spectra of SPTP in water at different temperatures: (a)  $0\text{ }^{\circ}\text{C}$ , (b)  $25\text{ }^{\circ}\text{C}$ , (c)  $50\text{ }^{\circ}\text{C}$ , (d)  $70\text{ }^{\circ}\text{C}$ .

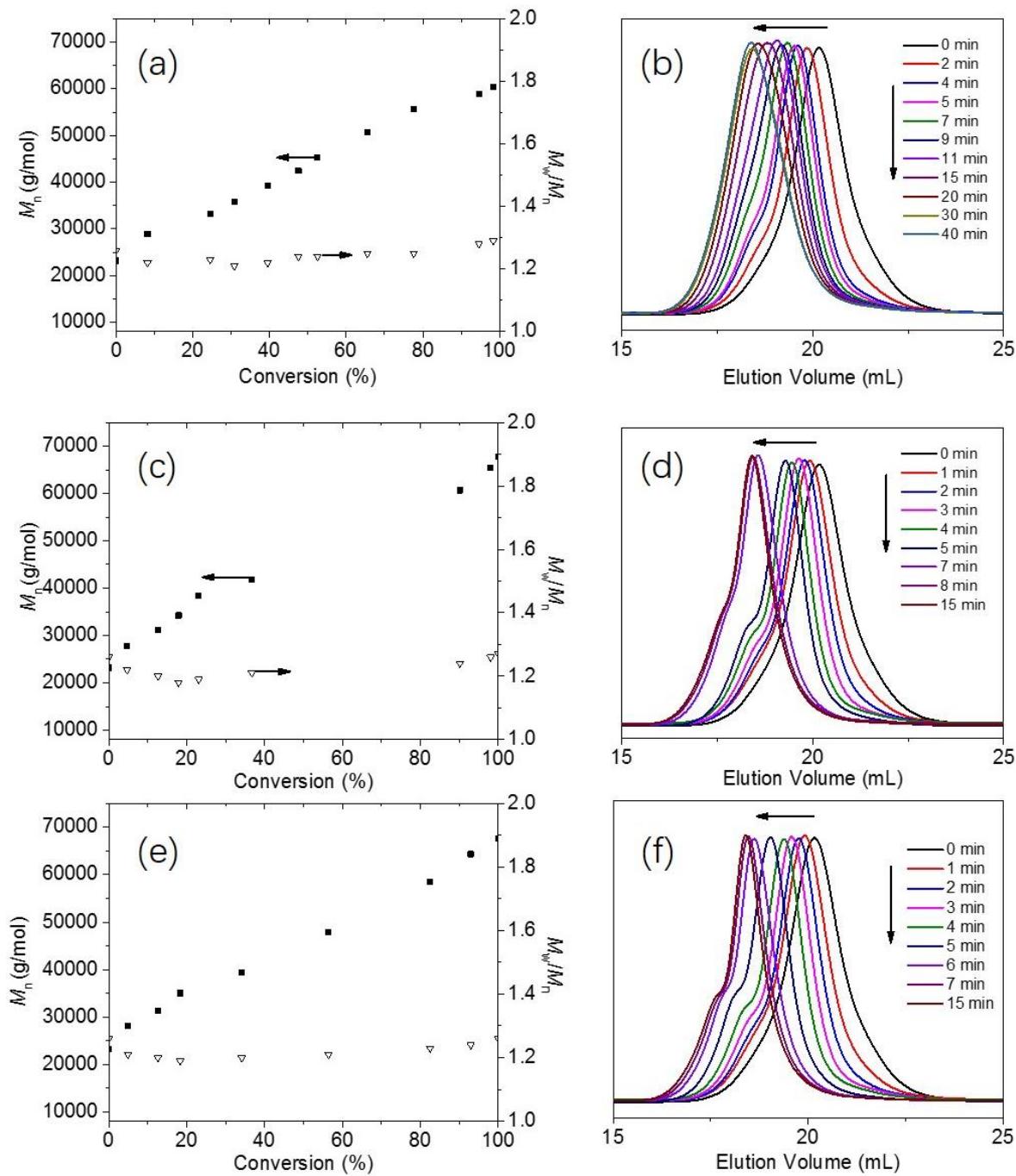


Figure S3. GPC traces recorded of PGMA<sub>68</sub>-PHPMA<sub>n</sub> diblock copolymers obtained from the kinetic study of photo-PISA of HPMA at different temperatures (10% w/w, target DP of 200): (a) 0 °C, (c) 50 °C<sup>2</sup>, (e) 70 °C; evolution of  $M_n$  and  $M_w/M_n$  with monomer conversion for photo-PISA of HPMA at different temperatures (10% w/w, target DP of 200): (b) 0 °C, (d) 50 °C, (f) 70 °C.

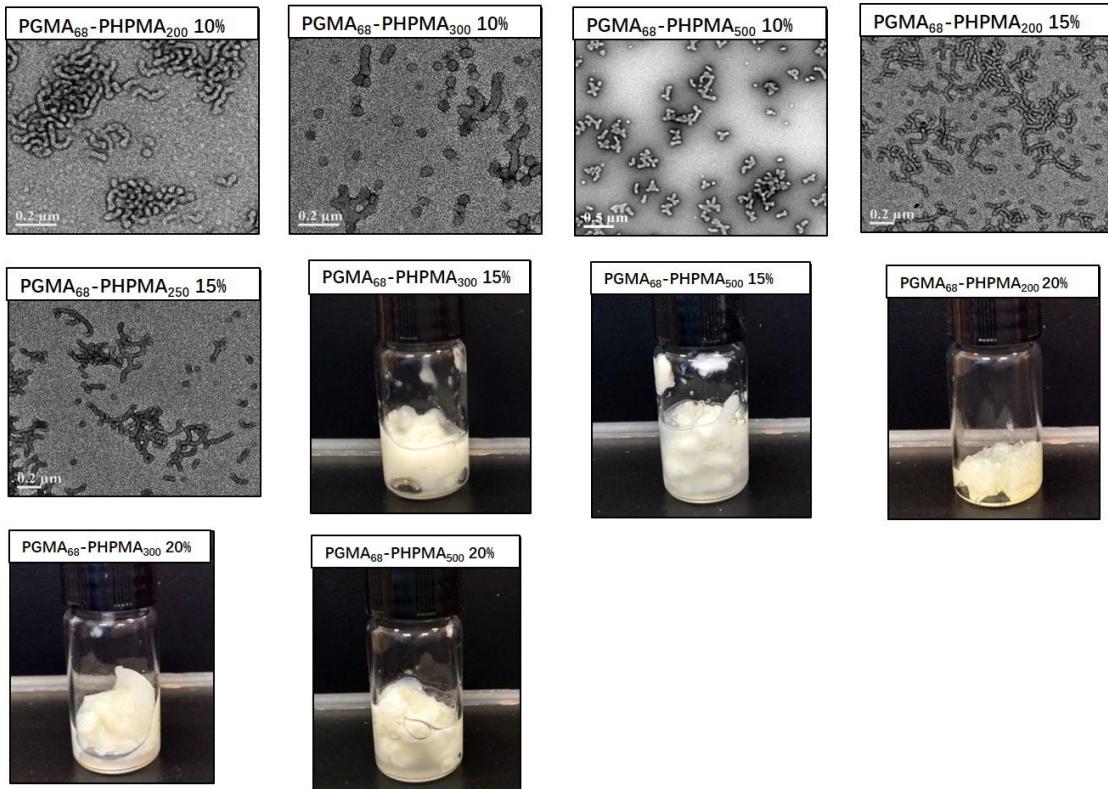


Figure S4. TEM images and optical images of PGMA<sub>68</sub>-PHPMAn diblock copolymers prepared via aqueous photo-PISA of HPMA at 0 °C.

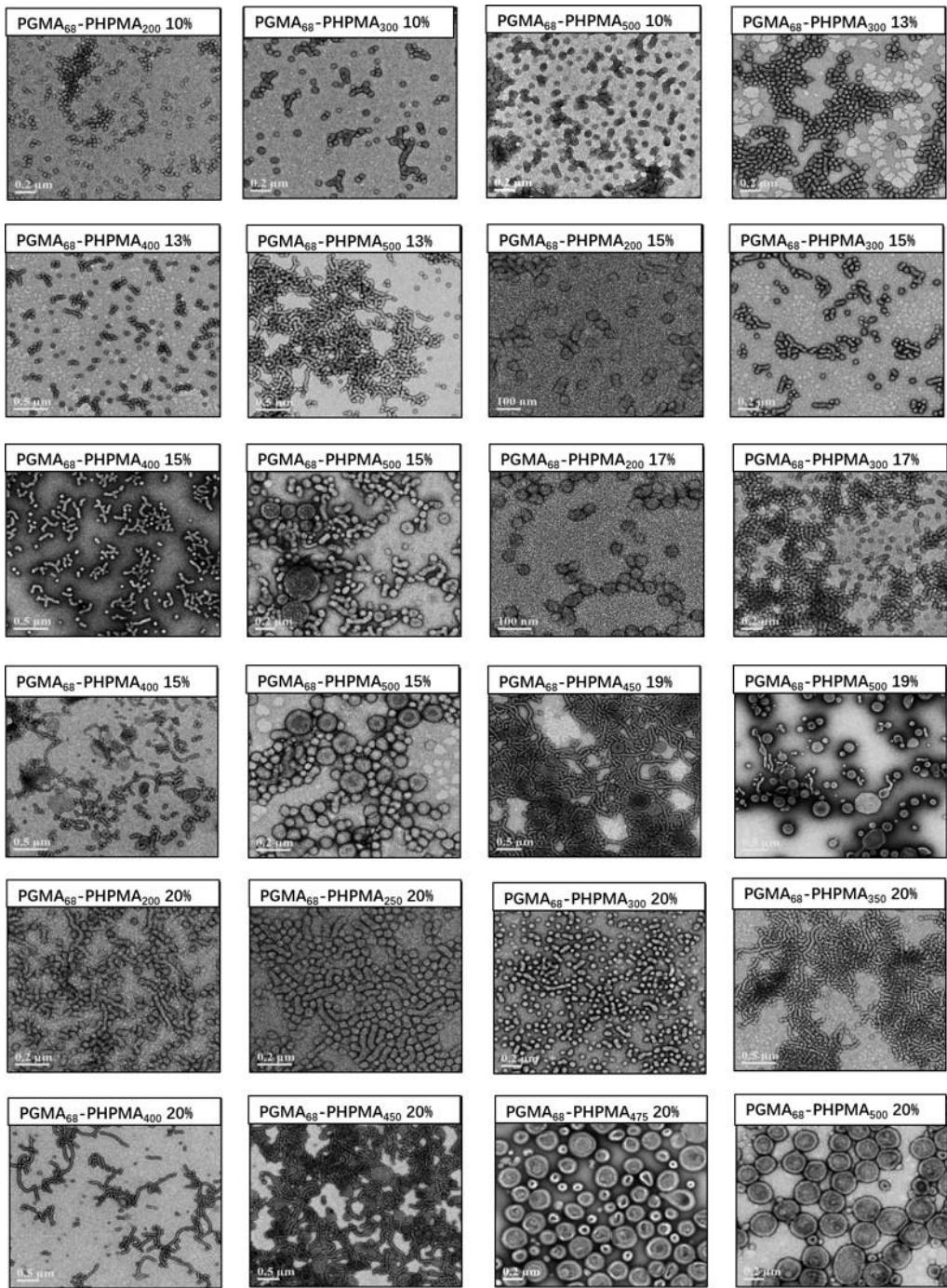


Figure S5. TEM images of  $\text{PGMA}_{68}\text{-PHPMAn}$  diblock copolymers prepared via aqueous photo-PISA of HPMA at 25 °C.

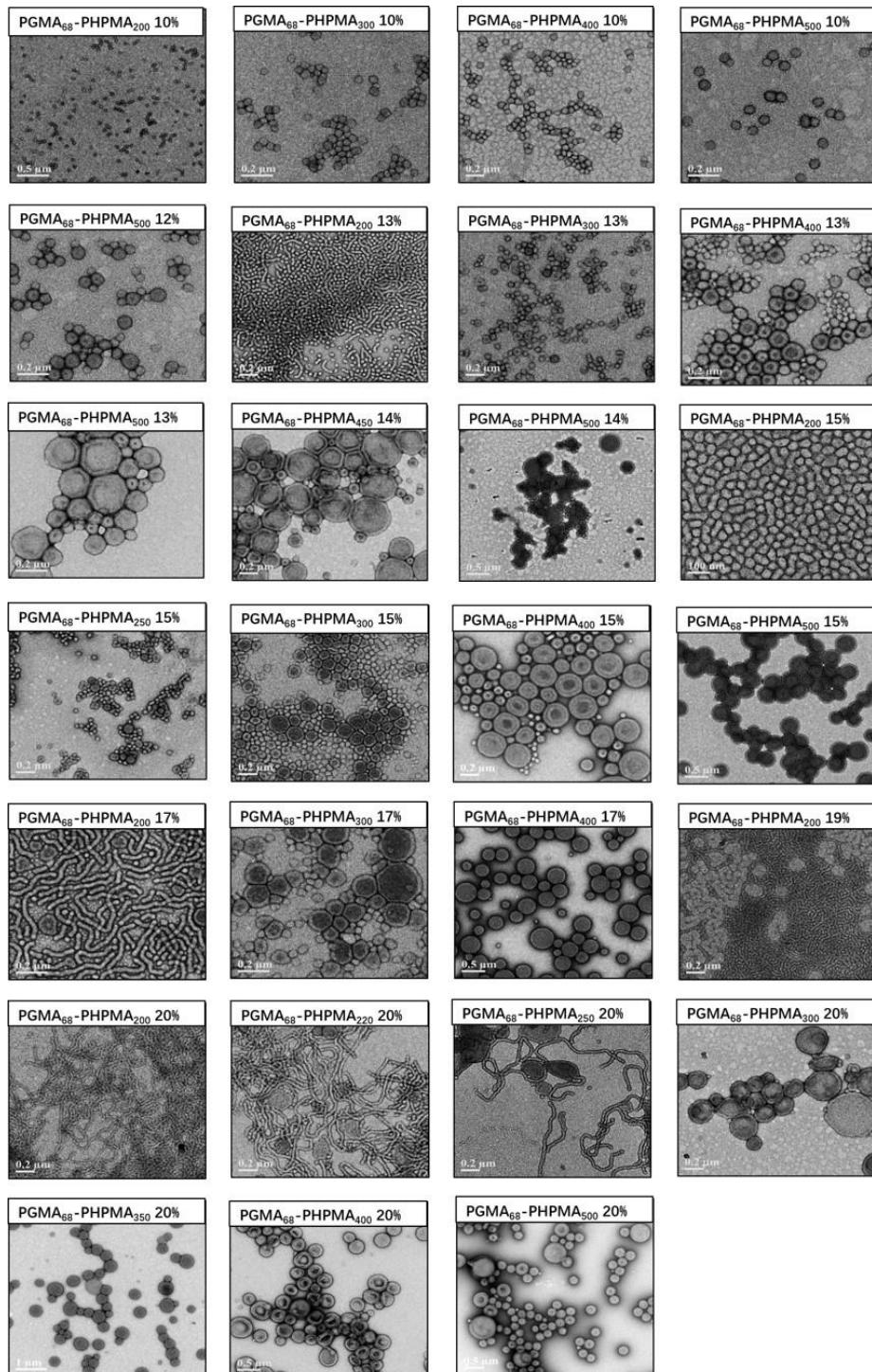


Figure S6. TEM images of PGMA<sub>68</sub>-PHPMAn diblock copolymers prepared via aqueous photo-PISA of HPMA at 70 °C.

Table S1. Summary of GPC data for a series of PGMA<sub>68</sub>-PHPMA<sub>n</sub> diblock copolymers prepared via aqueous photo-PISA at 0 °C.

Composition	Reaction Temperature (°C)	Monomer concentration	$M_n$ (g/mol)	$M_w/M_n$	Morphology
PGMA <sub>68</sub> -HPMA <sub>200</sub>	0	10%	63200	1.26	S, W
PGMA <sub>68</sub> -HPMA <sub>300</sub>	0	10%	68700	1.39	S, W
PGMA <sub>68</sub> -HPMA <sub>500</sub>	0	10%	88600	1.55	S, W
PGMA <sub>68</sub> -HPMA <sub>200</sub>	0	15%	62000	1.31	S, W
PGMA <sub>68</sub> -HPMA <sub>250</sub>	0	15%	67300	1.38	S, W
PGMA <sub>68</sub> -HPMA <sub>300</sub>	0	15%	79000	1.43	Precipitation
PGMA <sub>68</sub> -HPMA <sub>500</sub>	0	15%	109600	1.64	Precipitation
PGMA <sub>68</sub> -HPMA <sub>200</sub>	0	20%	62800	1.32	Precipitation
PGMA <sub>68</sub> -HPMA <sub>300</sub>	0	20%	80000	1.46	Precipitation
PGMA <sub>68</sub> -HPMA <sub>500</sub>	0	20%	113100	1.65	Precipitation

Table S2. Summary of GPC data for a series of PGMA<sub>68</sub>-PHPMA<sub>n</sub> diblock copolymers prepared via aqueous photo-PISA at 25 °C.

Composition	Reaction Temperature (°C)	Monomer concentration (w/w)	$M_n$ (g/mol)	$M_w/M_n$	Morphology
PGMA <sub>68</sub> -HPMA <sub>200</sub>	25	10%	65600	1.28	S
PGMA <sub>68</sub> -HPMA <sub>300</sub>	25	10%	85300	1.41	S
PGMA <sub>68</sub> -HPMA <sub>400</sub>	25	10%	103700	1.50	S
PGMA <sub>68</sub> -HPMA <sub>500</sub>	25	10%	116000	1.57	S
PGMA <sub>68</sub> -HPMA <sub>200</sub>	25	15%	63700	1.32	S
PGMA <sub>68</sub> -HPMA <sub>300</sub>	25	15%	83200	1.39	S, W
PGMA <sub>68</sub> -HPMA <sub>400</sub>	25	15%	100400	1.49	S, W
PGMA <sub>68</sub> -HPMA <sub>500</sub>	25	15%	123800	1.59	V, W, S
PGMA <sub>68</sub> -HPMA <sub>200</sub>	25	20%	64300	1.32	S, W
PGMA <sub>68</sub> -HPMA <sub>300</sub>	25	20%	79200	1.43	S, W
PGMA <sub>68</sub> -HPMA <sub>400</sub>	25	20%	103500	1.47	W, S

Table S3. Summary of GPC data for a series of PGMA<sub>68</sub>-PHPMA<sub>n</sub> diblock copolymers prepared via aqueous photo-PISA at 70 °C.

Composition	Reaction Temperature (°C)	Monomer concentration	$M_n$ (g/mol)	$M_w/M_n$	Morphology
PGMA <sub>68</sub> -HPMA <sub>200</sub>	70	10%	66500	1.24	S
PGMA <sub>68</sub> -HPMA <sub>300</sub>	70	10%	82300	1.33	S
PGMA <sub>68</sub> -HPMA <sub>400</sub>	70	10%	106900	1.36	S
PGMA <sub>68</sub> -HPMA <sub>500</sub>	70	10%	119000	1.49	S
PGMA <sub>68</sub> -HPMA <sub>200</sub>	70	15%	69200	1.29	S, W
PGMA <sub>68</sub> -HPMA <sub>300</sub>	70	15%	86600	1.33	V, S
PGMA <sub>68</sub> -HPMA <sub>400</sub>	70	15%	105700	1.43	V, S
PGMA <sub>68</sub> -HPMA <sub>500</sub>	70	15%	123100	1.54	V
PGMA <sub>68</sub> -HPMA <sub>200</sub>	70	20%	67400	1.26	W
PGMA <sub>68</sub> -HPMA <sub>300</sub>	70	20%	87500	1.35	V
PGMA <sub>68</sub> -HPMA <sub>400</sub>	70	20%	100000	1.48	V
PGMA <sub>68</sub> -HPMA <sub>500</sub>	70	20%	118600	1.44	V

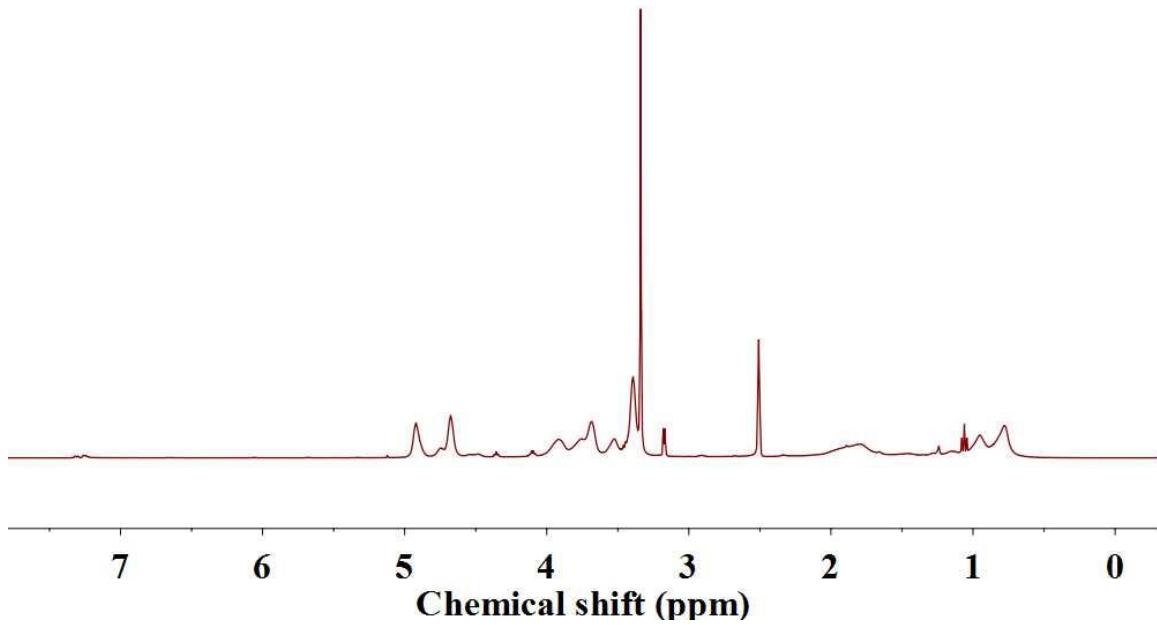


Figure S7. <sup>1</sup>H NMR spectrum of PGMA<sub>68</sub>-PETTC in DMSO-*d*<sub>6</sub>.