*Supporting Information for*

**Preparation and Characterization of Temperature/pH/CO$_2$-Triple-Responsive Homopolymers and Their Substituents Determined Response**

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**Characterization**

$^1$H NMR and $^{13}$C NMR spectra were tested by a Bruker Avance NEO 400MHz NMR spectrometer. Agilent PL-GPC50 (eluent: THF) was used to test the molecular weight and polydispersity index. Fourier transform infrared spectra were tested on a Nicolet IR200 FT-IR spectrometer using KBr-pellet. Transmission electron microscopy (TEM) images were tested using FEI TECNAI G2 F20 (200 kV). LCST, UCST and critical pH were tested on a UH-4150 UV-vis spectrophotometer equipped with a thermoregulator (±0.1 °C) at 500 nm. A NanoBrook Omni laser light scattering spectrometer was applied for dynamic light scattering (DLS) analysis. The fluorescence spectrum was observed by using a HITA CHI F-4600 fluorescence spectrophotometer.
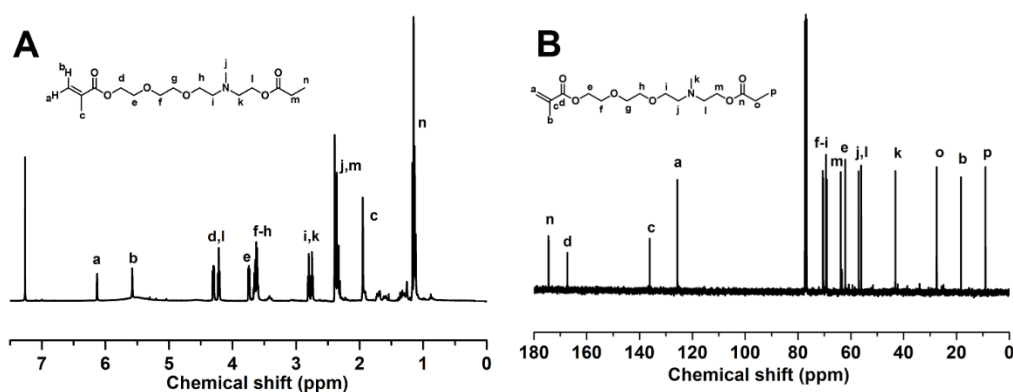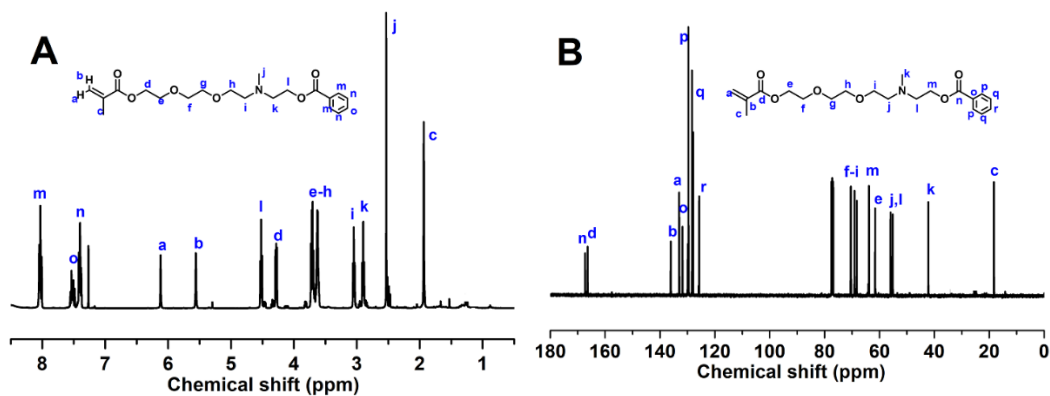
**Figure S1**. The $^1$H NMR spectra (A) and $^{13}$C NMR spectra (B) of Et-N-EO$_2$MA in CDCl$_3$.

$^1$H NMR (400 MHz, CDCl$_3$):δ 6.13 (s, 1H), 5.60 – 5.56 (m, 1H), 4.30 (dd, J = 5.5, 4.3 Hz, 2H), 4.22 (t, $J$ = 5.8 Hz, 2H), 3.74 (dd, $J$ = 5.5, 4.3 Hz, 2H), 3.69 – 3.56 (m, 6H), 2.80 (t, $J$ = 5.8 Hz, 2H), 2.75 (t, $J$ = 5.8 Hz, 2H), 2.42 – 2.28 (m, 5H), 1.95 (s, 3H), 1.15 (t, $J$ = 4.4 Hz, 3H). $^{13}$C NMR (101 MHz, CDCl$_3$):δ 174.4, 167.3, 136.1, 125.7, 70.7, 70.0, 69.4, 69.1, 63.8, 63.4, 43.1, 27.4, 18.2, 9.0.
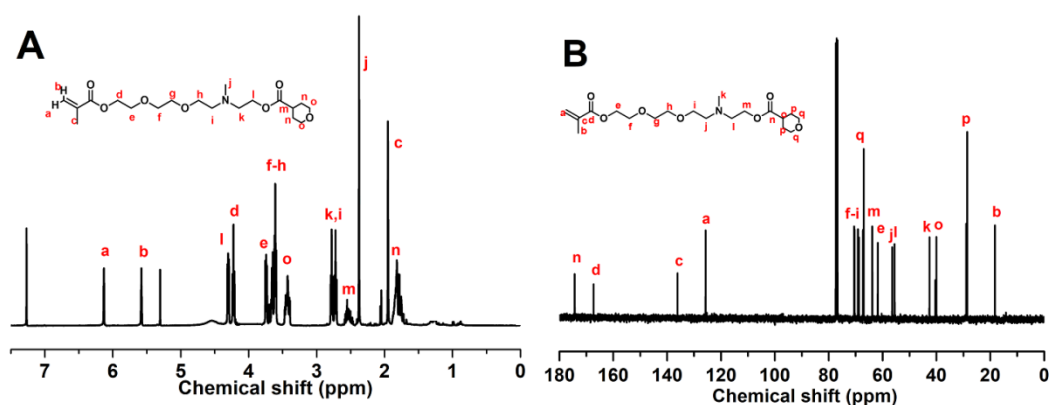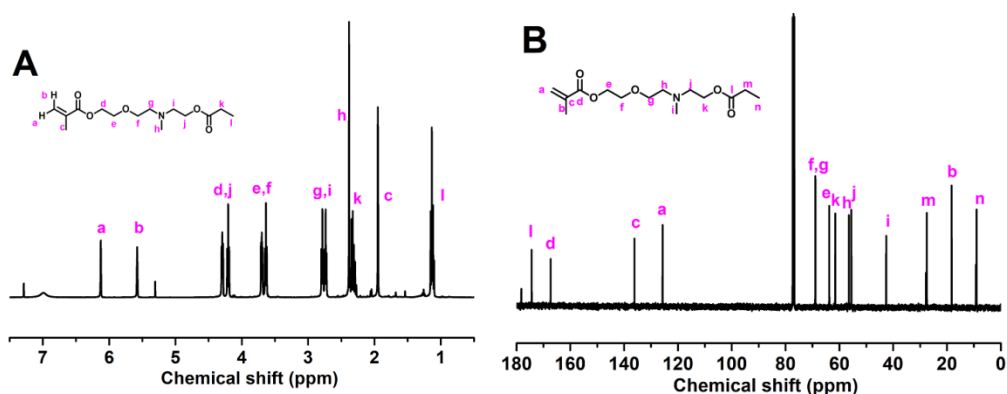


**Figure S2**. The $^1$H NMR spectra (A) and $^{13}$C NMR spectra (B) of THP-N-EO$_2$MA in CDCl$_3$.

$^1$H NMR (400 MHz, CDCl$_3$):δ 6.13 (s, 1H), 5.63 – 5.53 (m, 1H), 4.34 – 4.27 (m, 2H), 4.22 (t, $J$ = 5.9 Hz, 2H), 3.78 – 3.71 (m, 2H), 3.64 (ddd, $J$ = 15.2, 8.0, 4.4 Hz, 6H), 3.43 (tdd, $J$ = 11.4, 5.8, 2.8 Hz, 4H), 2.78 (t, $J$ = 5.8 Hz, 2H), 2.78 (t, $J$ = 5.8 Hz, 2H), 2.38 (s, 3H), 1.95 (s, 3H), 1.87 – 1.75 (m, 4H). $^{13}$C NMR (101 MHz, CDCl$_3$): δ 178.2, 174.3, 136.1, 125.7, 70.5, 69.1, 68.8, 67.2, 67.0, 63.8, 61.7, 56.4, 55.5, 42.6, 40.4, 28.6, 18.3.

**Figure S3**. The $^1$H NMR spectra (A) and $^{13}$C NMR spectra (B) of Ph-N-EO$_2$MA in CDCl$_3$.

$^1$H NMR (400 MHz, CDCl$_3$): δ 8.03 (tt, $J$ = 8.4, 1.8 Hz, 2H), 7.62 – 7.45 (m, 1H), 7.40 (ddd, $J$ = 7.4, 5.8, 1.9 Hz, 2H), 6.12 (s, 1H), 5.62 – 5.48 (m, 1H), 4.56 – 4.41 (m, 2H), 4.33 – 4.24 (m, 2H), 3.76 – 3.51 (m, 8H), 2.94 (t, $J$ = 5.6 Hz, 3H), 2.90 (t, $J$ = 5.6 Hz, 3H), 2.56 (s, 3H), 1.98 (s, 3H). $^{13}$C NMR (101 MHz, CDCl$_3$): δ 170.5, 167.2, 136.1, 132.9, 131.7, 129.9, 129.6, 128.3, 70.4, 69.1, 68.2, 63.8, 61.6, 56.0, 55.2, 42.2, 18.2.



**Figure S4**. The $^1$H NMR spectra (A) and $^{13}$C NMR spectra (B) of Et-N-EOMA in CDCl$_3$.

$^1$H NMR (400 MHz, CDCl$_3$) δ 6.13 (s, 1H), 5.58 (s, 1H), 4.36 – 4.26 (m, 2H), 4.21 (t, $J$ = 5.8 Hz, 2H), 3.74 – 3.68 (m, 2H), 3.64 (t, $J$ = 5.6 Hz, 2H), 2.79 (t, $J$ = 5.8
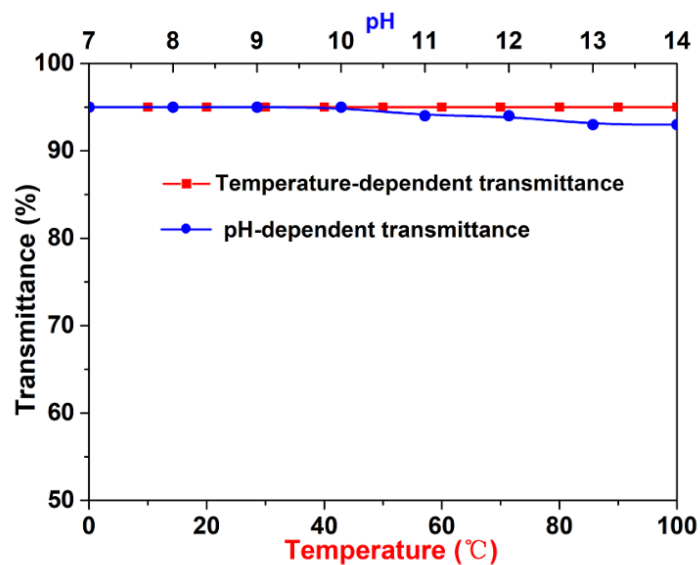
Hz, 2H), 2.74 (t, $J$ = 5.6 Hz, 2H), 2.38 (s, 3H), 2.37 – 2.26 (m, 2H), 1.95 (s, 3H). $^{13}$C

NMR (101 MHz, CDCl$_3$) δ174.3, 167.3, 136.1, 125.7, 68.9, 63.7, 61.5, 56.4, 55.5,

42.6, 27.5, 18.2, 9.2.
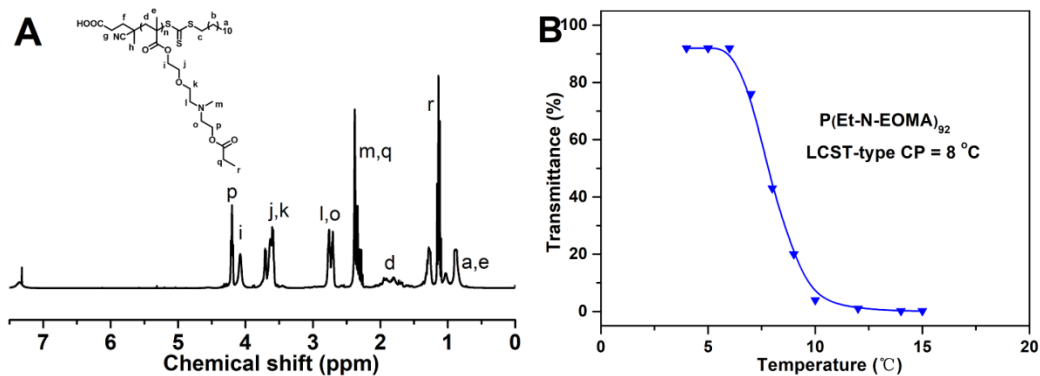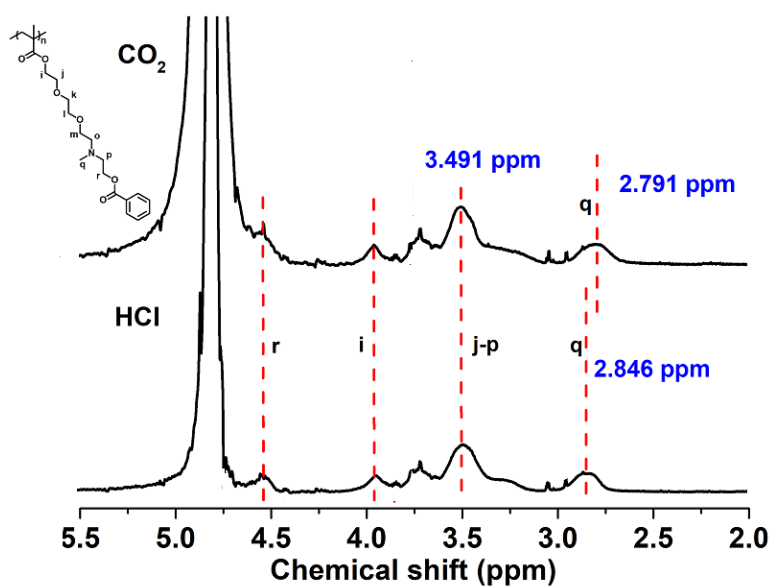


**Figure S5**. The $^1$H NMR spectra of P(Et-N-EOMA) in CDCl$_3$ (A) and thermoresponse of 1.0 wt % aqueous solutions of P(Et-N-EOMA)$_{92}$ (B).
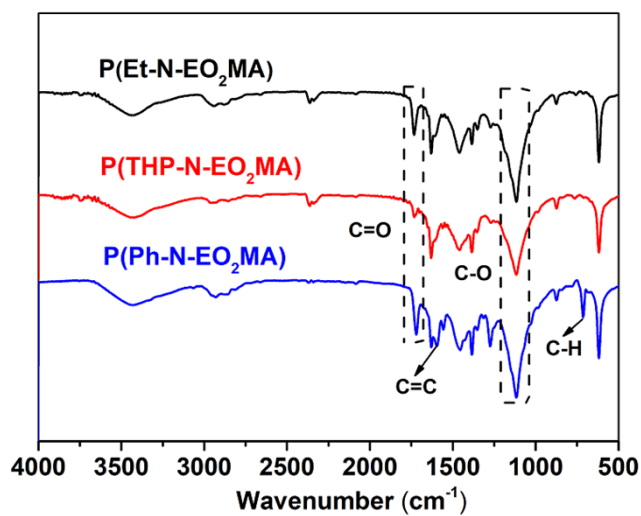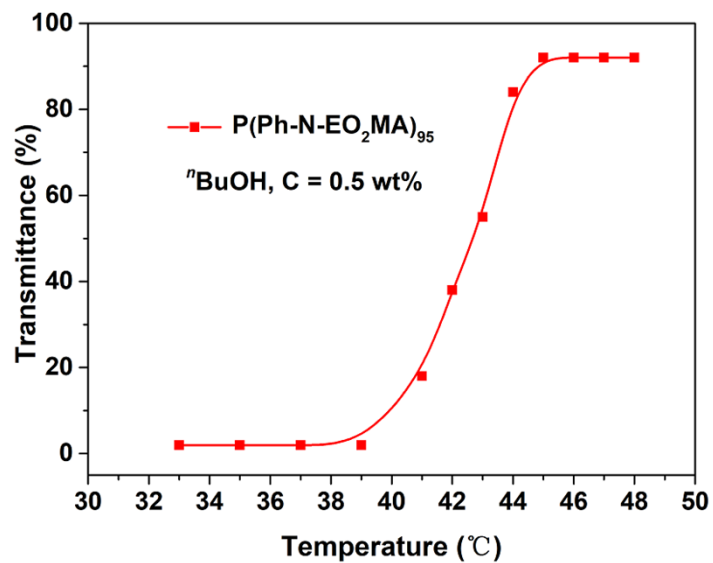


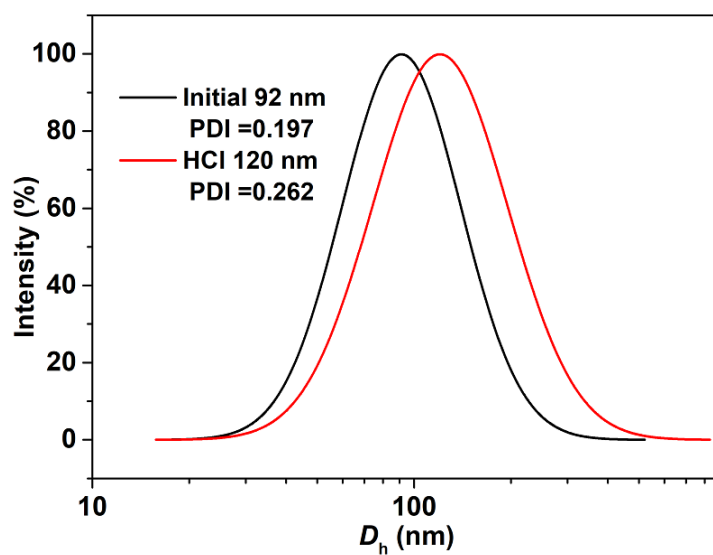**Figure S6**. The FT-IR spectra of P(Et-N-EO$_2$MA), P(THP-N-EO$_2$MA) and P(Ph-N-EO$_2$MA).

**Figure S7**. The temperature and pH-dependent transmittance of the 1.0 wt% aqueous solution of P(THP-N-EO$_2$MA).



**Figure S8** $^1$H NMR spectra of P(Ph-N-EO$_2$MA) after adding acid and bubbling CO$_2$. (pH=5.8).

**Figure S9**. Thermoresponse of P(Ph-N-EO$_2$MA)$_{95}$ in $^n$BuOH (0.5 wt%).



**Figure S10**. The $D_h$ of micelles of P(Ph-N-EO$_2$MA) and after treatment with HCl.