Electronic supplementary information for:

**Multiresponsive of highly water-soluble poly(3-hexylthiophene)-block-poly(phenylisocyanide) block copolymers**

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Fig. S2 $^1$H NMR spectrum of poly-2$_{30}$ in CDCl$_3$ at 25 °C (600 MHz).
Fig. S3 $^1$H NMR spectrum of poly(1$_{30}$-$b$-2$_{60}$) in CDCl$_3$ at 25 °C (600 MHz).

Fig. S4 FT-IR Spectrum of P3HT, PPI homopolymers and P3HT-$b$-PPI block copolymer at 25 °C using KBr pellets.
**Fig. S5** Size exclusion chromatograms of Ni(II)-terminated P3HT macroinitiators and the resulting P3HT-\(b\)-PPI block copolymers. (a) run 2, (b) run 3, (c) run 4, and (d) run 5 in Table 1 in main text.
**Fig. S6** UV-vis absorption spectra of P3HT, PPI, and P3HT-\(b\)-PPI block copolymer measured in CHCl\(_3\) at 25 °C \((c = 0.05\) g/L). 

**Fig. S7** UV-vis spectra of P3HT-\(b\)-PPI block copolymer poly(\(1_{30}\)-\(b\)-\(2_{60}\)) in the mixed solvents of THF and methanol at 25 °C \((c = 0.05\) g/L).
**Fig. S8** Photographs of P3HT-\(b\)-PPI block copolymer poly(1\(_{30}\)-\(b\)-2\(_{60}\)) in the mixed solvents of THF and methanol under UV light (365 nm) (\(c = 0.05\) g/L).

**Fig. S9** Fluorescent spectra of P3HT-\(b\)-PPI block copolymer poly(1\(_{30}\)-\(b\)-2\(_{60}\)) in the mixed solvents of THF and methanol with different volume ratios at 25 °C (\(c = 0.05\) g/L).
**Fig. S10** Dynamic light scattering curve of P3HT-\-b-PPI block copolymer poly(1_{30–b-2_{60}}) in THF (c = 0.1 g/L) measured at 25 °C.

**Fig. S11** Proposed self-assembly structure for P3HT-\-b-PPI block copolymer poly(1_{30–b-2_{60}}) in water at 25 °C.
**Fig. S12** Reversible emission changes of P3HT-\(b\)-PPI block copolymer poly(\(1_{30}-b-2_{60}\)) in CHCl\(_3\) (\(c = 0.05\) g/L) upon alternate addition of TFA (0.15 mM) and TEA (0.15 mM) at 25 °C with excitation at 365 nm.

**Fig. S13** UV-vis spectra of P3HT homopolymer poly-\(1_{30}\) in CHCl\(_3\) upon alternate additions of TFA and TEA at 25 °C (\(c = 0.05\) g/L).
**Fig. S14** $^1$H NMR spectrum of Me-TEG-Ts in CDCl$_3$ at 25 °C (600 MHz).

**Fig. S15** $^1$H NMR spectrum of compound 3 in CDCl$_3$ at 25 °C (600 MHz).
Fig. S16 $^1$H NMR spectrum of compound 4 in CDCl$_3$ at 25 °C (600 MHz).

Fig. S17 $^1$H NMR spectrum of compound 5 in CDCl$_3$ at 25 °C (600 MHz).
Fig. S18 $^{13}$C NMR spectrum of compound 5 in CDCl$_3$ at 25 °C (150 MHz).

Fig. S19 $^1$H NMR spectrum of compound 6 in CDCl$_3$ at 25 °C (600 MHz).
Fig. S20 $^{13}$C NMR spectrum of compound 6 in CDCl$_3$ at 25 °C (150 MHz).

Fig. S21 $^1$H NMR spectrum of compound 7 in CDCl$_3$ at 25 °C (600 MHz).
Fig. S22 $^{13}$C NMR spectrum of compound 7 in CDCl$_3$ at 25 °C (150 MHz).

Fig. S23 $^1$H NMR spectrum of monomer 2 in CDCl$_3$ at 25 °C (600 MHz).
Fig. S24 $^{13}$C NMR spectrum of monomer 2 in CDCl$_3$ at 25 °C (150 MHz).