

Supporting Information

Dual-responsive polypseudorotaxanes based on block-selected inclusion between polyethylene-*block*-poly(ethylene glycol) diblock copolymers and 1,4-diethoxypillar[5]arene

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1. ^1H NMR spectra and GPC traces of PE-*b*-PEGs

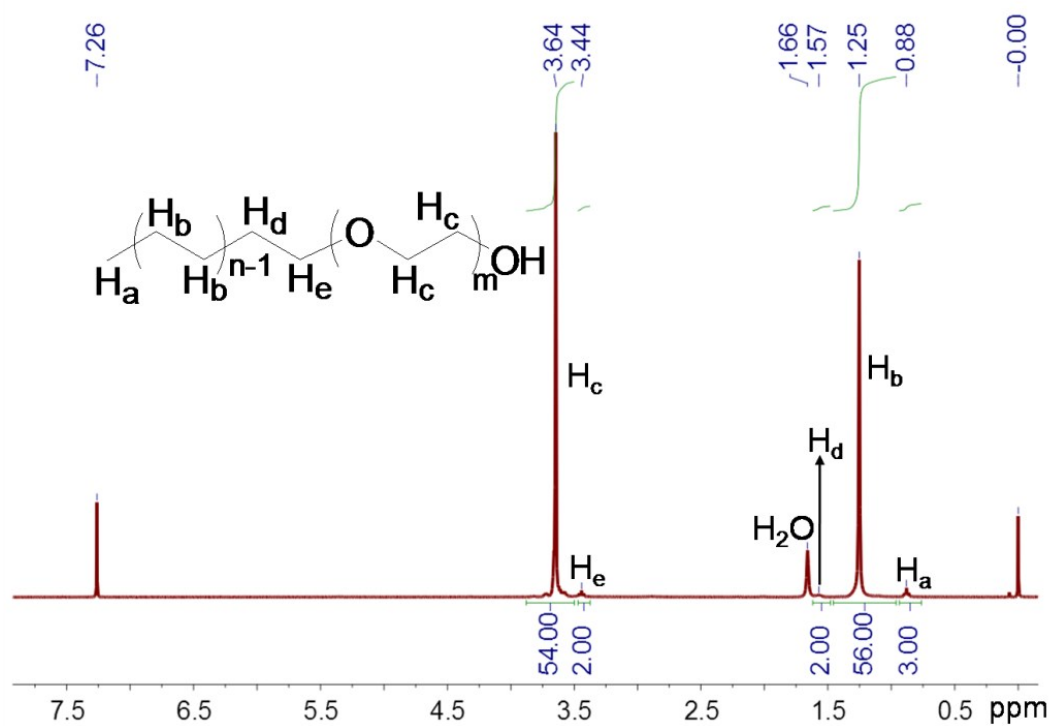


Fig. S1 ^1H NMR spectrum (500 MHz, CDCl_3 , 20 °C) of PE-*b*-PEG₁₄₀₀ (10.0 mg/mL).

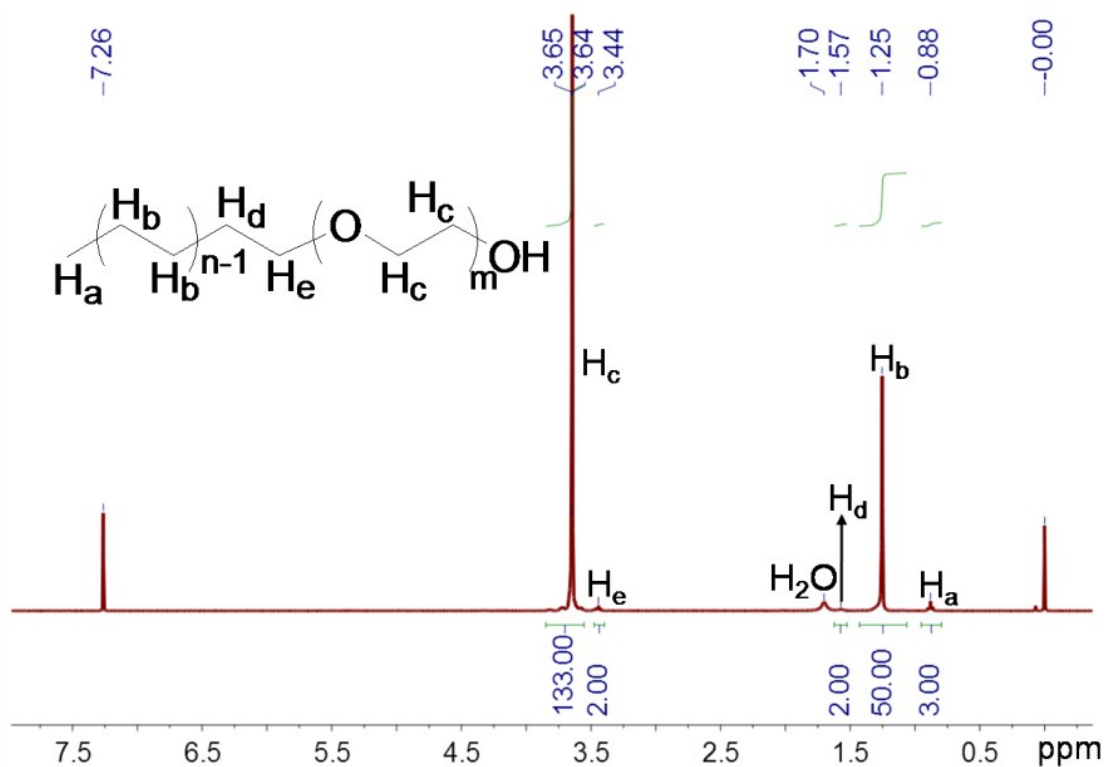


Fig. S2 ^1H NMR spectrum (500 MHz, CDCl_3 , 20 $^\circ\text{C}$) of PE-*b*-PEG₂₂₅₀ (10.0 mg/mL).

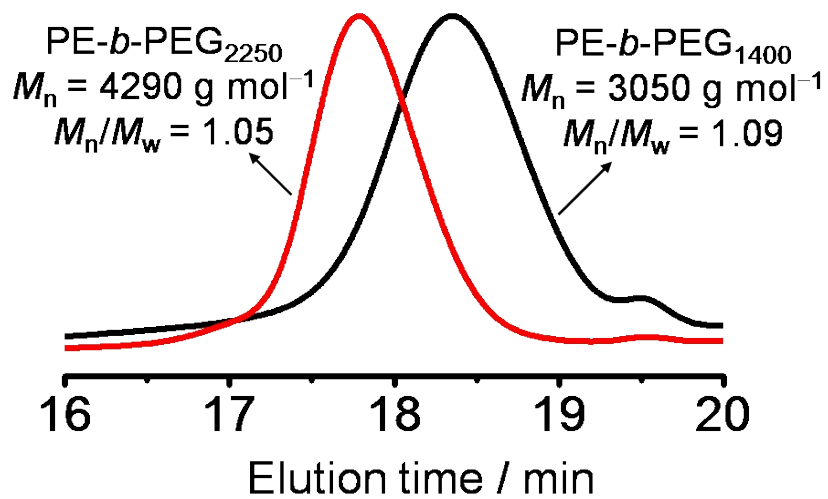


Fig. S3 GPC traces of PE-*b*-PEGs.

2. ^1H NMR spectrum of DEP5A

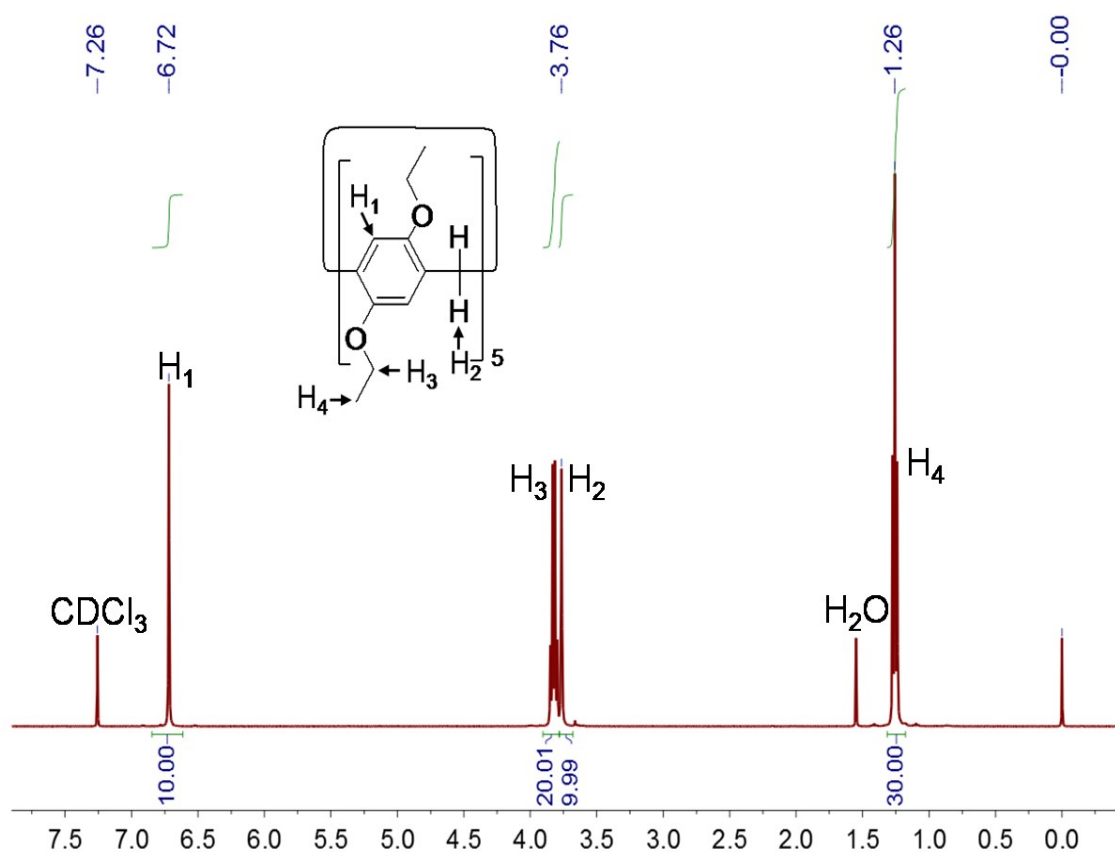


Fig. S4 ^1H NMR spectrum (500 MHz, CDCl_3 , 20 $^\circ\text{C}$) of DEP5A (12.0 mg/mL).

3. ^1H NMR titration of PE-*b*-PEG₂₂₅₀ with DEP5A

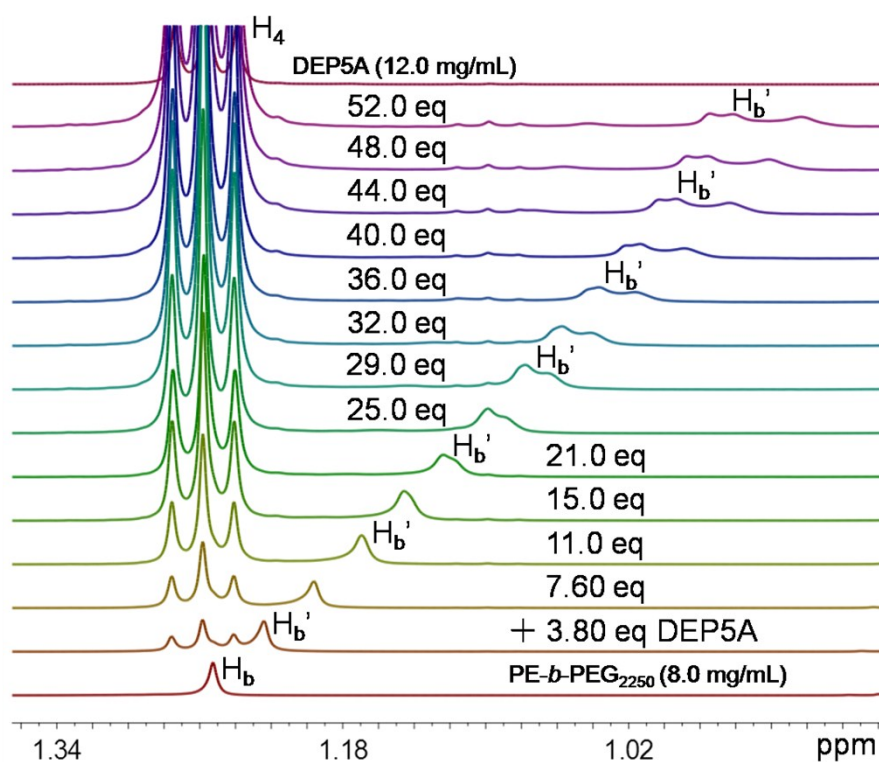


Fig. S5 Partial ^1H NMR spectra (500 MHz, CDCl_3 , 20 $^\circ\text{C}$) of PE-*b*-PEG₂₂₅₀ upon the addition of DEP5A.

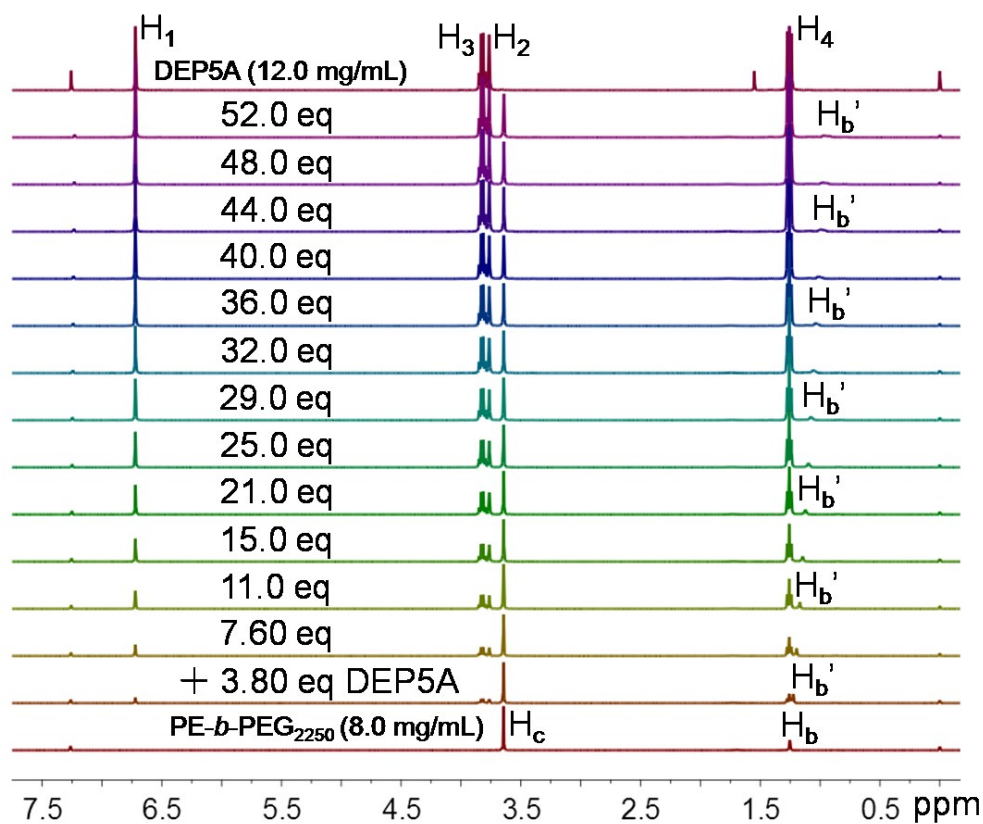


Fig. S6 ^1H NMR spectra (500 MHz, CDCl_3 , 20 $^\circ\text{C}$) of PE-*b*-PEG₂₂₅₀ upon the addition of DEP5A.

4. 2D NOESY NMR of PPR2

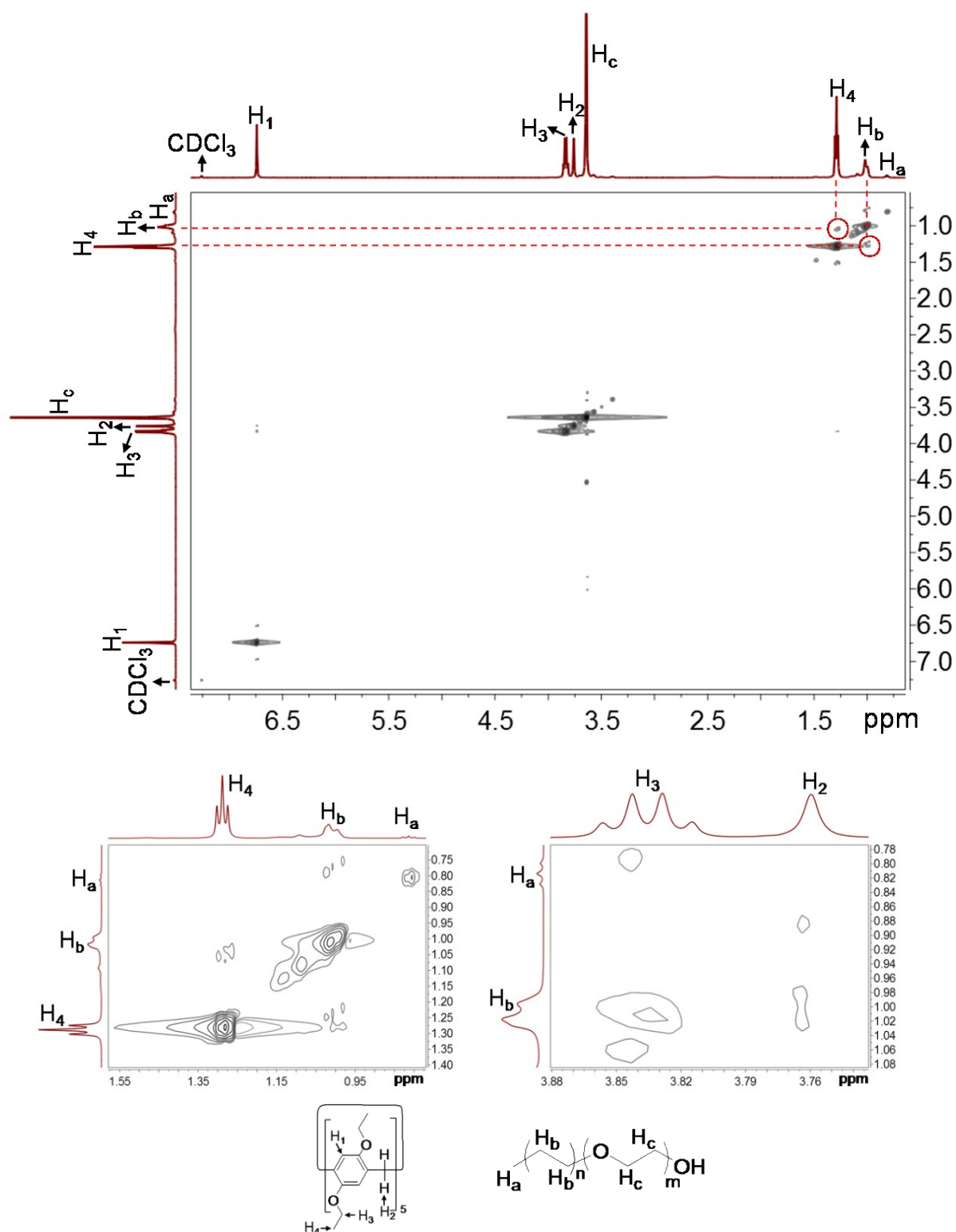


Fig. S7 2D NOESY study of PPR2 (DEP5A15.0 eq, PE-*b*-PEG₂₂₅₀ 8.0 mg/mL) (500 MHz, CDCl_3 , 20 °C).

5. ^1H NMR spectra of PPR2 at different temperatures

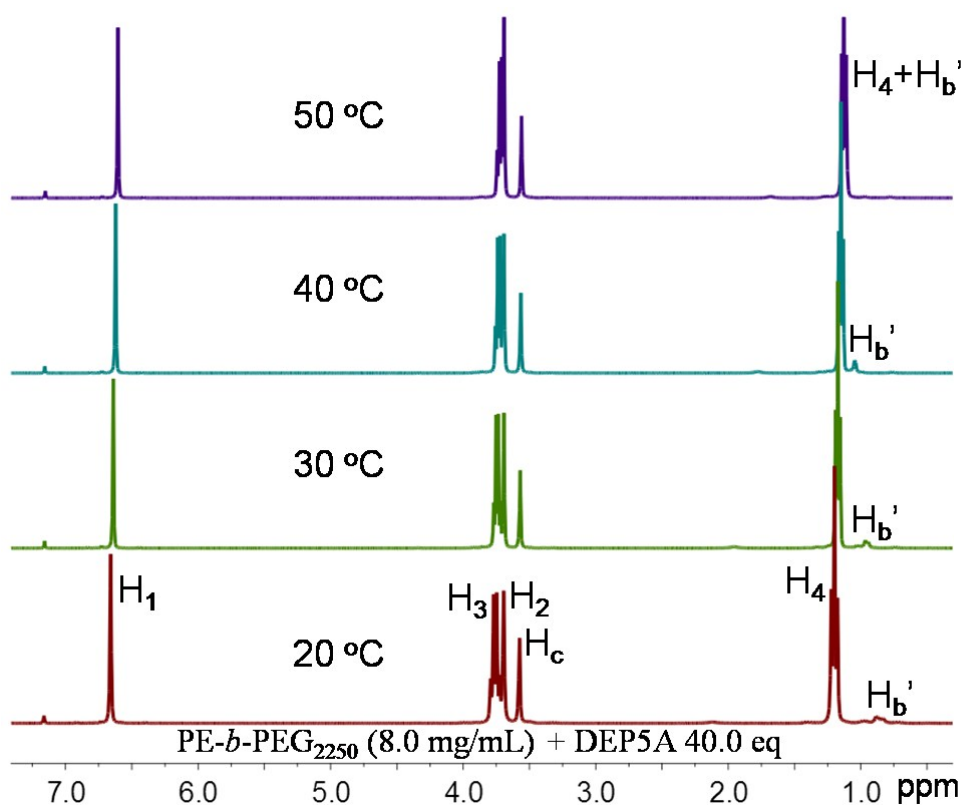


Fig. S8 ^1H NMR spectra (500 MHz, CDCl_3) of PPR2 at different temperatures.

6. Size distributions of PPR1 and PPR2 in CHCl_3 at different temperatures

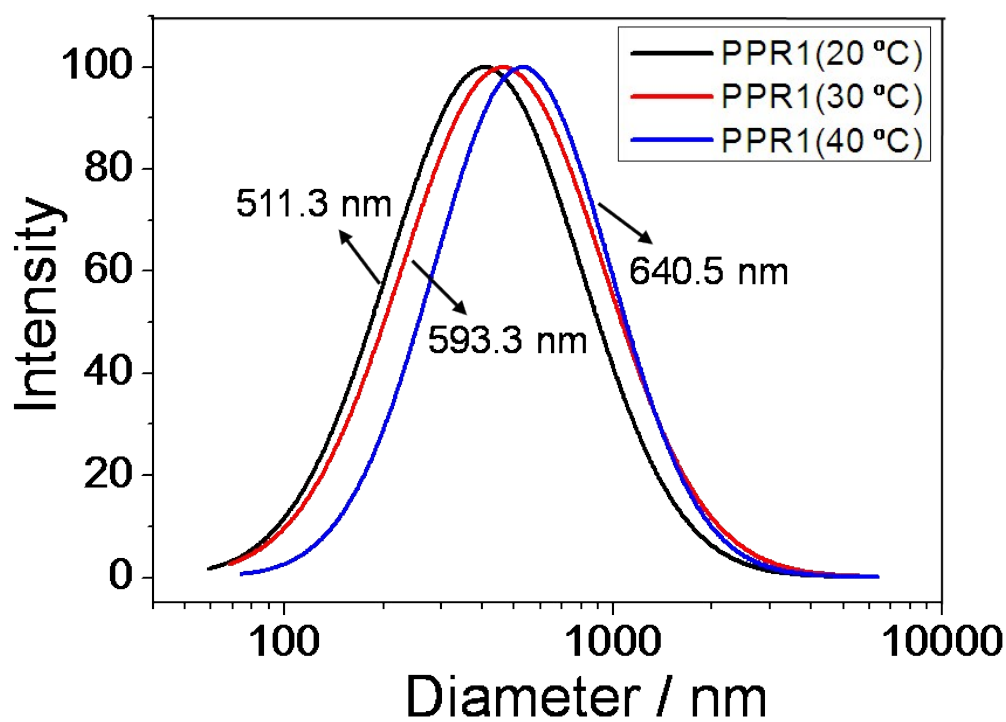


Fig. S9 Size distribution curves of PPR1 (DEP5A 40.0 eq, PE-b-PEG_{1400} 1.0 mg/mL) in CHCl_3 at different temperatures.

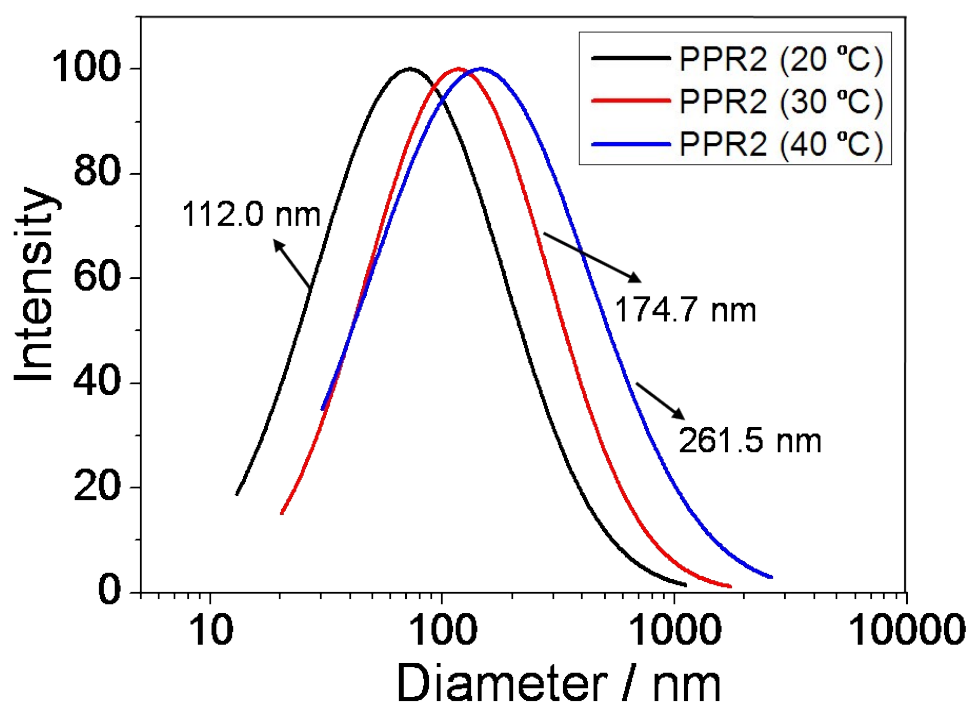


Fig. S10 Size distribution curves of PPR2 (DEP5A 40.0 eq, PE-*b*-PEG₂₂₅₀ 1.0 mg/mL) in CHCl₃ at different temperatures.

7. ¹H NMR titration of PPR2 with DBrBu and hexanedinitrile

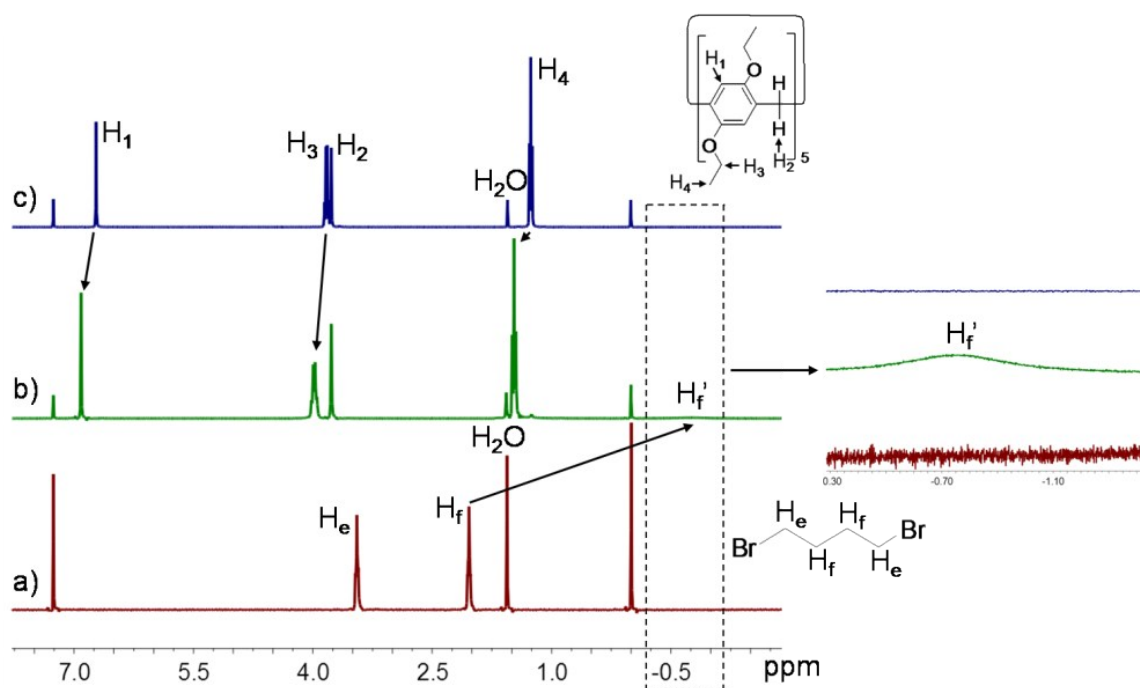


Fig. S11 ¹H NMR spectra (500 MHz, CDCl₃, 20 °C) of DBrBu a), DEP5A c), and their complex (1:1 (mol/mol)) b).

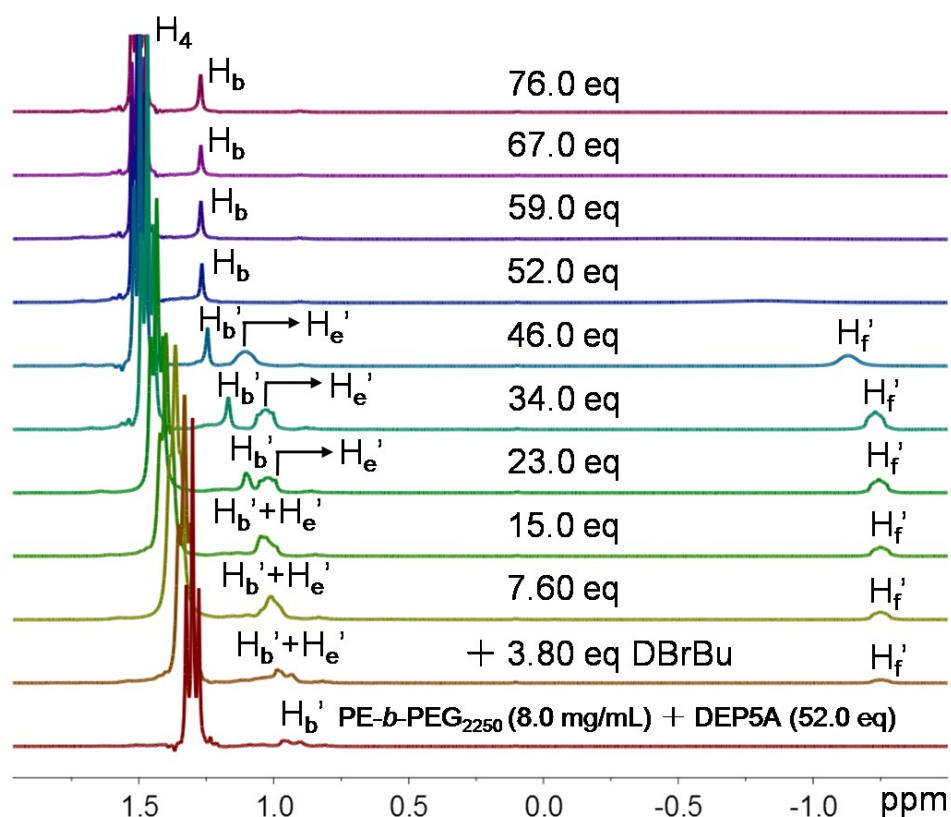


Fig. S12 Partial ^1H NMR spectra (500 MHz, CDCl_3 , 20 $^\circ\text{C}$) of PPR2 upon the addition of DBrBu.

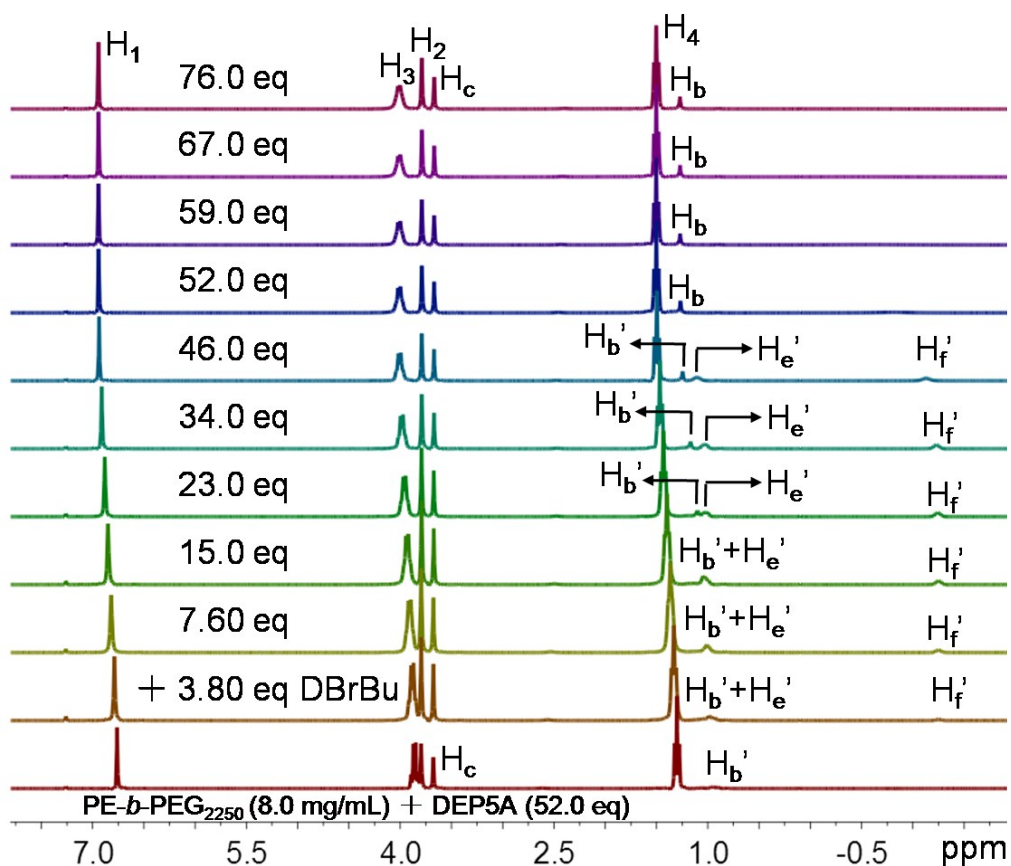


Fig. S13 ^1H NMR spectra (500 MHz, CDCl_3 , 20 $^\circ\text{C}$) of PPR2 upon the addition of DBrBu.

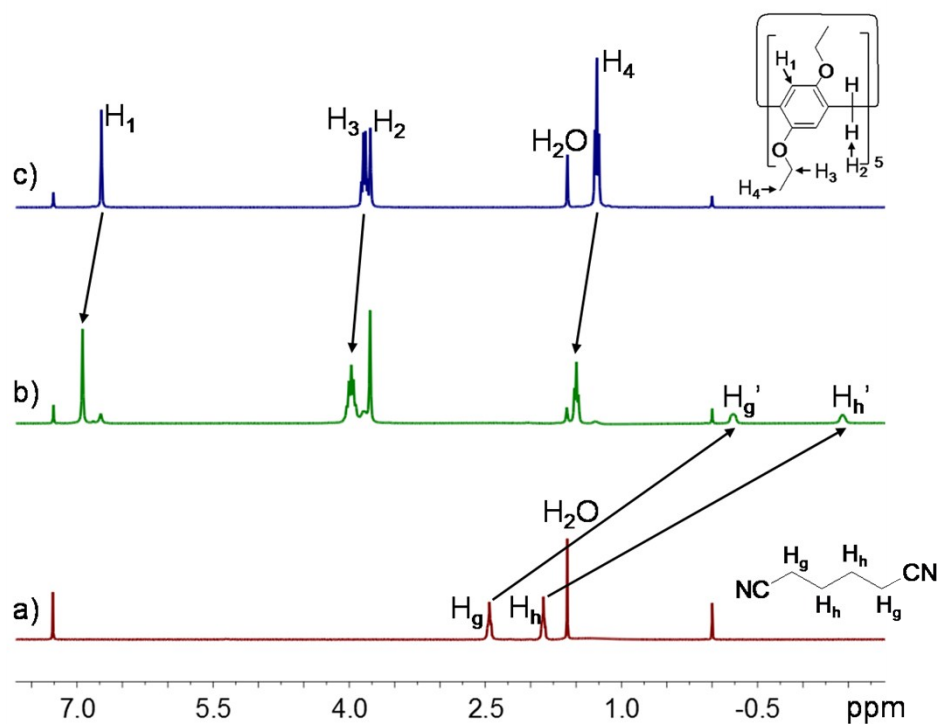


Fig. S14 ^1H NMR spectra (500 MHz, CDCl_3 , 20 $^\circ\text{C}$) of hexanedinitrile a), DEP5A c), and their complex (1:1 (mol/mol)) b).

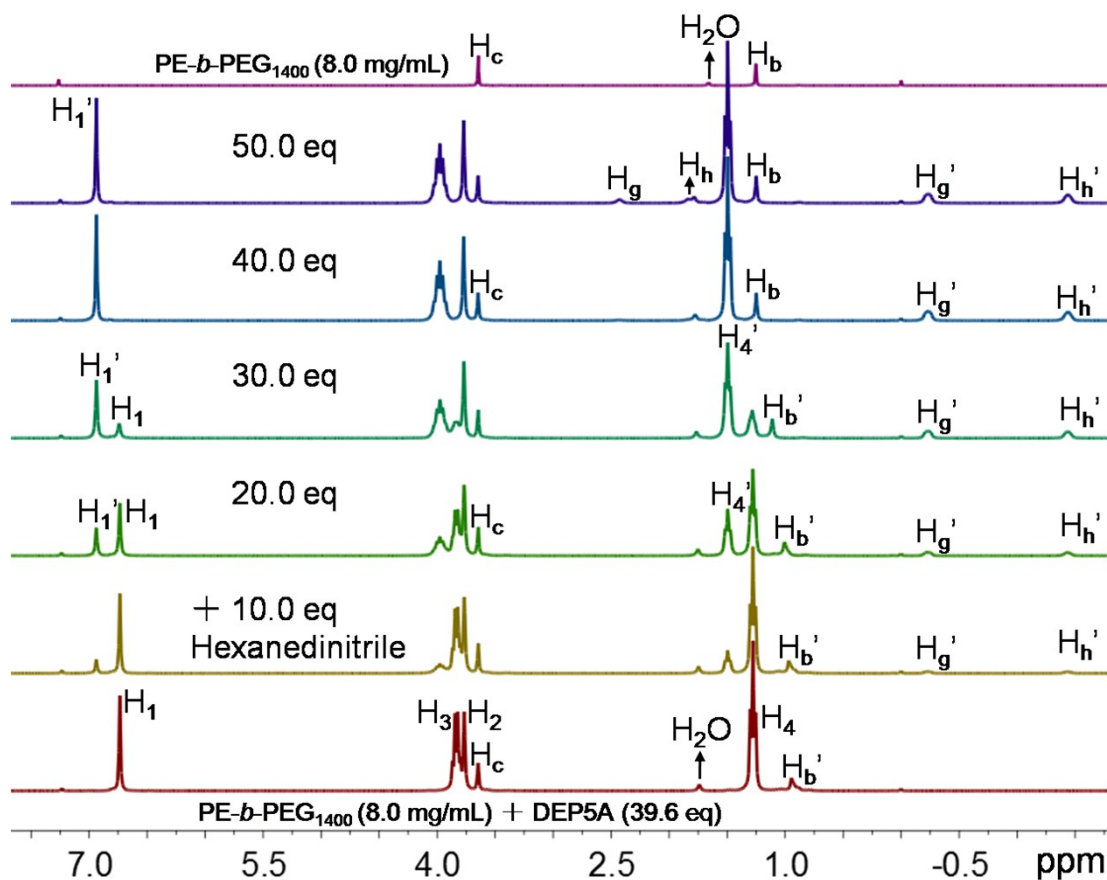


Fig. S15 ^1H NMR spectra (500 MHz, CDCl_3 , 20 $^\circ\text{C}$) of PPR1 upon the addition of hexanedinitrile.

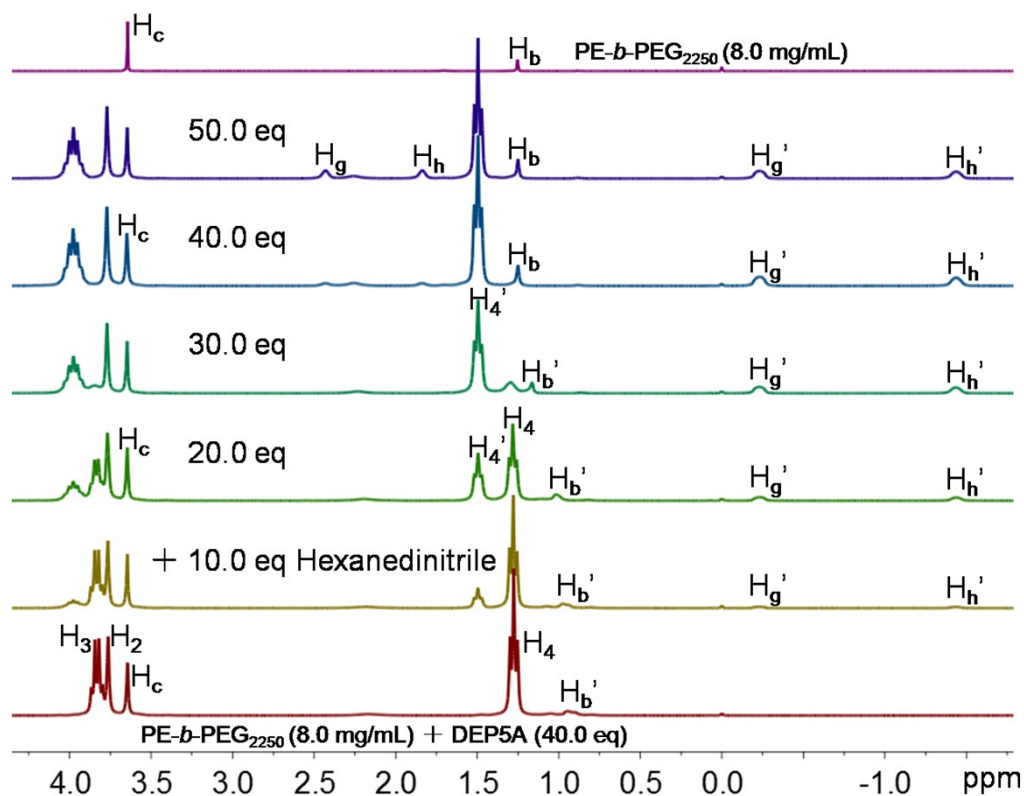


Fig. S16 Partial ^1H NMR spectra (500 MHz, CDCl_3 , 20 $^\circ\text{C}$) of PPR2 upon the addition of hexanedinitrile.

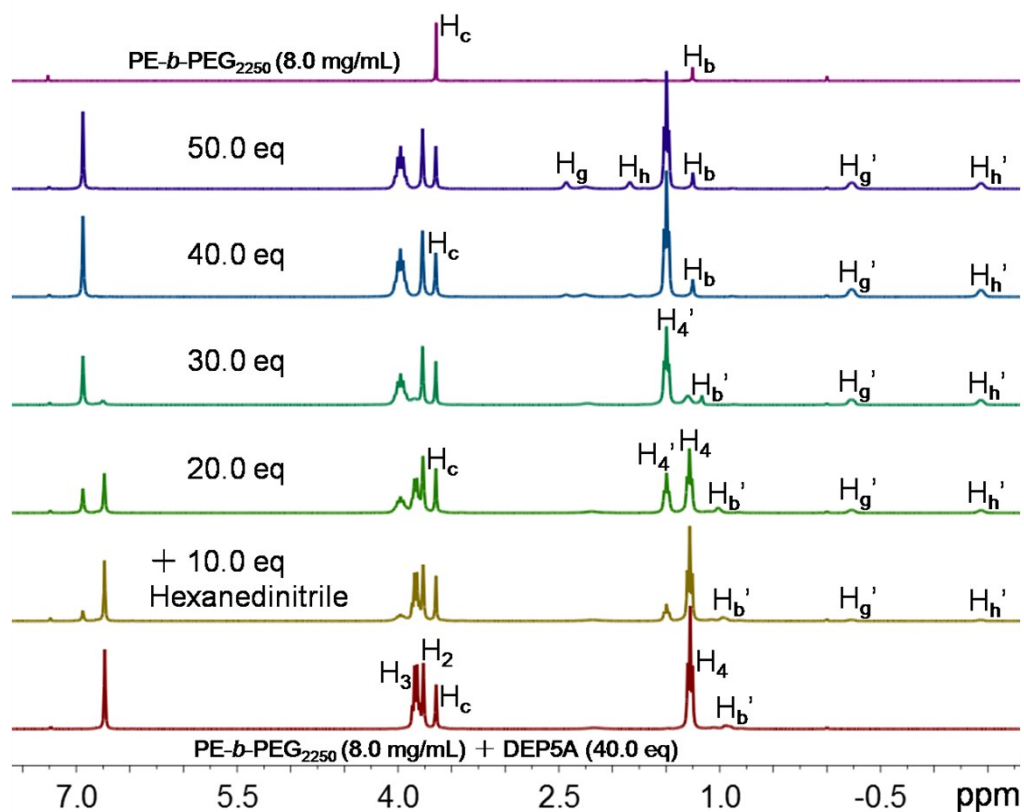


Fig. S17 ^1H NMR spectra (500 MHz, CDCl_3 , 20 $^\circ\text{C}$) of PPR2 upon the addition of hexanedinitrile.

8. FE-SEM images of self-assembled structures of PPRs and desassembled PPRs

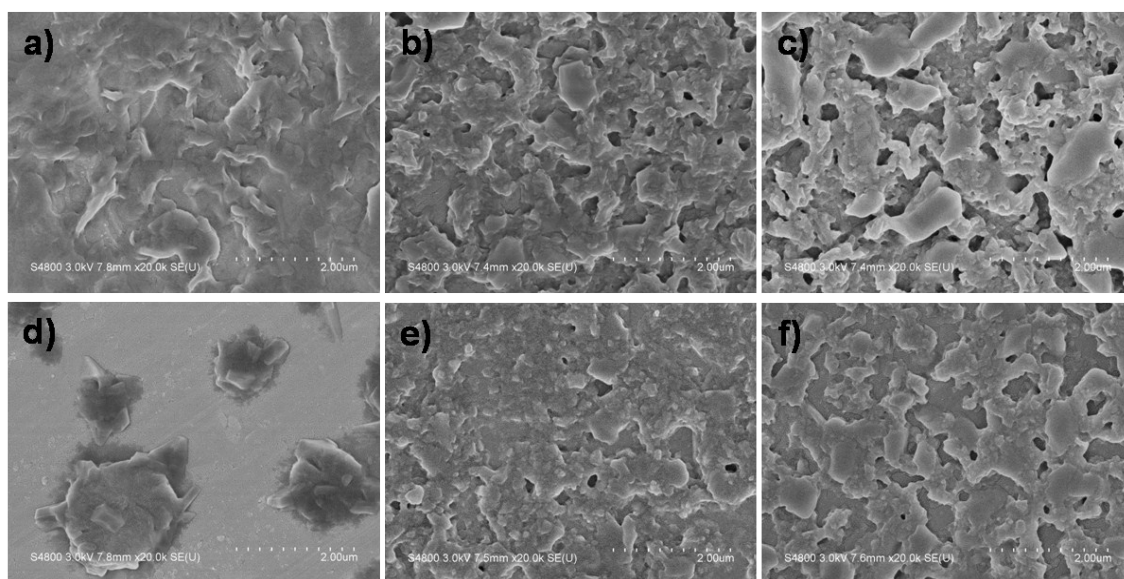


Fig. S18 SEM images of self-assembled structures in CHCl_3 at 20 °C: a) PPR1 (DEP5A 10.0 eq, PE-*b*-PEG₁₄₀₀ 1.0 mg/mL), b) PPR1 (DEP5A 20.0 eq, PE-*b*-PEG₁₄₀₀ 1.0 mg/mL), c) PPR1 (DEP5A 30.0 eq, PE-*b*-PEG₁₄₀₀ 1.0 mg/mL), d) PPR2 (DEP5A 10.0 eq, PE-*b*-PEG₂₂₅₀ 1.0 mg/mL), e) PPR2 (DEP5A 20.0 eq, PE-*b*-PEG₂₂₅₀ 1.0 mg/mL), f) PPR2 (DEP5A 30.0 eq, PE-*b*-PEG₂₂₅₀ 1.0 mg/mL). The scale bar of a)-f) is 2 μm .

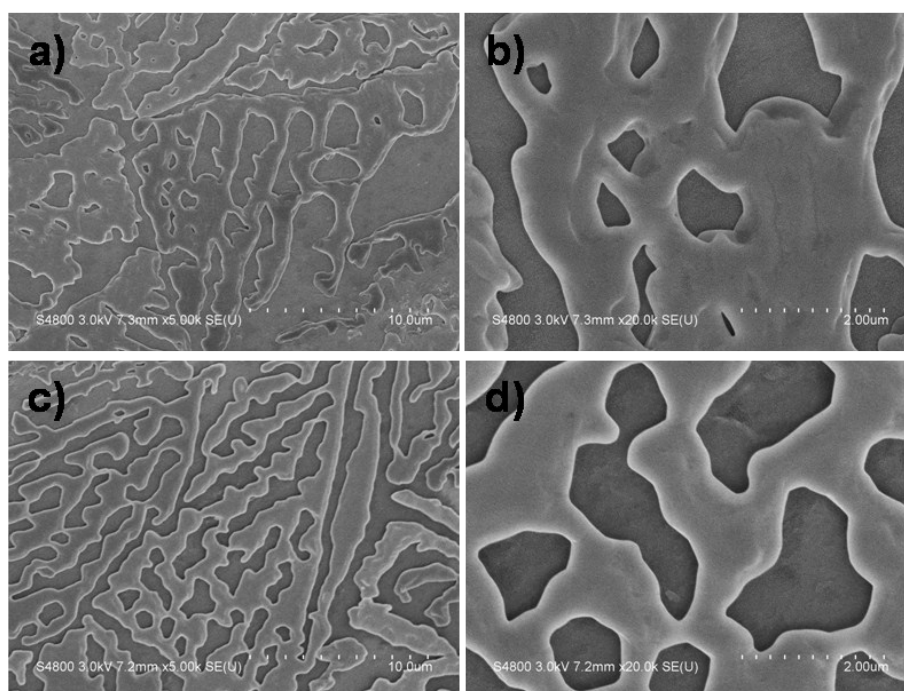


Fig. S19 SEM images of self-assembled structures in CHCl_3 at 20 °C: a) 40.0 eq hexanedinitrile was added to PPR1 (DEP5A 40.0 eq, PE-*b*-PEG₁₄₀₀ 1.0 mg/mL), c) 40.0 eq hexanedinitrile was added to PPR2 (DEP5A 40.0 eq, PE-*b*-PEG₂₂₅₀ 1.0 mg/mL), b) and d) are the magnified pictures of a) and c), respectively. The scale bar of a) and c) is 10 μm ; The scale bar of b) and d) is 2 μm .

9. FE-SEM and TEM images of electrosprayed structures of PPRs, DEP5A and desassembled PPRs

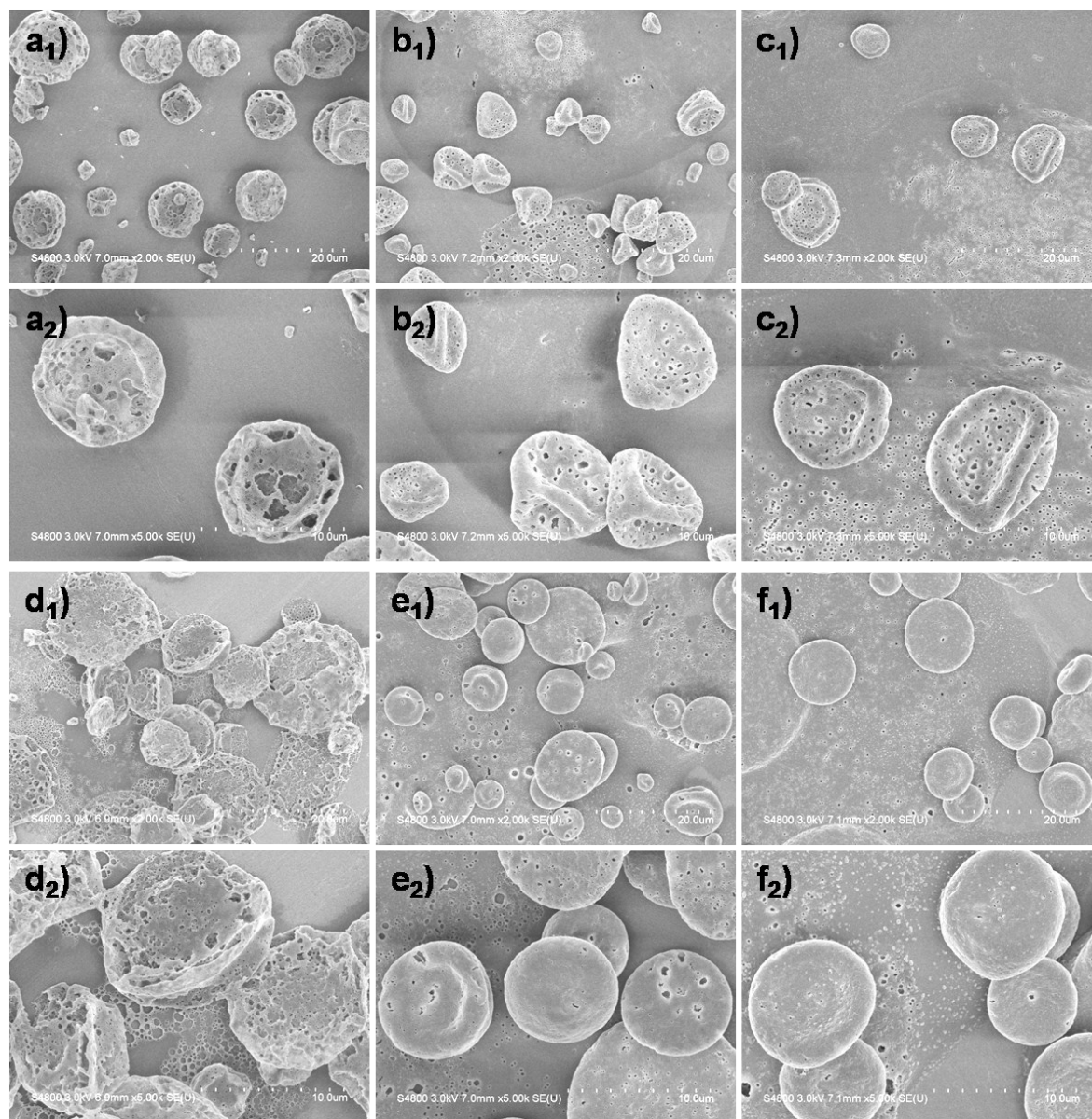


Fig. S20 FE-SEM images of electrosprayed structures: a₁) PPR1 (DEP5A 10.0 eq, PE-*b*-PEG₁₄₀₀ 5.0 mg/mL), b₁) PPR1 (DEP5A 30.0 eq, PE-*b*-PEG₁₄₀₀ 5.0 mg/mL), c₁) (DEP5A 40.0 eq, PE-*b*-PEG₁₄₀₀ 5.0 mg/mL), d₁) PPR2 (DEP5A 10.0 eq, PE-*b*-PEG₂₂₅₀ 5.0 mg/mL), e₁) PPR2 (DEP5A 30.0 eq, PE-*b*-PEG₂₂₅₀ 5.0 mg/mL), f₁) (DEP5A 40.0 eq, PE-*b*-PEG₂₂₅₀ 5.0 mg/mL). a₂), b₂), c₂), d₂), e₂) and f₂) are the magnified images of a₁), b₁), c₁), d₁), e₁) and f₁), respectively. The scale bar of a₁), b₁), c₁), d₁), e₁) and f₁) is 20 μ m. The scale bar of a₂), b₂), c₂), d₂), e₂) and f₂) is 10 μ m.

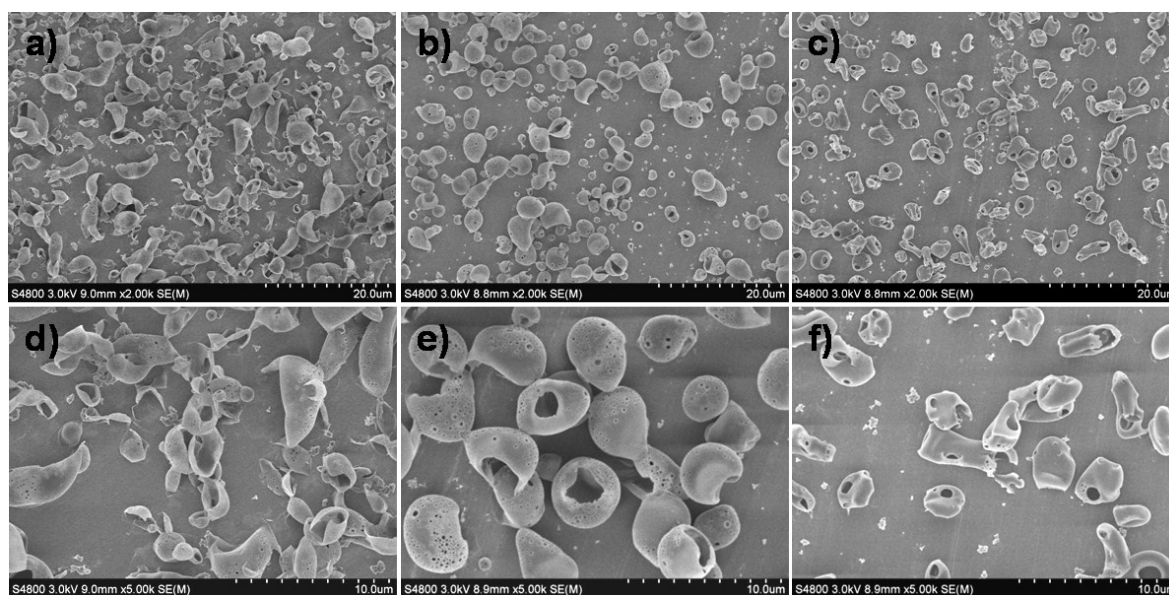


Fig. S21 FE-SEM images of electrospayed structures of DEP5A at different concentrations: a) 10.0 mg/mL, b) 20.0 mg/mL, c) 30.0 mg/mL. d), e) and f) are the magnified images of a), b) and c), respectively. The scale bar of a), b) and c) is 20 μm . The scale bar of d), e) and f) is 10 μm .

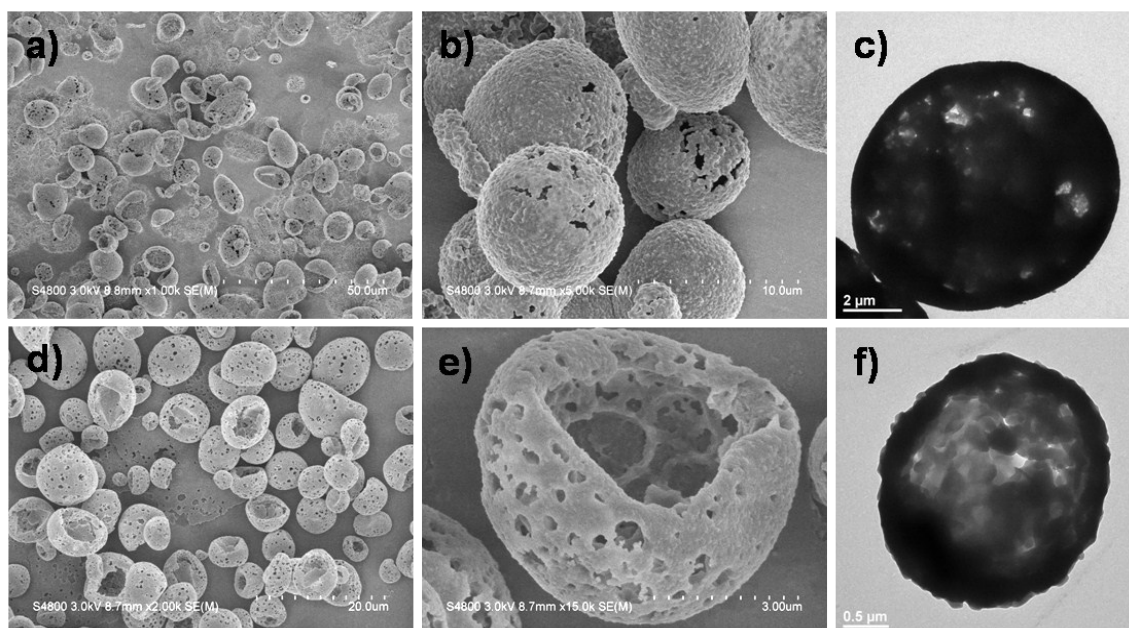


Fig. S22 FE-SEM images of electrospayed structures: a) 20.0 eq DBrBu was added to PPR1 (PE-*b*-PEG₁₄₀₀ 5.0 mg/mL, DEP5A 20.0 eq) (scale bar: 50 μm), d) 20.0 eq DBrBu was added to PPR2 (PE-*b*-PEG₂₂₅₀ 5.0 mg/mL, DEP5A 20.0 eq) (scale bar: 20 μm), b) and e) (scale bars: 10.0 and 3.0 μm) are the magnified images of a) and d), c) and f) (scale bars: 2.0 and 0.5 μm) are the TEM images of b) and e).

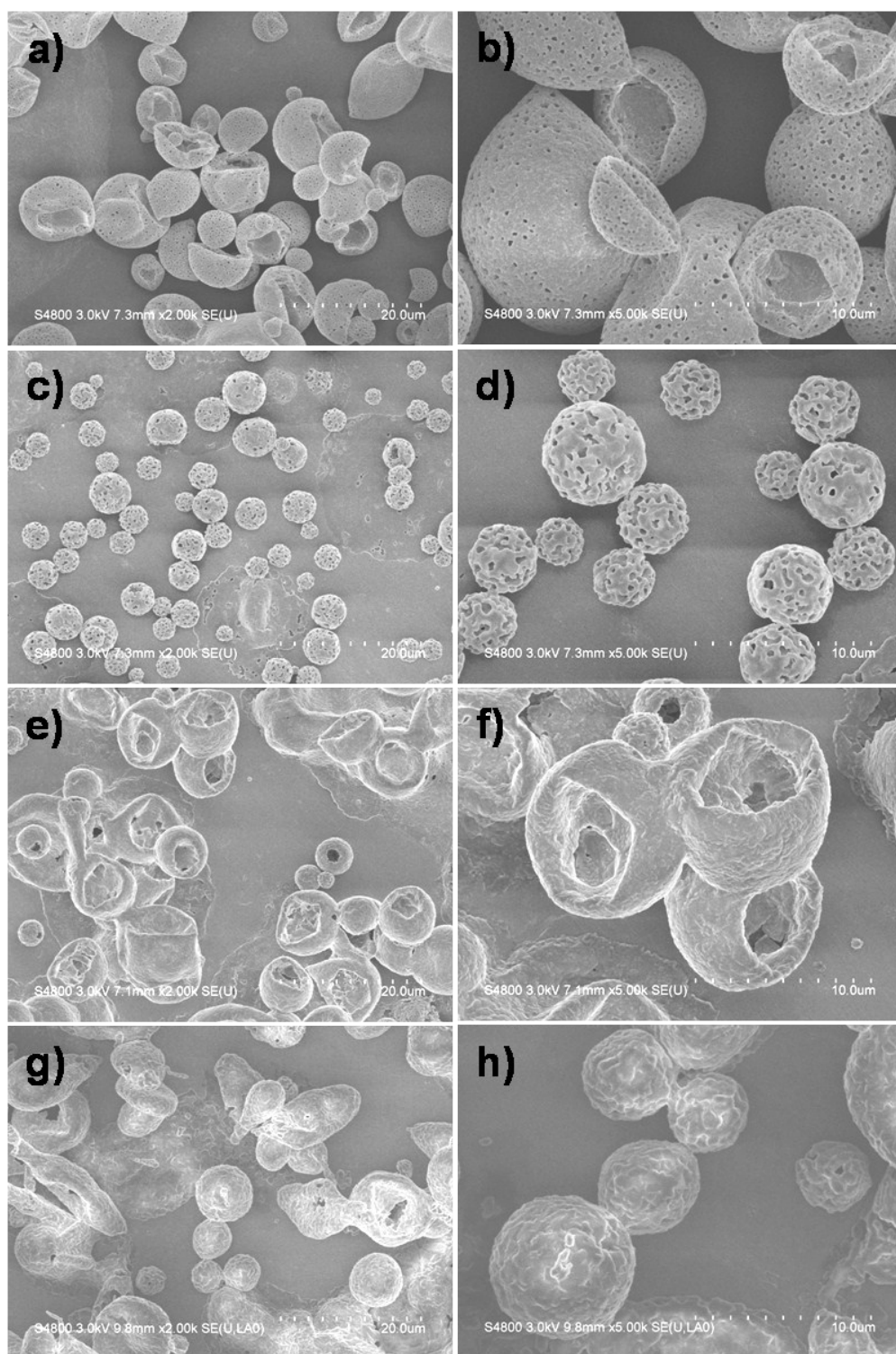


Fig. S23 FE-SEM images of electrospayed structures: a) 40.0 eq DBrBu was added to PPR1 (DEP5A 40.0 eq, PE-*b*-PEG₁₄₀₀ 5.0 mg/mL), c) 40.0 eq hexanedinitrile was added to PPR1 (DEP5A 40.0 eq, PE-*b*-PEG₁₄₀₀ 5.0 mg/mL), b) and d) are the magnified images of a) and c), respectively. e) 40.0 eq DBrBu was added to PPR2 (DEP5A 40.0 eq, PE-*b*-PEG₂₂₅₀ 5.0 mg/mL), g) 40.0 eq hexanedinitrile was added to PPR2 (DEP5A 40.0 eq, PE-*b*-PEG₂₂₅₀ 5.0 mg/mL), b), d), f) and h) are the magnified images of a), c), e) and g), respectively. The scale bar of a), c), e) and g) is 20 μm. The scale bar of b), d), f) and h) is 10 μm.