

## Supporting Information

### **Fabrication of poly(4-vinylpyridine)-*b*-polystyrene-*b*-poly(4-vinylpyridine) triblock copolymer particles via three-dimensional soft confined self-assembly**

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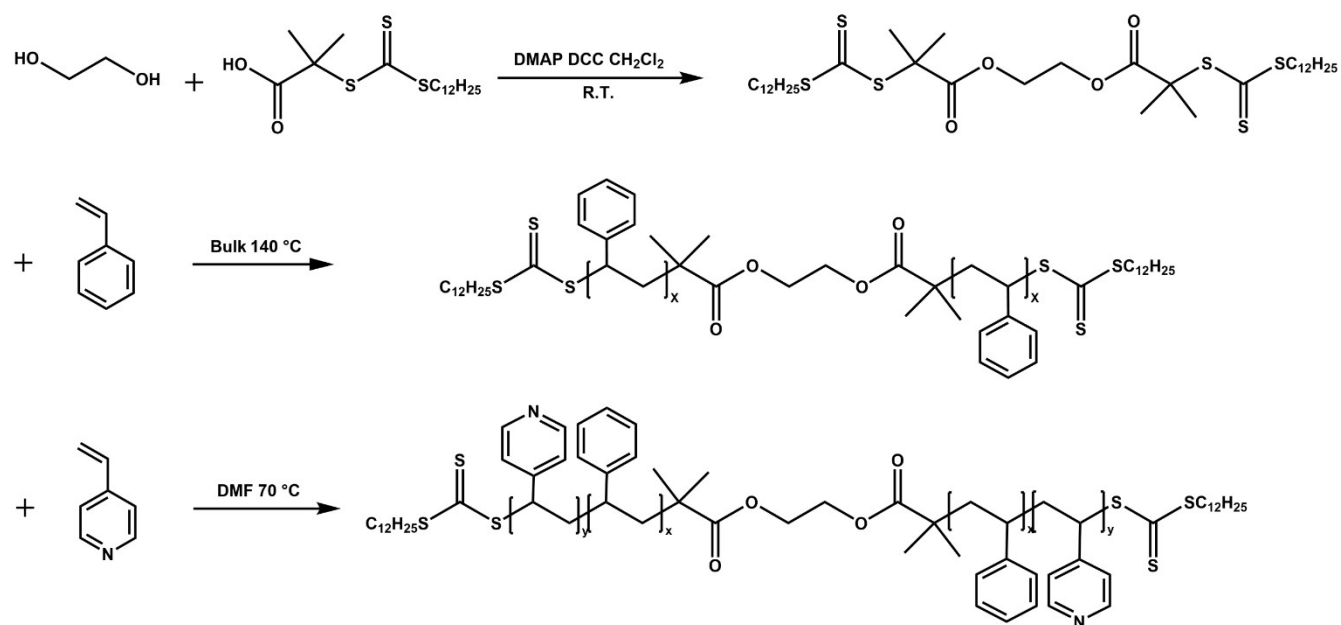
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## CONTENTS:

1. Synthesis routine of P4VP-b-PS-b-P4VP triblock copolymers (**Scheme S1**).
2. Characteristics of  $S_{12.2k}V_n$  diblock copolymers (**Table S1**).
3. GPC traces of CTA- $PS_{24.2k}$ -CTA (**Figure S1**),  $PS_{12.2k}$ -CTA (**Figure S2**) and  $S_{12.2k}V_n$  diblock copolymers (**Figure S3**).
4.  $^1\text{H-NMR}$  spectra of  $PS_{12.2k}$ -CTA and  $S_{12.2k}V_n$  diblock copolymers (**Figure S4**).
5. SEM images of  $S_{12.2k}V_{3.7k}$  triblock copolymer particles obtained via 3D-SCSA with various CTAB concentrations (mg/mL) of (a) 0.1, (b) 0.5, (c) 1.0 and SEM images of  $S_{12.2k}V_{12k}$  diblock copolymer particles obtained via 3D-SCSA with various CTAB concentration (mg/mL) of (d) 0.1, (e) 0.5, (f) 1.0 (**Figure S5**).

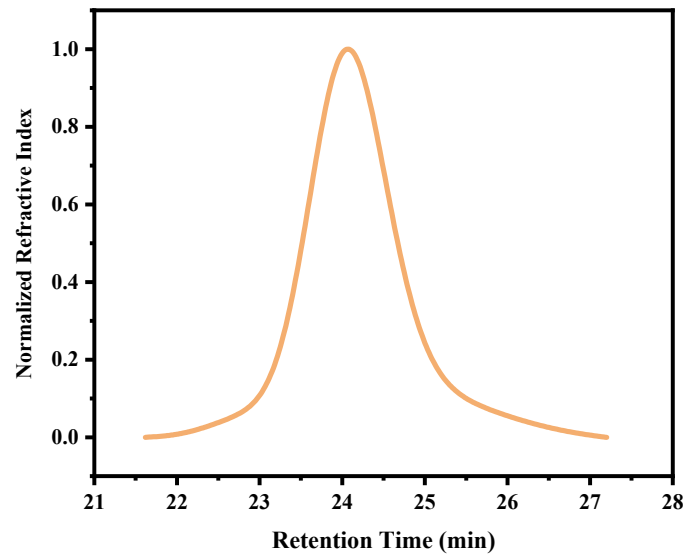


**Scheme S1.** Synthesis routine of P4VP-*b*-PS-*b*-P4VP triblock copolymers.

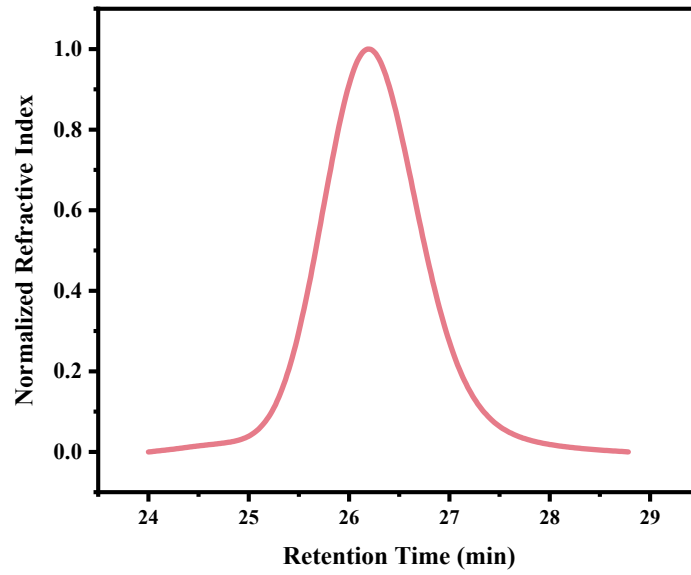
**Table S1.** Characteristics of  $S_{12.2k}V_n$  diblock copolymers.

Sample <sup>a</sup>	$f_{PS}^b$	$f_{P4VP}^b$	$\mathcal{D}^c$
$S_{12.2k}V_{3.7k}$	77.8	22.2	1.09
$S_{12.2k}V_{12.0k}$	51.5	48.4	1.17

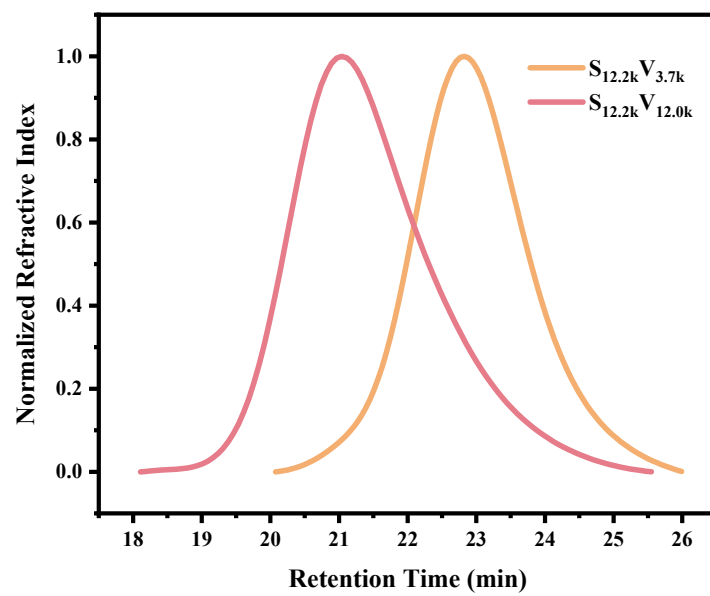
<sup>a</sup>Determined by <sup>1</sup>H NMR. <sup>b</sup>*f* represents the volume fraction of PS or P4VP blocks. The densities of PS and P4VP are 1.05 g/cm<sup>3</sup> and 1.10 g/cm<sup>3</sup>, respectively. <sup>c</sup>Polydispersity indexes ( $\mathcal{D}$ ) were obtained by GPC.



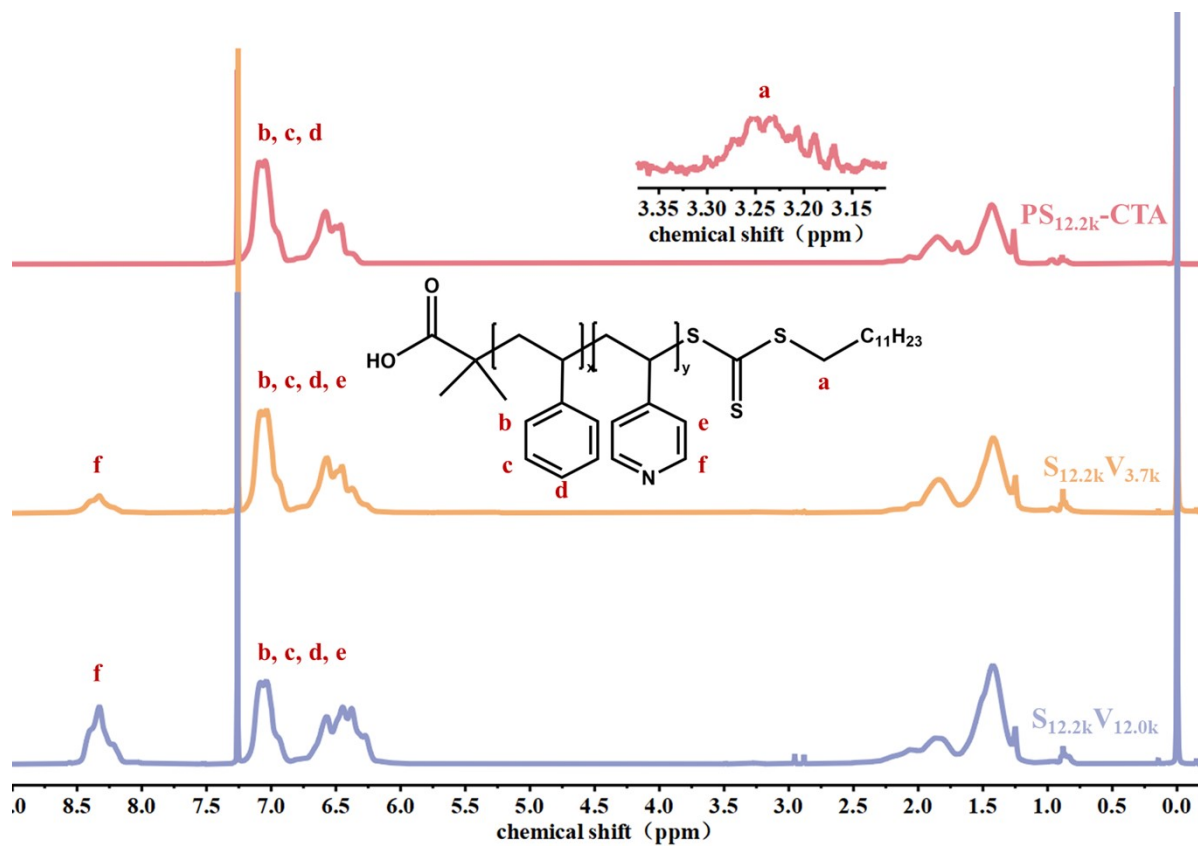
**Figure S1.** GPC trace of CTA-PS<sub>24.2k</sub>-CTA.



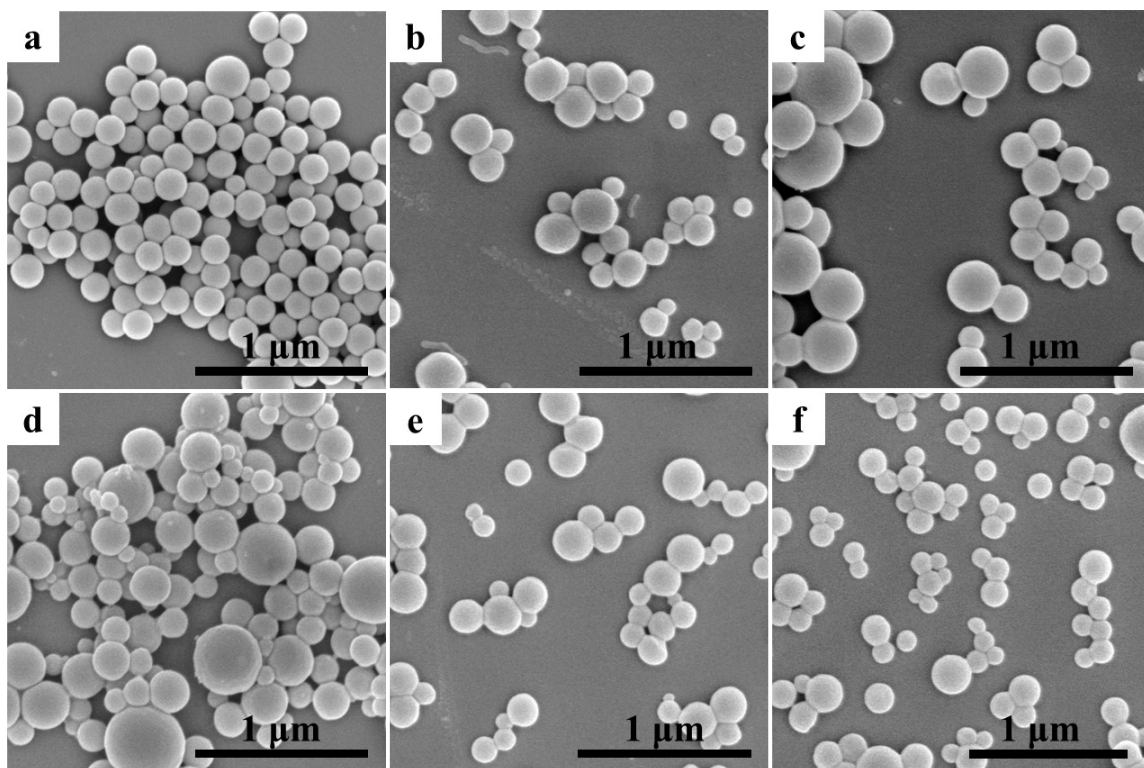
**Figure S2.** GPC trace of PS<sub>12.2k</sub>-CTA.



**Figure S3.** GPC traces of  $S_{12.2k}V_n$  diblock copolymers.



**Figure S4.** <sup>1</sup>H NMR spectra of PS<sub>12.2k</sub>-CTA and S<sub>12.2k</sub>V<sub>n</sub> diblock copolymer in CDCl<sub>3</sub>.



**Figure S5.** SEM images of  $S_{12.2k}V_{3.7k}$  diblock copolymer particles obtained via 3D-SCSA with various CTAB concentration (mg/mL) of (a) 0.1, (b) 0.5, and (c) 1.0. SEM images of  $S_{12.2k}V_{12.0k}$  diblock copolymer particles obtained via 3D-SCSA with various CTAB concentration (mg/mL) of (d) 0.1, (e) 0.5, and (f) 1.0.