Supporting Information

## Amino Acid Substituted Polyacetylene Based Chiral Core-Shell Microspheres: Helical Structure Induction and Application for Chiral Resolution and Adsorption

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About enantioselective crystallization: The prepared chiral composite microspheres are put into a simple separation column, which contains a hollow small test tube. There is a filter film at the bottom of the small test tube, and the bottom was sealed with a plastic film. Remove the preservative film and centrifuge after crystallization was completed. The crystals and microspheres remained in the small test tube, and the supernatant flows into the outer test tube. Chiral adsorption: put the prepared chiral microspheres into the filter paper, and then the steps are the same as above.



Fig. S1. Enantioselective crystallization and adsorption

A series of parallel experiments with different monomer concentrations of DVB-55 were carried out to obtain microspheres with uniform particle size and good morphology, as follows, anhydrous acetonitrile (29.4 mL, 29.2 mL, 28.9 mL, 28.8 mL, 28.6 mL), DVB-55 (0.6 mL, 0.8 mL, 1.1 mL, 1.2 mL, 1.4 mL; 2.0 vol.%, 2.7 vol.%, 3.6 vol.%, 4.0 vol.%, 4.6 vol.% relative to total volume), AIBN (12 mg, 16 mg, 22 mg, 24 mg, 28 mg, 2.0 wt.% relative to DVB55).



**Fig. S2.** The SEM and particle size distributions for PDVB microspheres with different volume fractions of DVB-55; 2 vol.% (A); 2.7 vol.% (B); 3.6 vol.% (C); 4.0 vol.% (D); 4.6 vol.% (E) and 2 vol.% (a); 2.7 vol.% (b); 3.3 vol.% (c); 3.6 vol.% (d); 4 vol.% (e); 4.6 vol.% (f)



Fig. S3. A comparison between the NMR spectra of P-M1-co-M2-Boc and P-M1-co-M2-NH<sub>2</sub>



Fig. S4. A comparison between the FT-IR spectra of P-M1-co-M2-Boc and P-M1-co-M2-NH $_2$ 



**Fig. S5.** The GPC curves of P-M1-*co*-M2-Boc (A) and P-M1-*co*-M2-NH<sub>2</sub> obtained from P-M1-*co*-M2-Boc (B)





**Fig. S7.** A, CD and UV-vis spectroscopy of the residual solution of enantioselective crystallization: PDVB (a); V-a (b); V-b (c); V-c (d); V-d (e)



Fig. S8. SEM images of Boc-L-alanine crystals growing induced by (L)-chiral core-shell microspheres.



**Fig. S9.** X-ray diffraction patterns of (a) pure Boc-L-alanine crystals and (b) Boc-L-alanine crystals were induced by (L)-chiral composite microspheres

Run	Optical rotation										
Pure amino acid	Boc-D-A	la (15 mg/n	nl)	Boc-D-Asn (5 mg/ml)			Met (5 mg/ml)		Pro (5 mg/ml)		
	0	.3692			-0.0671			0.0394		0.4745	
Time (h)	5	10	1	5	20	30		40		50	60
the Boc-Ala in	0.0332	0.0478	0.0	627	0.0812	0.125	5	0.2215	0.	3138	0.3507
excess in the											
residual solution											
(L-V-a)											
the Boc-Ala in	0.0443	0.0664	0.1	107	0.1476	0.262	l	0.328	0.	3510	0.3510
excess in the											
residual solution											
(L-V-b)											
the Boc-Ala in	0.0485	0.0728	0.0	956	0.1661	0.2879	)	0.3322	0.	3508	0.3508
excess in the											
residual solution											
(L-V-c)											
the Boc-Asn in	-0.0060	-0.0080	-0.0	150	-0.0190	-0.031	2	-0.0435	-0.	.0577	-0.0600
excess in the											
residual solution											
(L-V-c)											
the Met in	0.0035	0.0055	0.0	078	0.0102	0.015	7	0.0216	0.	0263	0.0307
excess in the											
residual solution											
(L-V-c)											

**Table S1.**The optical rotation of amino acid in excess in the residual solution

the Pro in	0.0379	0.0616	0.0901	0.1328	0.1945	0.3084	0.3368	0.3796
excess in the								
residual solution								
(L-V-c)								