Supplementary information

The separation characteristics and performance evaluation of the silica-based poly(pentabromostyrene) stationary phase in capillary electrochromatography

Zhihua Zhong, Zhanying Chu, Ziyi Dong, Weibing Zhang*, Lingyi Zhang*

Shanghai Key Laboratory of Functional Materials Chemistry, School of Chemistry and Molecular Engineering, East China University of Science and Technology, Shanghai, 200237, PR China

*Corresponding author
Prof. Weibing Zhang
School of Chemistry and Molecular Engineering
East China University of Science and Technology
Shanghai, 200237, PR China
E-mail: weibingzhang@ecust.edu.cn

Dr. Lingyi Zhang
School of Chemistry and Molecular Engineering
East China University of Science and Technology
Shanghai, 200237, PR China
E-mail: zhanglingyi@ecust.edu.cn
1. Synthesis of pentabromostyrene

1.6 g of FeBr$_3$ and 56 mL of Br$_2$ were added into a round-bottom flask in an ice bath. 16.4 mL of (2-bromoethyl)benzene was added dropwise under magnetic stirring and the reaction had continued for 24 h. Dichloromethane was added and the solution was washed thoroughly with sodium sulfite solution (1 mol/L). Then the organic phase was evaporated to obtained a white product, which is (2-bromoethyl)pentabromobenzene. Then, 25 g of the obtained (2-bromoethyl)pentabromobenzene and 200 mL of ethyl alcohol (EtOH) were refluxed at 86 °C for 2 h after the addition of 20 mL of KOH solution in EtOH (0.13 g/mL). The mixture was filtered, washed with water and dried in the oven to get the product pentabromostyrene.

The poly(pentabromostyrene) bonded silica (SiO$_2$@pPBS) was synthesized by a surface-initiated free-radical polymerization procedure. Firstly, 10 g of activated silica and 12 g of APTES were dispersed into 100 mL of toluene in a round-bottom flask and refluxed at 110 °C for 24 h. The solid product (named as SiO$_2$@NH$_2$) was washed with MeOH and dispersed into 100 mL of DMF solution containing 7.5 g of EEDQ and 4.2 g of ACV. The mixture was stirred for 12 h at room temperature. The white product (SiO$_2$@ACV) was washed with DMF and CH$_2$Cl$_2$ subsequently and dried at 25 °C in the oven. Finally, 3 g of SiO$_2$@ACV and pentabromostyrene were dispersed into 30 mL of EGMME and this reaction took place under refluxing at 70 °C for 24 h. The final product (SiO$_2$@pPBS) was washed with EtOH and then dried in the oven at 60 °C.

**Fig. S1** N$_2$ adsorption/desorption experimental results of SiO$_2$ (A) and SiO$_2$@pPBS (B).
**Fig. S2** The optical microscope image (A) and SEM image (B) of SiO$_2$@pPBS capillary packed column.

**Fig. S3** The influence of buffer pH on the EOF mobility of SiO$_2$@pPBS capillary packed column. Conditions: 40 cm capillary (22 cm effective length) × 75 μm i.d.; mobile phase, 5 mmol/L phosphate buffer with 60% ACN; applied voltage, 15 kV; injection, 5 kV × 5 s; detection wavelength, 214 nm.

**Fig. S4** The influence of buffer concentration on the EOF mobility of SiO$_2$@pPBS capillary packed column. Conditions: mobile phase, phosphate buffer with 60% ACN at pH 7; other
conditions were same as Fig. S3.

**Fig. S5** The influence of ACN concentration on the EOF mobility of SiO$_2$@pPBS capillary packed column. Conditions: mobile phase, 5 mmol/L phosphate buffer at pH 7; other conditions were same as Fig. S3.

**Fig. S6** Electrochromatogram of alkylbenzenes. Conditions: mobile phase, 5 mmol/L phosphate buffer with 60% ACN at pH 7; other conditions were same as Fig. S3.

**Fig. S7** Electrochromatogram of polycyclic aromatic hydrocarbons. Conditions: mobile phase, 5 mmol/L phosphate buffer with 90% ACN at pH 7; other conditions were same as Fig.
**Fig. S8** Electrochromatograms of five nucleosides under different ACN concentration.

Conditions: mobile phase, 5 mmol/L phosphate buffer at pH 7; other conditions were same as Fig. S3.

**Fig. S9** The chromatographic selectivity comparison of (A) SiO$_2$@pPBS capillary packed column and (B) C18 column. Conditions: (A) mobile phase: 5 mmol/L phosphate buffer with 80% ACN at pH 7; (B) mobile phase: 5 mmol/L phosphate buffer with 60% ACN at pH 7; other conditions were same as Fig. S3. Analytes: 1. Toluene, 2. Chlorobenzene, 3. Bromobenzene.
Fig. S10 Evaluation of stability of SiO$_2$@pPBS capillary packed column at pH 4 (A) and pH 7 (B). Conditions: 5 mmol/L phosphate buffer with 40% ACN, other conditions were same as Fig. S3. Analytes: 1. Acetone, 2. Thiourea, 3. Sulphanilamide, 4. Sulfaguanidine.