Supplementary information

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

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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_2Cl_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.

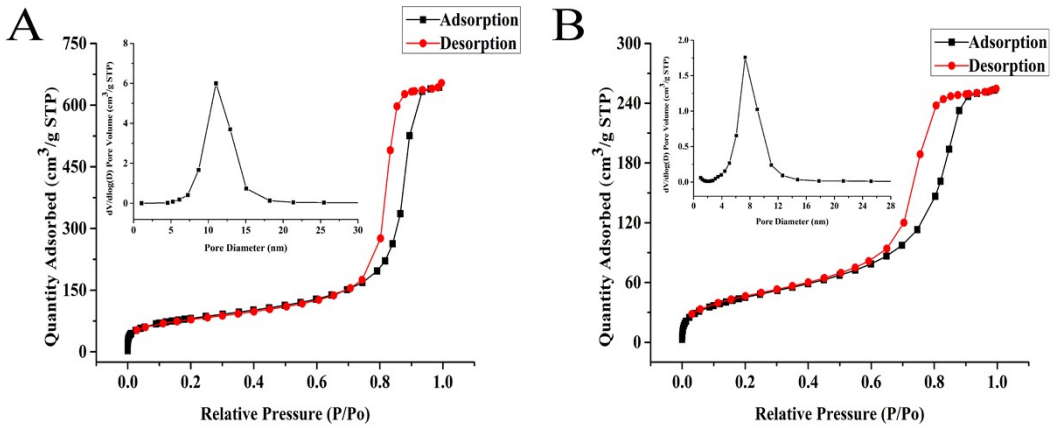


Fig. S1 N_2 adsorption/desorption experimental results of SiO_2 (A) and SiO_2@pPBS (B).

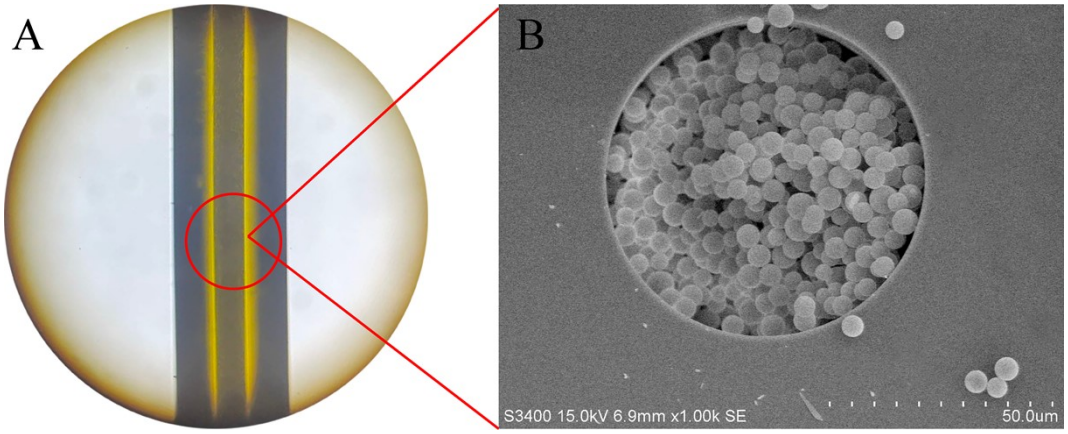


Fig. S2 The optical microscope image (A) and SEM image (B) of SiO_2@pPBS capillary packed column.

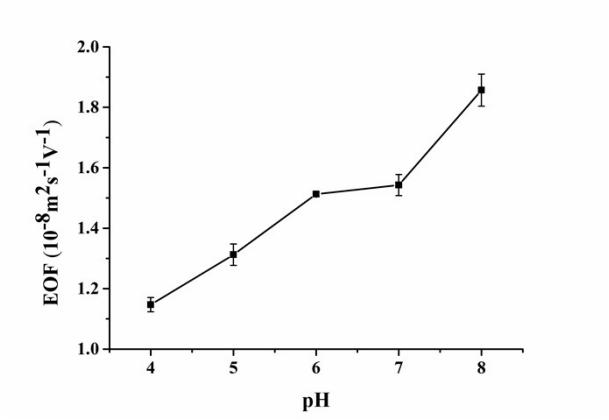


Fig. S3 The influence of buffer pH on the EOF mobility of SiO_2@pPBS capillary packed column. Conditions: 40 cm capillary (22 cm effective length) ×75 μm i.d.; mobile phase, 5 mmol/L phosphate buffer with 60% ACN; applied voltage, 15 kV; injection, 5 kV × 5 s; detection wavelength, 214 nm.

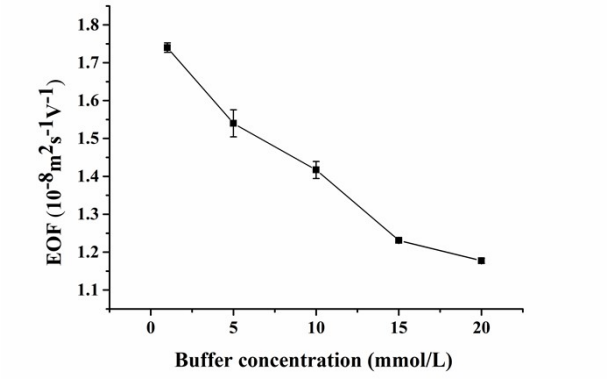


Fig. S4 The influence of buffer concentration on the EOF mobility of SiO_2@pPBS capillary packed column. Conditions: mobile phase, phosphate buffer with 60% ACN at pH 7; other

conditions were same as Fig. S3.

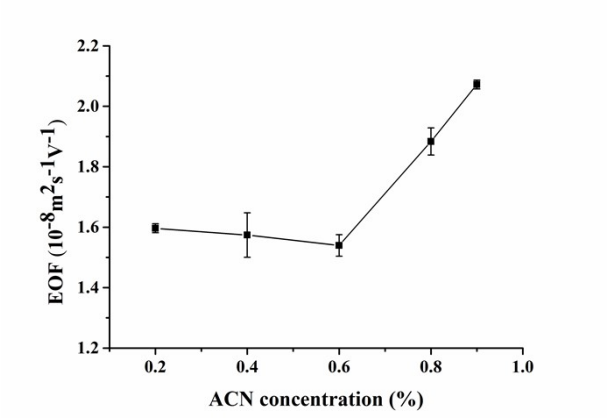


Fig. S5 The influence of ACN concentration on the EOF mobility of SiO_2@pPBS capillary packed column. Conditions: mobile phase, 5 mmol/L phosphate buffer at pH 7; other conditions were same as Fig. S3.

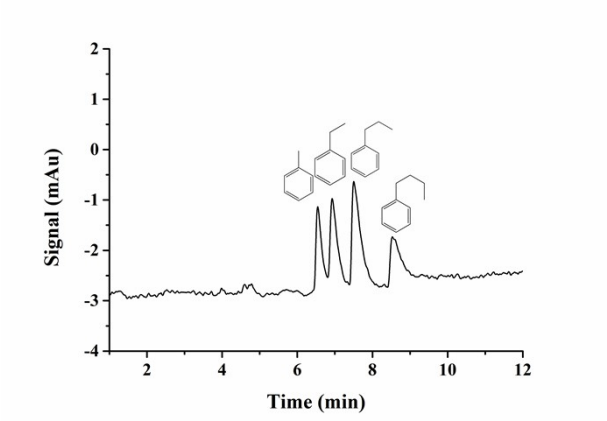


Fig. S6 Electrochromatogram of alkylbenzenes. Conditions: mobile phase, 5 mmol/L phosphate buffer with 60% ACN at pH 7; other conditions were same as Fig. S3.

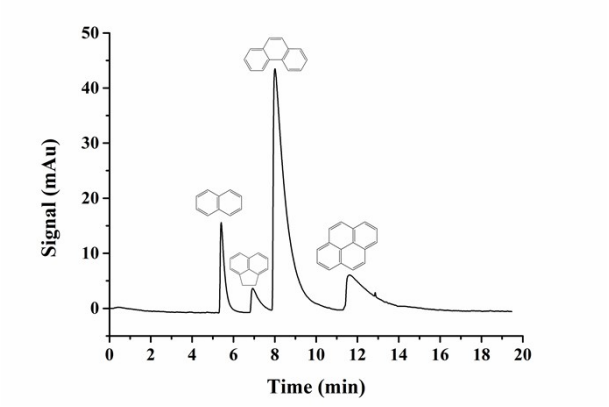


Fig. S7 Electrochromatogram of polycyclic aromatic hydrocarbons. Conditions: mobile phase, 5 mmol/L phosphate buffer with 90% ACN at pH 7; other conditions were same as Fig.

S3.

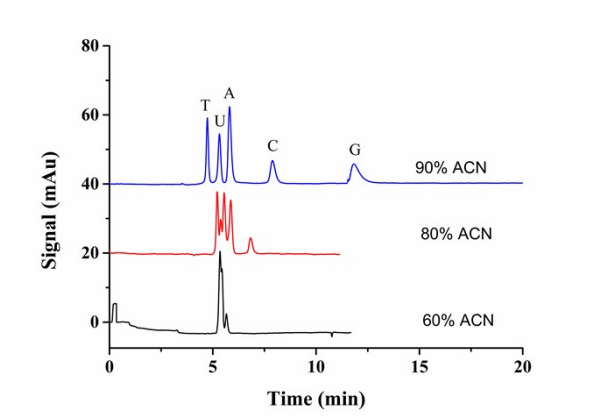


Fig. S8 Electrochromatograms of five nucleosides under different ACN concentration. Conditions: mobile phase, 5 mmol/L phosphate buffer at pH 7; other conditions were same as Fig. S3.

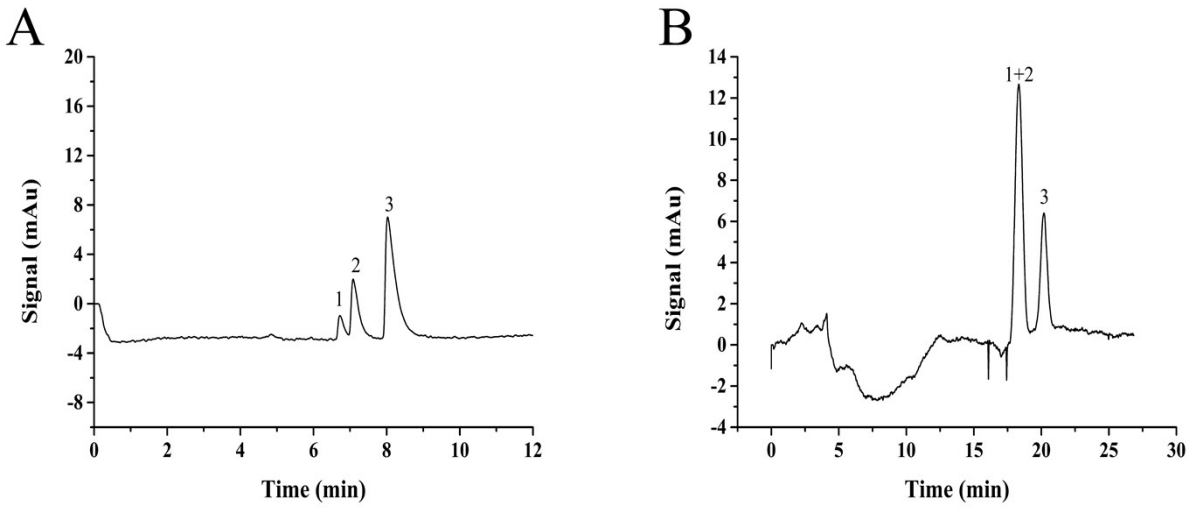
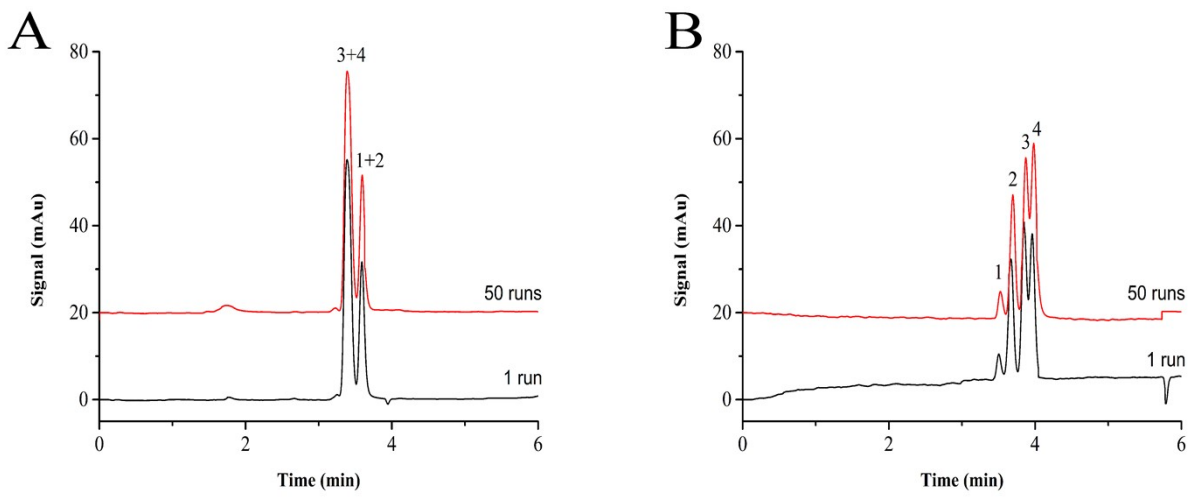


Fig. S9 The chromatographic selectivity comparison of (A) SiO_2@pPBS capillary packed column and (B) C18 column. Conditions: (A) mobile phase: 5 mmol/L phosphate buffer with 80% ACN at pH 7; (B) mobile phase: 5 mmol/L phosphate buffer with 60% ACN at pH 7; other onditions were same as Fig. S3. Analytes:1. Toluene, 2. Chlorobenzene, 3. Bromobenzene.



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78 **Fig. S10** Evaluation of stability of SiO_2@pPBS capillary packed column at pH4 (A) and pH

79 7 (B). Conditions: 5 mmol/L phosphate buffer with 40% ACN, other conditions were same as

80 Fig. S3. Analytes:1. Acetone, 2. Thiourea, 3. Sulphanilamide, 4. Sulfaguanidine.