Electronic Supplementary Information (ESI)

The use of PAMAM dendrimers as a dynamic coating for cyclodextrin mediated enantioseparation of selected basic drugs.

J. Znaleziona[a]*, V. Maier[a]

Experimental Part

Materials

Acetic acid (99.7 %), phosphoric acid (85.0 %), sodium hydroxide (0.1 M), PAMAM G 2.0, dimethylsulfoxide (DMSO), MES, MOPS, TAPS, *R,S*- and *S,R*- ephedrine, *R*- and *S*- isoproterenol were purchased from Sigma Aldrich (St. Louis, MO, USA). 2-hydroxypropyl-β-cyclodextrin (2-HP-β-CD, DS ~0.6) was provided by Fluka (Buchs, Switzerland). The *R,S*- and *S,R*-enantiomers of tapentadol were obtained from Zentiva k.s. (Prague, Czech Republic).
Background electrolytes (BGEs) used for the measurement of electroosmotic mobilities with/without PAMAM at different pH, were designed to have the fixed ionic strength set on 50 mM and the PeakMaster 5.3 was used for calculations[1,2]. The BGEs were prepared according to the presented table (Table 1). Then to each BGE the PAMAM dendrimers at 0.01 % (v/v) were added. The acidic components of BGEs were as follows: phosphoric acid (pH 2.5-3.0), acetic acid (pH 3.5, 4.0, 4.5, 5.0, 5.5), MES (pH 6.0, 6.5), MOPS (pH 7.0, 7.5), TAPS (pH 8.0, 8.5). The basic component of the BGEs was 0.1 M sodium hydroxide.
The standard solutions of racemic mixture of each drug were prepared separately in 1 $mg \cdot mL^{-1}$ concentrations by dissolution of the standard compound in deionized water. The working solution of racemic mixture of each drug was then forty times diluted with deionized water. All measurements were repeated five times, if not stated otherwise.
According to the precaution statements of the Producer wear protective gloves/protective cloths/eye protection/face protection when you working with PAMAM dendrimers.

Apparatus

The CE experiments were performed on a HP 3DCE and on an Agilent 7100 instruments (Agilent Technologies, Waldbronn, Germany) equipped with a photodiode array detector (DAD). Uncoated fused silica capillary was used (Polymicro Technologies, AZ, USA, 50 µm i.d., 365 µm o.d.) with total capillary length/effective capillary length 48.5/40.0 cm. The capillary was thermostated at 25 °C; the separation voltage was set to -20 kV/+20 kV according to the used capillary (coated/uncoated). Samples were injected hydrodynamically 50 mbar for 5 s. The capillary was preconditioned before the first use with 0.1 M sodium hydroxide for 20 min, deionized water for 15 min and with appropriate BGE for 15 min. Between analyses the capillary was washed with 1 min with 0.1 M sodium hydroxide, 1 min with deionized water and 2 min with the BGE.
When the PAMAM dendrimers were used for the first time the capillary was washed as follow: 10 min with 0.1 M sodium hydroxide, 10 min with deionized water, 10 min with dendrimer additive G 2.0 in the BGE at concentration of 0.01 % (w/v). Between analyses the coating capillary was washed with 1 min with 0.1 M sodium hydroxide, 1 min with deionized water and 2 min with the BGE.

References

1 M. Jaroš, V. Hruška, M. Štědrý, I. Zusková and B. Gaš, Electrophoresis, 2004, 25, 3080.

2 M. Jaroš, V. Hruška, M. Štědrý, I. Zusková and B. Gaš and Database of constituents by T. Hirokawa, PeakMaster 5.1, 2005.

TABLES

Table 1. BGEs composition. Calculations according to PeakMaster 5.3.

BGE acidic component concentration [mM]	BGE basic component concentration [mM]	Calculated pH	Measured pH	Analysis current at U = ± 20 kV[µA]
Phosphoric acid	**Sodium hydroxide**			
68.0	46.4	2.523	2.499	34.6
55.9	48.8	3.006	3.165	28.0
Acetic acid				
800.0	49.7	3.500	3.450	29.0
260.0	49.9	4.053	4.032	29.8
123.0	50.0	4.512	4.512	29.8
73.1	50.0	5.011	5.050	30.0
57.3	50.0	5.512	5.691	29.0
MES				
101.9	50.0	5.999	6.195	24.0
66.2	50.0	6.504	6.803	24.5
MOPS				
115.0	50.0	7.006	7.061	24.0
70.8	50.0	7.501	7.538	24.0
TAPS				
132.0	50.0	8.005	8.237	22.5
76.2	50.0	8.500	8.797	23.0

FIGURES

Fig.1. Chiral separation of basic drugs at pH 2.5 with PAMAM additive