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Journal of Chromatography A, 1091 (2005) 152–157
JOURNAL OF CHROMATOGRAPHY A
www.elsevier.com/locate/chroma

Enantioseparation of acidic enantiomers in capillary electrophoresis using a novel single-isomer of positively charged β-cyclodextrin: Mono-6^{A}-*N*- pentylammonium-6^{A}-deoxy-β-cyclodextrin chloride

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Received 19 April 2005; received in revised form 23 June 2005; accepted 6 July 2005
Available online 1 August 2005

Abstract

The new single-isomer of positively charged β-cyclodextrin, mono-6^{A}-*N*-pentylammonium-6^{A}-deoxy-β-cyclodextrin chloride (PeAM-β-CD), was employed for the first time for the enantioseparation of anionic and ampholytic analytes by capillary electrophoresis (CE). The synthesis and characterization of PeAM-β-CD were reported. The effect of background electrolyte (BGE) pH and selector concentration on the enantioseparation was investigated. Good separation was obtained at low BGE pH (ca. 5.0–6.0). The effective mobilities of all analytes were found to decrease with increasing CD concentration. PeAM-β-CD proved to be an effective chiral selector for most studied anionic analytes.

Keywords: Enantioseparation; Single-isomer; Positively charged β-CD; Capillary electrophoresis; Mono-6^{A}-*N*-pentylammonium-6^{A}-deoxy-β-cyclodextrin chloride

1. Introduction

Capillary electrophoresis (CE) has developed into an attractive and powerful technique for chiral separation and analysis in less than two decades. Cyclodextrins (CDs) and their derivatives remain the most widely used chiral selectors in CE. During the last decade, charged CDs have attracted much attention for their applications in chiral separations by means of capillary electrophoresis, as indicated by several recent reviews [1–5]. The application of positively charged CDs in chiral CE was launched by Terabe in 1989 [6]. Since then, a large number of cationic CDs, either randomly substituted or selectively substituted, have been developed and successfully used for the enantioseparation of anonic and neutral compounds. The use of randomly substituted cationic CDs may provide higher enantioselectivity with a stable electroosmotic flow (EOF) [7–9]. However, the degree and distribution of substitutions have significant effect on resolution of the enantiomers. Thus, the repeatability changed from batch to batch. Therefore, single-isomers of positively charged CDs are strongly recommended to be used for performing mechanistic studies and developing validated chiral CE assays [7,8]. Positively-charged CDs such as mono(6-aminoethylamino-6-deoxy)-β-CD [6], 6^{A}-methylamino-6^{A}-deoxy-β-CD, 6^{A}, 6^{D}-dimethylamino-6-deoxy-β-CD [10], heptakis(6-methylamino-6-deoxy)-β-CD [11], and mono(6-amino-6-deoxy)-β-CD [12] were the first described CD single-isomers. Other single-isomer examples of positively charged β-CDs in chiral CE have been published in literature [13–15] (per-substituted CDs) and [16–19] (mono-substituted CDs).

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0021-9673/$ – see front matter © 2005 Published by Elsevier B.V.
doi:10.1016/j.chroma.2005.07.045

Recently, a family of new single-isomers of positively charged CDs, consisting of mono-alkylimidazolium, pyridinium and alkylammonium cations on the C6- or C2 of the glucopyranose subunit of CD, were developed and successfully used for the enantioseparation of a wide range of anionic analyte [20]. In the present work, we report the synthesis, characterization and application of the new single-isomer, mono-6^A-*N*-pentylammonium-6^A-deoxy-β-cyclodextrin chloride (PeAM-β-CD), for the enantioseparation of a wide range of acidic enantiomers.

2. Experimental

2.1. Chemicals

Monobasic sodium phosphate (NaH_2PO_4), phosphoric acid and sodium hydroxide were purchased from Merck (Darmstadt, Germany). DMF and *n*-pentylamine were products of Fluka (Buchs, Switzerland). 2-methoxyphenylacetic acids, 2-phenoxypropionic acid, 2-(3-chlorophenoxy) propionic acid, 2-(2,4-dichlorophennoxy) propionic acid, 2-phenylbutyric acid and 3-phenylbutyric acid were pur-

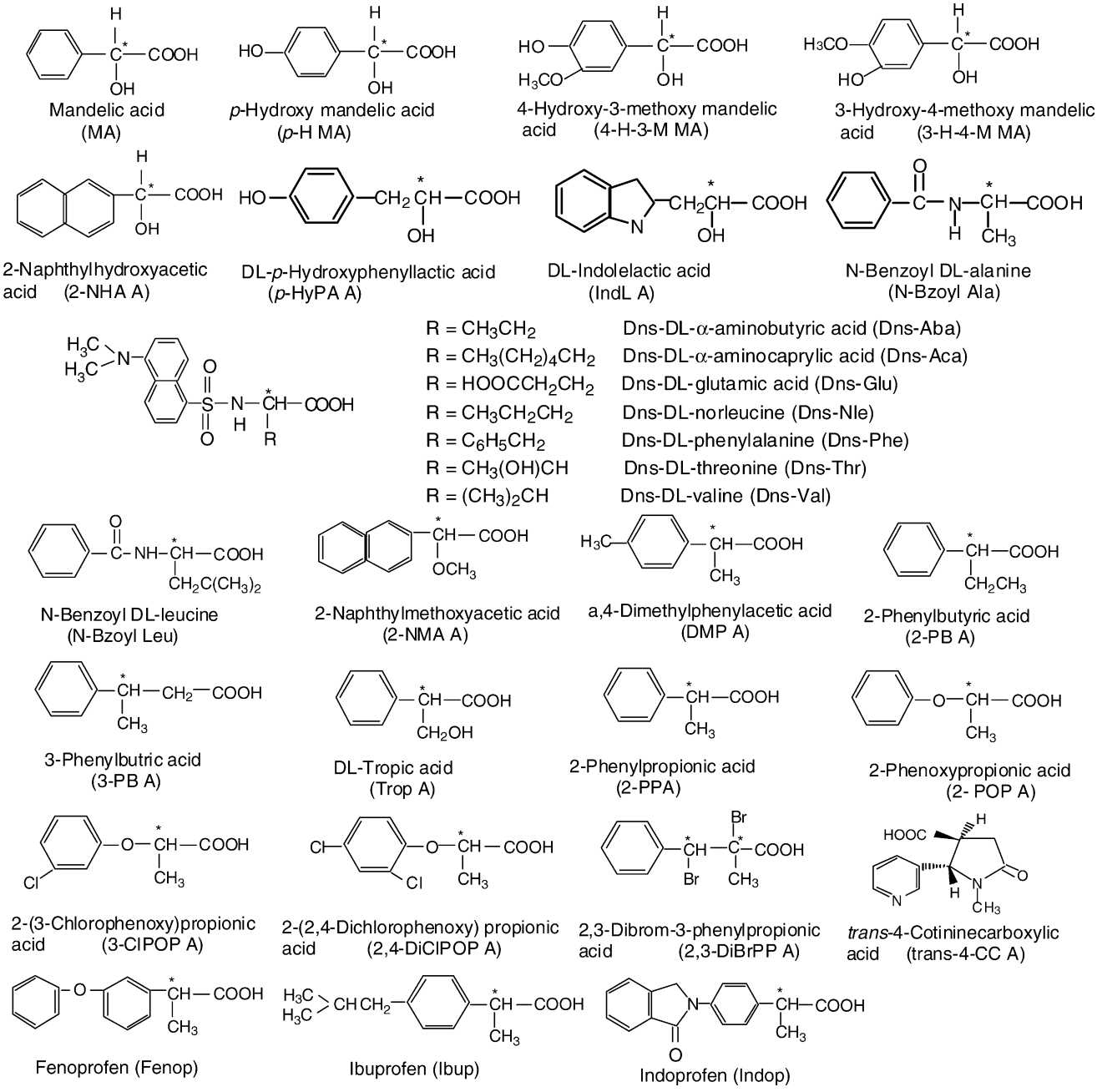

Fig. 1. Structure of hydroxy, carboxylic acids and amphoteric analytes studied.

chased form Lancaster Synthesis (Windham, NH, USA). 2-Naphthylhydroxyacetic acid and 2-naphthylmethoxyacetic acid were synthesized according to the report [21]. All other chiral acids were purchased either from Aldrich (Steinheim, Germany) or from Sigma (St. Louis, MO, USA). The structures of these acidic enantiomers are depicted in Fig. 1. Distilled water was prepared with a Nanopure water system (Omega Medical & Scientific Pte. Ltd., Singapore).

2.2. Synthesis and analytic characterization of PeAM-β-CD

6-Monotosyl-β-CD was prepared according to the reported procedure [22,23]. A solution of 6-monotosyl-β-CD and excess *n*-pentylamine in DMF was stirred at 80 °C for 5 h under nitrogen. The resultant solution was cooled to room temperature and poured into acetone. The white solid was filtered and recrystallized several times from ethanol to produce mono-6^{A}-*N*-pentylammonium-6^{A}-deoxy-β-CD tosylate (yield, 96.3%). The progress of reaction and purification was observed using a similar TLC method described in [22]. A further anion exchange process was performed to convert tosylate anion into chloride by the use of amberlite 900 (Cl) resin. The filtrate was collected and recrystallized three times from hot water to give the desired PeAM-β-CD.

The analytical data for PeAM-β-CD was as follows: pK_a: 8.74 ± 0.2; melting point: 266–267 °C; ^{1}H NMR (300 MHz, DMSO-$_{d6}$): δ 5.66–5.81 (OH-2,3), 4.86, 4.84 (H-1), 4.48 (OH-6), 3.75 (H-3′), 3.54–3.65 (H-3,5,6), 3.32–3.45 (H-2,4), 3.12 (H-4′), 2.85 (H-2′), 2.63 (CH_2), 1.47 (CH_2), 1.25 (CH_2), 1.24 (NH_2), 0.86 (CH_3); ^{13}C NMR (300 MHz, $DMSO_{d6}$): δ 101.9 (C1), 101.4 (C1′), 83.5 (C4′), 81.5 (C4), 73.0 (C2), 72.3 (C3), 72.0 (C5), 59.8 (C6), 48.4 (C6′), 46.4 (CH_2), 30.6 (CH_2), 28.5 (CH_2), 21.8 (CH_2), 13.8 (CH_3); Infrared IR (cm^{-1}, KBr): the asymmetric and symmetric SO_2– stretch of tosyl group at 1368 and 1157 disappeared in the spectra of PeAM-β-CD; elemental analysis: calculated for $C_{47}H_{82}NClO_{34}\cdot 6H_2O$ (1348.60) C: 45.50% H: 6.66% N: 1.13% Cl: 2.86%, determined C: 45.25% H: 6.58% N: 1.06% Cl: 2.82%. ESI MS (*m*/*z*): 1205.14 (cald) and 1205.40 (found) for [M$^+$]. The measured *m*/*z* value of M$^+$ agreed well with calculated value, which indicated the mono-substituted structure was really obtained.

2.3. Instrumentation and operating procedure

The NMR spectra were obtained on a Bruker ACF300 FT-NMR spectrometer. Mass spectra were performed on a Finnigan TSQ7000 mass spectrometer. All electrophoretic experiments were performed on a Beckman P/ACE MDQ CE unit (Fullerton, CA, USA), equipped with a variable-wavelength PDA (Photodiode Array, 190–300 nm) detector. Beckman CE software was used for data acquisition and system control. Separations were carried out with an uncoated fused-silica capillary (50 μm I.D., 59.2 cm length, 49 cm to the detector), obtained from Polymicro Technologies (Phoenix, AZ, USA).

Table 1
Influence of BGE pH on migration times (t_1, min), effective mobility (μ_1, ($\times 10^{-5}$) cm^2 V^{-1} s^{-1}), mobility difference ($\Delta\mu$, ($\times 10^{-5}$) cm^2 V^{-1} s^{-1}), selectivities (α) and resolutions (R_s) of racemic acids using PeAM-β-CD as chiral selector.

Acid analytes	pH 5.0					pH 6.0					pH 7.0					pH 8.0				
	t_1	μ_1	$\Delta\mu$	α	R_s	t_1	μ_1	$\Delta\mu$	α	R_s	t_1	μ_1	$\Delta\mu$	α	R_s	t_1	μ_1	$\Delta\mu$	α	R_s
MA	26.78	−18.7	11.72	1.11	4.62	18.50	−19.2	11.47	1.07	3.85	17.57	−19.5	10.11	1.06	1.88	16.45	−20.6	11.72	1.04	2.62
p-H MA	24.15	−15.1	14.09	1.12	3.41	16.22	−16.3	14.44	1.08	3.48	15.12	−16.7	12.92	1.06	2.44	14.79	−18.3	9.89	1.05	2.21
4-H-3-M MA	33.73	−17.2	3.94	1.04	1.87	17.36	−18.6	4.14	1.02	1.22	16.36	−18.4	3.78	1.02	0.91	15.71	−19.2	2.85	1.01	0.88
3-H-4-M MA	34.31	−13.7	7.41	1.09	3.36	16.03	−16.9	9.56	1.05	2.13	15.11	−16.9	8.8	1.04	1.76	14.93	−17.9	6.72	1.03	1.68
2-NHA A	11.96	−5.73	13.9	1.05	2.47	10.88	−6.62	13.64	1.05	2.29	12.29	−6.49	10.39	1.05	1.02	11.08	−8.12	12.18	1.04	1.51
2-NMA A	15.51	−6.08	5.71	1.03	1.13	10.66	−6.79	5.42	1.02	1.04	12.48	−7.07	4.67	1.02	0.88	11.19	−8.1	5.98	1.02	0.77
Dns-Aba	14.26	−6.83	6.54	1.03	1.25	10.73	−7.51	6.63	1.02	1.03	12.87	−7.69	6.14	1.03	1.02	11.95	−8.54	5.16	1.02	0.82
Dns-Glu	28.78	−15.1	5.46	1.03	1.34	18.15	−19.4	6.48	1.04	1.39	20.88	−19.6	2.91	1.05	1.32	20.71	−20.4	3.82	1.03	0.88
Dns-Phe	12.06	−2.29	3.03	1.01	0.61	9.30	−3.20	4.16	1.01	0.81	10.74	−3.32	4.45	1.02	0.93	10.17	−4.66	5.24	1.01	0.95
Dns-Val	17.06	−7.41	1.38	1.01	<0.5	11.26	−8.87	2.12	1.01	0.58	13.27	−8.87	2.27	1.01	0.71	12.41	−10	2.51	1.01	0.76

Conditions: 5 mM CD; 50 mM phosphate buffer (pH 5.0, 6.0, 7.0, and 8.0); temperature 25 °C; applied voltage 15 kV.

The applied voltage was +15 kV (normal polarity mode). Detection of analytes was carried out simultaneously at three wavelengths at 214, 254 and 280 nm at 25 °C.

All 50 μg/mL stock solutions of racemic samples were prepared with 50/50 (v/v) methanol/water mixture solution, filtrated with a 0.45 μm syringe type Millipore membrane and sonicated prior to use. Samples were introduced into the capillary by a 0.5 psi pressure injection (typically 5 s). The capillary was flushed between injections with 0.1 M NaOH, 1 M NaOH, water and running buffers for 2 min each.

Fifty millimeters phosphate buffers (pH 5–9) were used as background electrolytes (BGEs). Running buffers were prepared accordingly by dissolving appropriate chiral selector into BGEs. The electroosmotic flow (EOF) was measured with methanol as neutral marker. The separation selectivity (α), was calculated as $\alpha = t_2/t_1$, where t_1 and t_2 are the migration times of two enantiomers. The peak resolution values R_s, were calculated by dividing the migration time difference of the two enantiomers with half of the sum of their peak widths at the baseline.

3. Results and discussion

3.1. Effect of pH on enantioseparation with PeAM-β-CD

BGE pH is important for the separation of charged racemates because it directly affects the effective charge and the effective mobility of the racemic analytes. Also, BGE pH can affect the EOF [24] and the selective resolution ability of the cationic CDs [16,17]. In this study, the influence of pH on enantioseparation was examined in the range from 5.0 to 8.0 with 5 mM CD (Table 1). In general, the increase in BGE pH led to a decrease in the chiral R_s and α of all racemic acids. However, the increased pH usually led to shorter migration times, which is beneficial for fast separation. The decrease in migration times were mainly caused by the increased EOF. As shown in Table 1, good enantioseparation was achieved at low BGE pH (ca. 5.0 and 6.0). Considering both chiral resolution and migration time, we selected pH 6.0 for the following studies.

3.2. Effect of CD concentration on enantioseparation with PeAM-β-CD

All together 9 hydroxy, 9 amphoteric, 15 carboxylic acid enantiomers (Fig. 1) were selected as model analytes. The effect of CD concentration on enantioseparations of the selected analytes was examined with fixed BGE pH (6.0) and separation results are summarized in Table 2 (for hydroxy and amphoteric acids) and Table 3 (for carboxylic acids). Under the applied CZE conditions, the acidic analytes can be completely dissociated. Thus, the attractive electrostatic interaction between cationic CD and analytes were formed. The enantiomeric separation occurs mainly via a combined inclusion phenomenon and ion pair formation [12].

For most hydroxy acids (Table 2), the chiral resolution (R_s) increased with increment in CD's concentration in the whole range from 2.5 to 20 mM. Whereas, chiral R_s values of 2-NHA A decreased with increased CD concentration. Compared with the reported single-isomer aminated CDs [10,12,17], PeAM-β-CD is very powerful in resolving hydroxy acids. Satisfactory resolution values usually can be achieved over a wide range of CD concentration. The higher resolution of hydroxy acids may be attributed to their ability in forming secondary interactions (e.g. hydrogen bonding) with CD. The resolution ability of PeAM-β-CD was quite limited when separating dansyl (Dns) amino acids.

For carboxylic acids (Table 3), good separations were usually achieved at very low concentration of chiral selector (ca.

Table 2
Influence of chiral selector concentration on migration times (t_1, min), selectivities (α) and resolutions (R_s) of hydroxy and amphoteric acids using PeAM-β-CD as chiral selector

Acid analytes	2.5 mM			5 mM			7.5 mM			10 mM			20 mM		
	t_1	α	R_s	t_1	α	R_s	t_1	α	R_s	t_1	α	R_s	t_1	α	R_s
MA	18.06	1.04	1.7	18.50	1.07	3.85	19.58	1.09	3.32	20.13	1.11	4.14	20.29	1.13	5.2
p-H MA	15.69	1.05	1.77	16.22	1.08	3.48	16.96	1.10	3.59	14.37	1.09	3.24	17.33	1.12	4.79
4-H-3-M MA	16.07	1.01	0.73	17.36	1.02	1.22	20.52	1.04	1.53	21.55	1.05	2.21	24.35	1.09	3.55
3-H-4-M MA	15.34	1.03	1.32	16.03	1.05	2.13	17.94	1.08	3.16	18.13	1.09	3.48	18.99	1.12	5.42
2-NHA A	10.82	1.06	2.41	10.88	1.05	2.29	10.79	1.04	1.73	11.1	1.04	1.62	12.17	1.02	1.69
p-HyPA A	17.28	1.05	3.12	11.68	1.08	2.97	13.35	1.09	3.18	13.42	1.09	3.56	14.36	1.10	4.12
IndL A	18.43	1.12	1.34	14.28	1.04	1.74	15.98	1.05	1.57	16.11	1.06	2.12	17.54	1.08	2.32
N-Bzoyl Ala	21.24	1.04	<0.5	17.47	1.01	0.96	20.41	1.02	0.99	21.13	1.04	1.38	21.89	1.05	1.56
N-Bzoyl Leu	16.17	1.01	1.99	13.02	1.01	1.54	14.74	1.01	1.62	15.68	1.01	1.43	16.13	1.02	1.23
Dns-Aba	10.75	1.01	0.56	10.73	1.02	1.03	11.37	1.03	1.53	10.96	1.03	1.38	12.15	1.04	1.64
Dns-Glu	18.71	1.02	0.87	18.15	1.04	1.39	18.75	1.06	2.24	15.52	1.07	2.83	17.88	1.12	5.09
Dns-Nle	10.34	1.02	0.86	10.36	1.01	0.63	10.95	1.01	0.66	9.85	1.00	0	11.73	1.00	0
Dns-Phe	9.32	1.01	1.33	9.30	1.01	0.81	9.84	1.01	0.58		na			na	
Dns-Thr	11.59	1.02	0.72	12.17	1.01	0.53	12.95	1.01	<0.5	11.81	1.0	0		na	
Dns-Val	11.95	1.01	0.74	11.26	1.01	0.58	12.17	1.01	<0.5	10.73	1.0	<0.5		na	

Conditions: 50 mM phosphate buffer; pH, 6.0, containing different CD concentration; applied potential, 15 kV; 25 °C. na, not available; sample peaks overlapped with EOF.

Table 3
Migration times of the first enantiomers (t_1, min), separation selectivities (α) and measured peak resolution values (R_s) in pH 6.0 PeAM-β-CD BGEs for carboxylic analytes

Racemic acid analytes	2.5 mM			3.5 mM			5 mM			7.5 mM		
	t_1	α	R_s	t_1	α	R_s	t_1	α	R_s	t_1	α	R_s
2-NMA A	10.87	1.02	0.78	15.17	1.03	0.99	10.66	1.02	1.04	10.97	1.02	0.96
DMP A	14.63	1.03	0.76	17.65	1.03	0.84	11.02	1.02	0.89	12.31	1.02	0.62
2-PB A	17.01	1.02	0.71	19.37	1.02	<0.5	12.11	1.02	0.83	13.94	1.01	0.85
3-PB A	15.78	1.01	<0.5	20.88	1.01	0.88	11.32	1.01	<0.5	12.85	1	0
Trop A	26.19	1.02	0.75	21.99	1.02	0.95	16.35	1.02	1.13	18.65	1.08	1.02
2-PPA	23.98	1.05	1.68	19.04	1.05	2.14	15.55	1.03	1.56	17.36	1.07	1.42
2-POP A	19.11	1.06	1.48	15.42	1.07	2.8	15.68	1.06	2.3	17.77	1.03	2.41
3-ClPOP A	14.55	1.02	2.03	16.69	1.08	2.93	12.71	1.07	3.51	14.08	1.01	2.63
2,4-DiClPOP A	15.29	1.03	1.19	12.32	1.07	2.78	13.34	1.05	1.34	15.25	1.03	1.49
2,3-DiBrPP A	12.14	1.05	3.18	12.46	1.07	2.51	10.61	1.01	1.29	11.84	1.07	1.17
Trans-4-CC A	19.36	1.01	<0.5	18.29	1.01	<0.5	18.34	1.01	0.76	19.25	1.04	1.26
Fenop	10.59	1.01	<0.5	10.91	1.01	<0.5	10.83	1.01	1.45	12.18	1.11	1.71
Ibup	12.13	1.01	<0.5	12.43	1.01	0.8	10.32	1.01	0.53	10.34	1.01	0.53
Indop	11.93	1.01	0.77	12.26	1.01	0.99	10.96	1.01	0.86	10.95	1.01	0.91

Conditions: 50 mM phosphate buffer; pH 6.0, containing different CD concentration; applied potential 15 kV; 25 °C.

3.5 mM). Among them, five carboxylic acids (e.g. 2-PPA, 2-POP A and its derivatives) were baseline separated with CD concentration lower than 5 mM. Moreover, phenylpropionic acids (i.e. 2-PPA and 2,3-DiBrPP A) and phenoxypropionic acids (i.e. 2-POP A, 3-ClPOP 2, 4-DiClPOP) can be resolved well even at higher concentration of CD (i.e. 7.5 mM).

The effective mobilities of studied acids are dependent on CD concentration. The increase of PeAM-β-CD concentration generally led to decreased effective mobilities of all analytes, but only decreased to 1.99×10^{-5} cm^2 V^{-1} s^{-1}. The decrease in effective mobilities was mainly caused by the increased involvement of the analytes in complex with CD. The rapidly increasing ionic strength shifted the complexation equilibrium towards the forming of CD–analyte complexes, which moved much slower. Meanwhile the increased ionic strength and BGE viscosity also contributed for the decrease of effective mobility. For mandelic acid and its derivatives, their effective mobilitites were larger than -15×10^{-5} cm^2 V^{-1} s^{-1} at CD concentration lower than 10 mM. While some carboxylic acids such as Tropic A, 2-PPA, 2-POP A, 3-ClPOP A and 2, 4-DiClPOP A, maintained high effective mobilities ($>-15 \times 10^{-5}$ cm^2 V^{-1} s^{-1}) at CD concentration lower than 7.5 mM. Dns-Glu also maintained high effective mobilities (-14.7 cm^2 V^{-1} s^{-1}) even in the presence of 20 mM PeAM-β-CD.

The dependence of separation selectivities upon CD concentration is similar to chiral resolution. An increase in CD concentration generally led to an increased chiral selectivity of enantiomers such as MA and its derivatives, Dns-Glu, 3-ClPOP A and 2, 4-DiClPOP A. The separation selectivity of amphoteric acids, however, decreased with the increment of PeAM-β-CD concentration. Interestingly, the separation selectivity of most carboxylic acids presented a maximum at PeAM-β-CD concentration ranging from 3.5 to 5 mM.

In order to further demonstrate the chiral resolution power of PeAM-β-CD for these anionic analytes, a standard mixture containing six pairs of hydroxy acids and ampholytic analytes was baseline separated with 2.5 mM PeAM-β-CD within 20 min (Fig. 2). The migration order of analyte enantiomers in the standard mixture was verified by injecting each racemate individually.

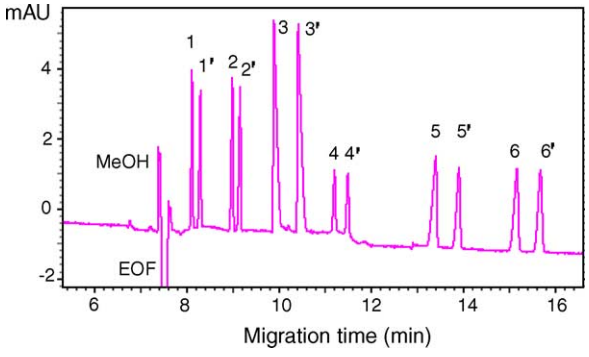

Fig. 2. Enantioseparation of a standard mixture of six chiral acids using PeAM-β-CD as chiral selectors. *Conditions*: 2.5 mM CD, pH 6.5, 50 mM NaH_2PO_4 buffer. *Legends*: (1) 1′: Dns-DL-α-aminocarpylic acid; (2) 2′: Dns-DL-phenylalanine acid; (3) 3′: 2-naphthylhydroxyacetic acid; (4) 4′: *N*-benzoyl-DL-leucine; (5) 5′: 3-hydroxy-4-methoxy mandelic acid; and (6) 6′: mandelic acid.

4. Concluding remarks

A novel single-isomer of positively charged β-CD, PeAM-β-CD, has been synthesized, analytically characterized and successfully used as chiral selector for the enantioseparation of anionic and amphoteric analytes. This cationic CD displayed different chiral resolution ability to various acids, which is strongly dependant upon BGE pH and CD concentration. For hydroxy acids, chiral resolution can be greatly improved by increasing CD concentration. Carboxylic acids, however, have a fair low optimum CD concentration. In conclusion, this positively charged CD showed powerful resolution abilities towards various acidic enantiomers due to

the introduction of positive charge and attractive electrostatic interactions with analytes.

Acknowledgements

Financial support for this work was provided by A*Star, Institute of Chemical and Engineering Sciences Ltd and the National University of Singapore (NUS). W.T. thanks NUS for awarding the research scholarship.

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