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Spliced capillaries for use in capillary electrophoresis with conductivity detection

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Abstract

Presently, commercial capillaries for use in capillary electrophoresis (CE) with conductivity detection have a permanently mounted sensor which forms part of the conductivity cell. Ability to splice different capillaries to the sensor would allow versatility in choice of capillary dimensions and bonded columns as well as simply replacing old capillaries. Various capillaries can be easily joined to the sensor using a zero dead volume stainless steel union and ferrule. The performance of a 60 cm×50 μm spliced capillary is compared to that of an original commercial capillary of the same dimensions through electropherograms of a mixture of anionic surfactants taken with a NaF buffer. The plate count (N) and resolution (R_s) of the spliced capillary averaged 93% and 82% of those for the unspliced capillary. The reproducibility of the peak height and peak mobility of the surfactants for the spliced capillary are generally 1–3% R.S.D. and <1% R.S.D., respectively. Characterization with respect to N and R_s of capillaries with different diameter and lengths for CE of anionic surfactants with conductivity detection is also investigated. An amine bonded capillary permitted the separation of anions or cations using the same electrolyte but just switching the polarity of the applied voltage.

Keywords: Capillary columns; Surfactants; Inorganic cations; Inorganic anions

1. Introduction

One main advantage of modern chromatography and capillary electrophoresis (CE) is the variety of columns available for use. For CE, the importance of choice in fused-silica capillary dimensions and possibility of using covalently bonded capillaries has been established [1,2]. However, commercial CE with conductivity detection does not provide for flexibility in column choice. Due to the design of the Crystal 1000 CE conductivity detector, a ConCap I fused-silica capillary must be used. Replacement of this capillary is somewhat costly and inconvenient. The inability to use capillaries of different dimensions or type such as bonded columns is also a limitation.

The purpose of this study is to show that a wide variety of capillaries can be reproducibly spliced to a short (5 cm) ConCap I fused-silica capillary which still fits onto the conductivity cell. Techniques for joining capillaries have been published previously. Capillaries were etched with hydrofluoric acid to form male and female ends which could then be joined together [3]. Although little loss in plate count or resolution was noted for the joined capillary, this joining method took considerable time and practice in order to be successful. Another previously published study used a ferrule based connector; a small diameter injection capillary for sampling of lung air way surface fluid in rats was connected to the

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standard 60 cm ConCap I separation capillary [4]. Separations of inorganic anions and cations were shown. However no quantitative evaluation of this connector with respect to its effect on efficiency, resolution, and peak response was made.

In this work, the performance of a bare spliced capillary to an unspliced (ConCap I) capillary with respect to effective mobilities, plate count (N), resolution, peak area, and linear response for the separation of anionic surfactants are compared. The ferrule based connector is used for all spliced capillaries. Amine and C_{18} bonded capillaries as well as bare spliced capillaries of different diameter and length are tested for the separation of surfactants. Using an amine bonded capillary, the separation of either inorganic cations or anions using the same running electrolyte is possible just by switching the polarity of the operating voltage. To the best of our knowledge, bonded capillaries have not been previously used with CE and conductivity detection.

2. Experimental

2.1. Chemicals

Reagent grade sodium nitrite ($NaNO_2$), potassium phosphate (K_2HPO_4), sodium carbonate (Na_2CO_3), sodium sulfate (Na_2SO_4), sodium fluoride (NaF), and sodium chloride (NaCl) originated from Fisher Scientific (Pittsburgh, PA, USA). The sodium bromide (NaBr), triethanolamine (TEA), sodium 1-hexanesulfonate [$CH_3(CH_2)_5SO_3Na$], sodium 1-decanesulfonate [$CH_3(CH_2)_9SO_3Na$], (2-chloroethyl)-trimethylammonium chloride ($ClETMA^+$), tetrahexylammonium bromide (THA^+), and tetraethylammonium bromide (TEA^+) were purchased from Aldrich (Milwaukee, WI, USA). The 2-[*N*-cyclohexylamino]ethanesulfonic acid (CHES), hexyldecyltrimethylammonium bromide (CTAB), lithium chloride (LiCl), and Triton X-100 were obtained from Sigma (St. Louis, MO, USA). The sodium 1-octanesulfonate monohydrate [$CH_3(CH_2)_7SO_3Na{\cdot}H_2O$] and sodium 1-dodecanesulfonate [$CH_3(CH_2)_{11}SO_3Na$] were procured from Lancaster Synthesis (Windham, NH, USA). The tetrabutylammonium iodide (TBA^+) was from Eastman (Rochester, NY, USA); the sodium nitrate ($NaNO_3$) was from Mallinckrodt (St. Louis, MO, USA) and the acetonitrile was from Baxter Healthcare (McGaw Park, IL, USA). The toothpaste samples were purchased at a local grocery store.

2.2. CE instrumentation

The CE instrumentation employed was a Crystal CE system made by Thermo Bioanalysis (Franklin, MA, USA) with a Crystal 1000 CE conductivity detector. The conductivity cell of the detector connects the ConCap I fused-silica capillary to the ConTip I sensor. The ConTip I sensor unites both electrodes concentrically on the same surface as the sensor. The ConTip I sensor has a detection surface that consists of a center Pt electrode (150 μm O.D.) surrounded by a polyimide insulator, then an electrode spacer, and finally the stainless steel second electrode (375 μm I.D.). The ConCap I connector houses the fused-silica capillary and the contact surface contains two grooves which allows rapid flushing with electrolyte. The distance between the ConCap I and ConTip I connectors has been optimized to be 24.1 ± 1.2 μm by the manufacturer and determines the detector cell volume (less than 3 nl) [5]. Due to the design of the ConCap I fused-silica capillary, the effective capillary length is equal to the total capillary length. Data acquisition was obtained with the Rainin Instruments (Woburn, MA, USA) interface connecting the CE system to a MacIntosh SE computer equipped with MacIntegrator I software.

2.3. Preparation of spliced capillaries

The ConCap I fused-silica capillary was spliced approximately 5 cm from the ConCap I capillary connector. Great care needs to be taken when splicing the capillary in order for the connection to be good. Smooth, perpendicular ends are needed so that the ConCap I capillary connector and the replacement capillary fit squarely together. This was possible by first removing the polyimide coating and then making the cut with a one inch square ceramic capillary cleaver. The capillary ends were examined using a 20× microscope. Next, the ConCap I capillary connector and the replacement capillary of desired length were connected using a Supelco

(Bellefonte, PA, USA) butt connector with a single ferrule. It was important to minimize the gap between the two capillaries by applying hand pressure as the ferrule was tightened. With the stainless steel union, the capillaries can be easily rejoined if necessary or changed which is a definite advantage over a glued glass union. After the spliced capillary was inserted in the instrument and buffer was flowing through the capillary, the butt connecter was loosened, until the buffer leaked out, and then the connector was tightened again. This reduced the chance of air gaps being formed which could cause excessive baseline drift and noise. This splicing technique was used in connecting the uncoated capillary obtained from Polymicro (Phoenix, AZ, USA) as well as the CElect Amine capillary and the C_{18} bonded capillary both obtained from Supelco.

2.4. Capillary conditioning

A bare capillary was flushed with water, 0.1 *M* NaOH and water again before first time use. At the beginning of the day, the conductivity detector cell was purged with the electrolyte; the capillary was also flushed with the electrolyte. The autocell cell voltage and the background electrolyte conductivity in microsiemens were recorded. The current across the capillaries ranged from 12–30 μA depending on capillary dimensions. In between runs, the bare and the amine bonded capillaries were just flushed with the NaF buffer for the separation of surfactants. However, for the separation of ions the bare capillary was conditioned with 1 m*M* CTAB prior to every run with CHES buffer [5] and once again the amine bonded capillary was just flushed with the buffer. At the end of the day or when changing the buffer, the capillary was flushed with water and the conductivity cell was purged with water.

2.5. Calculations

Plate count (N) was calculated using the equation that corrects for non-Gaussian peaks: $N=41.7\ (t_r/w_{0.1})^2/(b/a+1.25)$ where t_r=migration time, $w_{0.1}$= peak width 10% above the baseline, and b/a=peak asymmetry factor [6]. Resolution (R_s) was calculated using $R_s=1/4(N^{1/2})(\Delta\mu/\mu)$ where $\Delta\mu$=apparent mobility difference and μ=average apparent mobility for the peak pair [7].

3. Results and discussion

3.1. Comparison of unspliced (ConCap I) and spliced capillaries

The electropherograms of aliphatic sulfonates (Fig. 1) taken on both unspliced and spliced capillaries were very similar in appearance. The migration times were somewhat different; this was due to the unspliced electropherogram being obtained on an older capillary. Previously we have found this change in the electroosmotic flow (EOF) marker peak with capillary use [8]. However, the effective mobilities (μ_e) of the unspliced and spliced capillaries were very similar; the average μ_e values for C_6-, C_8-, and C_{12}-SO_3^- were respectively -0.019, -0.017, and -0.014 cm^2 (V min)$^{-1}$ for both the bare unspliced and spliced capillaries. The μ_e values

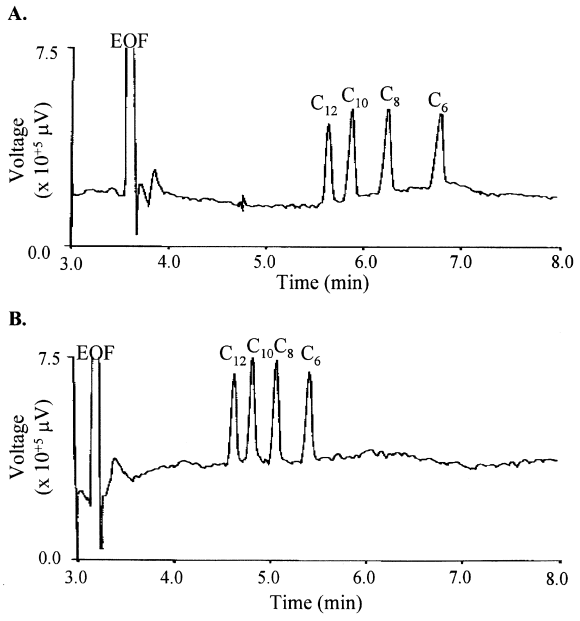

Fig. 1. Comparison of unspliced (A) and spliced (B) electropherograms. Electrolyte: 20 m*M* NaF, 1 m*M* TEA, water–acetonitrile (90:10); injection: pressure, 25 mbar for 12 s, 7.7 nl for 60 cm×50 μm I.D. capillary; voltage: +25 kV; indirect conductivity detection; Analytes: 80 ppm C_6-, C_8-, C_{10}- and C_{12}-SO_3^-.

for C_{10}-SO_3^- were −0.015 and −0.016 for the respective unspliced and spliced capillaries. For n=3, relative standard deviation (R.S.D.) ranged from 2.1–2.5% for the unspliced capillary and 0.5–0.7% for the spliced capillary. This R.S.D. difference is most likely caused by the difference in age of the capillaries.

A peak height and area reproducibility study was completed and it was determined that there was very little difference between the unspliced and spliced capillary for the same set of surfactants. Peak areas ranged from 1.0–1.2×10^6 μV and were identical for C_6-, C_8-, and C_{10}-SO_3^- on both capillaries; R.S.D.s (n=3) were from 2–8%. Peak heights were quite similar for all four analyte peaks ranging from 2.8–3.4×10^5 μV for the unspliced capillary and 3.3–3.9×10^5 μV for the spliced capillary. R.S.D. values (n=3) were from 0.6–3%. The peak height for the spliced capillary averaged 3.6×10^5 μV and was about 15% larger than that for the unspliced capillary.

Loss of overall performance through excessive band broadening and loss of resolution of closely separated peaks was found to be not of major concern when using the spliced capillary. A comparison of plate count (N) for the unspliced and spliced capillaries (60 cm×50 μm) showed on average 93% of the original plate count is maintained (Table 1). The percentage change in separation efficiency is comparable to other studies involving joined capillaries. The HF treated capillaries which were then joined together showed a loss of efficiency of only about 4% [3]. A porous joint required for interfacing electrochemical detection to CE was found to cause an average loss in separation efficiency of about 16% for six compounds studied [9]. The percent decrease in resolution between the unspliced and spliced capillaries was only 9% for the C_{12}–C_{10} peak pair (Table 1) and averaged 20% for the other two peak pairs. This variation in R_s values between the two capillaries may be due in part to differences in the average mobility of the peak pairs. Baseline resolution (>1.5) was still maintained for all peak pairs with R_s values ranging from 2.0–4.1. A linearity of response (Table 2) comparing spliced and unspliced capillaries was also completed using peak height of the surfactants from 40 ppm to 80 ppm. It was determined that the slope and the y-intercept values as well as correlation coefficients were quite similar for electropherograms taken on the two capillaries.

3.2. Effect of diameter and length of capillary

Capillaries of varying dimensions (both I.D. and length) were tested on the aliphatic sulfonate mixture obtained using the above mentioned NaF electrolyte with indirect conductivity detection. When the diameter of the capillary was increased to 75 μm I.D. (60 cm length) (Fig. 2) the migration time increased by about a factor of 1.6 and the sharpness of the peaks decreased by about the same factor. The plate count

Table 1
Average plate count (N) and resolution (R_s) data for different capillaries

Capillary	Number of plates (N) (n=12)[a]			Resolution (n=3) (between peaks C_{12}- and C_{10}-SO_3^-)		
	Average	S.D.	R.S.D. (%)	Average	S.D.	R.S.D. (%)
Unspliced 60 cm×50 μm I.D.	$4.1\cdot10^4$	$4.9\cdot10^3$	$1.2\cdot10^1$	2.2	$5.7\cdot10^{-2}$	2.6
Spliced 60 cm×50 μm I.D.	$3.8\cdot10^4$	$3.7\cdot10^3$	9.7	2.0	$1.6\cdot10^{-2}$	$8.3\cdot10^{-1}$
Spliced 45 cm×50 μm I.D.	$1.8\cdot10^4$	$2.2\cdot10^3$	$1.2\cdot10^1$	1.4	$1.1\cdot10^{-2}$	$7.3\cdot10^{-1}$
Spliced 75 cm×50 μm I.D.	$5.1\cdot10^4$	$9.5\cdot10^3$	$1.9\cdot10^1$	2.9	$1.3\cdot10^{-1}$	4.5
Spliced 60 cm×75 μm I.D.	$2.1\cdot10^4$	$6.7\cdot10^3$	$3.2\cdot10^1$	2.2	$1.3\cdot10^{-1}$	6.0

[a] Triplicate data for peaks representing C_{12}-, C_{10}-, C_8- and C_6-SO_3^- were averaged.

Table 2
Comparison of linearity response for the anionic surfactants on the bare unspliced and spliced capillaries

Unspliced Capillary (60 cm×50 μm I.D.)				
Analyte (RSO_3^-)	Data points	Linear range ($mg\,l^{-1}$)	Least squares equation $y=mx+b$	Correlation coefficient
C_6-	12	40–80	$m=2.21\cdot10^{+3}$ $b=1.03\cdot10^{+5}$	0.999
C_8-	12	40–80	$m=2.92\cdot10^{+3}$ $b=9.23\cdot10^{+4}$	0.999
C_{10}-	12	40–80	$m=3.16\cdot10^{+3}$ $b=8.87\cdot10^{+4}$	0.997
C_{12}-	12	40–80	$m=2.61\cdot10^{+3}$ $b=9.79\cdot10^{+4}$	0.988
Spliced Capillary (50 μm (I.D.), 60 cm length)				
Analyte (RSO_3^-	Data points	Linear range (mg/L)	Least squares equation $y=mx+b$	Correlation coefficient
C_6-	12	40–80	$m=3.01\cdot10^{+3}$ $b=9.64\cdot10^{+4}$	0.988
C_8-	12	40–80	$m=3.54\cdot10^{+3}$ $b=9.97\cdot10^{+4}$	0.996
C_{10}-	12	40–80	$m=3.47\cdot10^{+3}$ $b=1.15\cdot10^{+5}$	0.998
C_{12}-	12	40–80	$m=2.85\cdot10^{+3}$ $b=1.00\cdot10^{+5}$	0.999

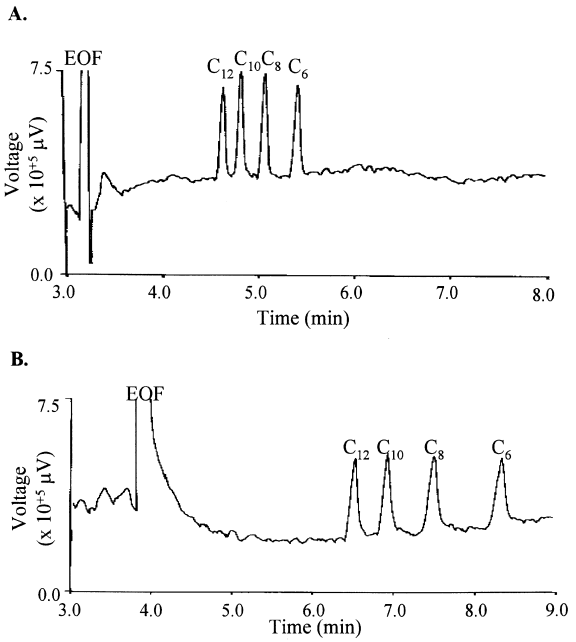

Fig. 2. Comparison of capillary diameter. Electrolyte: 20 m*M* NaF, 1 m*M* TEA, water–acetonitrile (90:10); injection: pressure, 25 mbar for 12 s, 7.7 nl for 60 cm×50 μm I.D. capillary (A) and 38.8 nL for 60 cm×75 μm I.D., capillary (B); voltage: +25 kV; indirect conductivity detection; analytes: 80 ppm C_6-, C_8-, C_{10}- and C_{12}-SO_3^-.

was almost half that for the 75 μm spliced capillary as compared to the 50 μm spliced capillary of the same length (Table 1). However the resolution of the C_{12}–C_{10} peak pair actually improved by 10% (Table 1) and averaged 19% better for the C_{10}–C_8 and C_8–C_6 peak pairs. Two explanations for this slight improvement in R_s for the wider diameter capillary are the slower average mobility of the peak pair and the lack of significant Joule heating. When the length was decreased to 45 cm (50 μm I.D.) (Fig. 3) the migration time decreased by a factor of 4 and the sharpness of the peaks increased by a factor of 2. For all the analytes, the plate count and resolution increased by a factor of 1.4 for the longer 75 cm capillary and decreased by an average factor of 1.7 for the shorter 45 cm capillary. This length of 45 cm was the minimum the instrument could accommodate. These trends were generally as expected and helps prove that the splicing technique is a feasible solution if sample load ability on a wide diameter capillary or faster throughput on a shorter capillary is important.

3.3. Comparison of bare capillary, bonded amine capillary, and C-18 capillary

A comparison of the CE separation of inorganic anions on a bare spliced and amine bonded (CElect Amine) capillary was made. One advantage of the CElect Amine capillary was no equilibration of an EOF modifier such as 1 m*M* CTAB was needed between runs. It was found that electropherograms taken on the CElect Amine capillary (Fig. 4) required longer migration times but interestingly peaks were actually sharper. The R.S.D.s of the apparent mobilities (Table 3) for the CElect Amine capillary were better than those of the bare capillary; this could be evidence that the bonded capillary is more reproducible than a chemically EOF modified bare capillary. The peak height (data not shown) and area

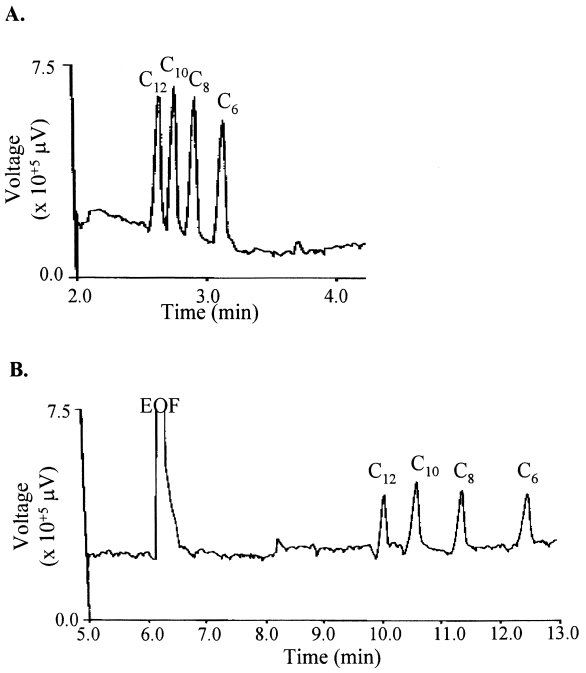

Fig. 3. Comparison of capillary length. Electrolyte: 20 m*M* NaF, 1 m*M* TEA, water–acetonitrile (90:10); injection: pressure, 25 mbar for 12 s, 10.2 nl for 45 cm×50 μm I.D. capillary (A) and 6.1 nL for 75 cm×50 μm I.D. capillary (B); voltage: +25 kV; indirect conductivity detection; analytes: 80 ppm C_6-, C_8-, C_{10}- and C_{12}-SO_3^-.

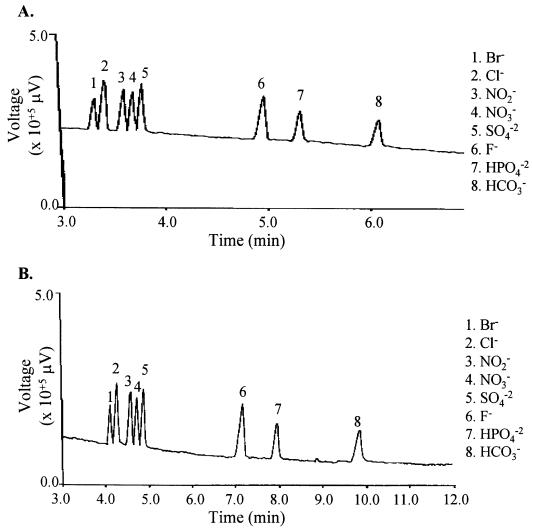

Fig. 4. Comparison of CTAB modified (A) and CElect Amine (B) electropherograms. Electrolyte: 50 m*M* CHES, 20 m*M* LiOH, pH=9.2, 0.03 %. (w/w) Triton X-100; injection: pressure, 25 mbar for 20 s, 12.8 nl for 60 cm×50 μm I.D. capillary; voltage: −25 kV; direct conductivity detection; analytes: 10 ppm Br^-, Cl^-, NO_2^-, NO_3^-, SO_4^{-2}, F^-, HPO_4^{-2} and HCO_3^-.

reproducibility study (Table 4) was completed and showed that both parameters for the CElect Amine capillary were slightly larger, on average about a factor of 1.2 for peak height and 2.2 for peak area. However, the R.S.D. values for this capillary were also poorer in every case except for carbonate in the peak height study and poorer in a few cases in the peak area study (Table 4). This could be due to the fact that the baseline for the bare capillary was very straight and the baseline for the CElect Amine capillary drifted downhill slightly causing more of an error in our integration software. Modification of the integration program was attempted; however, due to the simplicity of the integration software, alterations could not be made.

Another advantage of being able to use bonded

Table 3
Apparent mobility (μ_a) reproducibility study of anion standards on bare and bonded amine spliced capillaries

Anion standard (10 ppm) n=9	Bare Spliced			CElect Amine spliced		
	Average μ_a (cm^2 (V min)$^{-1}$)	S.D.	R.S.D. (%)	Average μ_a (cm^2 (V min)$^{-1}$)	S.D.	R.S.D. (%)
Br^-	0.0441	$7.24\cdot10^{-4}$	1.64	0.0352	$1.42\cdot10^{-4}$	0.404
Cl^-	0.0428	$7.22\cdot10^{-4}$	1.68	0.0339	$1.37\cdot10^{-4}$	0.404
NO_2^-	0.0406	$6.68\cdot10^{-4}$	1.65	0.0315	$9.98\cdot10^{-5}$	0.317
NO_3^-	0.0396	$6.27\cdot10^{-4}$	1.59	0.0305	$7.73\cdot10^{-5}$	0.254
SO_4^{2-}	0.0386	$7.31\cdot10^{-4}$	1.89	0.0296	$1.03\cdot10^{-4}$	0.346
F^-	0.0294	$5.23\cdot10^{4}$	1.78	0.0201	$7.84\cdot10^{-5}$	0.391
HPO_4^{2-}	0.0274	$5.01\cdot10^{-4}$	1.82	0.0180	$8.99\cdot10^{-5}$	0.498
HCO_3^-	0.0239	$3.68\cdot10^{-4}$	1.54	0.0143	$1.94\cdot10^{-4}$	1.36

Table 4
Peak area reproducibility study of anion standards on bare and bonded amine spliced capillaries

Anion standard (10 ppm) $n=9$	Bare spliced			CElect amine spliced		
	Average area (μV s)	S.D.	R.S.D. (%)	Average area (μV s)	S.D.	R.S.D. (%)
Br^-	$1.7\cdot10^{+5}$	$4.0\cdot10^{+3}$	2.3	$3.5\cdot10^{+5}$	$2.6\cdot10^{+4}$	7.4
Cl^-	$3.3\cdot10^{+5}$	$9.0\cdot10^{+3}$	2.7	$6.6\cdot10^{+5}$	$3.9\cdot10^{+4}$	5.9
NO_2^-	$2.7\cdot10^{+5}$	$7.1\cdot10^{+3}$	2.7	$5.4\cdot10^{+5}$	$3.3\cdot10^{+4}$	6.2
NO^{3-}	$2.2\cdot10^{+5}$	$1.2\cdot10^{+4}$	5.4	$4.4\cdot10^{+5}$	$2.6\cdot10^{+4}$	5.9
SO_4^{2-}	$3.0\cdot10^{+5}$	$1.8\cdot10^{+4}$	5.9	$5.7\cdot10^{+5}$	$2.6\cdot10^{+4}$	4.6
F^-	$4.3\cdot10^{+5}$	$1.2\cdot10^{+4}$	2.7	$9.9\cdot10^{+5}$	$3.1\cdot10^{+4}$	3.1
HPO_4^{2-}	$2.6\cdot10^{+5}$	$2.6\cdot10^{+4}$	9.8	$5.7\cdot10^{+5}$	$4.2\cdot10^{+4}$	7.3
HCO_3^-	$3.2\cdot10^{+5}$	$5.3\cdot10^{+4}$	$1.7\cdot10^{+1}$	$9.3\cdot10^{+5}$	$1.3\cdot10^{+5}$	$1.4\cdot10^{+1}$

capillaries is that the EOF can simply be modified increasing the cation window as compared to a bare capillary. Use of the bonded CElect Amine spliced capillary increased the cation window from 4 to 6 min. with better resolution of the cationic surfactants (data not shown). Use of the C_{18} capillary improved peak sharpness even though the cation window has increased to 10 min as compared to a bare capillary (Fig. 5). However, the THA^+ was no longer present when the C_{18} capillary was used; it is believed that the THA^+ was bonding to the capillary wall. The addition of Brij 35 and more acetonitrile were used as modifications of the electrolyte in an attempt to decrease the analyte capillary wall interactions. Both of these modifications of the electrolyte were not promising; the Brij 35 caused the baseline to become very noisy making identification of the peaks impossible and the additional acetonitrile (20% versus 10%) did not give reproducible evidence of the THA^+ peak. Use of the C_{18} capillary to resolve closely related short chain cationic surfactants should be feasible.

3.4. Analysis of real samples

Finally the analysis of toothpaste samples for cations and anions were completed on the CElect Amine capillary. Each variety of toothpaste was chosen for its specific analytes present. The active ingredient in Crest was sodium fluoride; those in Sensodyne were potassium nitrate and sodium monofluorophosphate and that in Topol was sodium monofluorophosphate. Representative electropherograms for the cations and anions in Crest and Sensodyne (Figs. 6 and 7) are presented. The cation electropherogram for Topol was similar to that of Crest and the Topol anion electropherogram included a monofluorophosphate peak and a large bicarbonate peak from the baking soda present. All of the cations

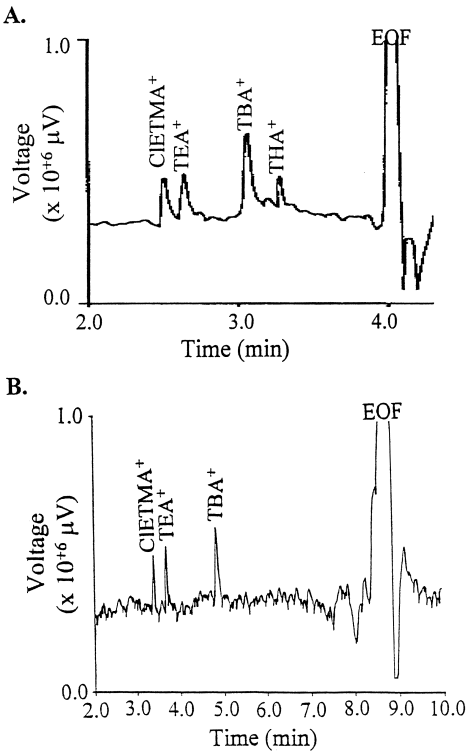

Fig. 5. Comparison of bare spliced (A) and C_{18} bonded (B) electropherograms. Electrolyte: 20 m*M* NaF, 1 m*M* TEA, water–acetonitrile (90:10); injection: pressure, 25 mbar for 12 s, 7.7 nl for 60 cm×50 μm I.D. capillary; voltage: +25 kV; indirect conductivity detection; analytes: 80 ppm $ClETMA^+$, TEA^+, TBA^+ and THA^+.

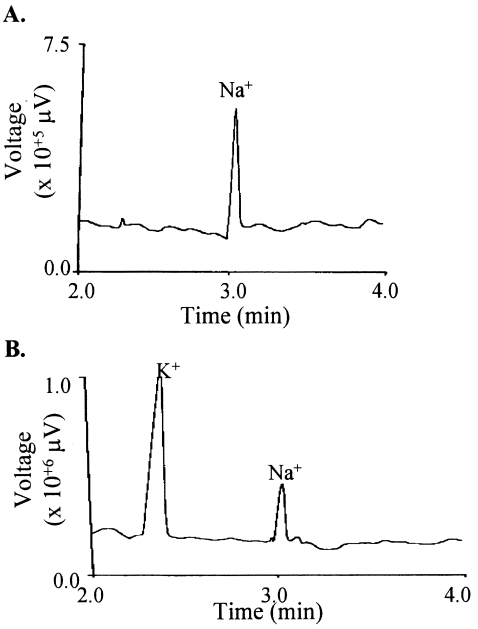

Fig. 6. Analysis of cations present in 0.21 g regular flavor paste Crest/50 ml water (A) and 0.25 g Fresh Mint Sensodyne/50 ml water (B). Electrolyte: 50 m*M* CHES, 20 m*M* LiOH, pH=9.2, 0.03 % (w/w) Triton X–100; injection: pressure, 25 mbar for 20 s, 12.8 nl for 60 cm×50 μm I.D. capillary; voltage: +25 kV and direct conductivity detection.

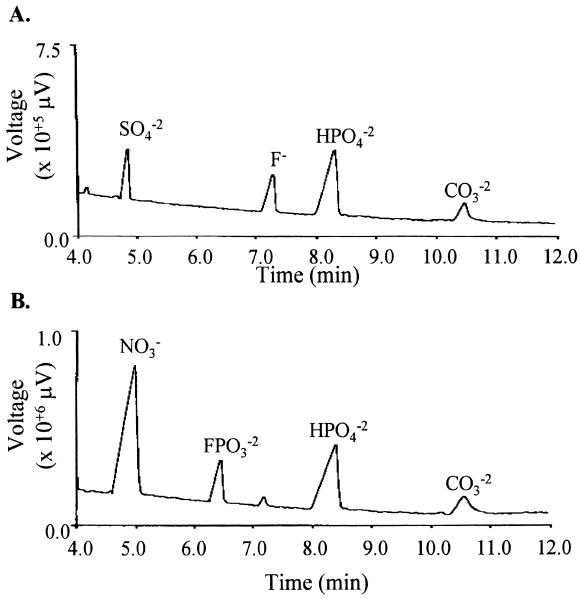

Fig. 7. Analysis of anions present in 0.21 g regular flavor paste Crest/50 ml water (A) and 0.25 g Fresh Mint Sensodyne/50 ml water (B). Electrolyte: 50 m*M* CHES, 20 m*M* LiOH, pH=9.2, 0.03 % (w/w) Triton X–100; injection: pressure, 25 mbar for 20 s, 12.8 nl for 60 cm×50 μm I.D. capillary; voltage: −25 kV and direct conductivity detection.

and anions expected were easily separated and detected except for the calcium present in the Sensodyne. It was believed that the calcium was not detected due to the high pH of the CHES electrolyte. A previous CE separation of cations including calcium at pH 8.0 required the use of EDTA as a complexing agent to prevent insolubility due to hydroxide precipitation or metal adsorption to the capillary wall [10]. Also, at this time a 30 m*M* 3-(*N*-morpholino)propanesulfonic acid (MOPS), 8 m*M* histidine, 0.03 % (w/w) Triton X-100 electrolyte was tried in an attempt to detect the calcium; however, the baseline was very erratic and not even sodium and potassium could be identified. However it can be concluded that this bonded amine capillary can allow the determination of either cations or anions in the real samples using the same run buffer but just switching the voltage polarity. This approach is comparable in ease of use to a recently published paper describing the simultaneous separation of small anions and cations with indirect detection [11].

3.5. Advantages and future work

It was determined that the splicing technique is not only feasible but reproducible. The spliced capillary allowed for versatility in the choice of capillary dimension and type such as bonded columns. Future work in capillary electrochromatography with conductivity detection is envisioned. In addition, a packed sample matrix cleanup capillary coupled to an open tubular separation capillary could aid in solving real world problems by CE.

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