Research Article

Polyacrylamide-based monolithic capillary column with coating of cellulose tris(3,5-dimethylphenyl-carbamate) for enantiomer separation in capillary electrochromatography

A hydrophilic chiral capillary monolithic column for enantiomer separation in CEC was prepared by coating cellulose tris(3,5-dimethylphenyl-carbamate) (CDMPC) on porous hydrophilic poly(acrylamide-co-N,N'-methylene-bisacrylamide) (poly(AA-co-MBA)) monolithic matrix with confine of a fused-silica capillary. The coating conditions were optimized to obtain a stable and reproducible chiral stationary phase for CEC. The effect of organic modifier of ACN in aqueous mobile phase for the enantiomer separation by CEC was investigated, and the significant influence of ACN on the enantioresolution and electrochromatographic retention was observed. Twelve pairs of enantiomers including acidic, neutral, and basic analytes were tested and nine pairs of them were baseline-enantioresolved with acidic and basic aqueous mobile phases. A good within-column repeatability in retention time (RSD = 2.4%) and resolution (RSD = 3.2%) was obtained by consecutive injections of a neutral compound, benzoin, on a prepared chiral monolithic column, while the between-column repeatability in retention time (RSD = 6.4%) and resolution (RSD = 9.6%) was observed by column-to-column examination. The prepared monolithic stationary phase showed good stability in either acidic or basic mobile phase.

Keywords:
Capillary electrochromatography / Cellulose tris(3,5-dimethylphenyl-carbamate) / Enantioseparation / Polyacrylamide / Monolithic capillary column

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1 Introduction

CEC, which combines the features of LC and CE, permits the extremely high efficiency separation of analytes due to the plug profile flow of mobile phase driven by the EOF in a capillary column. Additionally, as a miniaturized separation technique, CEC also shows its advantages in low consumption of sample, solvent, and stationary phases, especially for expensive chiral stationary phase (CSP). In the past decade, CEC has been proved as a potential technique for enantiomer separation. Various types of chiral CEC columns have been investigated, including open-tubular, conventionally packed, and monolithic capillary columns.

In open-tubular capillaries, the inside wall was modified with appropriate chiral selectors including CD derivative [1], proteins [2, 3], polysaccharide derivatives [4], etc. The chiral resolution ability was generally low due to the low phase ratios of open-tubular capillary columns. In particulates packed column, the CSPs were packed into the capillaries and restricted by the retaining frits. CSPs used in HPLC could be simply transferred to CEC, such as CD and its derivatives [5–7], protein [8], macrocyclic antibiotics [9], chiral polycarylamides [10], and Pirkle-type CSP [11]. But the bubble formation and poor repeatability caused by the supporting frits retarded the application of particulate packed column in CEC for the practical routine analysis of enantiomers. In recent years, monolithic stationary phase has attracted increasing attention in CEC. It can be prepared by in situ polymerization within the confines of capillaries, thus avoiding
both end-frits required for particulate columns to secure the packing materials in place [12]. Monolithic columns can be classified into two general categories: organic polymer-based and silica-based columns. Monolithic silica rods are porous monoliths consisting of a silica skeleton with interconnecting macropores. Chiral selectors have been incorporated into silica monolithic matrices by in situ encapsulation and entrapment of proteins [13, 14], or attached to silica monoliths by physical adsorption [15] and in-column derivatization [16]. The chiral selectors utilized for the preparation of enantioselective silica-based monoliths were covered from CD [16], chiral ion-exchanger [17], protein [15], and ligand-exchange-type phase [18] to cellulose derivatives [19]. However, the column stability under extremely low and high pH was the major limitation for the use of silica-based monolithic columns. CSPs based on the organic polymer monoliths for CEC have been prepared by immobilization of diverse chiral selectors such as proteins [20], CDs [21, 22], macrocyclic antibiotics [23, 24], crown ethers [25] and “Pirkle”-selector [26] onto the poly-methacrylates [26], and polyacrylamide rods [20–25].

Polysaccharide derivative CSPs have been widely applied in HPLC [27] with good chiral selectivity. In CEC, cellulose derivatives including cellulose tris(3,5-dimethylphenyl-carbamate) (CDMPC) [10, 28–30], cellulose tris(4-methylbenzoate) [31, 32], cellulose tris(3,5-dichlorophenylcarbamate) [33–35], and amylase(3,5-dimethylphenylcarbamate) [31, 32] have also been used for the separations of enantiomers via coating or bonding on silica-based particulate packed columns or monolithic column. Interestingly, little has been reported on the use of cellulose derivatives, such as CDMPC, for the preparation of hydrophilic organic polymeric monolithic CSPs in CEC.

In this work, a polyacrylamide-based chiral monolithic column with CDMPC coating was prepared for enantioseparation in CEC. By introducing a number of quaternary ammonium groups on the polyacrylamide-based monolith, the cellulose derivatives (CDMPC) could be tightly retained on the monolithic matrix via the strong hydrogen-bonding interaction. In addition, a hydrophilic matrix was preferable to a hydrophobic matrix by reducing the non-enantioselective interaction for the separation of enantiomers [36], so the coating of cellulose derivatives on the polyacrylamide-based monolithic capillary column is a promising combination for the preparation of CSPs in CEC with the advantages of easy preparation, powerful chiral recognition ability of polysaccharide derivatives, good pH stability, and substantial EOF over a wide range of pH.

2 Materials and methods

2.1 Chemicals and reagents

Microcrystalline cellulose and ACN were purchased from Merck (Darmstadt, Germany). Acrylamide (AA) and N,N’-methylene-bisacrylamide (MBA) were purchased from Acros (NJ, USA). γ-Methacryloxypropyltrimethoxysilane (γ-MAPS), 3,5-dimethylphenyl isocyanate, diethylamine, 2-(methacryloyloxy)ethyl trimethyl ammonium methyl sulfate (MEAMS), racemic benzoin, indapamide, praziquantel, Tröger’s base, alprenol, pindolol, propranolol, (R,S)-1,1’-bi-2-naphthol (BNI), and (R,S)-octahydro-1,1’-bi-2-naphthol (OHBN) were purchased from Sigma–Aldrich (Milwaukee, WI, USA). Racemic 4,4’-dimethoxy-5,6,6’-dimethylene-dioxy-biphenyl-2,2’-dicarboxylate (DDBD) was a gift from Lanzhou Institute of Chemical Physics (Lanzhou, China). Water used in all experiments was doubly distilled and purified by a Milli-Q system (Millipore, Milford, MA, USA). CDMPC was homemade as described in the literature [37].

2.2 Preparation of CDMPC-coated polyacrylamide monolithic columns

2.2.1 Preparation of polyacrylamide monoliths

A fused-silica capillary (75 μm id × 365 μm od), which was purchased from Yongnian Optic Fiber Plant (Hebei, China) was used to prepare a polyacrylamide-based monolithic capillary column. Briefly, the inner wall of a capillary was pre-vinylized by γ-MAPS as described by Dong et al. [38]. The approach of preparing the polyacrylamide-based monolith was similar as described by Xie et al. [39] with appropriate modification for CEC. A polymerization mixture containing MBA (40 mg), AA (20 mg), MEAMS (40 μL), AIBN (1 mg, 1 wt.% with respect to the monomers), and the porogenic solvent consisted of 1-dodecanol (200 μL) and DMSO (280 μL) was sonicated for 20 min to a homogeneous solution. Subsequently, the sonicated solution was infused into a vinyl pretreated capillary for full length with a syringe, and then both ends of the capillary were sealed with rubber stoppers. After that, the capillary was submerged into a water bath at 60°C for 10 h. The resultant polyacrylamide-co-N,N’-methylene-bisacrylamide) (poly(AA-co-MBA)) monolithic capillary column was first flushed with methanol for about 2 h to remove unreacted monomers, porogens, and other residues. Followed by an additional rinse with acetone, the synthesized polyacrylamide monolithic capillary column was dried overnight by passing through nitrogen at room temperature.

2.2.2 In situ coating of polyacrylamide monolith with CDMPC

The in situ coating of CDMPC on the dried polyacrylamide monolithic capillary column was carried out as following steps. At first, the in situ modification of the dried polyacrylamide monolithic capillary column was carried out using the approach described by Chankvetadze et al. [40]. Briefly, the dried polyacrylamide monolithic capillary column (35 cm) was connected to a stainless-steel column (50 mm × 4.6 mm id) filled with the prepared CDMPC solution, and then the opposite end of the stainless-steel column was connected to...
an HPLC pump. The HPLC pump was operated in a constant pressure mode of 10 MPa to drive the CDMPC solution through the capillary column. The HPLC pump pressure was remained at 10 MPa for additional 60 min after the capillary column was for sure filled with CDMPC solution determined by a laboratory microscope. Then the monolithic capillary column was detached from the stainless-steel column and left to dry at ambient pressure and temperature. After drying for 7 to 12 days at ambient condition, the monolithic capillary was further dried overnight in a vacuum oven at 40°C. The detection window was then made by burning off 1–2 mm of both the outer-coated polyimide and the inner polyacrylamide monolith of the capillary with a heating coil. The burned ashes of the polyacrylamide monolith within the capillary were flushed out with water using an HPLC pump.

2.3 Separation conditions in CEC

The separations of enantiomers by CEC were carried out on a CE instrument-P/ACE™ MDQ System (Beckman, Fullerton, CA, USA) equipped with a UV DAD, while the temperature for columns was set at 25°C and wavelength for detection was set at 214 nm. All analytes were dissolved in running mobile phase to give final concentrations ranged from 0.5 to 1.0 mg/mL. Electrokinetic injection was adopted to load samples by applying 10 kV voltage for 1 s. The chiral resolution factor (Rs) were calculated from the equation 

\[ R_s = \frac{2(t_{R2} - t_{R1})}{W_1 + W_2} \]

where \( t_{R1} \) and \( t_{R2} \) are the retention times of the 1st and 2nd eluted enantiomers, respectively, and \( W_1 \) and \( W_2 \) are the peak widths of these two enantiomers in elution order.

3 Results and discussion

3.1 Characteristics of the monolithic columns

The native poly(AA-co-MBA) monolithic matrix was prepared by in situ copolymerization of AA, MBA, and along with a positively charged comonomer of MEAMS in the presence of porogenic solvents of 1-dodecanol and DMSO using AIBN as the initiator. Due to the previnylization of the inner wall of the capillary by γ-MAPS, the prepared polyacrylamide monolithic matrix was covalently anchored onto the inner wall of capillary and the supporting frits were thus avoided in column preparation for CEC. Figure 1 shows the SEM photographs of the cross-section of a monolithic capillary column at two different magnifications (1000 × and 5000 ×), where the capillary was fully filled and attached by the copolymerized porous polyacrylamide monolithic matrix. Through this design, the prepared monolithic capillary column promised good stability without moving of the monolithic bed before and after the on-column modification of CDMPC, which was confirmed by applying a pressure of up to 20 MPa at one end of the column with an HPLC pump using an aqueous mobile phase containing 35% ACN.

In CEC, the generation of EOF is the essential need to drive the mobile phase through a capillary column. To achieve the separation of acidic, basic and neutral enantiomers, generally in CEC, a strong anode or cathode EOF are required. In this work, the positively charged comonomer of MEAMS with quaternary ammonium group was incorporated into the copolymerization of AA and MBA, and used as the generator of a strong anionic EOF in CEC. Even by the on-column coating of the native polyacrylamide monolith with the neutral cellulose derivative of CDMPC, the strong anionic EOF was yet maintained over a wide range of mobile phase pH values. Figure 2 presents the dependence of the anionic EOF on the pH values of the mobile phase on a CDMPC-coated polyacrylamide monolithic capillary column. It can be seen that the magnitude of EOF on the CDMPC-coated monolithic column remains in a range of \( 1.46 \times 10^{-8} \) to \( 1.02 \times 10^{-8} \) \( \text{m}^2 \cdot \text{v}^{-1} \cdot \text{s}^{-1} \) when the pH of mobile phase changes from 2.7 to 9.7. This should mainly benefit from the fully dissociation of the incorporated quaternary ammonium groups in the polyacrylamide monolith over the wide range of pH. The stable EOF indicated that the polyacrylamide-based monolithic capillary column by incorporation of positively charged ammonium groups could generate substantial anionic EOF over an extended pH range, which enabled the enantioseparation of acidic,
neutral, and basic chiral analytes with a single CEC column. The slight decrease of the anionic EOF with increase of mobile phase pH could be caused by the impairment of the zeta potential generated by the positively charge ammonium groups due to the partial dissociation of the negatively charged residual silanol groups on the inner wall of the fused-silica capillary.

The dependence of the applied voltage and the produced current was also investigated by using a CDMPC-modified monolithic capillary column (50 mg/mL of CDMPC) in CEC with a phosphate-buffered (2 mM, pH 2.7) mobile phase containing 35% ACN. The current linearly increased from 0.8 to 6.9 μA by increasing the applied voltage from 2 to 20 kV with a correlation coefficient (r) of 0.9993. This indicated that the Joule heating associated problems seemed not to be a cause for concern; thus, the fast separation of enantiomers could be achieved by applying high voltages on this monolithic capillary column.

### 3.2 Effects of coating conditions on enantioseparation

The chiral polyacrylamide monolithic capillary column for CEC was prepared by an in situ surface coating of the synthesized polyacrylamide-based monolith with the cellulose derivative of CDMPC within the confines of a fused-silica capillary. In this work, the coating factors such as coating time and concentration of CDMPC solution were investigated for obtaining the stable and reproducible CSP for the separation of enantiomers by CEC.

To avoid the possible damage such as the distortion and compression of polyacrylamide-based monolithic matrix within a fused-silica capillary under the high pressure of the HPLC pump for the coating of CDMPC, a coating pressure of 10 MPa was applied. Because of the high viscosity of CDMPC solution at the coating concentrations, the long enough coating time was required for obtaining the homogeneously CDMPC-coated monolithic column under the certain coating pressure. Thus, the coating of CDMPC within a capillary was monitored by inspecting the color change of the capillary monolithic column under a lab optical microscopy during the perfusion of CDMPC solution. Based on the microscopy monitoring of the CDMPC coating process, the darker and homogeneous cross-sections of monolithic columns along the axis of capillaries could be observed in stable when an additional time of 60 min remained for delivering CDMPC solutions through monolithic matrices right after the outflow of coating solutions out of columns.

In this approach, the neutral coated CDMPC was used as a chiral selector for resolving the enantiomers on the polyacrylamide monolithic support. The amount of CDMPC coated onto the monolithic support would result in different chromatographic properties of the column, such as the retention, resolution, and efficiency. Here, the influence of the concentration of CDMPC in coating solution was investigated for the separation of enantiomers. Figures 3a and b, respectively, show the obtained electrophorograms for enantioseparations of Tröger’s base and indapamide on the prepared columns coated with CDMPC at 30 and 50 mg/mL in acetone. It can be seen that the retention time and resolution of the enantiomers increased by increasing concentration of CDMPC in acetone coating solution, and the baseline separation of Tröger’s base could be achieved when the loading concentration of 50 mg/mL of CDMPC was used for both Tröger’s base and indapamide. However, the decrease of the column efficiency was also observed by increasing the coating concentration of CDMPC for enantioseparation in CEC which was similar with Chankvetadze et al. [41]. When the loading concentration of CDMPC increased to 90 mg/mL, the coating process became technically difficult because of the higher solution viscosity and the unacceptable long drying time (more than one month). Although shorter separation time and higher column efficiency could be obtained by loading lower concentration of CDMPC, the better chiral resolution was more appropriate with acceptable retention time and column efficiency. Thus, the coating concentration of 50 mg/mL of CDMPC in solution was used for the preparation of the chiral monolithic capillary column in this study.

### 3.3 Enantioseparation on the CDMPC-coated monolithic column

Polysaccharide derivatives have been widely used as chiral selectors for the separation of enantiomers in HPLC [10] and CEC [30] by bonding or coating on the particulate packed columns. On monolithic columns, the immobilization...
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Figure 3. Electrochromatograms of Tröger’s base and indapamide on CDMPC-coated monolithic columns. Experimental conditions: capillary columns, monolithic column coated with 30 mg/mL, or 50 mg/mL of CDMPC; mobile phase, 2 mM phosphate buffer containing 35% ACN at pH 2.7; applied voltage, 215 kV; injection, 10 kV x 1 s.

The enantioseparations of several basic enantiomers by CEC were also successfully carried out. Generally, basic enantiomers were positively charged in acidic or neutral aqueous solutions, and thus the directions of their electrophoretic mobilities of these basic analytes were opposite to the direction of the anionic EOF. Therefore, it is difficult for these basic analytes to be eluted out under acidic and neutral conditions, whereas, in a basic mobile phase, they were less positively charged and could migrate to the detection window when the EOF was greater than their electrophoretic mobilities in CEC. It was so-called the “counterdirectional mode” in CEC, which was suggested by Ericson and Hjertén [45]. In this mode, the electrostatic interaction caused irreversible adsorption of basic solutes on stationary phases was also avoided. By taking advantage of this CEC mode, two pairs of basic enantiomers, namely tetrahydropalmatine and pindolol, were completely resolved with a mobile phase of 2 mM phosphate buffer (pH 9.7) containing 35% ACN. Typical electrochromatograms are illustrated in Figs. 4f and
Table 1. Enantioseparation of test racemic compounds on the CDMPC-coated polyacrylamide-based monolithic column

<table>
<thead>
<tr>
<th>Compounds</th>
<th>Mobile phases</th>
<th>$t_1^*$ (min)</th>
<th>$t_2^*$ (min)</th>
<th>$N_1^*$ (plates/m)</th>
<th>$N_2^*$ (plates/m)</th>
<th>$R_s^*$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Benzoin</td>
<td>a</td>
<td>16.64</td>
<td>18.57</td>
<td>47 100</td>
<td>76 100</td>
<td>3.17</td>
</tr>
<tr>
<td>Benzoin rep</td>
<td>a</td>
<td>16.51</td>
<td>18.82</td>
<td>31 800</td>
<td>36 600</td>
<td>2.70</td>
</tr>
<tr>
<td>Indapamide</td>
<td>a</td>
<td>16.14</td>
<td>19.25</td>
<td>20 040</td>
<td>20 300</td>
<td>3.06</td>
</tr>
<tr>
<td>Pruziquantel</td>
<td>a</td>
<td>19.49</td>
<td>20.77</td>
<td>30 900</td>
<td>16 800</td>
<td>1.11</td>
</tr>
<tr>
<td>Tröger’s base</td>
<td>a</td>
<td>21.49</td>
<td>23.63</td>
<td>20 500</td>
<td>17 900</td>
<td>1.47</td>
</tr>
<tr>
<td>BNL</td>
<td>a</td>
<td>37.13</td>
<td>38.66</td>
<td>84 500</td>
<td>45 900</td>
<td>0.95</td>
</tr>
<tr>
<td>OHBN</td>
<td>a</td>
<td>39.02</td>
<td>49.07</td>
<td>15 100</td>
<td>21 700</td>
<td>3.55</td>
</tr>
<tr>
<td>Warfarin</td>
<td>a</td>
<td>23.51</td>
<td>28.32</td>
<td>28 000</td>
<td>56 000</td>
<td>4.14</td>
</tr>
<tr>
<td>DDBD</td>
<td>a</td>
<td>22.15</td>
<td>22.55</td>
<td>–</td>
<td>–</td>
<td>–</td>
</tr>
<tr>
<td>Tetrahydropalmatine</td>
<td>b</td>
<td>15.10</td>
<td>22.98</td>
<td>20 800</td>
<td>31 800</td>
<td>7.64</td>
</tr>
<tr>
<td>Tetrahydropalmatine rep</td>
<td>b</td>
<td>14.60</td>
<td>22.50</td>
<td>16 400</td>
<td>22 000</td>
<td>6.65</td>
</tr>
<tr>
<td>Pindololol</td>
<td>b</td>
<td>13.19</td>
<td>14.57</td>
<td>83 700</td>
<td>20 700</td>
<td>2.14</td>
</tr>
<tr>
<td>Alprenolol</td>
<td>b</td>
<td>11.03</td>
<td>11.23</td>
<td>–</td>
<td>–</td>
<td>–</td>
</tr>
<tr>
<td>Propranolol</td>
<td>b</td>
<td>16.51</td>
<td>16.93</td>
<td>–</td>
<td>–</td>
<td>–</td>
</tr>
</tbody>
</table>

Experimental conditions: column, 75 µm id poly(AA-co-MBA) monolithic column modified with 50 mg/mL of CDMPC; mobile phase, (a) 2 mM phosphate buffer containing 35% ACN at pH 2.7; (b) 2 mM phosphate buffer containing 35% ACN at pH 9.7; applied voltage, –15 kV; injection, –10 kV × 1 s; total length of capillary column, 31 cm, effective length, 20 cm.

*: $t$ represents the retention time of analytes; $N$ represents the plate number of column; $R_s$ represents the chiral resolution factor; subscripts of 1 and 2 designate the 1st and 2nd eluted peak of a pair of an enantiomer, respectively.

rep: repeat measurement of analytes on a column, which had been repeatedly used for 50 injections.

3.4 Effect of organic modifier in mobile phase on enantioseparation

The separation of enantiomers could be influenced by the content of the organic modifier in the mobile phase. An investigation of the effect of organic modifier (ACN) was subsequently carried out to evaluate the effect of organic modifier on retention factor, resolution, and efficiency for the prepared CDMPC-coated polyacrylamide monolithic capillary column. Benzoin was selected as a standard testing analyte for investigating the influence of the organic modifier in mobile phase, and the obtained results are shown in Fig. 5. The retention factor of benzoin enantiomers generally increased by decreasing the ACN content in mobile phase, which implied that the hydrophobic interaction between CDMPC and chiral analytes [30]. In chiral separation, this hydrophobic interaction is a kind of a “long-distance” interaction that pushes the chiral analytes close to the CSPs in RP mode, and cooperates with two “short-distance” interactions of π-π and hydrogen-bonding interactions for the chiral recognition. According to the retention factor tendency, the resolution value should increase, but it decreased when the content of ACN decreased from 35 to 30%. It may be due to the lower peak efficiency at 30% of ACN. The highest resolution value and efficiency were recorded when the mobile phase contained 35% ACN. The results, here, indicated that the organic modifier content had an important effect on both retention and resolution on our CSP and the concentration of 35% ACN was applied in our CEC enantioseparations. Although, the effect of the organic modifier in mobile phase on enantioseparation was obtained only based on the investigation of benzoin rather than all of the test enantiomers in this experiment, the fairly good separations of all test enantiomers were also realized under this percentage of ACN in mobile phase as presented in Table 1.
Figure 4. Separation of enantiomers on a CDMPC-coated polyacrylamide monolithic capillary column. Solutes: (a) praziquantel; (b) benzoin; (c) BNL; (d) OHBN; (e) warfarin; (f) tetrahydropalmatine; (g) pindolol. Experimental conditions were the same as described in Table 1.

Figure 5. Effect of ACN content in mobile phase on resolution, efficiency, and $k_1$ of the first eluted enantiomer of benzoin ($k_1 = t_1 - t_0/t_0$). All other experimental conditions were the same as described in Table 1.

3.5 Column repeatability and stability

Column stability and repeatability are two important parameters for chromatographic columns in general. To examine the repeatability of column preparation process, five CDMPC-coated polyacrylamide monolithic capillary columns were prepared. The RSDs of retention time and resolution are shown in Table 2. These RSDs indicated that the repeatabilities for column preparation of the CDMPC-coated monolithic columns as well as for the enantioseparation of analytes were acceptable. In addition, the prepared monolithic column coated with a concentration of 50 mg/mL of CDMPC was used with highly acidic (pH 2.7) and basic (pH 9.7) mobile phases for 50 injections. No column collapse and no significant decline of resolution factors, efficiencies were observed after the columns being repeatedly used for multiple injections, as shown in Table 1 (benzoin and tetrahydropalmatine as the test compounds). This indicated the good stability and pH resistance ability of CDMPC-coated polyacrylamide monolithic column in CEC.
Table 2. RSDs for run-to-run and column-to-column repeatabilities (n = 5)

<table>
<thead>
<tr>
<th>Mobile phases</th>
<th>Run-to-run (RSD, %)</th>
<th>Column-to-column (RSD, %)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Rt1</td>
<td>Rs1</td>
</tr>
<tr>
<td>Benzoin a</td>
<td>2.4</td>
<td>3.2</td>
</tr>
<tr>
<td>Warfarin a</td>
<td>2.8</td>
<td>3.6</td>
</tr>
<tr>
<td>Tetrahydro-</td>
<td></td>
<td></td>
</tr>
<tr>
<td>palmatine b</td>
<td>3.4</td>
<td>4.2</td>
</tr>
</tbody>
</table>

Experimental conditions and mobile phases a and b as described in Table 1.

4 Concluding remarks

An enantioselective poly(AA-co-MBA) monolithic capillary column was easily prepared by a radical polymerization followed by an on-column modification with CDMPC. The prepared monolithic capillary column showed good enantioselectivity with coated-CDMPC as the CSP on the hydrophilic polyacrylamide monolithic matrix, and also provided a substantial EOF over a wide pH range due to the incorporation of the charged comonomer MEAMS. Twelve pairs of enantiomers including acidic, neutral, and basic solutes were tested and nine of them were successfully resolved on this CDMPC-modified monolithic capillary column in aqueous mobile phases by CEC. The effect of organic modifier (ACN) content in mobile phases on separation of enantiomers was investigated, and the results indicated that the content of organic modifier had a significant effect on retention and resolution of enantiomers. The column also provided good repeatability and stability for the separation of enantiomers with acceptable RSDs of retention time and resolution in both acidic and basic mobile phase. Even for a long time use, no column collapse occurred during our experiment.

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5 References


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