



Acrylic-based high internal phase emulsion polymeric monolith for capillary electrochromatography

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ABSTRACT

The use of high internal phase emulsion polymers (polyHIPEs) for CEC applications has remained relatively unexplored. A few reports exist in the literature for the preparation of similar structures. In this study, polyHIPEs having high porosity, and interconnected open-cell structure, were introduced and evaluated as stationary phase for CEC. The polyHIPE monolithic columns were prepared by the in situ polymerization of isodecylacrylate (IDA) and divinylbenzene (DVB) in the continuous phase of a high internal phase emulsion (HIPE). Due to its well-defined polyHIPE structure with interconnected micron size spherical voids, the columns synthesized with different initiator concentrations were successfully used for the separation of alkylbenzenes. Furthermore, the columns indicated a strong electroosmotic flow (EOF) without any additional EOF generating monomer probably due to the presence of ionizable sulfate groups coming from the water-soluble initiator used in the preparation of polyHIPE matrix. The best chromatographic performance in the separation of alkylbenzenes was achieved by using 70% ACN in the mobile phase with high column efficiency (up to 200 000 plates/m).

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1. Introduction

In the recent years there has been significant progress in the development of macroporous monolithic materials for capillary electrochromatography (CEC). Since the problems of preparation of packed capillary columns for these systems such as inhomogeneous packing or bubble formation can be reduced by monolithic columns [1], they are very good alternatives to the conventional packed columns. Monolithic columns offer various advantages over the packed columns. The flow property of a liquid in monolithic column is totally different from that in packed columns. The inter-particle voids in the packed columns cause slower mass transfer because of diffusion between these voids and the pores within the particles [2]. On the other hand, the liquid in the monolithic column flows through the continuous phase of the macroporous material. The monoliths allow high flow velocities and superior mass transport at low back pressure due to their large pores and more open pore structure in comparison to packed columns [3,4]. As a consequence, they provide high permeability and fast separation. Moreover, since they bind to the wall of capillaries during

in situ polymerization, they do not require any retaining frits that lead to inhomogeneity in the separation media [5]. Another advantage of monolithic columns is the ease of the morphology control to optimize the chromatographic performance. The morphology of monolithic CEC columns is a very important parameter in order to control the flow velocity and the efficiency of the monolith [6]. The selectivity of the separation is controlled by the polarity of the monolith and the retention can be adjusted by the polarity of mobile phase and monolith surface [6].

Many studies on the effect of pore structure of the monoliths on chromatographic efficiency have been performed [7–9]. Ideally, a monolith should be a highly porous material that has interconnected large pores to facilitate flow [2]. In the conventional macroporous polymers, the porous structure is formed by the porogen which is a solvating or non-solvating inert solvent for the polymer. In addition to the type of porogen, type and percentage of monomer, crosslinker and initiator and polymerization temperature control the network structure and the porosity of these polymers.

An alternative approach to prepare macroporous monolithic polymers is to polymerize the continuous phase of a high internal phase emulsion (HIPE). HIPE is an emulsion system which has more than 74% droplet phase [10]. It consists of a monomeric continuous phase containing a surfactant and an aqueous dispersed phase. The polymerization of the monomer phase of a HIPE forms a macroporous material called polyHIPE. PolyHIPEs

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are highly porous materials with regular micron size spherical voids which are all interconnected through the windows. Due to these interconnections between the voids, polyHIPEs are highly permeable and so very attractive materials as chromatographic stationary phases. In the previous studies, a monolithic stationary phase for CEC was prepared by polymerization of bicontinuous microemulsions. However, SEM analysis of the monolith prepared shows that it does not have a polyHIPE structure. The morphological structure of the monolith was explained as “aggregate structure with empty channels” [11]. Poly(glycidylmethacrylate-co-ethyleneglycol dimethacrylate) monolithic supports were synthesized by radical polymerization of the continuous phase of water in oil high internal phase emulsions [12]. The monolith modified to bear weak-anion exchange groups was tested as a stationary medium for the separation of standard protein mixture. Recently, porous glycidylmethacrylate–divinylbenzene monoliths were synthesized using the HIPE approach [13].

In this study, a novel stationary phase was prepared by *in situ* polymerization of the continuous phase of a HIPE containing isodecylacrylate (IDA) and divinylbenzene (DVB) for CEC. IDA/DVB polyHIPE capillary monoliths were studied with respect to influence of organic modifier concentration, pH and voltage in the electrochromatographic separation of neutral molecules.

2. Experimental

2.1. Materials

The polyimide coated fused silica capillary with 100 μm , id and 360 μm , od was purchased from Polymicro Technologies (Phoenix, AZ, USA). The derivatization agent 3-(trimethoxysilyl)propyl methacrylate was obtained from Aldrich. The monomers used for polyHIPE synthesis were divinylbenzene (DVB) (80% Fluka) and isodecyl acrylate (IDA) (Aldrich). DVB was purified by distillation under vacuum and IDA was purified by using inhibitor remover replacement packing for HQ and MEHQ (Aldrich). The surfactant sorbitan monooleate (Span 80, Fluka), potassium peroxodisulfate (98%, Merck) and calcium chloride dihydrate (99%, Riedel), acetonitrile (ACN) (99.9%, Sigma–Aldrich), ethanol (99.8% Riedel) were used as supplied.

2.2. Preparation and characterization of polyHIPE monolithic column

Before the preparation of monolithic column, the silanization of the inner wall of a fused silica capillary tubing was carried out by following procedure: first, a capillary tubing 40 cm in length was washed with 0.2 M NaOH for 1.5 h, followed with distilled water for 30 min and methanol for 30 min at room temperature. Thereafter, the capillary was purged with nitrogen for 30 min. Finally 3-(trimethoxysilyl)propyl methacrylate–methanol solution (50/50, v/v) was flushed through the capillary for 20 min. This tubing filled with this solution was sealed by rubber septum and then kept in a water bath at 35 °C for 20 h. Then it was washed with methanol for 10 min and dried by nitrogen for 30 min.

The polyHIPE monolithic material produced was 90% porous based on aqueous phase content. The preparation was based on the procedure as described in [14]. The organic phase of the emulsion comprised isodecyl acrylate (IDA) (80 vol%), the crosslinker DVB (20 vol%), the surfactant Span 80 (20 vol% relative to total monomer volume) and the aqueous phase involved potassium persulfate (0.2 wt%) and $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ (1 wt%). Both phases were purged with nitrogen for 15 min. The HIPE was prepared by mixing organic phase at 320 rpm while the liquid phase was added slowly. After all aqueous phase has been added, the emulsion was stirred for

45 min. Then the HIPE was fed into the pretreated fused silica capillary (total length 40 cm) for 10 min by applying an external pressure of 2.5 bar onto one end of the column. The flow was then stopped and the capillary tube completely filled with HIPE was sealed at both ends by rubber septa and kept in a water bath at 60 °C for 48 h. After polymerization, the monolithic column was connected to a liquid chromatography pump to wash with absolute ethanol (2 h) then with distilled water (1 h) and ACN (1 h). The monolithic column was cut at both ends (i.e. 1.5 cm) and flushed with ethanol before and after the opening the detection window. The detection window was opened at 28 cm in length by burning off the 2–3 mm of coating at the end of polymer bed during the ethanol wash. Hence the separation of analytes was monitored in the polyHIPE column with an effective length of 28 cm and a total length of 37 cm. Prior to the connection of the capillary to the CEC system, the column was equilibrated by ACN/5 mM phosphate buffer (60/40, v/v) at the flow rate of 3 $\mu\text{L}/\text{min}$ overnight.

Scanning electron microscopy (SEM) analysis was performed to characterize the resulting monolith structure by SEM JEOL 6400 Model. For this purpose, surfaces were coated with gold and their microphotographs were obtained. The internal structure micrograph at a magnification of 750 \times was used to calculate average void and interconnecting window diameters. Voids and interconnecting windows are the large spherical cavities and the spherical holes connecting these cavities in the structure, respectively. At least 20 voids and 40 windows were measured on the SEM photograph to calculate the average values for void and interconnecting window diameters.

Pore size distribution was determined by using Quantachrome Corporation Poremaster 60 and samples were outgassed under vacuum before analysis. The intrusion mercury contact angle was 140°. The pressure range was 0.2–55 000 psi. The measurements were performed according to scan mode.

The surface area of the monolith was determined from nitrogen adsorption isotherms applying the Brunauer–Emmet–Teller (BET) model. The measurements were performed using a Quantachrome Autosorb 1 Automated Gas Sorption System.

The mechanical property of the monolith was tested by compression tests using Lloyd Instruments (LRX5K) equipped with 100 N load cell. Cubic samples (10 mm \times 10 mm \times 10 mm) were loaded at a rate of 0.5 mm/min.

2.3. Electrochromatography

Electrochromatographic experiments were performed by using Prince CEC 760 model capillary electrochromatography system equipped with photodiode array detector. To investigate the ability of the column as stationary phase, the column was connected to CEC system and equilibrated with ACN/5 mM phosphate buffer solution (60/40, v/v) at pH 7 by applying a voltage of 10 kV and an external pressure of 2 bar at the inlet of the column for 30 min. Then it was equilibrated further by applying a voltage of 20 kV without any external pressure for 15 min. A mixture of alkylbenzene derivatives including thiourea, benzene, toluene, ethylbenzene, propylbenzene and butylbenzene was used as sample for separation studies. The sample mixture was prepared at a concentration of 1% (v/v) in ACN/buffer solution (80/20, v/v). The detection wavelength was 200 nm. The electrochromatographic separations were carried out by using various volume fractions of ACN in mobile phase and different voltages from 10 to 30 kV.

The electroosmotic mobility, μ_{EOF} , was obtained by the following Eq. (1) where L_t was the total length of the column, L_e the distance from inlet to the detection point, V the applied voltage and t_R the retention time of the unretained analyte, thiourea.

$$\mu_{\text{EOF}} = L_e L_t / V t_R \quad (1)$$

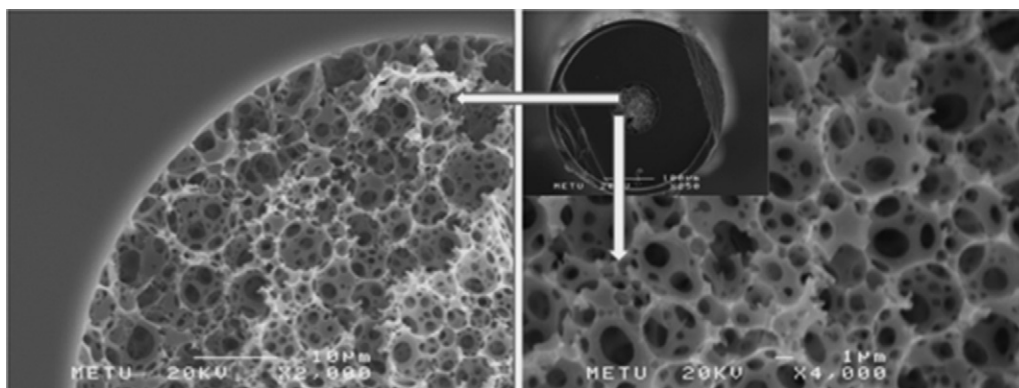


Fig. 1. SEM microphotographs of poly(IDA-co-DVB) polyHIPE monolith synthesized with the KPS concentration of 0.2% (w/w).

The theoretical plate number (N) and the plate height h were calculated by Eqs. (2) and (3) respectively. In Eq. (2), t_r and t_w were the retention time and the peak width at half-height, respectively.

$$N = 5.54(t_r/t_w)^2 \quad (2)$$

$$h = L/N \quad (3)$$

The retention factor (k) was calculated by the following Eq. (4) where t was the retention time of analyte and t_0 was the retention time of thiourea.

$$k = (t - t_0)/t_0 \quad (4)$$

3. Results and discussion

Special polyHIPE structure of poly(IDA-co-DVB) monolith in the capillary was confirmed by SEM analysis of the monolith sample obtained with the KPS concentration of 0.2% (w/w) (Fig. 1A). It was well attached to the capillary wall (Fig. 1B). SEM micrographs showed that the monolith had classic cellular structure of polyHIPEs that was a highly porous material with micron size voids, all interconnected through the windows. Due to the micron size voids and open pore structure of this 90% porous monolith, it provides a significant advantage to provide a higher mass transfer in comparison to conventional macroporous monoliths. The average void diameter and window diameter of the polyHIPE monolith were 8.9 ± 2.7 and 2.0 ± 0.9 μm respectively. The pore size distribution of the monolith determined by mercury intrusion porosimeter is represented in Fig. 2. As seen here, most of the pores were collected in the range of 2–5 μm . The surface area of the polyHIPE monolith determined by the BET was $5.44 \text{ m}^2/\text{g}$. Another advantage of this monolith is its elastomeric behavior. In the compression tests, the monolith demonstrated an elastomeric behavior up to stress of 1 MPa and recovered itself with little or no damage when the

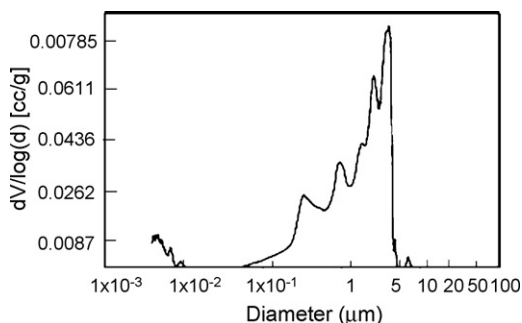


Fig. 2. The pore size distribution of poly(IDA-co-DVB) polyHIPE monolith synthesized with the KPS concentration of 0.2% (w/w).

stress is removed. An elastomeric polyHIPE has the advantage of that when a stress is applied to the monolith; it shows reversible elastic behavior without any permanent damage.

In CEC, the mobile phase and the analytes are driven by electroosmotic flow (EOF). The EOF velocity depends on zeta potential and so the charge density on the stationary phase surface [15]. Generally, an ionizable monomer such as 2-acrylamido-2-methylpropanesulfonic acid (AMPS) is used to generate EOF in the monolithic separation media used in CEC. It should be noted that the poly(isodecylacrylate-co-divinylbenzene), poly(IDA-co-DVB) monolith prepared in polyHIPE form did not require any EOF generating monomer which makes it superior than the other conventional monoliths prepared by using comonomers with strong ionizable groups. The reason is probably that the presence of ionizable sulfate groups generated by the decomposition of water-soluble initiator (i.e. $\text{K}_2\text{S}_2\text{O}_8$) tended to be on the surface of the material which surrounded by the dispersed aqueous phase. These groups should probably provide sufficient surface charge to generate the EOF.

The effect of ACN content in the mobile phase on EOF is shown in Fig. 3 for the polyHIPE monoliths obtained with different KPS concentrations. The electrophoretic mobility at a certain ACN concentration was higher for the monolith synthesized with higher KPS feed concentration. This result should be explained by the higher surface concentration of ionizable sulfate groups providing higher surface charge for the monolith synthesized with higher KPS concentration. On the other hand, the variation of EOF with the ACN concentration was similar for all monoliths. For each monolith, EOF mobility slightly decreased with the increasing ACN concentration particularly at the ACN concentrations higher than 65% (v/v). Similar behavior found in the previous studies [16,17] was explained by Schwer and Kenndler [18] with the decrease in zeta poten-

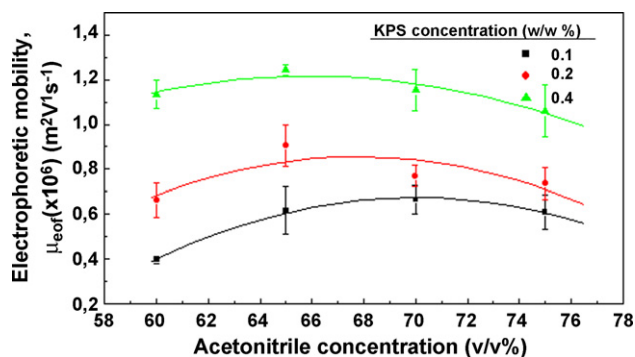


Fig. 3. The variation of electroosmotic mobility with ACN concentration in the mobile phase for the monoliths synthesized with different KPS concentrations, pH 7. The unretained marker: thiourea.

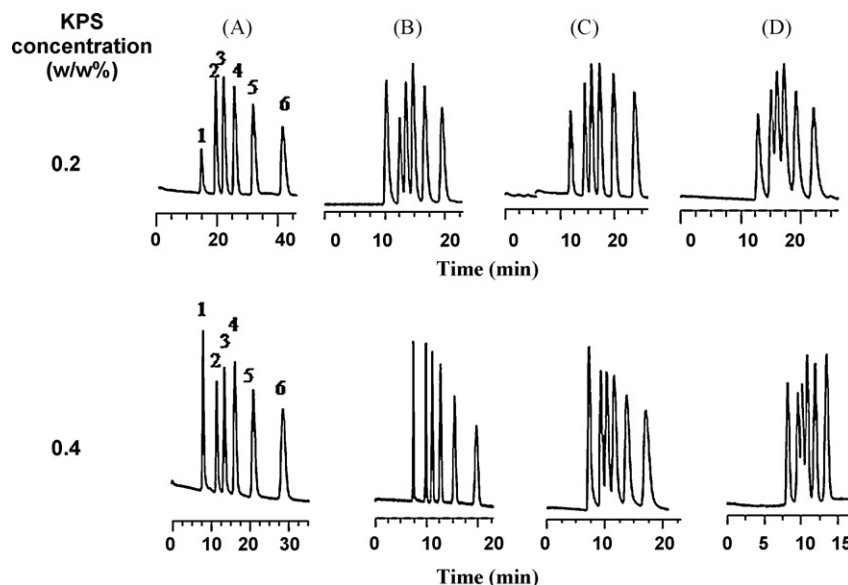


Fig. 4. The effect of ACN/buffer ratio on the electrochromatographic separation of alkylbenzenes by poly(IDA-co-DVB) monolithic columns produced with the KPS concentrations of 0.2% and 0.4% (w/w). ACN/buffer ratio (v/v): (A) 60/40, (B) 65/35, (C) 70/30, and (D) 75/25, mobile phase: ACN/5 mM phosphate buffer at pH 7, 22 °C, id: 100 μ m, effective column length: 28 cm, injection: 5 kV, 3 s; 200 nm. Order of peaks: (1): thiourea, (2) benzene, (3) toluene, (4) ethylbenzene, (5) propylbenzene, and (6) butylbenzene.

tial by addition of organic solvent. However, relatively lower EOF mobilities obtained at the ACN concentration of 60% (v/v) could not be explained. The influence of pH on the electrophoretic mobility was also studied and no appreciable change was observed in the pH range of 5–10 (not shown here). This result indicates that the monolith provides sufficient EOF over a wide pH range.

The chromatographic performance of the poly(IDA-co-DVB) monolith was investigated by the separation of selected alkylbenzenes in CEC. It is clearly known that separation behavior of the analytes are highly dependent on the mobile phase composition [19,20]. Therefore, first, the effect of ACN/buffer ratio on the electrochromatographic separation was evaluated and the representative electrochromatograms obtained by using ACN percentage in the range of 60–75% (v/v) are given in Fig. 4 for the polyHIPE monoliths synthesized with the KPS concentrations of 0.2% and 0.4% (w/w). Note that, the total analysis time suitable for practical use could not be obtained with the monolith synthesized with the KPS concentration of 0.1% (w/w). In most cases, it was higher than 1 h probably due to the lower EOF mobility as also shown in Fig. 3. In Fig. 3, it was seen that as the ACN concentration in the mobile phase was increased, the retention of the analytes decreased. 60% (v/v) ACN in the mobile phase provided a good separation, but it also caused a long analysis time for both columns. Note that the ACN/buffer ratio at which the best separation obtained was slightly different for both columns. The column synthesized with the KPS concentration of 0.2% (w/w) gave the optimum separation at 70% (v/v) of ACN concentration while the optimum separation was obtained at 65% (v/v) of ACN concentration with the column synthesized by the KPS concentration of 0.4% (w/w). The peak resolutions of electrochromatographic separation of the alkylbenzenes at different ACN/buffer ratios are indicated in Table 1 for both columns. As shown here, the resolution of the analytes were higher than 1.5 when the ACN percentage in the mobile phase was 60% and 70% (v/v). However further increase in ACN percentage caused decrease in the resolution of some analytes, especially for benzene and toluene. The highest resolutions were obtained at the ACN concentrations of 70% and 65% (v/v) for the columns synthesized by the KPS concentrations of 0.2% and 0.4% (w/w), respectively. The total analysis times were approximately the same in both cases. The higher surface charge of the column produced with the KPS

concentration of 0.4% (w/w) should be probably responsible for this behavior.

This trend can be seen also in Fig. 5 which shows the linear dependence of retention factor with increasing ACN concentration. This behavior is typical for the separation of alkylbenzenes in the reverse phase systems [16–21].

In CEC systems, the conductivity of mobile phase can be increased by increasing the applied voltage [22]. Thus, the retention of analytes can be adjusted by changing the applied voltage to improve separation performance. To investigate the effect of applied voltage on the electrochromatographic separation, the electrochromatograms of alkylbenzenes were obtained by using the voltages in the range of 10–30 kV at the ACN/buffer ratio of 70/30 (v/v). As it is seen from Fig. 6, the column afforded the separation of the analytes within 15 min without any significant change in separation power at the voltage of 25 kV. However, when 30 kV was applied, a decrease in the resolution was observed in the electrochromatogram (Fig. 6E). Table 2 shows the effect of applied voltage on the peak resolutions at the ACN/buffer ratio of 70/30 (v/v). From these data, it is seen that the resolutions decrease with increasing voltage for all analytes.

On the other hand no significant change was observed in retention factor with the applied voltage in the range of 10–30 kV at 70/30 ACN/buffer ratio, in the column obtained with the KPS con-

Table 1

The effect of ACN/buffer ratio on the peak resolution for the separation of benzene derivatives in the polyHIPE monoliths synthesized with the KPS concentrations of 0.2% and 0.4% (w/w).

ACN/buffer (v/v)	R(2/1)	R(3/2)	R(4/3)	R(5/4)	R(6/5)
KPS concentration: 0.2% (w/w)					
60/40	3.40	1.51	1.83	2.64	3.76
65/35	2.75	1.49	1.40	2.14	2.57
70/30	4.20	1.64	1.80	2.64	3.16
75/25	2.35	0.94	1.16	1.62	2.17
KPS concentration: 0.4% (w/w)					
60/40	5.64	1.55	3.43	3.92	4.61
65/35	6.28	2.92	3.43	3.92	4.62
70/30	2.48	1.06	1.21	1.96	2.41
75/25	2.35	0.80	0.88	1.47	2.10

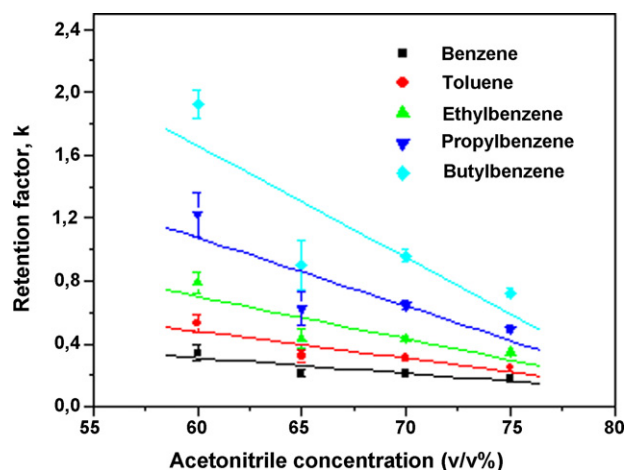


Fig. 5. The variation of retention factor with ACN concentration in the mobile phase for the column synthesized with the KPS concentration of 0.2% (w/w), mobile phase: ACN/5 mM phosphate buffer at pH 7, 22 °C, id: 100 μ m, effective column length: 28 cm, injection: 5 kV, 3 s; 200 nm.

Table 2

The effect of applied voltage on peak resolution for the separation of benzene derivatives in the polyHIPE monolith produced with the KPS concentration of 0.2% (w/w).

Voltage (kV)	R(2/1)	R(3/2)	R(4/3)	R(5/4)	R(6/5)
10	6.86	2.93	3.23	4.90	5.54
15	6.10	2.90	2.86	4.56	5.42
20	4.20	1.64	1.80	2.64	3.16
25	3.60	1.50	1.53	2.35	3.05
30	3.15	1.33	1.42	2.12	2.65

centration of 0.2% (w/w) (not shown here). The behavior was similar for all alkylbenzene derivatives except that lower retention factors were obtained with the increasing polarity of the analytes. As we expected the retention factor of the neutral analytes did not vary with the EOF.

The efficiency of the column was evaluated by theoretical plate number (TPN) and plate height values. The effect of linear velocity on the theoretical plate number and plate height is exemplified in Fig. 7 for the poly(IDA-co-DVB) column synthesized with the KPS concentration of 0.2% (w/w). The TPNs up to 200 000 and the plate heights with a minimum of 5 μ m were obtained by using ACN/buffer ratio of 70/30 (v/v). From Van-Deemter plot, the ideal value of the linear flow velocity was determined as 1.4 cm/min.

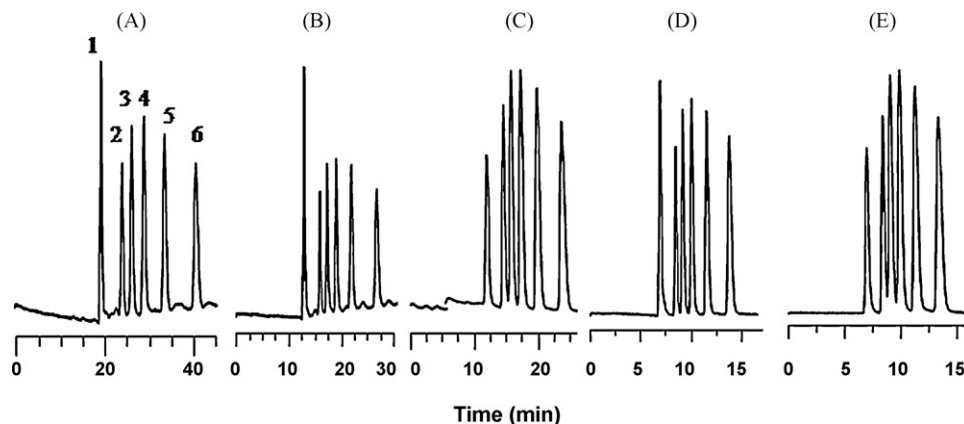


Fig. 6. The effect of applied voltage on the electrochromatographic separation of alkylbenzenes by poly(IDA-co-DVB) monolithic column synthesized with the KPS concentration of 0.2% (w/w). Applied voltage (kV): (A) 10, (B) 15, (C) 20, (D) 25, and (E) 30. ACN/phosphate buffer (pH 7, 5 mM) ratio: 70/30 (v/v), 22 °C, id: 100 μ m, effective column length: 28 cm, injection: 5 kV, 3 s; 200 nm. Order of peaks: (1) thiourea, (2) benzene, (3) toluene, (4) ethylbenzene, (5) propylbenzene, and (6) butylbenzene.

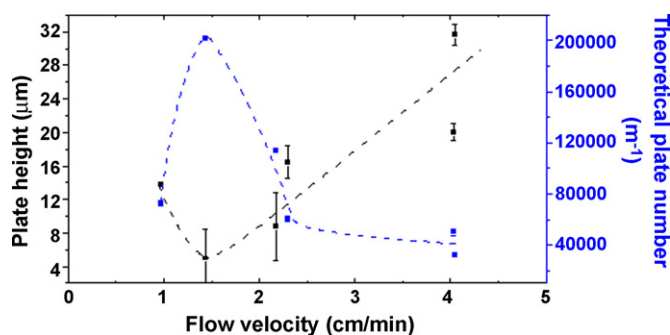


Fig. 7. The effect of linear velocity on the theoretical plate number and plate height of poly(IDA-co-DVB) column synthesized with the KPS concentration of 0.2% (w/w), ACN/5 mM Buffer: 70/30, analyte: thiourea, 22 °C, column id: 100 μ m, effective column length: 28 cm, injection: 5 kV, 3 s; 200 nm. Three measurements were performed for each data point. The error bars of TPN values are small and some of them could not be clearly shown in the figure.

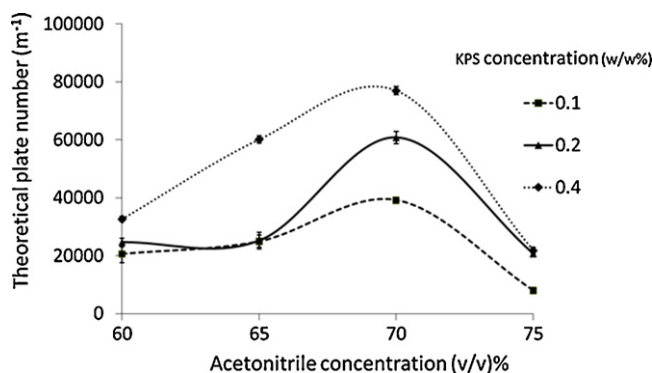


Fig. 8. The effect of ACN concentration on the theoretical plate number of poly(IDA-co-DVB) columns synthesized with different KPS concentrations, applied voltage: 20 kV, analyte: thiourea, 22 °C, column id: 100 μ m, effective column length: 28 cm, injection: 5 kV, 3 s; 200 nm. Three measurements were performed for each data point. The error bars of TPN values are small and some of them could not be clearly shown on the figure.

The theoretical plate numbers ranging between 170 000 and 210 000 plates/m were determined for poly(butyl methacrylate-co-ethylene dimethacrylate) monoliths with sulfonic acid functionalities [23,24]. The column efficiencies approximately 300 000 plates/m were found for hexyl methacrylate based monoliths [25,26]. The column efficiencies obtained with our polyHIPE monoliths are in the same order of magnitude with the acrylate

Table 3

The RSD values showing run-to-run reproducibility for polyHIPE monoliths produced with different KPS concentrations.

KPS concentration (w/w%)	Thiourea	Benzene	Toluene	Ethylbenzene	Propylbenzene	Butylbenzene
RSD values (%)						
0.1	10.40	10.58	11.17	11.31	11.63	11.80
0.2	4.68	4.62	4.57	4.43	4.35	4.01
0.4	2.25	1.57	1.72	1.75	2.08	2.12

based monolithic stationary phases with sulfonic acid groups in the literature.

The variation of theoretical plate number with the ACN concentration is given in Fig. 8 for the polyHIPE monoliths prepared with different KPS concentrations. As seen here, all monoliths gave the same behavior and exhibited a maximum at 70% ACN concentration. The highest TPN values were obtained for the polyHIPE synthesized with the highest KPS concentration (i.e. 0.4% w/w) having the highest electrophoretic mobility. For this column, the highest TPN and the lowest plate height were determined as $80\,000\text{ m}^{-1}$ and $12.5\ \mu\text{m}$, respectively.

As also seen in Fig. 5, the difference between the retention factors of alkylbenzene derivatives at a certain ACN concentration decreased with the increasing ACN concentration. The retention factors in a wider range with the longer analysis times were obtained for the ACN concentrations of 60% and 65% (v/v). On the other hand, the retention factors of alkylbenzene derivatives were so close at 75% (v/v) of ACN concentration. 70% (v/v) of ACN concentration allowed the good separation of alkylbenzene derivatives with satisfactorily different retention factors. This ACN concentration also gave the highest TPN value for the same polyHIPE monolith in Fig. 8.

To determine run-to-run precision in retention time, the analyte mixture containing each component at a concentration of 1% (w/w) was injected into the each column 5 times. The relative standard deviation (RSD) values of run-to-run reproducibilities obtained in CEC is given in Table 3. As seen here, RSD values were lower with the polyHIPE monolith synthesized by higher KPS concentration. Particularly RSD values around 2% obtained with the monolith synthesized by the KPS concentration of 0.4% (w/w) are acceptable. This result indicated that higher surface charge resulted in better stabilization of polyHIPE monolith in the electrochromatographic separation.

In our study, polyimide coated fused silica capillary tubings were used in all chromatographic separations. A study by Baeuml and Welsch about long term stability of fused silica capillaries showed that ACN as an inevitable organic modifier and was also used in our study caused a visible swelling even after 24 h storage [27]. Hence the lifetime of our column is mainly determined by the tubing life time. Therefore we did not consider it necessary to specify a long term stability data. On the other hand, the column was used up to 24 h of total analysis time without any change in its performance.

4. Conclusion

A polyHIPE monolithic column which had 90% porosity and a well-defined structure with interconnected micron size spherical

voids was successfully prepared within a capillary and it was evaluated as stationary phase for CEC. The polyHIPE monolith showed successful chromatographic performance in the separation of alkylbenzenes without the use of EOF generating monomer.

In summary, it is obvious that there is a lack of commercial monolithic columns in the market particularly due to the difficult preparation processes of monolithic silica columns. Emulsion-templating is a flexible, easy controlled method for the preparation of capillary column. Therefore prepared polyHIPE monolith can be a good alternative for controlling the fabrication process of the monolithic columns for CEC.

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