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Determination of the molecular mass distribution of synthetic polymers by size-exclusion electrochromatography

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Abstract

The performance of size-exclusion electrochromatography (SEEC) for the mass distribution analysis of synthetic polymers was studied and compared to conventional, pressure-driven size-exclusion chromatography (SEC). Electroosmotic flow control, within-day, day-to-day and column-to-column repeatability were determined for SEEC with respect to retention and separation efficiency. It was shown that by using the retention ratio instead of the migration time, the precision of the mass distribution calculations is sufficiently high, and that similar distributions were obtained for a sample analyzed by pressure-driven SEC and by SEEC. Furthermore, hexafluoroisopropanol was demonstrated to be a new and potent solvent for SEEC. It was used for the separation of narrow polymethylmethacrylate standards and several commercially important polymers such as polycarbonate, polycaprolactam and poly(ethylene terephthalate), using UV detection in the deep UV region (195–230 nm).

Keywords: Electrochromatography; Size-exclusion electrochromatography; Molecular mass; Hexafluoroisopropanol; Polymethylmethacrylate; Polycarbonate; Polycaprolactam; Poly(ethylene terephthalate); Polystyrene

1. Introduction

Capillary electrochromatography (CEC) is a separation technique which currently enjoys attention throughout the analytical science community. The attractive features of CEC, the fertile combination of the high separation efficiency of electrokinetic techniques with the high selectivity offered by various forms of liquid chromatography, have been extensively demonstrated in practice. In CEC an electric field is applied over a (packed) capillary column generating an electroosmotic flow (EOF) which carries the solvent and the solutes through the column. Limitations for particle dimensions with respect to flow development are less restrictive in electrochromatography than in HPLC. Highly efficient separations have been shown on columns packed with particles of 1.5-μm diameter and smaller [1,2]. Moreover, since the EOF velocity is virtually independent of the flow channel width, a homogeneous flow velocity profile over the column cross section is obtained, resulting in a reduced peak broadening compared to pressure-driven chromatography [3–5]. The occurrence of a significant liquid flow through the pores of stationary phase particles may further improve the separation efficiency [6–11]. A general advantage of capillary separation

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methods such as CEC is the low amount of stationary phase material required and low solvent consumption. This may facilitate the use of special stationary phases for, e.g. chiral separations [12] or the use of expensive or toxic solvents.

Different selectivity modes have been applied in CEC, such as reversed-phase [3–4,10–11], normal-phase [13] and (dynamic) ion-exchange [14,15] systems.

Recently, size-exclusion electrochromatography (SEEC) has been introduced [6–10]. It was shown that SEEC can offer high-speed and high-efficiency molecular-size separations of (synthetic) polymers, with minimal consumption of organic solvent. So far, SEEC has been studied only in the separation of well defined, narrow polystyrene (PS) standards using *N,N*-dimethylformamide (DMF) as the mobile phase. Since PS is UV active and DMF has a relatively high dielectric constant, this combination was a logical first selection to explore the potential of SEEC for polymer separations. However, before SEEC can be regarded as a valuable alternative for classical size-exclusion chromatography (SEC), its potential and performance in practical applications has to be studied further.

In this paper results are presented of a study on the practical applicability of SEEC for the characterization of various synthetic polymers. The quality of flow control, injection repeatability and column-to-column repeatability have been assessed. Molecular mass distributions of real-life polymer samples as obtained by SEEC have been compared with SEC results. Special attention has been given to the application of 1,1,1,3,3,3-hexafluoroiospropanol (HFIP) as solvent. HFIP is a potent solvent for a variety of polymer types that are insoluble in common organic solvents. However, the toxicity and excessively high price of HFIP are serious drawbacks for its application as a solvent in conventional SEC and the low solvent consumption of SEEC may be a real advantage here.

2. Experimental

2.1. Chemicals

DMF, non-stabilised tetrahydrofuran (THF), methanol and toluene were obtained from Acros (Geel, Belgium). HFIP, tetra-*n*-butylammoniumtetrafluoroborate (TBATFB) and lithium chloride came from Merck (Darmstadt, Germany). The narrow polystyrene standards were obtained from Merck, Polymer Labs. (Church Stretton, UK) and Polyscience (Warrington, PA, USA). The poly(methylmethacrylate) (PMMA) standards were obtained from Polymer Labs. All standards had a polydispersity <1.2, as stated by the suppliers. Two broad PS samples with different mass (PS 1 and PS 2) were prepared in the laboratory by free radical polymerization in toluene as described elsewhere [16]. The two polycarbonate (polybisphenol-A) samples with different molecular mass (PC 1 and PC 2) where a kind gift from Dr. E. Venema (General Electric Plastics, Bergen op Zoom, Netherlands). The poly(ethyleneterephthalate) (PET) sample was scraped from a plastic bottle and the poly(caprolactam) sample was bought from Polyscience. Of each of the polymer samples a stock solution was prepared in the appropriate solvent at a concentration of 10 mg ml^{-1}. A stock solution of toluene in DMF was prepared at a concentration of 100 $\mu l\,ml^{-1}$. Samples were prepared by mixing appropriate volumes of the stock solutions and pure solvent to obtain concentrations of approximately 1.0 mg ml^{-1}. Polycarbonate was injected at a concentration of 10 mg ml^{-1} in DMF and at 1.0 mg ml^{-1} in HFIP. Either toluene (in DMF) or acetone (in HFIP) served as the totally permeating markers, added to all samples at a concentration of 10 $\mu l\,ml^{-1}$.

For the SEEC experiments, the mobile phase consisted either of DMF to which LiCl was added at a concentration of 0.1 m*M*, or HFIP to which TBATFB at a concentration of 1.0 m*M* was added.

Unmodified silica particles with a diameter of 5 μm and a nominal pore size of 30 nm (Nucleosil 5-300; Macherey–Nagel, Düren, Germany) were used as the stationary phase for separations in DMF. With HFIP as the mobile phase, 10-μm sulfonic acid modified particles (Nucleosil SA-10, nominal pore size 10 nm Macherey–Nagel) were used.

2.2. Instrumentation for size-exclusion electrochromatography

All electrokinetic experiments were performed on a $HP^{3D}CE$ system (Hewlett-Packard, Waldbronn, Germany). During separation a pressure of 10 bar

was applied at both ends of the column, which was thermostatted at 20°C. UV detection was performed at 260 and 270 nm (DMF) or at 195 and 220 nm (HFIP).

Injections were performed electrokinetically through the application of 5 kV for 10 s unless specified otherwise. Experiments were performed in duplicate unless otherwise stated and the mean values were used for further calculations. Migration times and plate numbers were calculated using the CHEMSTATION software (Hewlett-Packard).

2.3. Instrumentation for conventional size-exclusion chromatography

Two different instrumental set-ups were used for conventional pressure-driven SEC (PD-SEC). With both instruments non-stabilized THF was used as the mobile phase.

The first system (SEC 1) consisted of a Spectroflow 400 solvent delivery system (ABI, Ramsey, NJ, USA) operated in the constant flow mode at 1.0 ml min^{-1} connected to a Rheodyne type 7010 injector (Rheodyne, Berkeley, CA, USA) equipped with a 20-μl sample loop. The column was a single 600× 7.5 mm PL-GEL 5 μm Mixed-C column from Polymer Labs. Detection was performed using a Spectroflow 757 variable-wavelength UV detector (ABI) operated at 254 nm (polystyrenes) or 265 nm (polycarbonate). The detector signal was recorded on a flat-bed recorder (BD-41, Kipp en Zonen, Delft, Netherlands) and was simultaneously digitised using a Smartlink model KNM-DCV 12-RS232-C D/A converter (Keithley Instruments, Cleveland, OH, USA). Data analysis was performed using custom-made software. The second system (SEC 2) consisted of a Waters Alliance 2690 separations module (Milford, MA, USA) operated at a flow-rate of 0.35 ml min^{-1} connected to a Waters 410 differential refractometer as the detector. With this system a Waters Styragel HR-4E and a Styragel HR-S column were used in series. Both columns were 30 cm×7.8 mm I.D. and were thermostatted at 35°C.

2.4. Column preparation for size-exclusion electrochromatography

The preparation of the capillary columns for SEEC has been described in detail elsewhere [8]. Briefly, at one end of a 40-cm length of fused-silica tubing (100 μm I.D.×375 μm O.D., Polymicro Technologies, Phoenix, AZ, USA) a temporary frit was prepared through heating of a 1–2 mm plug of bare silica particles (Nucleosil 100-5) with a small gas flame. A slurry containing 10 mg ml^{-1} of the respective packing material in methanol was prepared and placed in the slurry chamber, which was a 20 cm length of stainless steel tubing (I.D. of 1/16 in.; 1 in.=2.54 cm) The capillary was connected to the slurry chamber and high pressure was used to drive the particles into the column.

High pressure was delivered by a high-pressure membrane pump operated at a maximal pressure of 500 bar. After 1 h, the pressure was relieved from the column, which was subsequently flushed with water at a pressure of 150 bar for 1 h. Next, permanent frits were prepared at a distance of approximately 25 cm from each other, by heating the packed section using an electrically resistively heated metal strip device. Then the pressure was relieved carefully from the column, which was then reversely connected to the pump in order to remove the excess of particles. Next a 3-mm wide detection window was prepared adjacent to the outlet frit, by burning off the protective coating. When DMF was used as the mobile phase the column was connected to a standard HPLC pump (Spectroflow 400, ABI) equipped with a laboratory-made flow splitter and flushed with the mobile phase at a constant pressure of ~50 bar. The column was then cut to the desired length and installed into the SEEC instrument.

When HFIP was used as the mobile phase a pressure of 10 bar was applied onto the column inlet for 60 min to flush the column with the mobile phase. The columns were electrokinetically conditioned by the application of a ramped voltage gradient of up to 15 kV across the column over 30 min.

3. Results and discussion

3.1. Repeatability studies

Many factors affect the magnitude and direction of the EOF: the sign and density of the charge on the surface of the silica particles, the ionic strength of the mobile phase and the dielectric constant of the

Table 1
Run-to-run repeatability ($n=20$) of the migration time, the retention ratio and the separation efficiency of SEEC; mobile phase: 0.1 m*M* LiCl in DMF; stationary phase: 5 μm Nucleosil 300; UV detection at 260 nm

Standard		Migration time		Retention ratio		Plate height	
Compound	M_r	Average (min)	RSD (%)	Average	RSD (%)	Average (μm)	RSD (%)
PS	675 000	5.13	0.9	0.671	0.5	40	6.1
PS	43 900	5.76	1.3	0.754	0.1	30	7.9
PS	2100	7.22	1.3	0.946	0.1	25	2.0
Toluene		7.64	1.3	N/A	N/A	8.3	1.3

solvent [5]. The general experience in CEC is that it is often difficult to maintain a constant EOF over a large number of experiments.

To test the EOF repeatability for the SEEC system, a sample containing three different PS standards and toluene was injected repeatedly. The mobile phase consisted of DMF to which 0.1 m*M* LiCl was added and a separation voltage of 15 kV was applied. Soon it was found that a single buffer vial could not be used repeatedly when precise flow control is desired. The observed drift of migration times can be explained by the lack of buffering capacity of the mobile phase. During electrophoresis a change of the pH of the solution in the inlet vial will be induced by the electrochemical processes at the electrode. Therefore, in further experiments the mobile phase was refreshed after each single experiment.

With a fresh mobile phase solution in the inlet vial for each run, a large number ($n=20$) of repeated runs was performed. Both the retention times of the injected components were recorded as well as the plate heights of the separated components. The data obtained are summarized in Table 1. The variance in the migration time is relatively low at approximately 1%. Still, for mass calibration of SEEC columns using only the migration times of standards such a precision is too low. When the migration times were corrected for the migration time of the completely permeating marker (toluene), the spread in the results was strongly reduced. The imprecision of the retention ratio was reduced to approximately 0.1%, which generally suffices for the determination of polymer mass distributions.

The run-to-run repeatability of the separation efficiency was worse than for the migration times, with RDS values up to ~8.0% for the plate heights. Still, with reduced plate heights in the order of 5–8 for the PS standards the efficiency of the SEEC system was completely satisfactory.

Next, the day-to-day repeatability of the migration times and the separation efficiency was tested by injection of a sample mixture onto the same column five times repeatedly on 6 different days (Table 2). Again a relatively large variance in the migration time is observed and a substantial improvement of the repeatability can be obtained by using the retention ratio instead. The day-to-day repeatability of the separation efficiency is not worse than the within-day repeatability.

The column-to-column repeatability, which is often considered to be the most problematic factor to

Table 2
Day-to-day repeatability (5 consecutive runs on 6 different days) of the migration time, the retention ratio and the separation efficiency of SEEC; mobile phase: 0.1 m*M* LiCl in DMF; stationary phase: 5 μm Nucleosil 300; UV detection at 260 nm

Standard		Migration time		Retention ratio		Plate height	
Compound	M_r	Average (min)	RSD (%)	Average	RSD (%)	Average (μm)	RSD (%)
PS	675 000	5.34	21.1	0.696	0.3	34	39
PS	43 900	5.96	2.1	0.777	0.1	36	2.8
PS	7600	6.78	2.0	0.884	0.1	38	0.7
PS	2100	7.26	1.9	0.946	0.1	24	0.7
Toluene		7.67	2.0	N/A	N/A	8.2	2.0

control in electrochromatography, was also tested. This was done by injecting the same sample mixture six times repeatedly on six different columns on different days (Table 3). The variance in retention times from column-to-column is in the order of 10%. For the retention ratio it is seen that the column-to-column repeatability is in the order of only 2%. The largest variation was observed for the separation efficiency which was in excess of 25%. However, when a closer look was taken at the rough data it was found that one of the columns (column 3) was performing less efficiently than the others. When the data for this column where left out, the divergence in the separation efficiency decreased significantly (Table 3). Surprisingly, the deviating column showed no abnormalities with respect to the migration times or the retention ratios. Apparently, the most problematic factor in column preparation for SEEC is not to make columns with the same retention properties, but with the same high separation efficiency and both factors need to be tested for in each column before it is used.

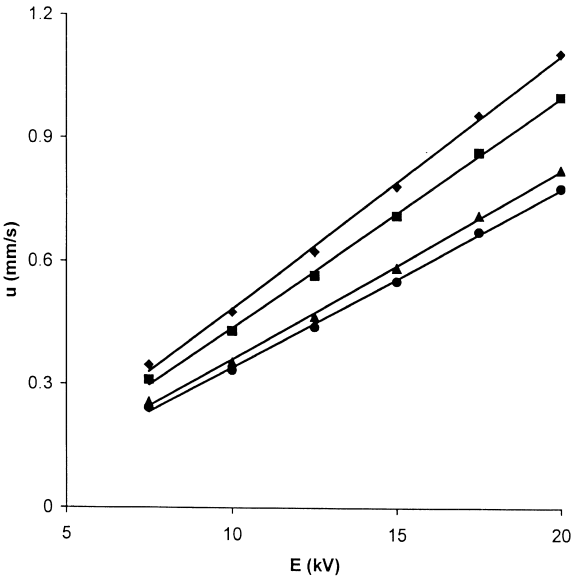

Fig. 1. Migration velocity of several PS standards and toluene as a function of the separation voltage. Solutes: ■, PS 160 000; ♦, PS 39 500; ▲, PS 2100 and ●, toluene. Mobile phase: 0.1 m*M* LiCl in DMF; stationary phase: 5 μm Nucleosil-300.

3.2. Separation voltage and injection procedure

The EOF and the migration velocity of the standards were tested for linearity with respect to the applied electric field strength (Fig. 1). The mobile phase was 0.1 m*M* LiCl in DMF and was refreshed after each single run. The applied electric field strength was varied between 7.5 and 20 kV.

The migration velocity of the components is linear with the applied field strength. With the low ionic strength mobile phase used, Joule heating does not affect the separation even when high electric field strengths are applied. The high mobile phase velocities that can obtained even at relatively low electric field strengths allows the potential use of longer columns. This in return would lead to the generation of higher plate counts, providing the ability to determine the mass distribution of a polymer sample more accurately. Also columns packed with particles with different pore size may be used, the so-called linear columns, extending the mass range of the particular column.

Next, plate height curves were plotted for the different PS standards and toluene (Fig. 2). The plate height of PS is only marginally dependent on the mobile phase velocity, which is due to the enhanced

Table 3
Column-to-column repeatability ($n=6$) of the migration time, the retention ratio and the separation efficiency of SEEC; mobile phase: 0.1 m*M* LiCl in DMF; stationary phase: 5 μm Nucleosil 300; UV detection at 260 nm

Standard		Migration time		Retention ratio		Plate height[a]	
Compound	M_r	Average (min)	RSD (%)	Average	RSD (%)	Average (μm)	RSD (%)
PS	97 200	5.44	9.0	0.706	2.6	30	21
PS	39 500	5.98	8.6	0.777	1.9	36	15
PS	2100	7.29	7.8	0.947	0.4	24	12
Toluene		7.70	7.6	N/A	N/A	8.3	7.5

[a] Data without column 3.

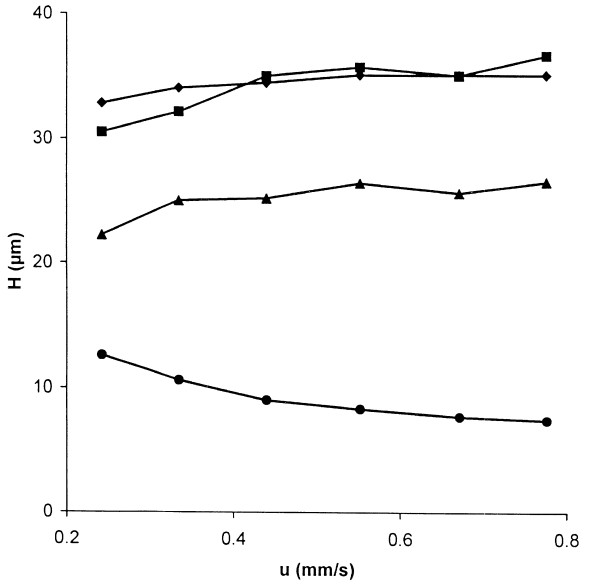

Fig. 2. Plate height curves for several of the PS standards and toluene. Solutes: ■, PS 160 000; ♦, PS 39 500; ▲, PS 2100 and ●, toluene. Mobile phase: 0.1 m*M* LiCl in DMF. Stationary phase: 5 μm Nucleosil-300.

separation efficiency as a result of faster mass transfer kinetics [9–11].

Since both selectivity and separation efficiency do not diverge with the applied electric field strength, SEEC may be performed relatively quickly compared to PD-SEC, where the application of higher mobile phase velocities strongly affects the separation efficiency and separations are commonly performed using low mobile phase velocities resulting in extended separation times.

For quantitative analysis, injection procedures should be very precise and no sample discrimination should occur. Also overloading of the column should be prevented. With electrokinetic injection, which is commonly employed in electrochromatography, the injected sample amount can be varied by changing the electric field strength and the injection time. Both these factors where tested for linearity with respect to peak height and peak area. Fig. 3 shows that all tested factors are linear with the injection voltage and duration up to at least 7 kV for 10 s (70 kV s). The estimated injection plug length for such a large injection time is approximately 0.5 cm, corresponding to an injection volume of approximately 7.5 nl. The injection plug length is 2.0% of the column length. However, the separation efficiency is still not affected by such a prolonged injection.

3.3. Determination of molecular mass distributions

Using the preferred specified experimental conditions and a 0.1 m*M* LiCl in DMF as the mobile phase, a mass calibration curve for PS was constructed for SEEC. Mixtures of narrow PS standards were injected three times onto the column and the averaged retention ratios were used (Fig. 4). A third order polynomial function was fitted through the data points that was used for further calculations. Next, the broad PS and PC samples were injected. For calculation of the mass distribution of the PS samples the UV trace at 260 nm was taken, while for the PC samples the UV trace at 270 nm was used. For both polymers it holds that the UV signal is directly proportional to the number concentration of monomers of the respective polymer, thereby allowing simple calculation of the concentration of the PS at each time interval.

In Fig. 5 the calculated mass distributions of the broad synthetic polymers shown. Parameters characterizing these distributions, such as the M_{top}, M_n and M_w, which correspond to the most abundant molecular mass, the number-averaged molecular mass and the mass-averaged molecular mass, respectively, and the polydispersity, *P*, were calculated from the chromatograms. Similar calculations were performed using data obtained by PD-SEC on either one or both available SEC instruments to allow comparison with the SEEC results (Table 4). For all samples approximately the same numbers are found for the mass distribution parameters obtained by SEC or electrochromatography. The differences observed between the SEEC and the SEC results were of the same order of magnitude as the differences found when the two SEC instruments were compared. Equivalent differences in mass distribution parameters were found when in another study different PD-SEC instruments and columns were compared [17].

3.4. Experiences with HFIP as a mobile phase for SEEC

One of the primary advantages of SEEC is that the

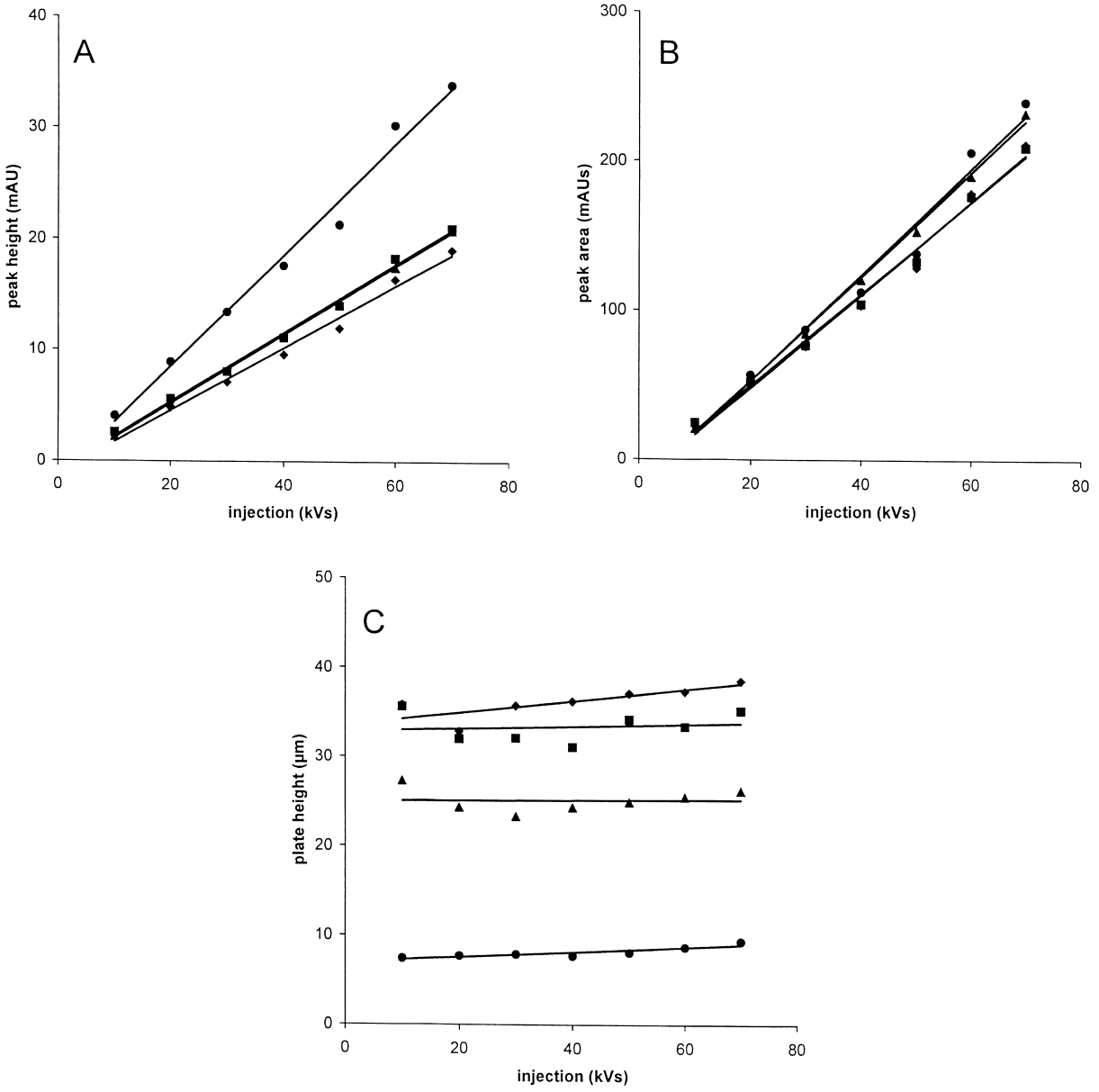

Fig. 3. Effect of injection conditions (time and voltage) on the peak height (A), peak area (B) and the separation efficiency (C) of some polystyrene standards. Solutes: ■, PS 97 200; ♦, PS 39 500; ▲, PS 2100 and ●, toluene. Mobile phase: 0.1 m*M* LiCl in DMF. Stationary phase: 5 μm Nucleosil-300.

capillary dimensions of the column provides a significant reduction of the solvent consumption compared to conventional SEC. For solvents such as HFIP, which is a toxic and expensive, but common solvent for a variety of synthetic polymers, this may be a real advantage. HFIP may be a good solvent for SEEC, since it has very good UV transmission properties (UV cut-of <190 nm) and it has a relatively high dielectric constant ($\varepsilon_r = 20$).

First several salts were tested for solubility in

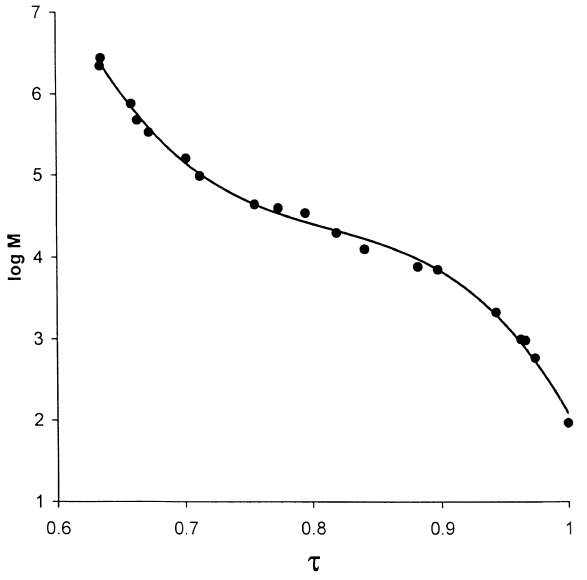

Fig. 4. Mass calibration curve for polystyrene in SEEC. Mobile phase: 0.1 m*M* LiCl in DMF; stationary phase: 5 μm Nucleosil-300; separation voltage: 20 kV.

HFIP. The linearity between current conduction in capillary electrophoresis and the concentration of TBATFB demonstrated that this salt dissociates in HFIP. In all further experiments TBATFB was added to HFIP at a concentration of 1.0 m*M*.

It was found that the EOF in open capillaries and in columns packed with bare silica particles was too low to be used for separation by electrochromatography, when HFIP containing 1.0 m*M* TBATFB was used as the mobile phase. This may be due to the acidity of the solvent itself, or to the presence of relatively high concentrations of impurities such as hydrofluoric acid, which prevents silanolic acid groups from dissociating and developing charge on the surface. When sulphonic acid modified (strong cation-exchange) particles, which are more easily charged at low pH, were used as the stationary phase, a relatively high EOF was obtained. The EOF velocity measured at 20 kV was 0.38 mm/s, which is still 3–4 slower than the EOF mobility found for the same particles with DMF as the mobile phase under otherwise identical conditions. However, it proved to be much more difficult to generate a stable EOF, even when the mobile phase was refreshed after each run or when the retention ratio was used instead of

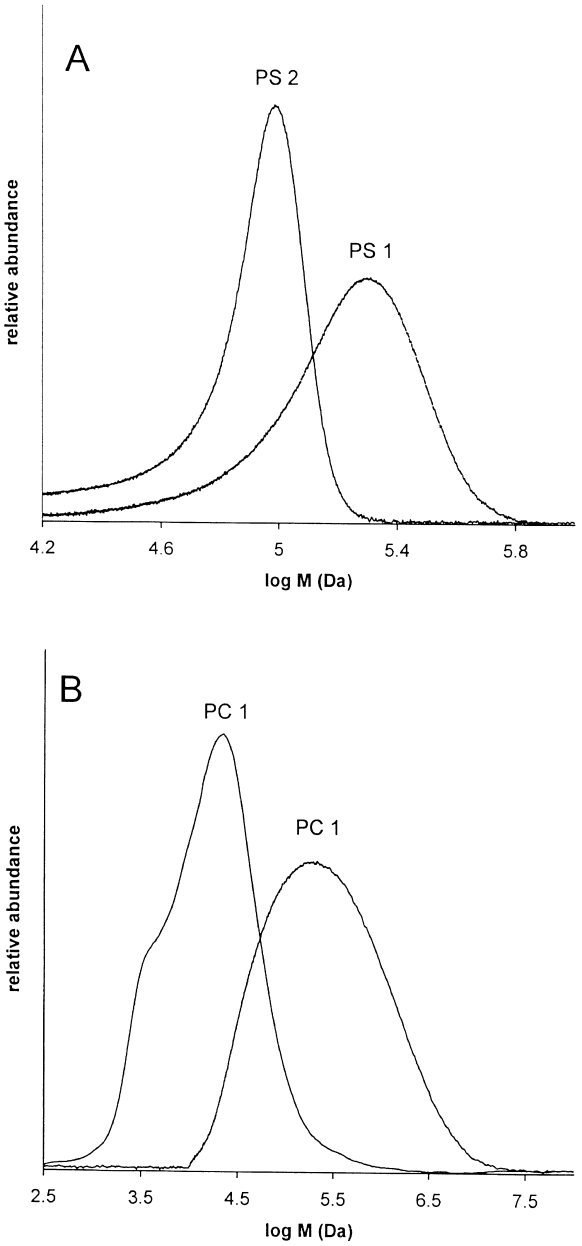

Fig. 5. Mass distributions of the two broad polystyrene samples (A) and the polycarbonate samples (B) as determined by SEEC. Mobile phase: 0.1 m*M* LiCl in DMF; stationary phase: 5 μm Nucleosil-300; applied electric field: 20 kV; samples: 5.0 mg ml^{-1} in DMF; separation voltage: 20 kV; UV detection at 260 nm (A) and 270 nm (B).

Table 4
Mass distribution parameters calculated for the broad synthetic polymers as obtained by the different techniques

Technique	M_{top}	M_n	M_w	P
Polystyrene sample 1 (PS 1)				
SEEC	137 850	162 710	226 130	1.39
SEC 1	130 710	149 010	226 200	1.52
SEC 2	133 120	173 390	232 540	1.34
Polystyrene sample 2 (PS 2)				
SEEC	92 830	115 650	149 280	1.29
SEC 1	95 850	105 720	146 350	1.38
SEC 2	81 000	119 620	176 510	1.48
Polycarbonate sample 1 (PC 1)				
SEEC	20 090[a]	22 090[a]	34 240[a]	1.55
SEC 1	20 040[a]	21 040[a]	31 380[a]	1.49
SEC 2	–	–	–	–
Polycarbonate sample 2 (PC 2)				
SEEC	63 050[a]	65 230[a]	101 110[a]	1.55
SEC 1	75 760[a]	75 410[a]	112 250[a]	1.49
SEC 2	–	–	–	–

[a] In polystyrene units.

the actual retention time. Still, the feasibility and attractiveness of using HFIP as a solvent for SEEC could be demonstrated.

A mass calibration curve was constructed for PMMA through the separation of a number of PMMA standards (Fig. 6). The retention ratios of the PMMA standards were calculated using acetone as the totally permeating marker. The curve shows that the mass (size) range of PMMA standards that can be separated on the column material is relatively small compared to the mass range available with the 300 Å bare silica material used for the separations with DMF as the mobile phase. This is due to the larger pore diameter of the latter particle types.

An example of a separation of three PMMA standards of different mass by SEEC using HFIP as the mobile phase can be seen in Fig. 7. Using HFIP as the mobile phase, the PMMA standards were nicely resolved and could be detected at 220 nm. At this wavelength the acetone produced a negative peak, therefore the migration time of acetone was determined from the UV trace at 254 nm where acetone is highly UV active.

Examples of SEEC separations of some commercially relevant polymer samples are shown in Fig. 8.

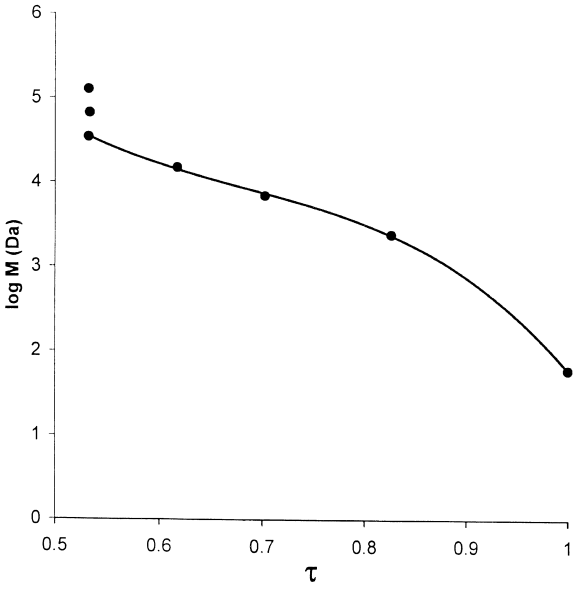

Fig. 6. Mass calibration curve for PMMA in SEEC. Mobile phase: 0.1 m*M* TBATFB in HFIP. Stationary phase: 10 μm Nucleosil SA-100; separation voltage: 25 kV; UV detection at 220 nm.

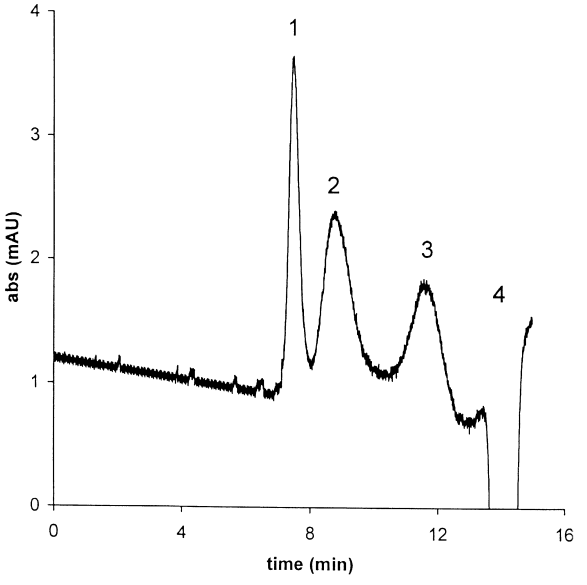

Fig. 7. Separation of three different PMMA standards by SEEC using 1.0 m*M* TBATFB in HFIP as the mobile phase. Solutes: (1) PMMA 67 000; (2) PMMA 15 100; (3) PMMA 2400 and (4) acetone. Mobile phase: 0.1 m*M* TBATFB in HFIP; stationary phase: 10 μm Nucleosil SA-100; separation voltage: 25 kV; UV detection at 220 nm.

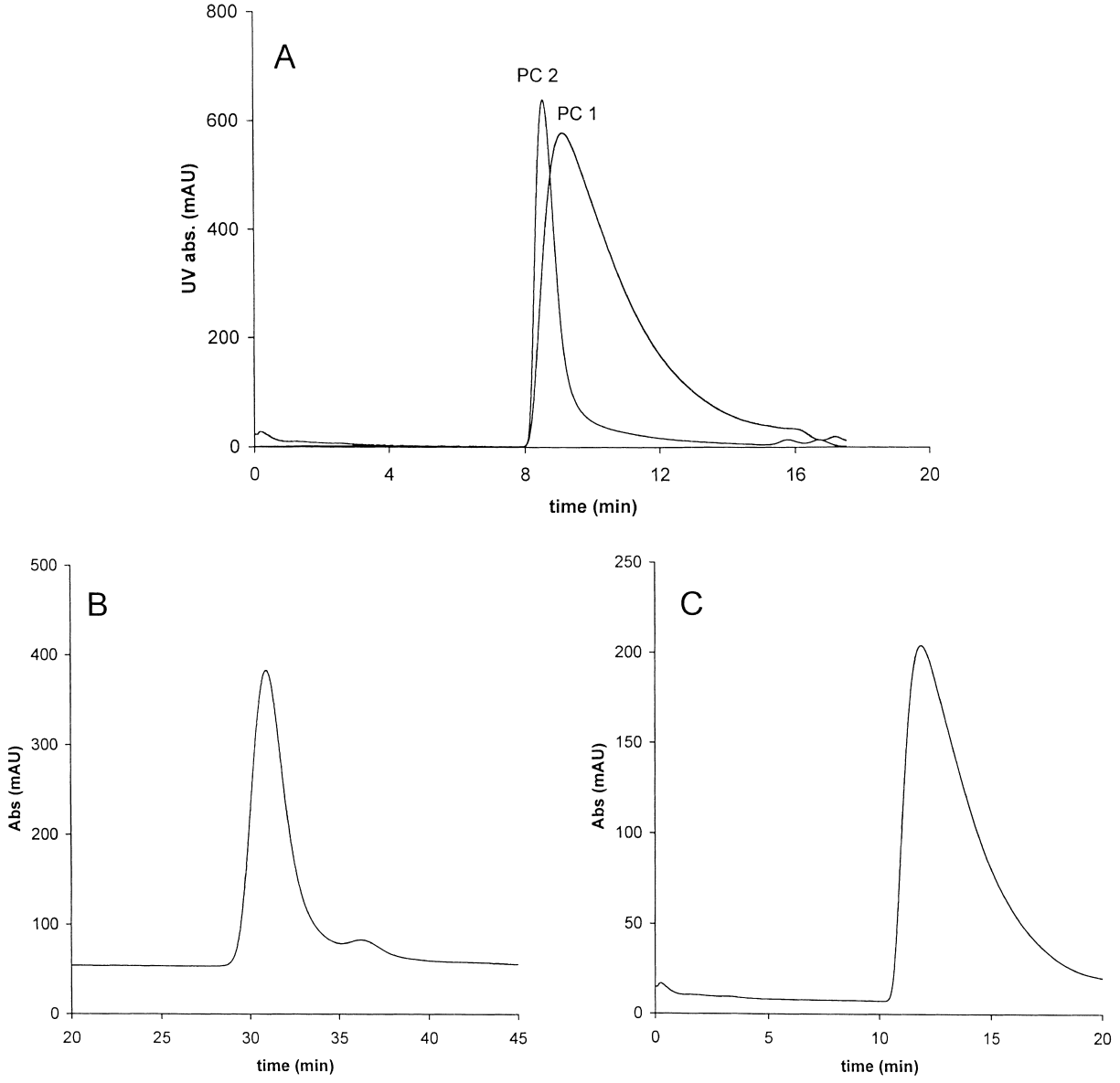

Fig. 8. Separation of several commercially important, HFIP soluble synthetic polymers by means of SEEC. Samples: (A) PC; (B) PET and (C) polycaprolactam. Mobile phase: 0.1 m*M* TBATFB in HFIP. Stationary phase: 10 μm Nucleosil SA-100; separation voltage: 25 kV; UV detection at 195 nm.

Again, the high UV transmission properties of HFIP allow for a high sensitivity and the ability to use low UV wavelengths for detection. For example the two PC samples were also subjected to SEEC analysis using HFIP instead of DMF as the solvent. The very good UV transmission properties of HFIP allowed detection at 195 nm resulting in an ~500-fold increase of the sensitivity compared to UV detection at 265 nm as applied with DMF as the solvent. Using UV detection at 195 nm highly sensitive detection could also be performed for PET and polycaprolactam samples.

4. Conclusions

It has been established that EOF can be well controlled in SEEC. In addition, the injection procedure and the the repeatability of the retention and efficiency are precise enough to allow SEEC to be used for the quantitative analysis of mass distributions of synthetic polymers. Comparison of mass distributions as determined from SEEC and PD-SEC data show that there is no significant difference between the two methodologies. The main advantages of SEEC over PD-SEC are the ability to perform relatively fast separations, and the small dimensions of the separation system.

The feasibility of using SEEC for the mass analysis of a variety of synthetic polymers is demonstrated by using HFIP as a new mobile phase for SEEC. Here, the potential of SEEC is even higher due to the small mobile phase consumption, resulting in a large reduction of analysis costs. Future development of SEEC will mostly rely on demonstrating new solvents and applications for SEEC and the development of miniaturized detectors, other than UV, suitable for (synthetic) polymers.

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