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Journal of Chromatography A, 709 (1995) 227–239

JOURNAL OF CHROMATOGRAPHY A

Study of the operating conditions of axial compression columns for preparative chromatography

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First received 24 February 1995; revised manuscript received 12 April 1995; accepted 12 April 1995

Abstract

The packing behavior and the stability of a spherical-particle, silica-based packing material and the operation of an axial compression column are discussed. The coupling between the axial compression pressure and the inlet pressure at constant flow-rate was investigated. The results demonstrated also an excellent column efficiency, a rapid packing consolidation, and a long-term stability of the column performance. Reduced height equivalents of theoretical plate (HETPs) around 2 were routinely obtained and the column did not show any sign of degradation of its efficiency or stability after 700 h of continuous use. These results are attributed primarily to the spherical nature of the material used. Finally, a procedure for opening the top flange of a column packed under axial compression without loosing any stationary phase nor degrading the column efficiency is described.

1. Introduction

For over twenty years, a slow debate regarding the relative superiority of spherical- and irregular-shaped particles has been held without yet reaching a conclusion. This problem seems to have been of more serious concern to vendors and manufacturers than to the scientific community, and very few systematic comparative studies have been published. Admittedly, the differences between the hydrodynamic and the kinetic performance observed in analytical applications did not seem very significant compared to the differences of retention properties reported between any two brands of silica material. As reliable, accurate information is nearly priceless, there was little incentive in taking the particle shape into account when separation factors are of so paramount importance.

Things have changed somewhat with the development of preparative chromatography. The cost of the packing material, although not the most critical component of production costs [1–3], has become important. The cost of bulk packing materials is higher when they are made of spherical particles than when made of irregular-shaped particles. For production purposes, several elements other than the separation factors must be taken into account. Therefore, the issues of the relative technical advantages

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0021-9673/95/$09.50
SSDI 0021-9673(95)00474-2

and disadvantages of the two types of packing materials is resurfacing. Although not always convincingly, it is often claimed that the performance of spherical particles is superior to that of irregular-shaped materials.

The most important advantages claimed for spherical particles are as follows. First, because of the conditions under which they are prepared, there is a better control over their particle-size distribution. Whether a narrower size distribution is a real advantage, however, is still open for discussion. It is said that the small particles determine the column permeability whereas the large ones control the column efficiency, but systematic experiments are lacking. Second, spherical particles can tolerate higher compression pressures than irregular particles and they would be less susceptible to fragmentation into smaller particles during handling, packing, and consolidation. This mechanical superiority is suggested by the preeminent strength of the eggshells and cupolas. Packings do suffer during consolidation [4] and materials made of spherical particles seem to consolidate faster, under lower pressures, and into a more compact, more resilient bed than those made of irregular-shaped particles [5]. The practical extent of these advantages remains unclear and several systematic studies are needed to understand the different aspects of this complex issue. It should be underlined that, even in preparative chromatography, the issues of primary importance remain (i) the separation factor and (ii) the saturation capacity. The final choice of a packing should be made between those materials which have the best characteristics for these two properties and give columns with a long useful life.

In previous publications [6–8] we have studied the performance of dynamic radial and axial compression columns packed with irregular-shaped particles. The goal of this paper is the description of experimental results obtained with a material made of spherical particles when using the methods we have previously developed for the packing of dynamic axial compression columns and for testing of these columns for long-time durability.

2. Experimental

2.1. Dynamic axial compression column

The axial compression unit was an LC.50.VE.500.100 Column Skid obtained from Prochrom (Champigneulles, France). The column is made of a stainless steel cylinder (5.0 cm × 59.0 cm) with a maximum working pressure of 100 bar. The axial compression is applied by a hydraulic piston operated by a Haskel pump (Burbank, CA, USA). The Haskel pump in turn is assisted by compressed nitrogen from a cylinder.

2.2. Solvent delivery system

A Kiloprep 100 HPLC pump was obtained from Biotage (Charlottesville, VA, USA). The pump can deliver solvents at flow-rates up to 500 ml/min, at a maximum pressure of 138 bar. The flow-rate is set manually. The system includes also two solvent ports and an injection valve with a 1.5-ml injection loop.

2.3. Stationary phase

All the columns used in this work were packed with Kromasil C_8 (Eka Nobel, Bohus, Sweden). This chemically bonded spherical silica has an average particle size of 13.0 μm and an average pore size of 100 Å. The axial compression column was packed using the procedure previously described [8], at a pressure of 61.5 bar. All experiments were carried out at this axial compression pressure, except for those during which the influence of the compression pressure was studied.

Several analytical columns of different lengths, all with an internal diameter of 0.46 cm, were packed in our laboratory using the same stationary phase, and a conventional slurry packing method at 483 bar, in agreement with the recommendations of the manufacturer. The characteristics of these columns are summarized in Table 1.

Table 1
Characteristics of the columns

Column properties	Axial compression column	Analytical column #1	Analytical column #2	Analytical column #3
Dimension (cm)	20.0 × 5.0	20.0 × 0.46	20.0 × 0.46	10.0 × 0.46
Total porosity (solvent A)	0.72	0.76	0.73	0.71
Total porosity (solvent B)	0.67	0.75	not done	0.70
Total porosity (from mass)	0.74	0.74	0.75	not done
Phase ratio (solvent A)	0.39	0.32	0.37	0.40
Phase ratio (solvent B)	0.48	0.34	not done	0.43
Phase ratio (from mass)	0.35	0.35	0.33	not done
k' (acetone)	0.37 ± 0.01	na[a]	na	0.30 ± 0.01
k' (phenol)	2.82 ± 0.20	na	na	2.84 ± 0.05
k' (cresol)	6.04 ± 0.46	na	na	6.18 ± 0.12

[a] na = not available.

2.4. Chemicals

Acetone, *m*-cresol, phenol, methyl benzoate, toluene, acetonitrile, nitric acid, ammonium bifluoride, isopropanol, and methanol were all purchased from Baxter (Atlanta, GA, USA) and were 99.9% pure. Phenethyl alcohol was purchased from Fluka (Buchs, Switzerland). Distilled water from the chemistry department plant was filtered on a 1.2-μm membrane before use.

2.5. Detector

A UV–Vis detector (Model 203-7083, Linear Scientific, Reno, NV, USA) equipped with a variable path length preparative cell was used to collect chromatograms. With a short cell path length the detector response remains linear up to much higher concentrations than with conventional HPLC detectors. The cell can be operated under high pressures and can easily accommodate a 500 ml/min flow-rate. For HETP measurements, the cell path length was kept to its maximum (3 mm), so that a reasonable response could be obtained with analytical-size injections.

2.6. Pressure sensor

The column inlet pressure was measured with an Omega pressure transducer Model PX603-2KG5V (Omega, Stamford, CT, USA). This transducer gives a 1–5 V d.c. linear output. Its response time is 1 ms. The output was adjusted to read 0.402 V d.c. for 0 bar and 2.022 V d.c. for 138 bar, for compatibility with the data acquisition system. Calibration shows that the inlet pressure [P (bar)] and the voltage output [V (V)] are related by $P = (V - 0.402)/1.1745 \cdot 10^{-2}$). The reading of the sensor at a zero inlet pressure (no flow-rate) was checked periodically.

2.7. Displacement sensor

Dynamic changes of the column length were measured with an Electro-Mike displacement sensor Model PAA1555 (Reagan Controls, Charlotte, NC, USA). This sensor includes a displacement transducer and a transmitter with analog output of 2 to 9 V d.c. for a displacement between 2.0 and 9.0 mm. The output voltage was

attenuated to 2.2 V d.c. for our data system. Calibration of the sensor output with known targets shows the response to be linear in the +2 to +9 mm range, d (distance between target and sensor in mm) and V (output in V) being related by $d = (0.20 \pm 0.01)V + (0.18 \pm 0.06)$.

Unlike in our previous work [8], the sensor was hanged from a stand and the steel target was clamped to the compression piston, in such a way that an increase in target distance means an increase in the column length. Depending on the movement of the piston, the distance between the sensor and the target increases or decreases. Because of the nature of the device, a change of 0.01 mm in the column length can be detected easily, while the actual column length is known within only 0.1 cm. The actual length of a packed column was measured by attaching a long, thin electric wire to the piston rod, just below the piston head, stretching it with a small weight, and measuring the position of a small mark on this wire. The zero reference is obtained by raising the piston in an empty column until it touches the top flange. This device permits a measurement of the column length with an accuracy of 0.1 cm.

2.8. Data acquisition system

The data system consists of a Waters System Interface Module (SIM) with two A/D convertors (Milford, MA, USA). This SIM is capable of the simultaneous monitoring of four sensors and/or detectors and can control three HPLC pumps. The digitized data from the SIM was collected by Waters Maxima 820 version 3.31 software loaded on a NEC computer. All the data files were translated to ASCII format for further use and uploaded to the computer network of the University of Tennessee. For treatment of these data, various DOS- and VMS-based software was developed in our laboratory.

2.9. Methods

In the experiments reported here, two solvents were used as the eluent. These were methanol (solvent A) and a mixture of 40% methanol and 60% water (v/v, solvent B). The test samples were low-concentration solutions of acetone (1.6 ml/l), phenol (0.4 g/l), and *m*-cresol (0.5 g/l) in the eluent. Sample volumes for HETP measurements were 1.5 ml, injected by filling an appropriate loop. For economic and waste management reasons, the solvents were pumped in closed circuit, with a 15–20 l reservoir on the solvent line. The solvent was replaced when the baseline absorbance became significantly higher than that of fresh solvent.

Three outputs were recorded in most of the experiments, the UV-detector signal and the outputs of the displacement and the pressure sensors. The data from the pressure transducer were converted to pressure units (bar), and the output from the sensors to changes in the column length (cm) and the pressure (bar). The chromatographic data were used to calculate the column efficiencies, from the width at peak half-height, and the retention factors. The reduced velocities ($\nu = u\, d_p/D_m$, with u the linear velocity, d_p the average particle size, and D_m the molecular diffusivity) and reduced plate heights ($h = H/d_p$, with H the actual column HETP) were fitted to the van Deemter equation [9], using a nonlinear least-squares fit. The classical Wilke–Chang [10] equation was used to estimate the diffusion coefficients of the compounds used. It gives as the molecular diffusivities of acetone, phenol, and cresol, in solvent B, at 25°C, $7.39 \cdot 10^{-6}$, $6.59 \cdot 10^{-6}$, and $5.98 \cdot 10^{-6}$ cm^2/s, respectively.

3. Results and discussion

The characteristics of the columns studied are summarized in Table 1. The results of a long-term stability test of the axial compression column are reported in Table 2. During this study, several similar determinations of the dependence of the column efficiency on the mobile phase flow-rate were completed. Flow-rates and column efficiencies were converted into reduced parameters, viz., reduced velocity and reduced plate height, and fitted to the classical van

Table 2
Compiled Van Deemter parameters for the axial compression columns

Column no.	Solvent	Sample	a	c	h_{min}	ν_{opt}	Hours used
11	A	acetone	1.62	0.089	2.42	4.50	10
11	A	acetone	1.55	0.098	2.39	4.29	20
11	A	acetone	1.68	0.080	2.44	4.74	46
11	B	acetone	2.18	0.042	2.73	6.55	
		phenol	3.03	0.012	3.32	12.25	72
		cresol	2.81	0.015	3.14	10.95	
11	B	acetone	2.09	0.052	2.70	5.88	
		phenol	2.80	0.025	3.22	8.49	123
		cresol	2.86	0.016	3.20	10.61	
11	B	acetone	1.96	0.047	2.54	6.19	
		phenol	2.42	0.028	2.87	8.02	238
		cresol	2.90	0.010	3.17	13.42	
11	B	acetone	1.96	0.051	2.57	5.94	
		phenol	2.67	0.026	3.10	8.32	263
		cresol	2.47	0.024	2.89	8.66	
11	A	acetone	1.63	0.088	2.43	4.52	371
11	A	acetone	1.62	0.084	2.40	4.63	391
11	A	acetone	2.07	0.050	2.67	6.00	
		phenol	1.83	0.047	2.41	6.19	497
		cresol	2.49	0.030	2.96	7.75	

Deemter equation [9]. Since few data points were collected at low flow velocities, the coefficient b of this equation was taken arbitrarily as equal to 1.8, as in our previous study [8]. The values of the coefficients a and c are shown in Table 2. Table 2 also contains the values of the minimum reduced plate height (h_{min}) and the optimum reduced velocity (ν_{opt}).

As seen in Table 2, the column performance did not change significantly over a period of 500 h. In fact, several other experiments, different from the conventional testing done during the first 500 h, were performed with this same column for an additional 200 h (experiments reported later, in Sections 3.4–3.8). After 700 h, the column was briefly tested again and dismantled. Although a more limited data set was acquired, the final test showed that the column efficiency had not changed.

3.1. Column performance with pure methanol

As in previous papers [7,8], a preliminary study of the column performance was done using pure methanol as the mobile phase and a 1.5% acetone solution as the sample. The results are given in Table 2. Acetone was used as an unretained compound for the determination of the void volume [8], hence of the total porosity, which is reported in Table 1. The total porosity of analytical columns was also determined by washing the column with pure methanol, unpacking it and weighing the packing material recovered, after drying it in an oven at 110°C for 12 h. The stationary phase volume was determined by assuming the density of silica to be 2.2 g/cm^3.

Fig. 1 shows a typical plot of the reduced plate height versus the reduced mobile phase velocity for both the axial compression column and an analytical column. The lines are derived from the best fit of the respective data to the van Deemter equation. The axial compression column showed better efficiency than the analytical column (Fig. 1). This is not uncommon for spherical particles [11] and might be explained either by a smaller relative contribution of extra-column band broadening or by a genuinely better packed

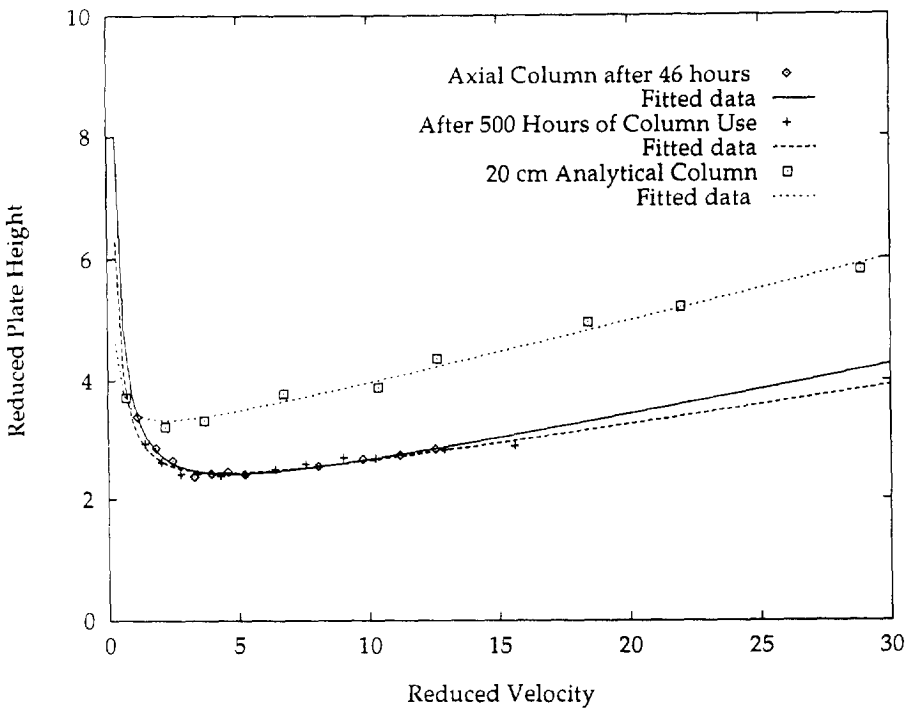

Fig. 1. Typical Van Deemter plot obtained. Plot of the reduced plate height versus the reduced velocity for an axial compression column (20.0 × 5.0 cm) and an analytical column (20.0 × 0.46 cm). The eluent was methanol and the sample a solution of acetone in methanol.

column. Fig. 1 also contains data comparing performance of the axial column after 46 and 500 h of use, respectively. As seen in Fig. 1, the column performance was essentially the same after 500 h of use as immediately after packing. For this column the van Deemter parameters were $a = 1.61$, $b = 1.93$, $c = 0.086$, $h_{min} = 2.42$, $v_{opt} = 4.74$ after 46 h and $a = 1.84$, $b = 1.34$, $c = 0.068$, $h_{min} = 2.44$, $v_{opt} = 4.44$ after 500 h of column use. For the analytical column the van Deemter parameters were $a = 2.86$, $b = 0.53$, $c = 0.105$, $h_{min} = 3.33$, and $v_{opt} = 2.22$.

The data in Table 1 show that the values of the total porosity and the phase ratio of the different columns are consistent. Although there is some scatter of the data for the analytical columns, the axial compression column gives on the average a systematically lower total porosity and a larger phase ratio than the analytical columns.

3.2. Column performance with methanol–water (40:60, v/v)

Solvent B was used for the long-term stability study of the axial compression column. A sample containing acetone ($k' = 0.37$), phenol ($k' = 2.82$), and *m*-cresol ($k' = 6.04$) was used to monitor the efficiency of the column at long time intervals (see Table 2). These values of the retention factors of our standard test compounds are slightly higher than those found with the C_{18} stationary phases previously used [7]. A high surface density of the bonded alkyl chains on the stationary phase could explain this change. It is not due to an error in the mobile phase composition. The values obtained for the retention factors on the axial compression column and the analytical columns are in excellent agreement. It is clear from the data in Table 1 that the

performance of the axial compression column did not degrade significantly after 500 h of use.

3.3. Column permeability and average particle size determination

During the course of this study, the inlet pressure of the axial compression column was determined at different flow-rates, while determining the column efficiency. The values obtained for the inlet pressure were corrected for the contributions from the inlet and outlet frits and the connecting tubes, as done in our previous study [8]. As shown by the plot of a typical set of corrected inlet pressures versus the flow-rate in Fig. 2, these data fit well to a straight line. Introducing the slope of this straight line into the Darcy equation gives a particle size of 12.2 μm. The average particle size determined independently by Coulter counter was 11.5 μm for the material used in the axial compression column and recovered afterward and 11.2 μm for the virgin material. The nominal particle size was 13.0 μm.

3.4. Inlet pressure and axial compression pressure

Several sets of experiments were carried out to investigate the effect of the axial compression on the packing density and the permeability, hence the inlet pressure of the axial compression column. If the axial compression increases the packing density, the permeability should decrease and, in turn, the inlet pressure required to sustain a given flow-rate should increase. In these studies we have set the axial compression pressure to a particular value and measured the inlet pressure at different flow-rates. The results obtained are illustrated in Fig. 3, which shows a

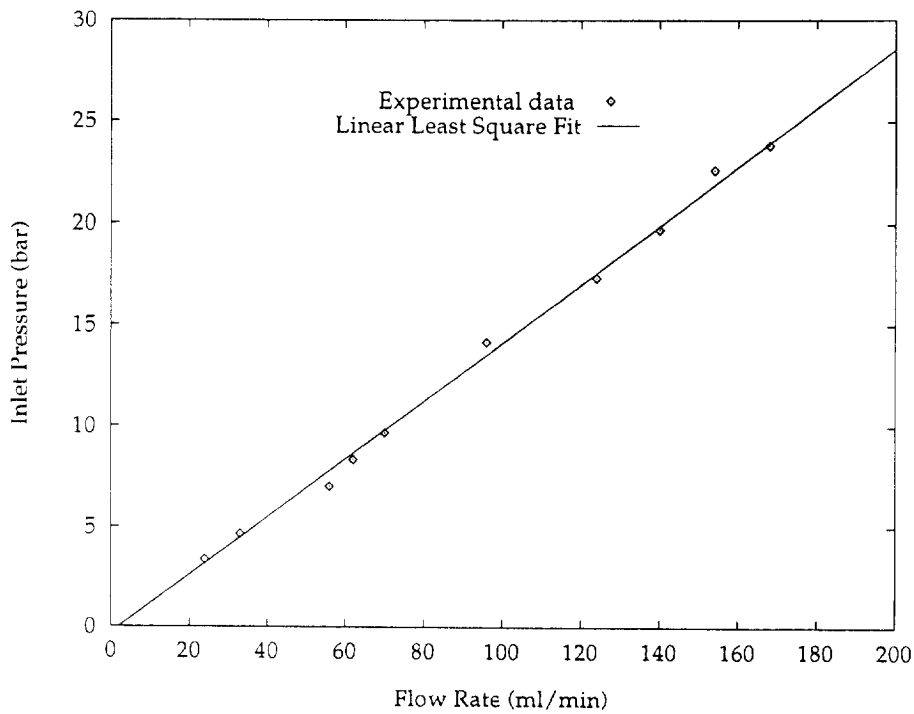

Fig. 2. Plot of the column inlet pressure versus the flow-rate for the axial compression column. The solvent was methanol–water (40:60, v/v). The average particle size obtained from this set of data was 12.2 μm.

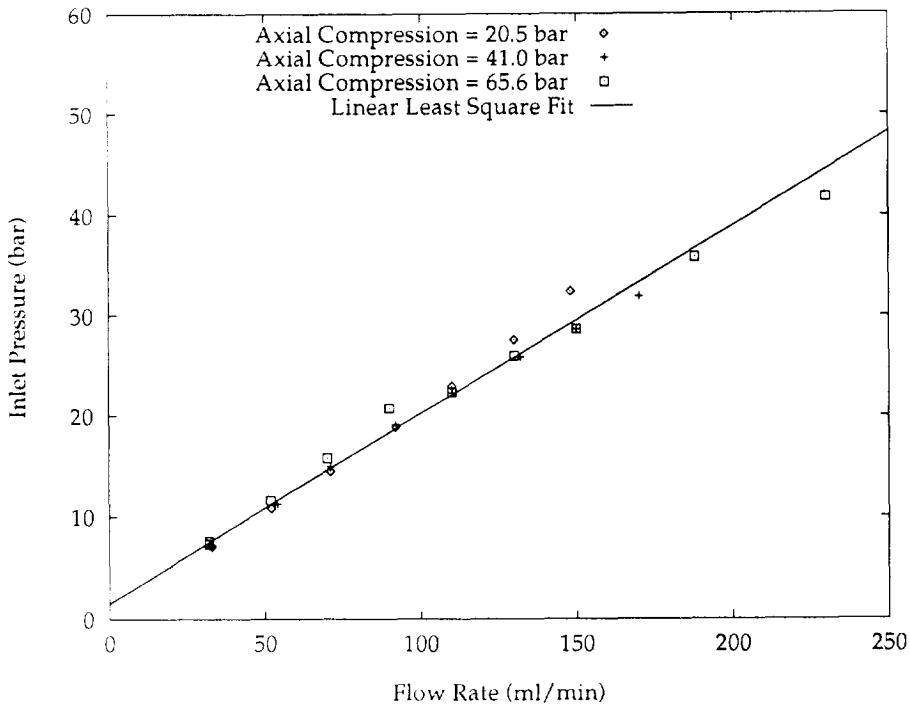

Fig. 3. Plot of the column inlet pressure versus the flow-rate at different axial compression pressures. Solvent methanol–water (40:60, v/v).

plot of the inlet pressure versus the flow-rate at three different axial compression pressures between 20 and 66 bar. The permeability does not change significantly within the range of compression pressures applied. This suggests that the consolidation of the bed has already been achieved when the first experiment was started and that the compressibility of the consolidated bed is small. Indeed, the packed bed had been consolidated under 66 bar during the initial consolidation of the column, prior to these experiments. Both results are in agreement with other experimental findings which will be reported soon [5]. Introducing the slope of the straight line in Fig. 3 into the Darcy equation gives an average particle size of 9.98 μm.

3.5. *Effective axial compression pressure (EACP) versus flow-rate*

The effective axial compression pressure is the stress applied by the piston against the top of the packing divided by the piston cross-section area. Because a solid, the packing material, is stressed in these compression experiments, the local strain can vary widely across the column. It is useful, however, to follow the variation of this effective compression pressure during the operation of the column. Since the column inlet pressure acts against the axial compression, the effective compression pressure is the difference between the applied axial compression pressure and the inlet pressure. It depends on the flow-rate if, as recommended by Prochrom, the axial compression is kept constant. Results are shown in Fig. 4. The linear decay of the effective compression pressure with increasing flow-rate is expected from what was reported above (Fig. 3). However, it is interesting to note that at high flow-rates the EACP can become negative. This is technically possible because the equipment used in this work for the dynamic axial compression of the column bed is designed in such a way that the piston cannot be pushed back if the

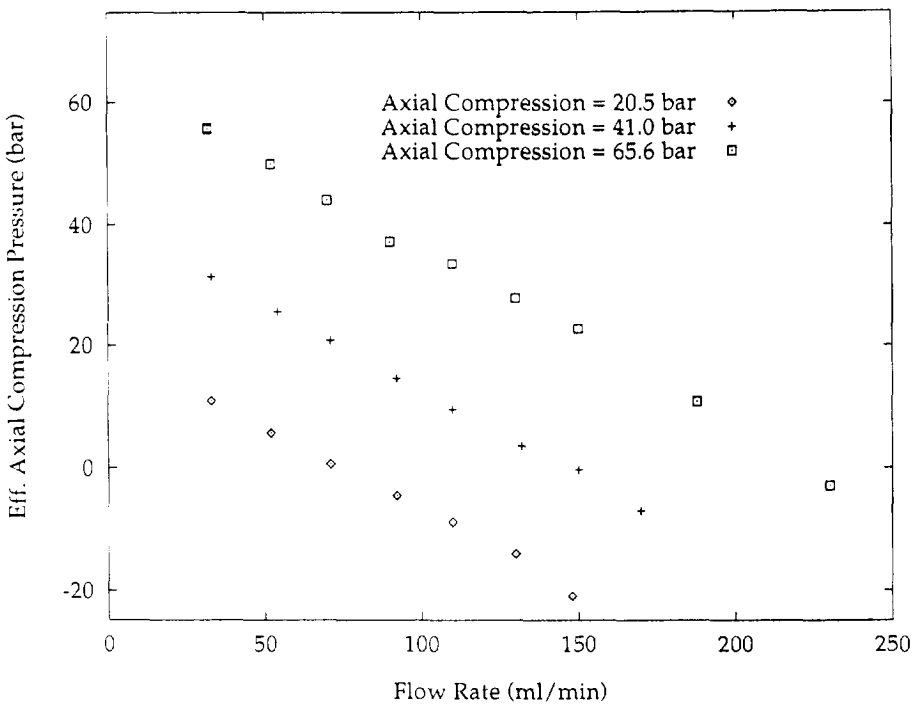

Fig. 4. Plot of the effective axial compression pressure versus the flow-rate for the axial compression column. The initial compression pressures set were 20.5, 41.0, and 65.5 bar. Solvent: methanol–water (40:60, v/v).

compression pressure falls below the inlet pressure. The bed remains compressed like the gas in a tire as long as the pressure in the driving jack is not released. Small leaks are possible, however, and there is a slight compression release of the bed, as we see now.

3.6. Column length and effective axial compression pressure

The column length measured during the experiments just described is plotted against the effective axial compression pressure (EACP) in Fig. 5. In this case, the EACP is varied by varying the flow-rate at constant applied compression pressure. As expected, the column length decreases with increasing EACP and tends toward a constant limit. The higher the axial compression pressure applied, the lower this limit. Fig. 6 shows the result of a complementary experiment. It gives a plot of the column length versus the EACP, at constant flow-rate (110 ml/min) and with different values of the axial compression. A similar behavior is observed. Finally, the plot of the inlet pressure versus the EACP shown in Fig. 7 illustrates the relationship between the effective compression pressure, that is the stress actually applied to the bed, and the inlet pressure for a constant flow-rate. The increase of the column inlet pressure at constant flow-rate corresponding to an increase of the EACP of 30 atm is 6% (31.5 bar to 33.5 bar).

3.7. Replacement of the top flange

It is frequently necessary to open the column and change the top flange. Done without special care, this operation results in a loss of stationary phase, which can cause a problem, for example when the exact amount of packing material used needs to be known. This operation can be done without loss or any disturbance of the packing by the following procedure. A solvent which wets

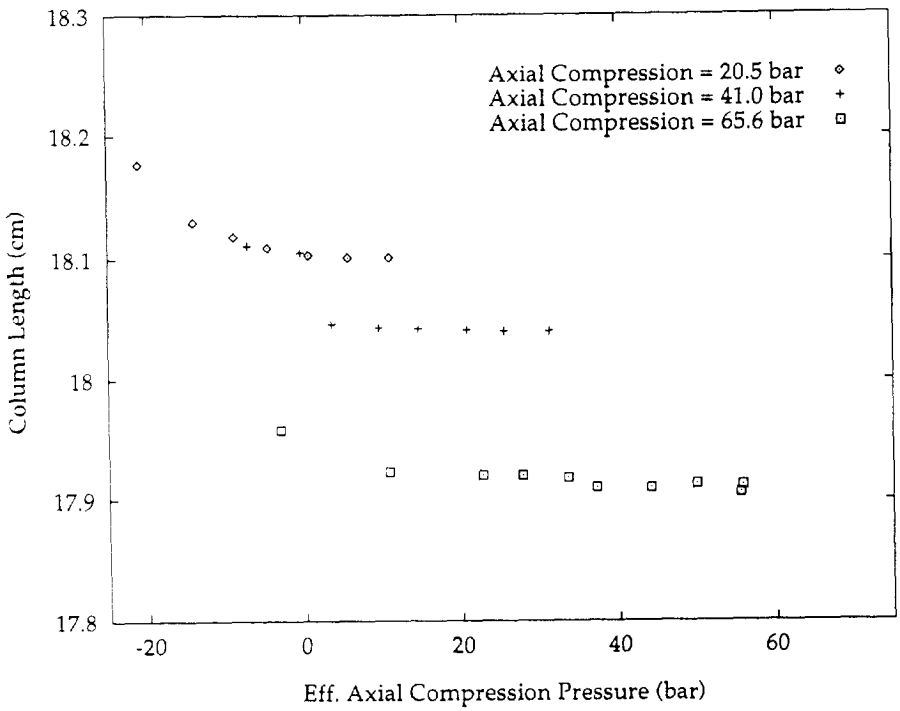

Fig. 5. Plot of the column length versus the effective axial compression pressure during the compression experiments in Fig. 4.

well the packing, methanol in the case of RPLC packings, must be used. First the flow direction of the mobile phase during the experiment is reversed by pumping the solvent into the top flange side. The piston is lowered by 2 cm and held in the neutral position. Solvent is run through the column for five minutes. After flowing several column volumes of solvent while using the top flange as the column inlet, it is safe to open the column top. The top flange is unfastened and opened. A large void, ca. 2 cm deep, is found but the top of the packing is flat. The piston is then moved up slowly by applying a low axial pressure and is stopped when the top flange would barely touch the bed if in position. At this point the top flange is secured in place and the solvent pump is restarted. This procedure is simple. Reversal of the flow direction does not seem to be a problem for a dynamic axial compression column. This procedure allows rapid changes of the top flange or clean/change of the frit.

3.8. Repairing a bad column by axial compression

A void tends to form more or less slowly at the inlet of conventional chromatographic columns, causing a marked degradation of their efficiency. In this last set of experiments, we tried to simulate the formation of a void at the column inlet and to measure its effect on the column efficiency and inlet pressure. The solvent used was methanol and the sample a 3% solution of acetone in methanol. The flow-rate was 151 ml/min. The procedure described in the section above was used to open and close the top of the column and move its top flange. After six hours under a constant stream of mobile phase, the column was tested. The results obtained are shown in row 1 of Table 3. The subsequent operations are also summarized in Table 3. The next day the piston was lowered again by 1 cm as shown in row 3. At this stage, the column efficiency appeared to have been degraded quite

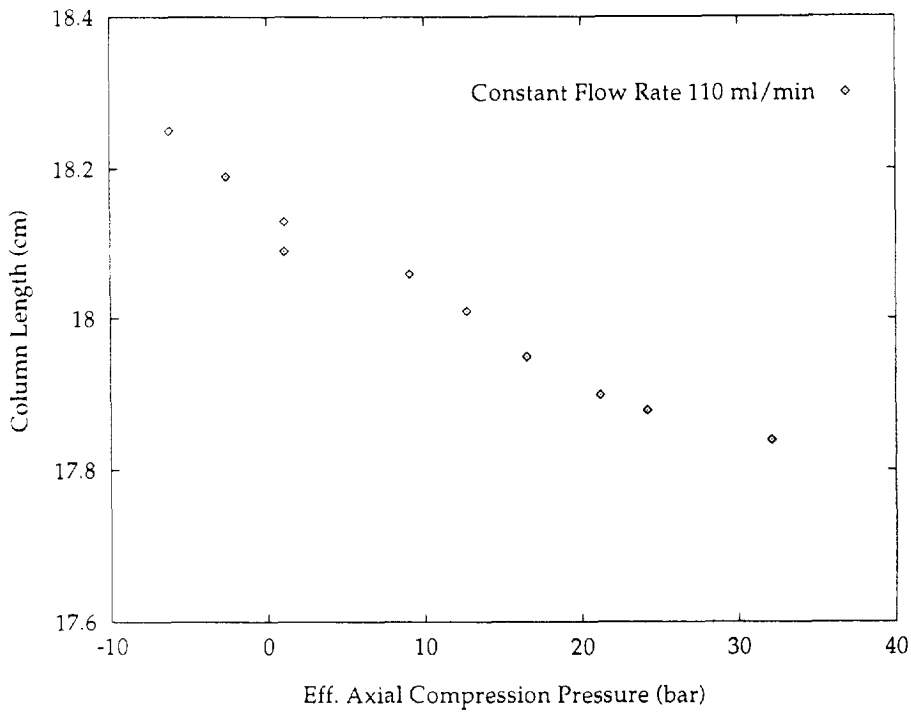

Fig. 6. Plot of the column length versus the effective axial compression pressure at constant flow-rate. The solvent was methanol–water (40:60, v/v).

significantly after eight hours of operation (Table 3, row 5). The previous level of column performance was recovered by a succession of upward movements of the piston (i.e. by decreasing the void space above the piston). The maximum efficiency ever achieved with this column was then obtained (row 9). Finally, the piston was lowered again by 2 cm from the position it occupied when the data in row 9 were being recorded. Comparing the data in rows 5 and 9, we see that an efficiency gain of 69% (from 1488 plates to 2521 plates) in number of theoretical plates was achieved by restoring a sufficiently large, positive effective axial compression and by eliminating the void at the column entrance.

The single most serious disadvantage of the procedure is the increase of inlet pressure by 19%. It was surprising to observe that at no time the column did exhibit any signs of channel formation, even when a 2-cm long void had been created at the column entrance by following a procedure which, in many respects could be qualified as operational failure if it had not been carried out on purpose. The formation of large cavities at the column inlet and their sealing demonstrate the effectiveness of the dynamic compression principle. It permits a change of the flow direction in a dynamic compression column when needed, without any apparent consequences on the column performance.

Acknowledgements

This work has been supported in part by Grant CHE-9201662 of the National Science Foundation and by the cooperative agreement between the University of Tennessee and the Oak Ridge National Laboratory. We acknowledge the long-term free loan from Prochrom (Champigneulles, France) of the 5-cm I.D. stainless-steel column

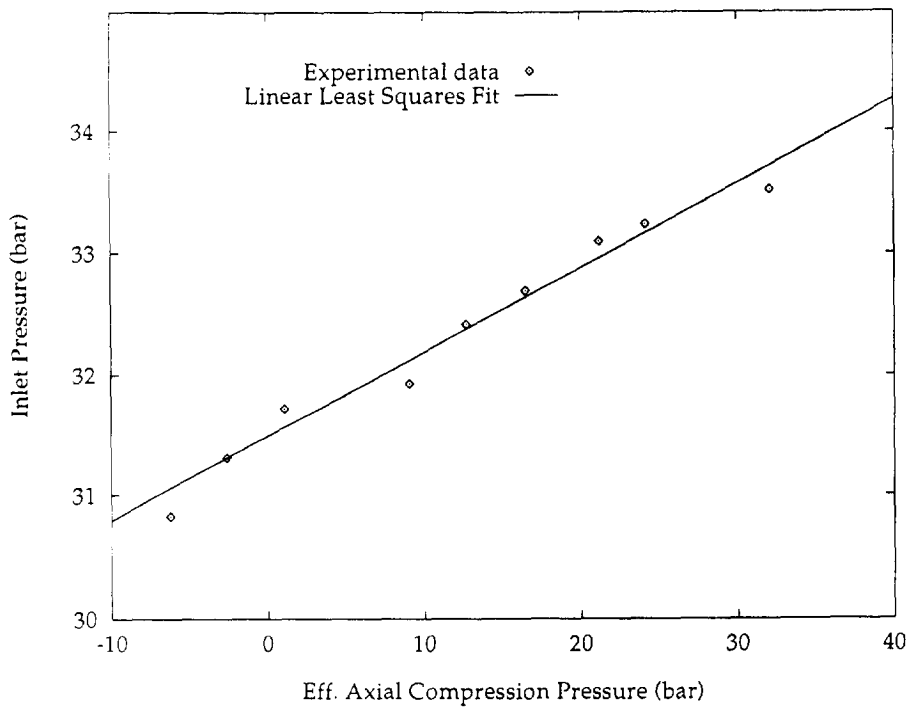

Fig. 7. Plot of the column inlet pressure versus the effective axial compression pressure at constant flow-rate.

and the axial compression unit. We thank Biotage (Charlottesville, VA, USA) for the long-term free loan of their Kiloprep 100 solvent-delivery system and the Linear Scientific Model 204 UV detector equipped with a preparative scale cell. We thank BTR Separation, Wilmington, DE (formerly The PQ Corporation) for their gift of a semi-preparative Linear Scientific Detector and Eka Nobel for their gift of 2 kg of C_8 bonded spherical silica for this study.

Table 3
Repairing a bad axial column by applying axial compression

No.	Condition of experiment	Inlet pressure (bar)	Efficiency of column
1	No ACP[a] applied, piston just touching the bed and the piston held at neutral	13.05	2279
2	Same as #1 after over night (pump not in operation)	13.22	2181
3	Piston position lowered 1 cm from the position at #1	13.05	1534
4	Same as #3, after 2 h of pumping operation	12.88	1517
5	Same as #3, after 8 h of pumping operation	12.80	1488
6	Piston brought up 1 cm with low ACP and held at neutral	12.88	1919
7	Piston brought up 1 cm with low ACP and held at neutral	12.88	2362
8	Piston with ACP = 63 bar. After 2 h of operation, the column length was 17.2 cm	15.35	2482
9	Same as #8, after 6 h of pumping operation	15.18	2521
10	Piston brought down 2 cm from #9	13.22	2017

[a] ACP = axial compression pressure.

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