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Coiled High-Efficiency Liquid Chromatography Columns

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Abstract

Columns having a compact shape are highly desirable in liquid chromatography, allowing better and easier thermostatting. Packing a conventional high resolution liquid chromatography column in its straight configuration, followed by coiling, has not always resulted in a retention of efficiency. We have recently described a novel polymeric packing material, open-pore polyurethane, where high resolution–low pressure liquid chromatography columns are prepared *in situ*. These columns have been tested in their linear configuration and then coiled to a variety of coil sizes. Using 2.16 mm i.d. stainless steel tubes, coil diameters as small as 2 in. have been examined and no loss of efficiency was detected. This result has not been achieved before with conventional packings. Additionally, it has been shown that these columns may be coupled in series and the total number of theoretical plates may be calculated. This allows a high resolution series arrangement of columns with minimum column space requirements. This is generally not possible with conventional columns because of their high back-pressure.

INTRODUCTION

In an attempt to effect a reduction in column size in chromatography, coiling has often been attempted. These efforts have been mostly unsuccessful. When a 40-in. column, 4 mm i.d. was coiled to a 4.3-in. radius, the

HETP increased 800% (*1*). Less drastic coiling, for example, bending a 3 m × 2.1 mm i.d. column into a trombone shape having 3 × 1 m straight sections and the 2 U bends having a 3-in. radius, led to a 50% decrease in the number of theoretical plates (*2*). A recent examination of this problem and a summary of the previous, sometimes contradictory, results has appeared (*3*). The previous controversy was whether the observed decrease in efficiency was due to the disruption of the packing geometry or particle crushing upon coiling, or the flow velocity profile difference in a coiled tube compared with a straight tube. The latter authors showed that a column could be packed in a coiled manner with the same efficiency as one which was packed into a straight tube and then coiled. However, at a column diameter (internal) of 1.9 mm, simple coiled columns could not be made which had the same efficiency as straight columns. Various, more complicated, coil configurations were discovered which did have the same efficiencies as the straight columns.

We have recently described (*4*) the preparation and properties of open-pore polyurethane (OPP) columns for liquid chromatography. These are similar to those reported (*5, 6*) for use in gas–liquid chromatography. These authors noted that these columns could either be prepared in the coiled configuration or packed in a straight configuration and then bent to any desired shape after the polymerization was complete. It was reported that the column should be filled with liquid while the coiling was performed. Because of the inconvenience of long straight columns, particularly with respect to temperature control, we have investigated the highly desirable possibility of coiled liquid chromatographic columns. An OPP column, designated column 1, was prepared using a 2:1 excess molar concentration of polyol (LA475 Union Carbide) to diisocyanate (Mondur MR) in the preparation of the polyurethane. The total reactant concentration was 18.0 g/100 ml of solution, and the solvent was carbon tetrachloride/toluene, 40/60 (v/v). Two volumes of polyol and one volume of diisocyanate solutions were mixed and poured into a 1-m column, 2.16 mm i.d. Polymerization was visible after 15 min, and unreacted starting materials were flushed out with the chromatographic solvent, 20% isopropanol in *n*-heptane, after 24 hr. This column was then tested in the linear conformation using 2,5-dichloraniline as a test solute ($k' = 0.48$) and 20% isopropanol in *n*-heptane as solvent. This same column, while still containing solvent, was coiled and tested, sequentially, in the following coil diameters (in inches): 12, 6, 4, 2.5, and 2. In no case could we detect a measurable change in elution time or peak width at the base for the linear column or the coiled columns. The flow rate was set at 0.54

ml/min. At this flow rate the pressure drop along this column was only 30 psi, indicating that very long columns, having a high efficiency and allowing high sample loads, can be utilized with only moderate pressure drops. Chromatograms at three representative coil diameters, 12, 6, and 2.5 in., are shown in Fig. 1. The number of theoretical plates calculated from these chromatograms is 470 ± 10 plates/m. The straight column operated under these conditions exhibited 467 plates/m. This effect, of exactly similar performance whether in the straight or simple coiled state, has been verified for other columns having this diameter. We have also examined the

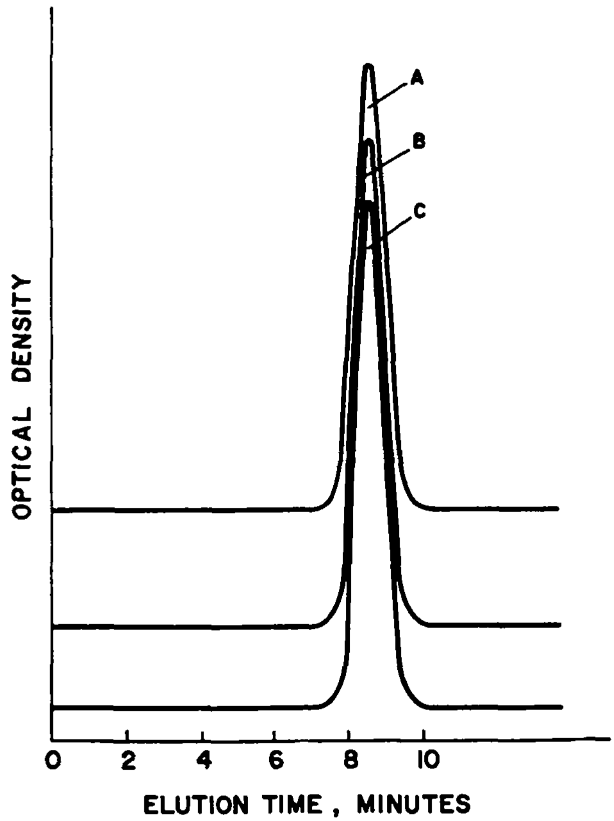

FIG. 1. Chromatograms of 2,5-dichloroaniline using column 1, an open-pore polyurethane packed column, coiled to various diameters: (A) 12 in., (B) 6 in., and (C) 2.5 in. Solvent: 20% isopropanol in *n*-heptane.

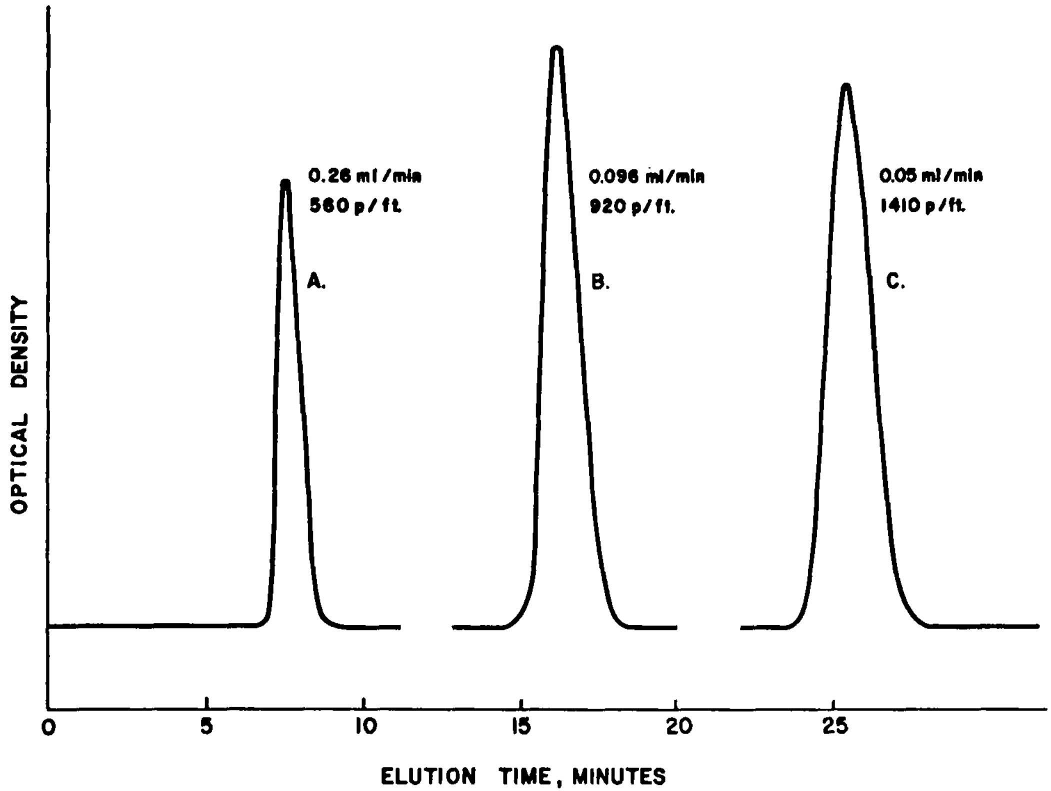

FIG. 2. The effect of flow rate on column efficiency for column 1, an open-pore polyurethane packed column, coiled to 2 in. diameter. Solute: 2,5-dichloroaniline. Solvent: 20% isopropanol in *n*-heptane.

effect of flow rate on the efficiency of this column when it is coiled in the smallest diameter used, 2 in. As shown in Fig. 2, the columns increase in efficiency as the flow rate is decreased in an manner exactly similar to the straight columns. No peak distortion is evident at any flow rate used.

The OPP columns consist of nonporous spheres which are chemically bonded together and also are firmly attached to the wall. Presumably this structure is able to accommodate the column coiling by compression and expansion without significantly changing the nature of the interstitial volume or the velocity flow profile.

Compact coiled columns offer tremendous advantages in liquid chromatography, particularly for long column construction and temperature controlled separations. For this reason we have investigated the effects of joining these columns in series. The results are shown in Table 1.

As may be seen from Table 1 for 2,5-dichloroaniline, the number of plates for the series combination of columns 2 and 3 is equal to the result of simply adding the values for the individual columns. This has been predicted to occur when the component columns have the identical values of the number of theoretical plates (*7*). However, for the case of 2,6-dichloroaniline the values of the number of theoretical plates is quite different for each column. In this case the following equation must be used to calculate the number of theoretical plates expected from the series combination (*7*):

$$n = \frac{(\Sigma V_i)^2}{\Sigma (W_{bi}/4)^2}.$$

TABLE 1

The Effect on the Number of Theoretical Plates of Joining Coiled OPP Columns in Series

Solute	Flow rate (ml/min)	k'	Number of theoretical plates, n		
			Column 2[a]	Column 3[b]	Series combination of columns 2 and 3
2,5-Dichloroaniline	0.55	0.48	289	308	603
2,6-Dichloroaniline	0.18	0.27	592	1010	1421

[a]2:1 ratio OH:NCO, 20 g/100 ml of solution.
[b]2:1 ratio OH:NCO, 18 g/100 ml of solution.

where V_i is the elution volume for column i for a given solute and W_{bi} is the base peak width for column i for a given solute.

Using this equation, the number of theoretical plates is calculated to be 1417 for the series combination, in good agreement with the observed value.

CONCLUSIONS

Open-pore polyurethane columns may be made compact by a simple coiling technique. Using 2.16 mm i.d. tubing, a coil diameter of 2 in. may be used with no loss of efficiency compared with the linear configuration. This has not been achieved before for this diameter column and a simple coil geometry with conventional packing materials. The unique property of the OPP being bonded to the column wall is believed to be responsible for this behavior. This prevents detrimental flow velocity profiles from developing, which appears to be the reason for decreased performance with conventional packings. These compact coiled columns may be joined together in series combination to achieve a high plate count system occupying a minimal volume. If the plate count for a given solute is equal for each column, the numbers of theoretical plates are additive. If the individual plate counts are different for a given solute for different columns, the plate count is less than the arithmetic total but may be calculated.

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