High-Throughput GPC Analysis of Phenolic Resin Using TSKgel SuperHZ Columns

TSKgel
APPLICATION NOTE

Abstract

Polymer-based TSKgel SuperHZ columns shorten analysis time and reduce mobile phase consumption for high throughput analysis of organic soluble polymers. The packing material consists of 3µm polystyrene divinylbenzene particles.

Introduction

Conventional gel permeation chromatography (GPC) is known for long run times, often requiring several columns in series to achieve the required resolution. Recently, there has been a push for a combinatorial-like platform for the high throughput analysis of a large number of polymer samples. TSKgel SuperHZ columns are now available in a 3µm particle size, providing equivalent resolution to conventional 5µm GPC columns in half the time.

Experimental Conditions

The performance of 3µm TSKgel SuperHZ columns was compared with that of 5µm TSKgel H_{XL} columns containing particles of the same chemical and physical composition. As TSKgel SuperHZ columns are available in either 4.6mm or 6.0mm inner diameter housings for decreased solvent consumption, flow rates were adjusted to ensure equivalent linear velocity as that obtained with 7.8mm ID conventional columns. Similarly, sample volume was proportionally adjusted to avoid volume overload on the narrower bore columns.

Results

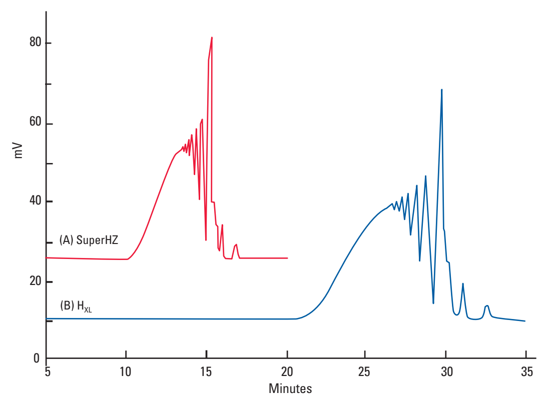
The link between efficiency and operational variables in chromatography was first described by van Deemter et al. in the 1950's and later refined by Giddings and, more recently, Knox. Column efficiency equations describe the various contributions to band broadening that take place when sample components travel through the column. The processes include (1) longitudinal diffusion, which plays a negligible role in practical HPLC, (2) dispersion of the sample band due to the velocity profile between particles, (3) dispersion due to the presence of the packed bed (often referred to as Eddy diffusion), which forces sample bands to go around the particles, and dispersion from resistance to (4) mobile phase and (5) stationary phase mass-transfer inside the particle. Except for longitudinal diffusion, terms (2) - (5) are all affected by particle size.

Smaller particle size columns have narrower flow paths between particles and the mean velocity in each flow path is lower, which results in less dispersion. Similarly, Eddy diffusion is reduced because of the shorter distance a solute has to travel to get around a particle. The smaller particle diameter also results in less time to traverse the stagnant mobile phase volume in the pores, which in SEC can be looked at as the stationary phase. The end result is that the sum of all dispersion processes leads to narrower bands, which the advantage, of course, is paid for at the price of higher column back pressure.

Researchers are given a choice when diffusion is minimized by smaller sized particles; either utilize the extra resolving power or shorten the analysis time by reducing column length. *Figure 1* shows the latter approach. Resolution of a phenolic resin sample on the TSKgel SuperHZ column(s) is the same as for the larger particle size TSKgel H_{XL} column(s), but this result was obtained in half the time.

The shorter column length of a TSKgel SuperHZ column, combined with a narrow bore, reduces solvent consumption and disposal cost. *Figure 2* shows the column efficiency versus flow rate relationship in a slightly different way. The efficiency of a small molecule separation on 3µm packing material achieves its optimum levels at higher flow rates than conventional particles. Additionally, optimal efficiencies are maintained over a greater range of flow rates than the 5µm material.

Figure 1. GPC Comparison of TSKgel SuperHZ and H_{XL}



Columns:	(A) TSKgel SuperHZ (4000, 3000, 2500) (4.6mm ID x 15cm x 3)
	(B) TSKgel H_{XL} (4000, 3000, 2500) (7.8mm ID x 30cm x 3)
Eluent:	THF
Flow rate:	(A) 0.35mL/min
	(B) 1.0mL/min
Temperature:	40°C
Detection:	RI
Sample:	Phenolic resin
Inj. volume:	(A) 5µL (B) 30µL

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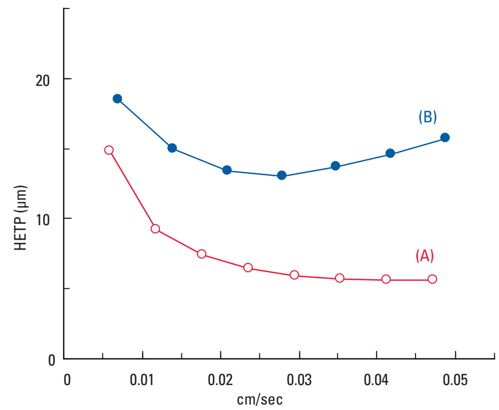
Figure 2. Comparison of the Effect of Linear Velocity on HETP in TSKgel SuperHZ2500 and TSKgel G2500HXL Columns



Columns:	(A) TSKgel SuperHZ2500 (6.0mm ID x 15cm)
	(B) TSKgel G2500H_{XL} (7.8mm ID x 30cm)
Eluent:	THF
Temperature:	25°C
Sample:	dicyclohexyl phthalate (DCHP)
Inj. volume:	(A) 2µL (B) 20µL

Conclusion

TSKgel SuperHZ columns demonstrate excellent resolution in less time than conventional 5µm columns, which makes these columns ideal for high-throughput GPC applications.

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TOSOH Bioscience LLC
3604 Horizon Drive, Suite 100
King of Prussia, PA 19406
Orders & Service: (800) 366-4875
Fax: (610) 272-3028
www.separations.us.tosohbioscience.com
email: info.tbl@tosoh.com

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