Columns for High Temperature Liquid Chromatography: Present Status

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Introduction

Temperature is an overlooked variable in most HPLC separations. Performing HPLC analyses at higher temperatures results in faster and more efficient separations. Instrumentation that allows isothermal and temperature programmed HPLC at temperature up to 200° have been available for several years, however the selection of columns that can withstand those temperatures are limited.

Most people think of Zirconia based stationary phases for high temperature work, but because of excessive column bleed they are not good candidates for for temperature programmed HPLC.

Introduction

Polymeric stationary phases based on polystyrene-divinylbenzene (PSDVB) and graphitic carbon have been used successfully for temperature programmed HPLC at elevated temperatures (1).

This work evaluates a number of polymeric stationary phases that are based on divinylbenzene (DVB). Three columns were evaluated: a DVB column, a DVB column with a C_{18} bonded phase, and a hydroxylated DVB column.

Experimental

Columns and their characteristics are summarized in Table 1. Each column was tested using a 50:50 acetonitrile:water mobile phase with a three component test mix consisting of uracil, phenol, and benzene at 50°C before being exposed to high temperatures. The retention time, theoretical plates, peak width, and asymmetry were recorded. Next, each column was flushed with 1000 column volumes of 50:50 acetonitrile:water at 1.0 mL/min starting at 100°C. This was repeated at 20° intervals until the column showed signs of stationary phase degradation. After each high temperature exposure, the column was cooled to 50°C and the three component test mix was analyzed, then the same parameters (retention time, etc) were recorded. All three columns were evaluated in this manner.

Table 1.
Jordi Columns Evaluated

Column	Packing	Particle Size (micron)	Pore Size (Angstroms)	Dimensions (mm)
DVB	DVB divinylbenzene	5	300	100 x 4.6
C_{18}	DVB bonded with C_{18}	5	500	100 x 4.6
Hydroxylated (OH)	Hydroxylated DVB	5	500	100 x 4.6

Results and Discussion

Figure 1 shows the change in theoretical plates of the Jordi DVB column with temperature. Figure 2 shows the change in theoretical plates with temperature for the Jordi DVB C_{18} column, and Figure 3 shows the change in efficiency with temperature with the Jordi hydroxylated DVB column.

All three columns show good efficiency until a temperature of 160°C. At this temperature, there is a significant drop in theoretical plates. This was also accompanied by a decrease in retention time. This was taken as an indicator that the stationary phase had suffered some damage. Further study indicated that the columns were stable to 150°C.

Figure 1.

Effect of Temperature on Efficiency of Jordi DVB Column

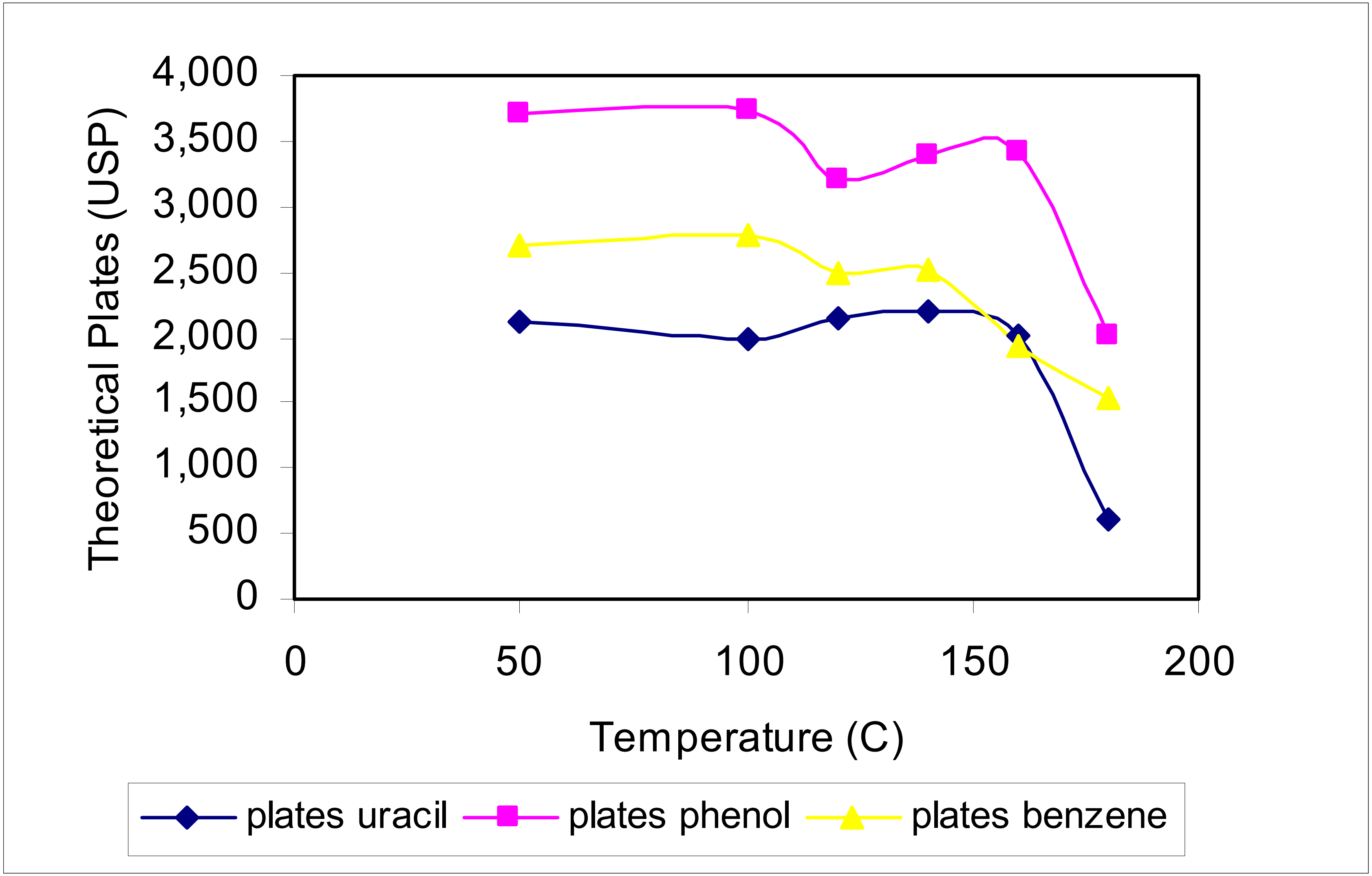


Figure 2.

Effect of Temperature on Efficiency of Jordi DVB C_{18} Column

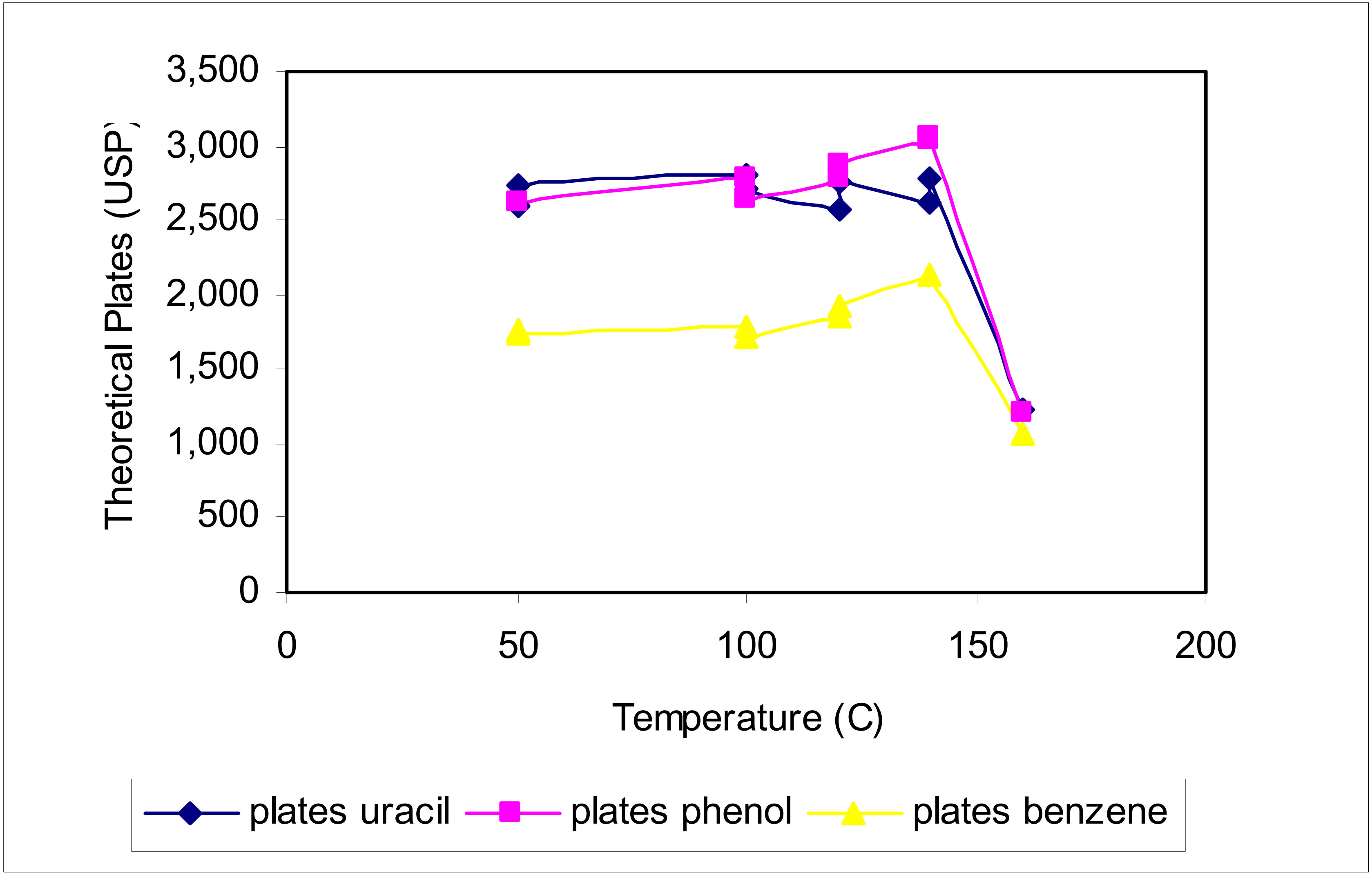
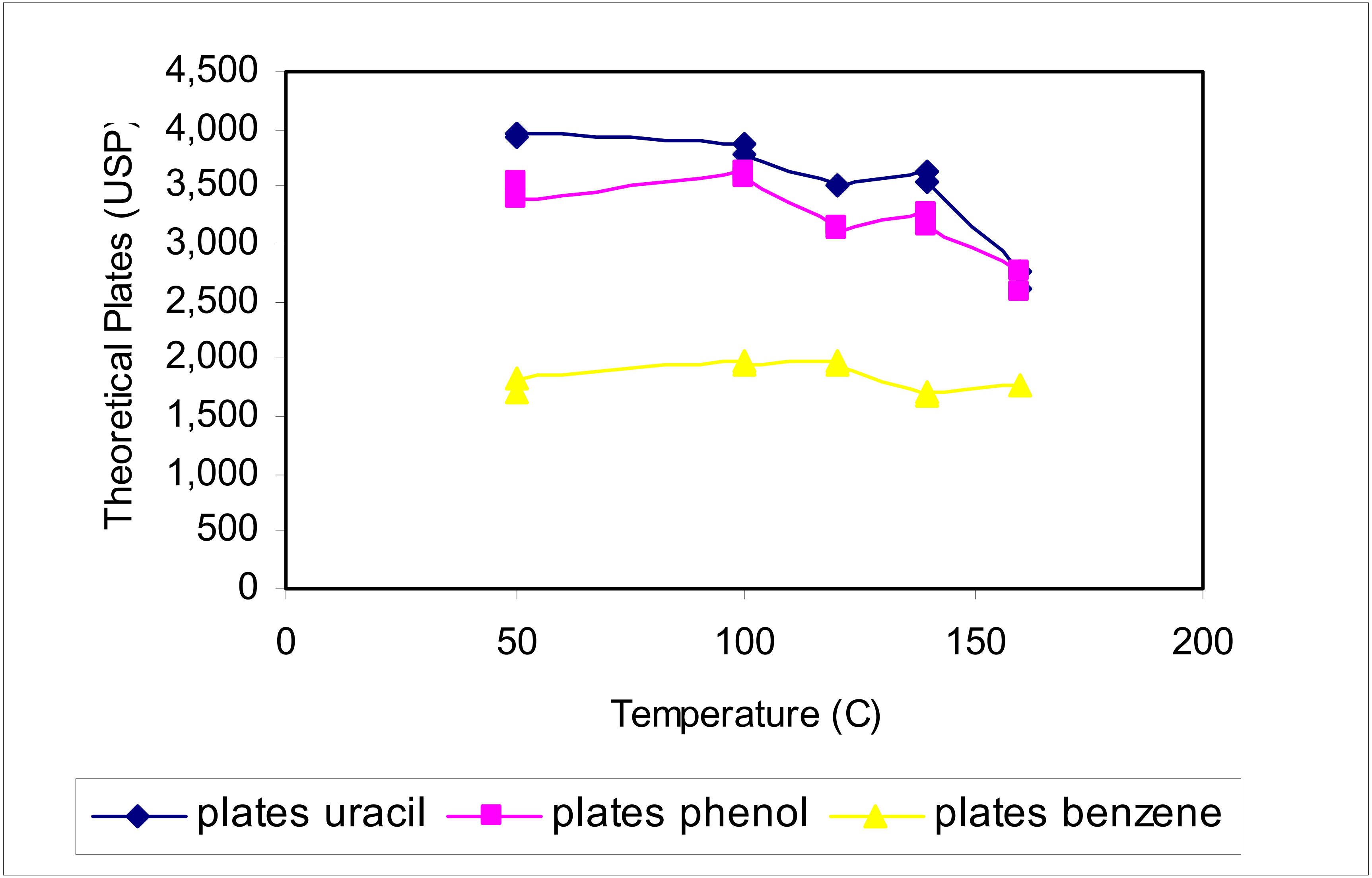


Figure 3.

Effect of Temperature on Efficiency of Jordi DVB OH Column



Conclusions

- Stationary phases based on divinylbenzene (DVB) can be used at temperatures up to 150°.

Acknowledgements

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References

1. S J Marin, et al, *J Chrom A*, **1030**, 255-262, (2004).

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