TOWARDS A SOLUTION FOR VISCOUS HEATING IN ULTRA-HIGH PRESSURE LIQUID CHROMATOGRAPHY

K. Broeckhoven (1), J. Billen (1), M. Verstraeten (1), K. Choikhet (2), M. Dittmann (2), G. Rozing (2) and G. Desmet (1)

(1) Vrije Univ. Brussel, Dep. of Chemical Engineering, Pleinlaan 2, 1050 Brussels, Belgium, kbroeckh@vub.ac.be
(2) Agilent Technologies, Hewlett-Packard-Str. 8, 76337 Waldbronn, Germany

The recent years have seen the development of chromatographic instruments that can operate at pressure up till 1200 bar. There have however been no solutions found to resolve certain fundamental problems occurring during the operation at ultra-high pressures, despite numerous publications investigating the problem. The research into the high pressure effects on the chromatographic conditions, however managed to identify and quantify these important drawbacks of the use of ultra-high pressures.
The most important fundamental effect (not taking into account limitations on instrumentation and column packing) that limits the applicability of even higher operating pressure in LC, is the occurrence of viscous heating due to the dissipation of the viscous forces in the liquid when passing through the column bed.
The generation of heat results in an increase in mobile phase temperature along the column axis. Removing this heat to the environment through the column wall is not applicable since this will result in unacceptable loss of column performance [1]. On the other hand, removing the heat only with the eluting solvent (adiabatic operation) is also limited if one wants to keep the increase of solvent temperature within acceptable limits, since it can easily top 60°C at 2000 bar operating pressure for organic solvent. Operating the column in still air conditions will reduce the temperature increase by 25-50%, but this will result in significant loss of column performance due to radial velocity biases in the column bed [2]. Even in adiabatic conditions, the backflow of heat through the column wall of a single column will introduce a similar effect.
The proposed novel way to counteract the effect of viscous heating consists of separating the column bed in separate columns and connecting these through thin capillaries which are actively cooled to the temperature of the incoming liquid in the first column. This results in a limited pressure drop over each column, reducing the maximal increase in temperature due to the intermediate cooling. In addition, the amount of heat transported from columns ends to front is reduced and the remixing of liquid after each column reduces the presence of radial concentration differences due to any occurring radial velocity differences.

[1] A. de Villiers, H. Lauer, R. Szucs, S. Goodall, P. Sandra, J. Chromatogr. A 1113 (2006) 84-91.
[2] K. Broeckhoven, K. Choikhet, G. Rozing and G. Desmet, Poster presented at the 32nd International Symposium on High Performance Liquid Phase Separations and Related Techniques, Baltimore, MD, USA, P03.25, 10-16 May 2008.