

ORIGINAL PAPER

Performance and lifetime of slurry packed capillary columns for high performance liquid chromatography**Martin Franc, Jiří Vojta, Jana Sobotníková, Pavel Coufal*, Zuzana Bosáková***Charles University in Prague, Faculty of Science, Department of Analytical Chemistry,
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Fused silica capillary columns of the internal diameter of 320 μm were packed with the Nucleosil C18 stationary phase of 5 μm using the slurry packing method. The time of the bed compaction phase, packing pressure, and the use of ultrasound varied to study their influence on the column performance. Van Deemter curves were measured and separation impedance values were calculated in order to assess both separation efficiency and kinetic performance of the columns. Selected columns were tested again after nine months to evaluate the stability of their beds. Separation efficiencies of all columns were similar, but a major difference, caused by the use of ultrasound, was observed in the bed stability. Columns sonicated for 25 minutes during the bed compaction phase exhibited unchanged performance in the course of several months, while the performance of non-sonicated columns decreased.

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Micro separation methods, being environmentally friendly and requiring only a sample volumes of tens of nanoliters to analyze, are extensively used these days. Although a wide selection of capillary columns for HPLC is now commercially available, preparation of own columns has still advantages, especially within the academic environment. For instance, self-packed capillary columns can be customized to study a given separation problem and the costs of such a column can be substantially lower than those of a commercial one. Packing of analytical columns can be simply described as a filtration of stationary phase particles on the column's outlet frit. An empty column is connected to a reservoir filled with the stationary phase particles. The particles are then pushed into the column by a so called packing phase and retained inside by the outlet frit. As a packing phase, gas (Crescentini et al., 1988), supercritical CO_2 (Robson et al.,

1996; Roulin et al., 2000) or liquid (Keller et al., 1977; Bristow et al., 1977; Borra et al., 1987; Ehlert et al., 2008a) can be used. After the column is filled, the obtained stationary phase bed is compacted by rinsing with a suitable solvent in order to achieve a more stable and efficient column. The simplest and thus the most attractive technique is packing by liquid. The stationary phase is in form of a slurry in the reservoir; therefore, this technique is usually called slurry packing. This method is widely used for column preparation and it is also possible to employ it to pack an HPLC chip (Xie et al., 2005; Yin et al., 2005; Lazar et al., 2006; Gaspar et al., 2007; Ehlert et al., 2008b; Jung et al., 2009). This, however, requires special equipment as well as an empty chip, which usually has to be self-manufactured. On the other hand, the only equipment required for capillary column packing is an isocratic pump and an empty HPLC column serving as the slurry reservoir. However, even in this simple setup, there is a number of variables

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