CONTROLLED UV PHOTO-INITIATED FABRICATION OF MONOLITHIC POROUS LAYER OPEN TUBULAR (MONOPLOT) CAPILLARY COLUMNS FOR CHROMATOGRAPHIC APPLICATIONS

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Porous layer open-tubular capillary columns (PLOT) possess a porous layer of stationary phase covering the inner surface of the capillary tubing, preserving an open-tubular structure after the completion of all column preparation steps. One of the main difficulties in the fabrication of UV-initiated monolithic PLOT (monoPLOT) columns is achieving a uniform porosity in the monolith, or across the porous layer. In most commercially available UV reactors, the light source is commonly located on the top of the oven, thereby providing UV light from one direction only. Such ovens rely heavily on diffuse reflection and scatter within the oven to effectively polymerise the target. For such UV reactors, it has been shown that the layer thickness and polymer density of the monolith are higher on the side of the column facing the light source[1].

In the work presented herein, an automated column fabrication technique, based upon a UV LED array oven, and providing precisely controlled 'in-capillary' UV initiated polymerisation at 365 nm, is presented for the production of monoPLOT columns of varying length, I.D. and porous layer thickness. Through controlled UV power and exposure time, the developed approach allows the preparation of columns of different length, due to an automated capillary delivery approach, providing precisely controlled and uniform layer thickness and monolith morphology. The relationships between direct exposure times, intensity and layer thickness were determined, as were the effects of capillary delivery rate (indirect exposure rate), and multiple exposures on the layer thickness and axial distribution. Layer thickness measurements were taken by scanning electron microscopy (SEM), with the longitudinal homogeneity of the stationary phase confirmed using scanning capacitively coupled contactless conductivity detection (sC^4D). The new automated UV polymerisation technique presented in this work allows the fabrication of monoPLOT columns with a very high column-to-column production reproducibility, displaying a longitudinal phase thickness variation within ± 0.8% RSD.

[1] Eeltink S. et al., Electrophoresis 27 (2006) 4249