Study on Packing Structures in Liquid Chromatography (LC) Columns Based on X-Ray CT

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Introduction

It is well known that the efficiency of LC-columns strongly depends on the homogeneity of the packed bed. Measurement of local system parameters related to the column cannot be achieved easily without the deterioration of the flow field, as the separation takes place inside closed columns. CT provides the opportunity to measure local packing properties in a non-destructive way. Axial compression columns packed with different Silica-Gels were investigated with a CT-Scanner at several axial positions. Potassium iodide (KI) solutions in Methanol were conveyed through the column. The high X-Ray attenuation of KI rendered the determination of intra-column breakthrough curves possible. By fitting the Equilibrium-Dispersive-Model (EDModel) to the experimental, local breakthrough curves, information about the spatial variation of the column efficiency and the velocity distribution was obtained. The Kozeny-Kármán-equation allowed to estimate local interstitial void fractions. It was found that the packing can be divided into a homogeneous core region, where porosity and efficiency vary only slightly and into a wall region with decreasing porosity and efficiency, respectively. The results in terms of local porosities and dispersion coefficients were then used to develop CFD-Models of the investigated columns. The simulations were performed assuming a rotational symmetry (2D-Model) of the flow field in order to keep the computational effort reasonable. Work in progress covers the impact of different packing materials, e.g. spherical and irregular materials and different packing modes upon the packing properties. The final aim is to get a better understanding of the packing structure and its dependency on different packing parameters.

Theory of Computed Tomography

The ability of x-rays to pass matter is the basic principle of X-ray CT. While passing through an object the beams are attenuated, the attenuation is material dependent. This can be expressed by the law of Lambert-Beer.

$$\ln\left(\frac{I}{I_0}\right) = -\int_0^L \mu(x,y)dz \qquad (1)$$

The attenuation of the beams after passing the column is monitored from different directions. This allows for the reconstruction of an image of the column cross section. The different grey-shades represent different local attenuation coefficients or CT-Numbers, respectively. From this image the information about the spatial variation of the local void fraction or local saturation can be extracted.

Experimental Setup

The setup used for experiments including all peripherals is depicted in figure 1.

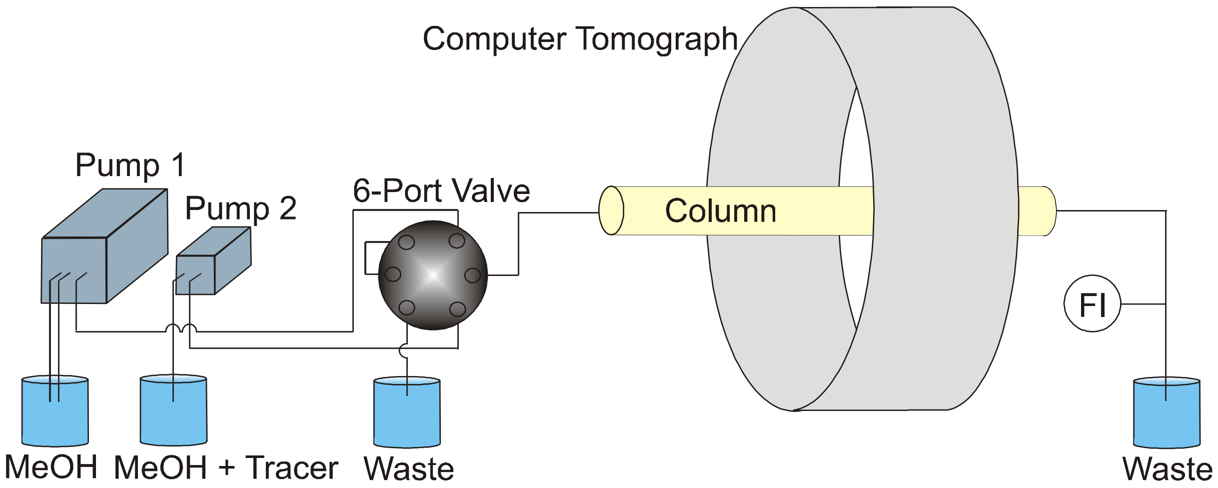

Figure 1: Experimental setup

Two HPLC pumps conveyed either pure solvent (Pump 1) or a solvent/tracer mixture (Pump 2). A six-port valve made a fast interchange between the two different mobile phases possible. As the column was fixed on the moveable patient table, the scan of different axial positions of the column could be achieved easily. A non-polar hydrophobic ODS phase was used, in order to prevent interactions between the ionic tracer and the stationary phase. The columns were made of glass and included in a plastic jacket, for the reason that steel columns cannot be used in X-ray CT without severe difficulties. The scanner used, was a medical Siemens Somatom Plus Scanner.

The breakthrough fronts of the tracer at different axial positions were then monitored online. Afterwards the column was equilibrated with pure solvent again. The influence of several parameters, like packing pressure or solid phase particle shape on the chromatographic systems was studied.

Results

The breakthrough of tracer at one axial position in a 50 mm inner diameter column is shown in figure 2. One can clearly see that the breakthrough has not necessarily to occur exactly in the center of the column, which is caused by different hydrodynamic effects. Afterwards breakthrough curves inside the column could be calculated from the CT-Images. These experimental values were then fitted to the commonly used Equilibrium-Dispersive-Model. The fitted parameters were the retention time t_0 and the Peclet number, which describes the ration between convection and dispersion. This can be seen in figure 3.

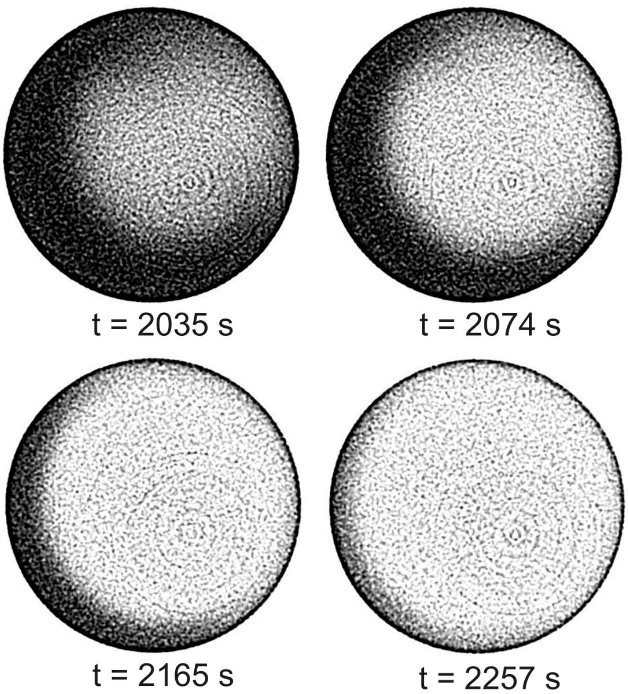

Figure 2: Intra column tracer breakthrough

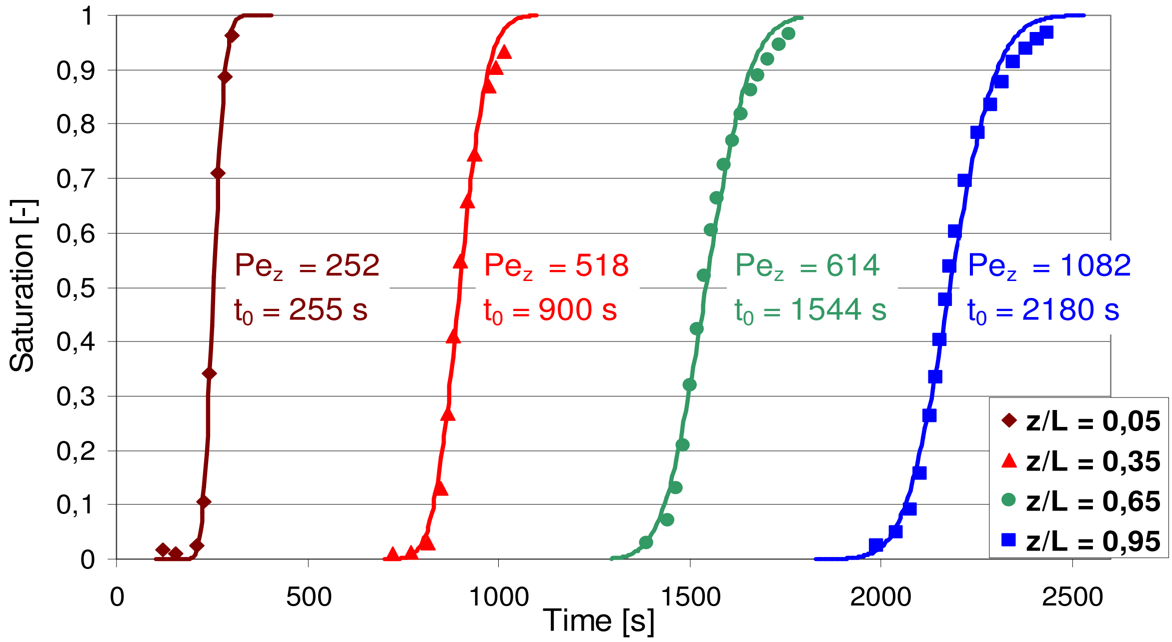

Figure 3: Experimental (points) and fitted breakthrough curves (lines) at different axial positions z/L

The EDM describes the experimental findings quite well. An exception from this is found at the end of the graphs, which corresponds to the outer region of the column cross section, i.e. the column wall. As the column packing is often described as not homogenous in this region [1], this might be one reason for the deviation from the idealised model.

Conclusion

It was shown that X-ray CT is a reasonable method for investigating the packing structure of adsorption columns in a non-invasive way. The necessary experimental setup proved to be straightforward and the data acquisition was achieved easily compared to other methods [2]. Evidence was found, that the packing of LC-columns is not necessarily homogenous, but can be divided in a core and a wall region.

References

[1] G. Guiochon et al.: J. Chromatogr. A 762 (1997) 83-88

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