Pillar-structured microchannels for liquid chromatography

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Summary

Liquid Chromatography is perhaps the most widely used technique in analytical chemistry. Most of the analytical laboratories worldwide have a chromatographic apparatus, and the resources involved with chromatography are huge. Pharmaceutical, and biotechnology industries, environmental analysis, food analysis, are just few of the important examples where liquid chromatography is an essential tool. Every single small technological improvement in this technique would potentially yield in massive benefits in terms of resources saving at a global level. The efforts in improving liquid chromatography are reflected in some of the evolutions of this technique. For instance, the use of multidimensional separations to resolve particularly complex mixtures; the development of monolithic stationary phases to improve the permeability to flow of the chromatographic column; the successful commercialization of UPLC to improve chromatographic resolution and reduce solvent and total analysis time. Continuous studies on the development of new more selective stationary phases are part of the same scenario.

One approach is to use microfabricated chromatographic supports, instead of using a disordered packing material or monolithic media. The main advantages are related to the higher homogeneity which leads to higher resolution and less flow resistance with respect to the traditional supports.

The main target of this work is to perform a fundamental study from an experimental standpoint on the possibility of performing pressure-driven separation in channels structured with micropillars.

In Chapter 1 the concept and the mechanisms of liquid chromatography are introduced. The relation between band dispersion phenomena and resolution power of a chromatographic system is briefly reviewed. The correlation between stationary phase support spatial homogeneity and band dispersion is explained, and the possibility of increasing the homogeneity of the chromatographic support via microfabrication is described.

Chapter 2 gives a review on the design, development and use of micromachined devices for separations in the liquid phase, where the presence of topographic structures
or chromatographic supports was essential for the separation. This overview is not limited solely to chromatography, but also considers separation based on other principles.

In Chapter 3 the band dispersion measurements of unretained species inside microchannels structured with cylindrical pillars are given. The measurements were performed on silicon devices. The size of the cylindrical structures (5 and 10 µm) and the overall porosity (roughly 40%) were comparable to the correspondent parameters of standard packed columns. By comparing the band dispersion of unretained compounds in the microfabricated device and columns packed with non-porous spherical particles, the substantial reduction of band dispersion gained with the use of homogeneous supports was clearly demonstrated. In this part also the detrimental effect of the band dispersion occurring in the side-walls of the channels was observed and described.

In chapter 4 the chromatographic permeability for microfabricated channels structured with non-porous cylindrical and diamond-shaped micropillars was measured. The channels tested have different voidance values. The superior permeability of homogeneous microfabricated structures in comparison with the packed columns was experimentally proven. Extremely low chromatographic permeability values, unmatched by packed or monolithic columns, could be measured. Based on the data available, some predictions are given of the performances possibility offered by the microstructured channels.

A normal phase separation in the microfabricated channels is reported in Chapter 5. The bare surface of the (oxidized) silicon pillars could be used as stationary phase. The separation of the three fluorescent markers was extremely fast in spite of the low retention factors of the molecules. The band dispersion observed for the retained compounds was comparable to that for the unretained peak at moderate and low flow velocities.