Design and fabrication through additive manufacturing of devices for multidimensional LC based on computational insights

Adamopoulou, T.

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Chapter 6

Future Outlook
Abstract
This chapter provides a discussion on the challenges on the way towards a peak capacity of a million, as well as some of the obstacles faced during this study. The main challenges include obtaining suitable devices for two- and three-dimensional spatial liquid chromatography, flow control, stationary-phase materials, orthogonality and detection. Only the first two aspects were examined in this thesis. The remaining aspects will be included in the next volumes of the STAMP series. Finally, a few suggestions for future research and promising options are included.
Chapter 6

Current and future challenges

The challenges along the way towards a peak capacity of one million include the need for suitable devices, flow control in each compartment, incorporation of stationary-phase materials inside these devices as well as their localization within the desired compartment, orthogonal retention mechanisms, and detection. In Fig. 6.1 the aforementioned aspects are presented, along with some subsections of investigation. In this book the first two aspects are addressed, while the remaining challenges will be addressed in following volumes.

**Fig. 6.1.** Schematic representation of the challenges to be addressed within the STAMP project.

In Fig. 6.2 the challenges addressed in this thesis, viz. devices for two- and three-dimensional liquid chromatography and flow control in these types of devices, are shown as well as the studied aspects per category.
Fig. 6.2. Schematic representation of the aspects addressed in this volume.

Regarding the devices, suitable concepts and designs had to be produced. The design process involved an iterative cycle that included designing, simulating, fabricating and testing. Different arrangements were created, both single-piece and modular devices. Examples of single-piece devices are those studied in Chapter 2, while the assembly presented in Chapter 3 is modular. Another example of a modular device is the SLIT (Simple Liquid Transfer), which is described in Chapter 5 (see Fig. 5.11). In order to bring these designs to life, 3D-printing was used as fabrication method. Different 3D-printing techniques can be used and within the STAMP project selective laser melting, fused-deposition modelling and stereolithography were used for the creation of the devices bodies. The majority of the devices described in this thesis were manufactured using stereolithography, while examples of selective laser melting include the freeze-thaw valve (see Fig. 6.3) [1] and a titanium 3D-printed device for studying the localized thermal polymerization of monolithic stationary-phases [2].
Another aspect in the context of fabrication is the suitability of materials. Desired properties are solvent compatibility and pressure resistance. In Fig. 6.4, pieces with minimal, medium and severe damage caused by solvents are depicted. Developing chemically inert and pressure resistant materials form an ongoing effort in other groups and developing new materials was not an explicit goal within our project. 3D-printing material solutions that may also be used in the future in the field of separation science include 3D-printing of glass [3], 1H,1H,6H,6H-Perfluoro-1,6-hexyl diacrylate (PFHDA) resins [4] and polyethylene glycol diacrylate (PEG-DA) [5].

**Fig.6.4.** Pieces with minimal (A), medium (B) and maximum solvent damage.
Another desired aspect concerns the miniaturization to micro-scale features. With the available 3D-printing techniques it is possible to fabricate devices with features down to 100 μm. So far, each effort to increase printing resolution has resulted in an increase in the printing time. A novel solution was proposed within the STAMP project by Dr. Suhas Nawada, the so-called hybrid stereolithography ("Stereolithographic 3D-printing assembly and stereolithographic 3D-printing method" – patent pending). This technique is a combination of stereolithography and photomasks. Depending on the layer that is projected from the printer, a photomask with features is being used to block the UV light at specific locations, creating minimal features down to 5 μm or possibly less, while the printing time is no longer than for a device with larger features. This type of printing may feasibly be used to fabricate devices with really small features, potentially incorporating frits or even 3D-printed stationary phases. In this case one can anticipate that the entire device (housing and stationary phase) can be fabricated directly. That would be highly favorable not only from a fabrication, but also from a chromatographic point of view. It would enable relatively fast printing of optimally ordered structures for analytical-scale devices, heralding a new era in chromatography. Advantages of devices with optimally ordered stationary-phases have been demonstrated by pillar array columns [6] and by 3D-printed columns with homogeneous packing (preparative scale) [7].

To study the performance of fabricated devices, flow tests and pressure tests were performed to assess the printability, band broadening and pressure resistance. An example of a device used in pressure testing is shown in Fig. 6.5. The evaluation of the separation efficiency will be assessed in a future volume.

Fig. 6.5. Device before (left) and after (right) a pressure test.
Concerning the subject of simulations, computational fluid dynamics were employed to assess design and chromatographic aspects of spatial multi-dimensional devices. The chromatographic aspects included band broadening, analyte transfer between dimensions and flow confinement for each dimension. The aspect of flow distribution in $^{3}$LC×$^{3}$LC has previously been assessed in [8]. Future prospects regarding the design and simulation of devices include shape optimization, which may be used to reduce dead zones created at the corners of the devices, without interfering with the velocity profile, while in some cases even improving the performance of the device (see Fig. 6.6).

![Velocity-magnitude contour plots from 90° angle corners (top) to modified design with topological improvements (bottom).](image)

Additionally, other factors affecting the separation performance may also be first assessed through simulations. Examples may be the effect of fabrication inaccuracies in terms of flow-distribution and band-broadening or the use of an external on-chip temperature gradient, similar to the procedure described in [9]. The latter arrangement may be fabricated via multi-material 3D-printing, for example using inkjet or fused deposition modelling printing.

**Epilogue**

The aim of the work reported in this thesis was to improve the existing design guidelines and procedures for the creation of devices for spatial two- and three-
dimensional Liquid Chromatography as well as other applications in Analytical Sciences. Future volumes will target the aspects of retention mechanisms and separation optimization, materials for 3D-printing and stationary phases, performance of 3D-printed devices for multi-dimensional LC, and detection methods and concepts for spatial multi-dimensional LC.

References