



Investigation, comparison and design of chambers used in centrifugal partition chromatography on the basis of flow pattern and separation experiments[☆]



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ABSTRACT

In centrifugal partition chromatography (CPC) the separation efficiency is mainly influenced by the hydrodynamic of mobile and stationary phase in the chambers. Thus, the hydrodynamic has to be investigated and understood in order to enhance a CPC separation run. Different chamber geometries have been developed in the past and the influence of several phase systems and CPC operating conditions were investigated for these chambers. However, a direct comparison between the different chamber types has not been performed yet. In order to investigate the direct influence of the chamber design on the hydrodynamic, several chamber designs – partially similar in geometry to commercial available designs – are investigated under standardized conditions in the present study. The results show the influence of geometrical aspects of the chamber design on the hydrodynamic and therewith, on the separation efficiency. As a conclusion of the present study, some ideas for an optimal chamber design for laboratory and industrial purpose are proposed.

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1. Introduction

Counter current chromatography (CCC) is a kind of liquid–liquid chromatography, where two immiscible liquid phases are used as stationary and mobile phase. Thereby, the separation mechanism is based on differences in the sample component's distribution behavior between both liquid phases. Thus, when two components are injected into a CCC system, the component with a higher distribution to the stationary phase will elute later than a component with a lower one [1].

In order to immobilize one liquid phase as stationary phase, while the other liquid phase is pumped through as mobile phase, two different approaches using centrifugal forces have been developed. In hydrodynamic CCC, invented by Ito [2] in 1966, a coil of (Teflon) tubing rotates in planetary like movement around a central sun axis and its own planetary axis. The resulting centrifugal force field varies in intensity and direction time-dependently [3]. Hence, “undefined” zones of immobilized stationary phase are

generated in the tubing. In hydrostatic CCC, better known as centrifugal partition chromatography (CPC) and used in this study, the liquid stationary phase is immobilized in several chambers placed on a rotor with a single central axis of rotation, generating a time-independent centrifugal field. The chambers on the rotor are connected by ducts to form a chamber cascade. Hence, the mobile phase is pumped in at one side of the chamber cascade and leaves it at the other end, while the stationary phase is kept in the chambers [3]. Thus, “clearly defined” zones of immobilized stationary phase are present in each CPC chamber. Inside the chambers a specific hydrodynamic is established with regard to the flow of mobile phase through the stationary phase and the circulation of stationary phase itself in the chambers. Understanding the hydrodynamic is important, as it directly influences the separation efficiency in CPC. For example, the contact between both phases and thus, the mass transfer at the interfacial area generated can be enhanced by a fine dispersion of the mobile into the stationary phase. At the chamber outlets in turn, a fast separation of both phases is necessary to retain the stationary phase in the chambers in order to receive high resolution and capacity during a CPC separation run. Hence, the hydrodynamic behavior of a phase system in the CPC chambers at certain operating parameters needs to be known or predictable for each separation task to yield high performance.

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The hydrodynamic in CPC itself is influenced by the following [3,4]:

- (1) Physical properties of the phase system, such as densities, viscosities and interfacial tension.
- (2) Operating parameters like rotational speed of the rotor, volume flow of mobile phase and mode of operation – i.e. either upper or lower phase is used as mobile phase.
- (3) Chamber parameters like shape, size, volume, and material of chambers and channels.

The influences of solvent system's physical properties and CPC operating parameters on the hydrodynamic and separation efficiency in CPC have widely been investigated within several studies. For example, Armstrong et al. [5] used van Deemter-type plots to demonstrate the influence of operating conditions like rotational speed and volume flow of mobile phase on the separation efficiency with CPC. He found maximum separation efficiency at either very low or high mobile phase volume flow, what is contrary to common gas or liquid chromatography were usually a maximum at one specific flow rate exists only. A connection between the resulting separation efficiency and the hydrodynamic behavior like dispersion and stationary phase retention was drawn by Foucault et al. [6] using a Stokes model in dependency of the operating parameters. Thereby, the mobile phase was considered as moving droplets in the stationary phase. The stability of a phase system, namely the ability of the stationary phase to be retained in the chambers, was correlated to the physical properties, which were varied in a wide range in his study. First pictures and a description of characteristic flow regimes under variation of operating conditions and phase system's properties were presented by van Buel et al. [7]. Marchal et al. [8] continued this work and Adelman et al. [9] used images in combination with computational fluid dynamic (CFD) simulations to further investigate the hydrodynamic and the selection of suitable operating conditions [10,11]. Considering the importance of CPC chamber geometry and size, several different designs have been developed in the last decades: After the first cartridge-type CPC built by Sanki Engineering Co. Ltd. (Kyoto, Japan) and presented in 1982 by Murayama et al. [1], the FCPC (Fast CPC) chamber was patented by de La Poype et al. [12] probably guided by the first flow visualization results. Additionally, Foucault et al. [13] introduced the TwinCell design that is frequently used nowadays. Further approaches of chamber design are the asymmetric shaped TwinCells, which are particularly designed for use of slow coalescing solvent systems like aqueous-two-phase systems (ATPS) [14], and the Partitron 25 by Margraff et al. [15] for large scale application. Recently, the aspect of chamber design seems to be considered by ROTACHROM Technológiai Kft. (Magyarország, Hungary) using CFD simulations. Although all chamber types have been investigated intensively, these studies have been performed under non-uniform conditions, e.g. varying phase systems, operating conditions and apparatus types. Thus, a direct and useful comparison between the different chambers has not been performed yet. Furthermore, a direct comparison between these state-of-the-art and new designed chambers is not easily feasible, since all chambers vary in size and amount of chambers on a rotor. Therefore, it is unknown which kind of chamber in dependence of the operating conditions will be most efficient for a specific separation task.

That's why the influence of the chamber geometry and the resulting hydrodynamic and separation performance is investigated in this study. Based on the results gained from this and earlier studies [9–11,16], new ideas for a chamber design were phrased and based on that a customized chamber was built. This chamber was compared to the FCPC design and two kinds of TwinCell chambers. To ensure comparability of hydrodynamic and separation

experiments for the different chambers investigated, all experiments were performed under unified conditions, e.g. same phase system and sample compounds. All chamber types were reconstructed by the same production method and from same material and run in the same CPC device. Furthermore, reference values for the comparison of different chambers independently from their volume and number were developed.

2. Materials and methods

2.1. Phase system and sample compounds

The Arizona N system composed of n-heptane, ethyl acetate, methanol and water (1:1:1:1, v/v) was used for all experiments presented. Organic solvents were purchased in analytical grade from VWR International (Radnor, PA, USA) and deionized water was used. The system's upper phase was always used as stationary phase and the lower phase as mobile phase, i.e. the descending mode of the CPC was selected. Methylene blue (Merck KGaA, Darmstadt, Germany) was used for dyeing the mobile phase and as tracer compound within sample injection. (+)-Carvon (Sigma-Aldrich, St. Louis, MO, USA), that is a compound of the GUESS-Mix according to Friesen and Pauli [17], was used as sample compound for the separation experiments. The partition coefficient that is defined as the quotient of equilibrium concentration in upper and lower phase was measured in 15 mL falcon tubes. Concentrations in both phases were analyzed by gas chromatography (type: Agilent Technologies 7890 A; Santa Clara, CA, USA).

2.2. CPC experiments

As CPC device, a FCPC[®] by Kromaton (Annonay, France) was used. Originally the device was equipped with a separation rotor with 1320 chambers and 200 mL total volume. For all experiments within this study, this rotor was replaced by a single disc rotor with a certain chamber design to be investigated. The single disc rotors were constructed as described in earlier studies [9–11,16]. The peripheral setup does not differ substantially from these publications, with the exception of two additional pressure indicators (type: dTrans p30, 0–25 bar; JUMO GmbH & Co. KG, Fulda, Germany), one at the inlet and one at the outlet of the CPC, to measure the pressure drop in the CPC device. To decrease the detector noise, the temperature of the CPC flow out was changed before UV-detection, as described in Schwienheer et al. [16]. Therefore, a second water bath (JULABO GmbH, Seelbach, Germany) with a coil of steel tubing was used, through which the CPC flow out was pumped before entering the detector. As shown in [16], where aqueous two-phase systems (ATPS) were used, a cooling device in front of the detector decreased the detector noise. For the aqueous–organic system used in the present study, it was found, that a similar improvement in detector signal was achieved when heating up the CPC flow out before entering the detector. This can be explained by the temperature dependency of the miscibility gap of aqueous–organic systems, which, contrary to ATPS, mostly decreases with increasing temperature. Even if the system in a CPC chamber is in hydrodynamic equilibrium (i.e. in a measurable range) a small amount of stationary phase will probably be discharged, e.g. within a micro emulsion of stationary in mobile phase. This does not significantly change the stationary phase retention during the experimental time and a significant change in stationary phase retention was never observable with the online images received during the experiments. However, the micro emulsion will increase the detector noise. By heating up the liquid flow out to 40 °C, it was possible to decrease the detector noise and improve the quality of the recorded chromatograms and thus, their evaluability.

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