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Systematic optimization and experimental validation of ternary simulated moving bed chromatography systems

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ABSTRACT

Over the past several decades, many modifications have been proposed in SMB chromatography in order to effectively separate a binary mixture. However, the separation of a multi-component mixture using SMB is still one of the major challenges. Recently, a computational study was performed which compared various existing isocratic ternary separation operating schemes (including the JO process) in terms of the maximum throughput attained, and Generalized Full Cycle strategy was proposed based on a systematic design, which was found to have significant improvement over existing strategies [Agrawal and Kawajiri (2012)]. Nevertheless, the operating strategies were not experimentally validated. In this study, we validate both JO and Generalized Full Cycle SMB systems experimentally. A simultaneous optimization and model correction scheme has been implemented to arrive at the optimal operating condition which satisfies the optimal productivity as well as the desired purity and recovery of products experimentally.

1. Introduction

Since its development in the 1960s, simulated moving bed (SMB) chromatography has been widely discussed in the literature and also applied in numerous industries such as petrochemical, sugar and pharmaceutical industry [1,2]. The SMB process is based on a flow scheme that takes advantage of continuous and counter-current movement of the liquid and stationary phases without actual movement of the solid. As shown in Fig. 1, the standard SMB unit consists of multiple chromatographic columns which are interconnected in a cyclic conformation. The feed and desorbent are supplied continuously and at the same time extract and raffinate streams are withdrawn through the outlet ports. The feed mixture consists of two components which are separated utilizing the difference in their affinity toward the adsorbent phase. The faster moving component is recovered from the raffinate outlet while the slower moving component is recovered through the extract outlet. The counter-current motion of the liquid and adsorbent phases is achieved by switching both inlet and outlet ports simultaneously at a regular interval in the direction of liquid flow. Since SMB is a continuous and cyclic operation, it enables higher throughput and incurs less desorbent consumption compared to the batch chromatography. However, the standard SMB operation is limited to the separation of binary mixtures.

In chemical or bioprocessing industry, on the other hand, we often encounter multi-component mixtures when purifying any natural or biological product [1]. Here the feed mixture consists of a large number of components of similar chemical structures and the target product is located somewhere in between the fastest and the slowest eluting components. Although a number of approaches have been suggested to perform the separation of multicomponent mixtures, the application of SMB for multi-component separation is still considered one of the major challenges. Since SMB enables high throughput and reduces desorbent consumption, there has been a continuous effort to find modified SMB schemes that allow for higher productivity yet meeting the same product specifications. Examples of such modifications are the processes called the SMB cascade, where the two standard SMB systems are connected in series [3], Eight-zone SMB, where the two standard SMB systems are integrated into one single SMB unit [3], Five-zone SMB, where the zone prior to the feed location is split into two sections and an additional outlet is provided for the recovery of the intermediate eluting component [4,5], four-zone SMB, where the flow connection upstream of the extract outlet is broken while the intermediate and the slowest moving components are recovered through the extract outlet at different instants of time [5,6], ISMB where multiple components are withdrawn separately at different time instances within a step [7], and JO process, where the intermediate eluting component is fractionated only during Step 1 while Steps 2, 3 and 4 are similar to the standard SMB operation but without feeding [8–12]. In a recent computational study, various existing isocratic ternary separation strategies were compared

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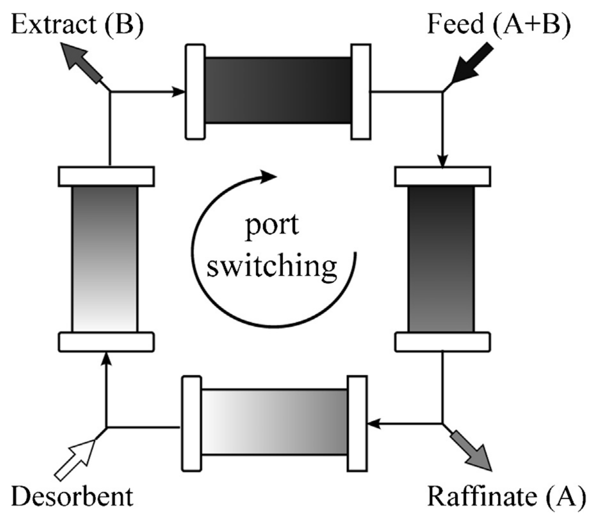

Fig. 1. Standard four-zone SMB configuration for the separation of a binary mixture.

in terms of the maximum throughout attained and the desorbent to feed ratio required [13]. This study had further investigated finding the best ternary separation strategy from the various available SMB configurations by considering a Generalized Full Cycle (GFC) formulation based on a systematic design. It was concluded that the JO process and the GFC operations have significant improvement over existing strategies. Nevertheless, the operating strategies were not experimentally validated.

The goal of this study is to demonstrate the GFC operation experimentally for the first time, and compare its performance to the JO process. We implement a simultaneous optimization and model correction (SOMC) scheme in order to resolve the model mismatch [14–16]. In addition, we would also show a systematic comparison of both JO and GFC operations by presenting a Pareto plot of the productivity achieved against the desired purity of the intermediate eluting component experimentally.

The organization of this paper is as follows: Section 2 describes the JO and the GFC operating strategies for the separation of ternary mixtures. Section 3 presents the ternary separation system used in this study. Section 4 explains the modeling of the SMB system. Section 5 elaborates on the optimization strategy used in order to find the optimal operating strategies. Section 6 presents the experimental system considered in this study. Section 7 discusses the simultaneous optimization and model correction (SOMC) scheme to systematically remove the model-mismatch from the SMB system. Section 8 presents the experimental results with regard to the JO and the GFC operating schemes and discusses the efficacy of SOMC scheme. Section 9 concludes the paper and presents the scope of future work.

2. Operating strategies

2.1. JO process

The JO process is a unique pseudo-SMB operation compared to the other isocratic modifications of the standard Four-zone SMB. In this operating strategy, the entire cyclic operation of SMB is modified. A base case operation is shown in Fig. 2(a), while many modifications are also proposed in the patent by Masuda et al. [8]. Fig. 2(b) shows the normalized concentration profiles inside the SMB columns when the JO operation is implemented. These concentration profiles are plotted at the beginning of each step of the JO operation after reaching cyclic steady state. In Step 1, the flow connection between column 2 and 3 is broken so that the intermediate eluting component can be recovered upstream of the shut-off valve. The feed mixture is simultaneously fed to the downstream side to load the SMB system. Steps 2, 3 and 4, on the other hand, are similar to the standard SMB operation with no feed inlet. In these steps, only the fastest and the slowest eluting component are recovered while feeding the fresh desorbent during the remaining steps. Moreover, the desorbent velocity and the switching time of Steps 2–4 are allowed to be different from Step 1 to further add the flexibility in the collection of fastest and the slowest eluting component. The inlet and outlet streams are switched as in the standard SMB operation in the clockwise direction to simulate the counter-current motion of the stationary phase. Steps 1–4 completes the cycle and this operation is repeated constantly in order to recover the pure products. The number of independent parameters that affect the performance of the JO operation are seven including the two desorbent velocities, two switching times, feed, extract and the zone 1 velocity in Step 2. The more detailed information regarding the design of the JO operation and the determination of operating conditions can be found elsewhere [8–10].

It is also important to note that the JO process can be implemented experimentally on any SMB system which can implement the standard SMB operation (shown in Fig. 1) without any major hardware modification. We may require an additional binary valve to break the flow connection during Step 1. The JO process was commercialized by Organo corporation for the separation of raffinose, sucrose, betains and salts [17], which was also applied to isolation of raffinose from beet molasses [18]. Another ternary SMB process implemented on an industrial scale is the sequential SMB [19,20], which is not considered in this work.

2.2. Generalized Full Cycle (GFC) process

It is well known in the literature that the performance of SMB system can be dramatically improved by changing the operating conditions such as flow rates and switching time or the operation itself [13,21–24]. Moreover, numerous SMB configurations can be created by changing the relative positions of feed and desorbent inlets, or the extract, raffinate and the intermediate stream outlets. Therefore, it is very important to find the best operating strategy among various SMB configurations. The GFC process is based on this idea of identifying the best separation strategy from various different alternatives [13].

In this strategy, we generalize the JO process by introducing additional inlet and outlet streams as shown in Fig. 3. In the GFC formulation, each step of the GFC formulation consists of two inlets: one for feed and the other for the desorbent, and three outlets: one for each of the product. These inlet/outlet flow rates and the switching time are allowed to change in the different steps and thus each step can be operated in a distinct way. In addition, the inlet and outlet flow rates can also be turned off whenever required. Hence, the GFC formulation is a framework that encompasses numerous ways of operating SMB and the flow rates and the switching times are nothing but the decision variables of an optimization problem. Unlike other operating strategies, the structure of the SMB operation is not chosen here a priori. Instead, the optimizer extracts the best operating strategy that optimizes the objective function while meeting the product constraints at the same time. Since there are four columns and four steps per cycle of the GFC process, the number of independent parameters that affect the performance of the GFC operation are 24; six in each step including the switching time, desorbent, feed, extract, raffinate and the zone 1 velocity.

It is interesting to note that, in the GFC formulation, the connection between any two columns can be broken in any of the steps to recover a particular product similarly to the first step of the JO process. Hence, the JO process can be derived as a special case of GFC formulation. Since the optimal solution is obtained from this inclusive and general structure, the optimal operating scheme derived