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Backfill-simulated moving bed operation for improving the separation performance of simulated moving bed chromatography

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ABSTRACT

The Backfill-SMB (BF-SMB) strategy was designed to improve the separation performance of simulated moving bed (SMB) chromatography. In the BF-SMB operation, a limited amount of products from raffinate and/or extract was re-fed to the SMB circuit as a backfill-feed. Two additional operating variables, backfill ratio (*BR*) and backfill length (*BL*), were suggested to determine the amount and injection length of backfill-feed. This strategy was applied to a four-zone SMB with one column (1-1-1-1) and two columns (2-2-2-2) per zone using a binary mixture with a nonlinear isotherm. Various BF-SMB operational methods were designed to supply backfill-feed to the feed node and/or intermediate node. The separation performances of conventional SMB and BF-SMB were compared in terms of purity, recovery, and eluent consumption. The BF-SMB successfully improved the separation performance of the conventional SMB because backfill-feed led to the rich condition of the main component at each product withdrawal node. Due to the 'TMB effect' caused by backfill-feed, the BF-SMB operation was more efficient in the 1-1-1-1 configuration than in the 2-2-2-2 configuration, showing maximum improvement of 5–7% purity and recovery from the performance of conventional SMB. In addition, partial recycling of eluent by backfill-feed resulted in a decrease in eluent consumption up to 10% even with improved purity and recovery in BF-SMB.

1. Introduction

The simulated moving bed (SMB) consists of multi-columns with an appropriate sequence of inlet and outlet port switching designed to simulate a countercurrent flow. It has high productivity and low eluent consumption compared to batch chromatography due to continuous and countercurrent flow. It has been applied since the 1960s by Universal Oil Products for large-scale separations in the petrochemical industry, and its adoption has since been widened to fine chemical and pharmaceutical applications [1–4].

Many operating strategies have been studied to improve the separation performance of SMB. Some of the strategies focused on modulating operation parameters between two consecutive switches: the VariCol operation uses the asynchronous switching [5], the PowerFeed, Partial-feed, and ModiCon methods vary feed flow-rate or concentration [6–8], the Partial-Discard method discards a certain portion of the product [9], and the Outlet Streams Swing (OSS) operation modifies outlet flow-rate [10]. In conventional SMB, the impurity component comes out at the initial stage and the last stage of the switching period in the extract and raffinate withdrawal node, respectively. The general concept of the above strategies is to extend the time that the impurity at the initial and last stages needs to reach product withdrawal [6–8] or avoid product withdrawal by closing the port, moving the node, or discarding part of the product [5,9,10].

As another strategy, additional equipment was applied to the SMB, such as an additional column before the feed node of the SMB circuit in FeedCol [11] and additional tools for recycling the contaminated part of the product to the feed node in Fractionation and Feedback SMB (FF-SMB) [12]. In the previous study, the Recycling Partial-Discard (RPD) operation was introduced as an improved Partial-Discard (PD) strategy [13]. The raffinate discarded portion and extract discarded portion could contribute to improved SMB performance by recycling to the feed node at the initial and last stages of the switching period, respectively. In addition, practically, the product recycling step has also been applied to the PAREX process (industrial scale SMB unit for p-xylene separation) for flushing the transfer lines before the extract product node [14,15].

A more efficient operation strategy for improving the separation performance of SMB is needed due to strict regulations regarding high purity products in the fine chemical and pharmaceutical industries. As a representative adsorptive cyclic process, pressure swing adsorption (PSA) is currently used to separate a wide range of gas mixtures. Because their processes are very similar,

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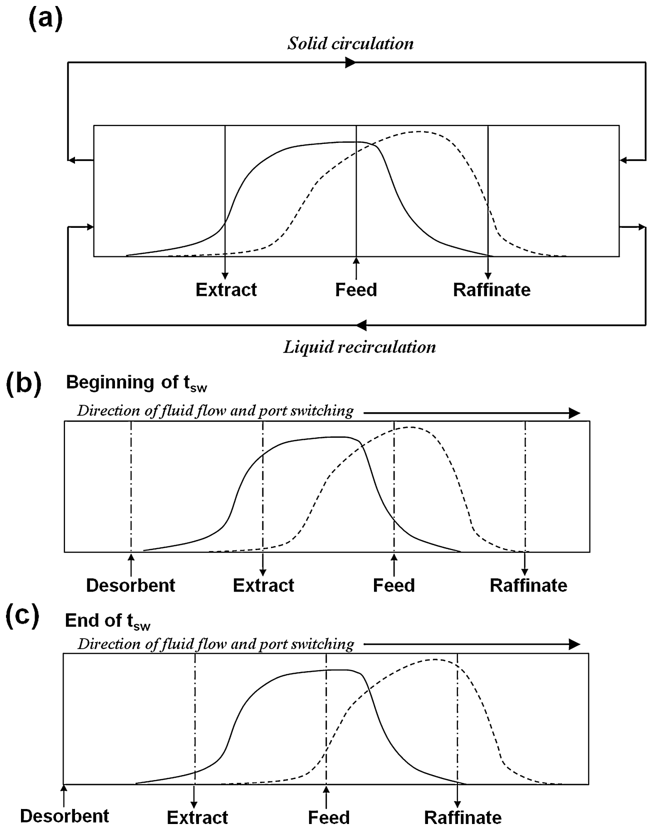

Fig. 1. General internal concentration profiles of (a) TMB, (b) SMB at the beginning of the switching period, and (c) SMB at the end of the switching period.

operational strategies used in PSA may be applied to SMB. Using the backfill step with a portion of product is often applied to the PSA process to obtain a high purity product. Various backfill methods were introduced to the cyclic step sequence of PSA to improve the purity and recovery of product [16,17].

The purpose of the backfill step in PSA is to clean the adsorbent bed using product-rich gas and to overcome the limitation of product purity. Therefore, PSA with a backfill step results in high purity production as the feed is supplied. Like PSA processes, it is possible that a portion of the product can be supplied to the SMB circuit as part of the feed. This may become a raffinate-rich or extract-rich condition at a certain part of the column and result in improved SMB performance. In the Multicolumn Continuous Chromatography (M3C) process, the true moving bed (TMB) was integrated with a concentration loop at the extract node and the fraction of the solution recycled from zone I to zone II was concentrated before reinjection [18]. A high concentration of the more retained compound created a displacement effect on the less retained compound and induced the desorption. In this study, this method was called "Backfill-SMB (BF-SMB)". The BF-SMB strategy was designed to improve the separation performance of simulated moving bed (SMB) chromatography. Various operational methods of backfill were applied to a four-zone SMB with one (1-1-1-1) and two columns (2-2-2-2) per zone using a binary mixture with a nonlinear isotherm. Compared to conventional SMB, the BF-SMB showed improved separation performance in terms of purity, recovery, and eluent consumption.

2. Principles of backfill-simulated moving bed

Fig. 1 shows a schematic of the internal concentration profiles in the true moving bed (TMB) (Fig. 1(a)), SMB at the beginning of the switching period (Fig. 1(b)), and SMB at the end of the switching period (Fig. 1(c)). Although the mixture actually moves along the eluent flow in the SMB operation, Fig. 1(b) and (c) shows that each node moves to the opposite direction of the fluid flow with equivalent fluid velocity.

As shown in Fig. 1(a), the composition of the mixture near the feed node remains constant due to countercurrent flow of the fluid and solid at the same velocity in the TMB operation. However, node switches occur in the SMB operation so that the composition of the mixture around the feed node changes consistently during a switching period. In other words, the feed is injected to the raffinate-rich position at the beginning of the switching period (Fig. 1(b)) and to the extract-rich position at the end of the switching period (Fig. 1(c)) in the conventional SMB. This difference between the TMB and SMB operations is a main factor accounting for relatively lower separation performances of the SMB. The TMB effect could be added to SMB by the raffinate-rich composition of the initial part of the feed and the extract-rich composition of the last part of the feed, as reported in previous studies [11,13].

The purpose of the Backfill-SMB is to generate a TMB effect to improve the separation performance of the SMB operation. In this study, the TMB effect was implemented to the BF-SMB by re-feeding a limited portion of product in the SMB. Raffinate and extract products can be supplied to the SMB circuit as backfill-feeds. In addition, backfill-feed can be injected to various nodes (Feed and Intermediate nodes) in the BF-SMB.

The raw feed and backfill feed supplied from the product reservoir were called original-feed and backfill-feed, respectively. The following four cases of BF-SMB operation were categorized as representative backfill methods. All BF-SMB operations were compared to the same throughput of the feed as the conventional SMB operation. Therefore, the concentration of the original-feed in the Backfill-SMB was higher than the concentration of the feed in the conventional SMB because the original-feed, which has higher concentration than the raw feed, was injected within the limited duration in the Backfill-SMB operation. And it was also reported that the pulse injection of concentrated feed improves the separation performance in literature [8].

- Case I (BF-I in Fig. 2(a)): In the BF-SMB operation, limited amounts of raffinate and extract were re-fed to the feed node at the initial and last stages of the switching period, respectively, in the BF-I operation (Fig. 2(a)). Backfill-feed and original-feed were injected to the feed node and their injection times were partitioned for feeds in the BF-I operation. Therefore, the TMB effect can be produced by injecting each feed. The original-feed, within the limited injection time is used in the BF-SMB because the comparison between BF-SMB and conventional SMB is made at the same throughput of original-feed in SMB operations. And, with respect to the recycling method, the BF-I operation in Fig. 2(a) is the same as the RPD operation in the previous work [13] because the raffinate-rich portion and extract-rich portion are recycled to the feed node at the initial and last stage of the switching period, respectively.

  However, the purity and concentration of the recycled portion is different between the two strategies. Since a portion from a reservoir for collecting discarded portion is recycled to the feed node in the RPD operation, the purity and concentration of the recycled portion are the average of the partial-discarded portion. On the other hand, the backfill-portion in the BF-SMB operation has the average of the purity and concentration of each product because it comes from a product reservoir.
- Case II (BF-II in Fig. 2(b)): In the BF-II operation, extract backfill-feed was injected to the intermediate node between the feed and extract nodes (E–F node) while the raffinate backfill-feed was fed to the intermediate node between the feed and raffinate